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Vol. 25, No. (1), March 2026

CONTENTS

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Review Research Paper

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Torrefaction of Commonly Disposed Agricultural Waste Biomass for an Improved and Sustainable Energy Future: A Review

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ABSTRACT

Scientists and policymakers are continuously making techno-economic efforts to close the loop in the agricultural value chain by utilizing and maximizing agricultural wastes and their products. The rising issues of agricultural waste management significantly impact the ecosystem and impede environmental sustainability. Untreated and wrongly disposed agricultural residues are a major threat to health (human and animal), the economy, and a significant contributor to greenhouse gas emissions. However, this review extrapolates a resource efficiency technology to address the energy deficit by converting these sustainable waste resource sources to sustainable energy through a sustainable energy system. The torrefaction technique is a more energy-efficient thermochemical process to upgrade the biomass fuel quality. Studies on readily available and commonly disposed agricultural wastes valorised with their energy values, energy density and physicochemical properties were reported in this study, and their performances were compared with fossil fuel (coal and sub-bituminous coal) properties. The assessment brings to the submission that many agricultural wastes can be upgraded to comparable quality in performance via the torrefaction process. It further discovers that the synergy of certain additives and the optimization of process conditions, such as residence time, temperature, pressure, and gas carrier, could better upgrade the biofuel quality without major compromise on product yield.

INTRODUCTION

Global warming concerns about the continuous rise in greenhouse gas emissions and massive depletion of fossil fuels such as coal, petroleum, and natural gas are major challenges of industrialization. In coal-fired plants, for instance, fossil fuel air pollution from the burning of a ton of coal generates 3.67 tons of CO_2, which is disastrous to human health and the environment. As predictions to hit 14 billion $t.y^{-1}$ by 2050 heighten, the adoption of alternative sources of energy that are sustainable and renewable has become a necessary demand (Acharya et al. 2012). Subsequently, a report from the International Energy Agency (IEA), shows that the press for bioenergy has risen four times over the decades, which is expected to capture over 17% of global energy by 2060 (Cross et al. 2021, Röder et al. 2020) The ready availability of bio-feedstock has given bioenergy a leverage over other renewable and alternative energies such as solar, wind, hydropower and geothermal. It is also seen to have the potential to mitigate the greenhouse effect, giving a win-win carbon credit and reducing biomass waste. Hence, combating the solid waste (SW) complex problem due to improper waste management practices causes environmental and health concerns (Abdullah et al. 2022).

BIOMASS AND THE CONCEPT OF TORREFACTION-THERMOCHEMICAL PROCESS

Biomass is referred to as the biological material of plant and animal origin, alongside their waste and residues (Chew and Doshi, 2011). Biomass, an acclaimed carbon-neutral fuel, engages in the bio-cycle, and the CO_2 from its combustion is reinjected into the growth of new crops. It is a choice of sustainable fuel able to reduce net carbon emissions instead of fossil fuel (Chew and Doshi, 2011). However, this biomass is broadly divided into woody and non-woody biomass. Its short carbon cycle makes it a renewable energy source with low greenhouse gas emissions based on its CO_2 captured during photosynthesis. However, biomass is a good substitute/ alternative to coal for sustainable energy production. Its drawbacks are its non-homogeneity, moisture content, alkality, cost of mobility, grindability, and reduced energy density relative to coal.

The transformation of biomass to energy can be achieved through several routes, such as biochemical, mechanical, and thermochemical. Thermochemical processing is attractive and efficient in transforming biomass to energy as it captures a broader fuel feedstock. Its lower temperature requirement makes it a suitable energy technology (Chew & Doshi 2011). However, the process of torrefaction, known as mild pyrolysis, could mitigate the shortcomings of biomass (Acharya et al. 2015). According to Acharya et al. (2012), it is the decomposition of biomass leading to the release of volatiles, having its final product as solid fuel known as torrefied biomass/fuel. The thermochemical transformation of biomass helps to bring down the NOx and SOx emitted relative to fossil fuels (Gilbert et al. 2009).

According to Kumar et al. (2020), the two primary thermal pretreatment techniques to enhance biomass quality and improve their properties are wet and dry torrefaction. Although wet torrefaction is less frequent, both processes can be used to obtain hydrophobic, uniform, high-carbon, and densified energy solid fuel (Acharya et al. 2015). Dry torrefaction (DT) occurs within a low oxygen environment at temperatures ranging from 200 to 300°C for a duration of 30 to 60 min. (Acharya et al. 2013), at atmospheric pressure, whereas WT is a thermochemical conversion process in subcritical water. WT is the thermal treatment of biomass in water at temperatures of 180-265°C, spanning from about 5 min to hrs at pressures above 1 MPa (Yan et al. 2009).

All lignocellulosic biomass has relatively similar patterns of cell walls, although they might differ slightly based on their specific composition and biomass. It mostly comprises 20% fixed carbon and 80% volatile content on a dry basis. It is made up of (40-60 wt.%) cellulose, (10- 25 wt.%) lignin, and (20-40 wt.%) hemicellulose (Acharya et al. 2012). However, the process of torrefaction changes the composition of the biomass under varying conditions such as temperature, time, pressure, and the nature of the gas. This process alters the chemical composition and causes the breakdown of hemicellulose, cellulose, and lignin, as shown in Fig. 1, where the hydroxyl (OH) compound is breaking down, and the hydrophilicity is improved (Bridgwater et al. 2000).

The torrefaction process can be broadly categorized into three phases: size reduction, drying, and roasting (torrefaction) phase (Acharya et al. 2012). The uniform and fine biomass

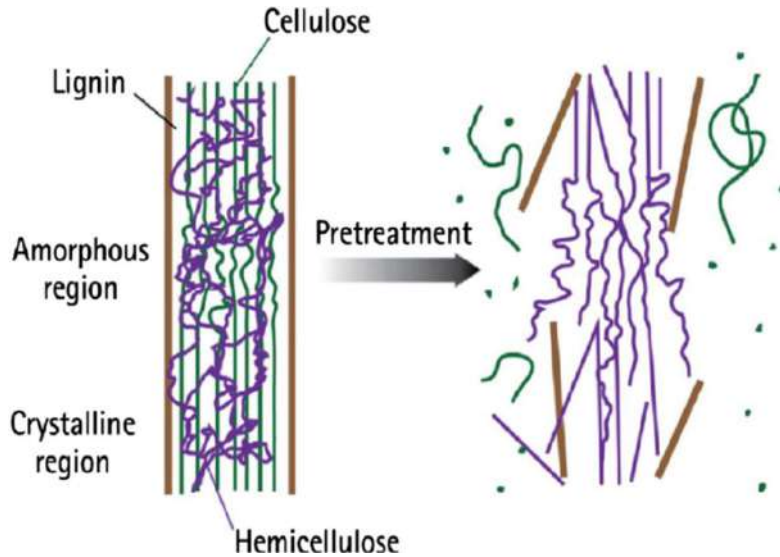

Fig. 1: Structure of biomass and thermal pretreatment(Acharya et al. 2012).

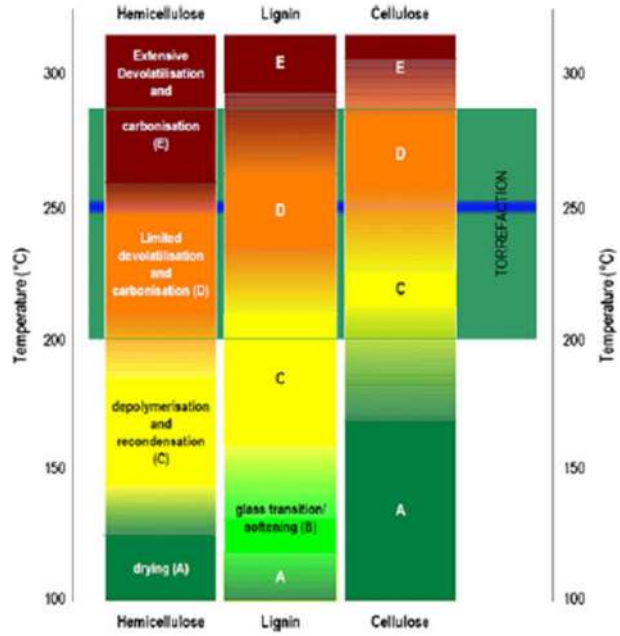

Fig. 2: Decomposition regimes of lignocellulosic material during thermal treatment (Uslu et al. 2008).

is subjected to drying to reduce moisture considerably and to liberate condensable and non-condensable gases and volatiles, before feeding into the torrefaction reactor. However, the extent of gases and volatiles liberated depends on the condition of torrefaction, especially temperature, leaving behind a solid product known as char or torrefied biomass (Ciolkosz et al. 2011). This process enhances the combustive characteristics of biomass for an attractive solid fuel suitable for heat energy applications.

Furthermore, it is worth noting that a series of decomposition reactions occurs in torrefaction, leading to a series of gaseous compound releases. This process alters the elemental compositions of the biomass and reduces the H/C, O/C ratio as hydrogen and oxygen content decline. It is also characterized by the destruction of hydroxyl (OH) groups in the decomposed biomass polymer structure, making it hydrophobic. At about 110°C, a major percentage of moisture is lost, and further temperature increases lead to polymeric structural decomposition, mainly hemicellulose. Between 250-300°C, more hemicellulose is decomposed, leading to massive weight loss at this stage with slight lignin and cellulose decomposition (Uslu et al. 2008, Rousset et al. 2011). Fig. 2 provides a typical representation of these processes with respect to temperature.

Typical biomass is constrained in widespread energy applications due to its inherent excessive moisture, volatile, oxygen content, lower density, low calorific value, and grindability (Singh et al. 2020). However, the torrefaction process helps to improve its physicochemical properties, such as achieving a reduction in volatile matter under a relatively lower heating temperature while most of the fixed carbon content remains. Fig. 3 provides a typical schematic of a torrefaction setup in a tube furnace, where a weighted biomass sample is loaded in the ceramic boat, and a nitrogen flow is supplied at a specified flow rate to the furnace for an assigned residence time. The hemicellulose content is the main and most reactive volatile matter decomposed during torrefaction, than the other two components, cellulose and lignin. As the torrefaction temperature advances, the mass yield is often seen to decline, and this is principally due to two major factors, which are moisture loss and thermal decomposition to form a volatile gaseous product such as H_2O, CO, CO_2, acetic acid and other organics (Poudel et al. 2015). This thermal decomposition occurs mainly on hemicellulose and lignin at torrefaction temperatures below 250°C; the partial decomposition of the cellulose later occurs as torrefaction conditions become more severe.

Energy yield is the measure of the ratio of actual energy retained after the torrefaction process to the initial energy content of the biomass (Bridgeman et al. 2008). Also, the combustibility of biochar can be assessed by using the fuel ratio, which is defined as the ratio of fixed carbon content to volatile matter content. This is a principal index to evaluate the potential/capacity of biochar fuel properties to replace coal. Also, according to Lin et al. (2025).

$$\text{Energy density} = \frac{\text{HHV of the torrefied sample}}{\text{HHV of the raw sample}} \quad \ldots(1)$$

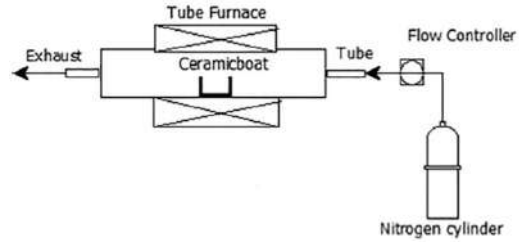

Fig. 3: Schematic of torrefaction setup (Patidar & Vashishtha 2021).

$$\text{Mass yield} = \frac{\text{Mass of torrefied sample}}{\text{Mass of raw sample}} \times 100\%. \quad ...(2)$$

$$\text{Energy yield} = \text{Energy density} \times \text{Solid yield} \quad ... (3)$$

Increased production of agricultural waste due to population growth and expanded agricultural practices has created an inexhaustible and sustainable agro-waste feedstock which can be converted into useful forms, such as biochar, biofuels, bio-coal pellets and other structural products. However, these wastes have not been effectively managed as they majorly litter the environment as pollutants instead of a bioresource for energy generation and a means for job creation. This review helps to appreciate the vast abundance of agro-waste in its varieties in the environment. It also showcases torrefaction as a low-cost and less energy-intensive route for bioenergy generation with emphasis on its operational process/ boundary conditions to achieve desirable efficient energy and optimal mass-energy yield balance. Furthermore, it also captures the techno-economic implications of selected torrefied agro-wastes.

The techno-economic analysis discusses the economic feasibility and technical performance of converting agricultural residues into more energy-dense and stable forms of biomass. This includes energy insecurity and environmental sustainability. The subject of temperature control, residence time and process design, carbon emission, waste reduction, cost-benefit analysis, market potential, agricultural waste type (rice husk, corn stalk and wheat straw, etc are among the contributors to the techno-economic feasibility of the torrefied agro-waste. Biomass torrefaction increases the energy content per unit weight (mass), and subsequent pelletization markedly improves the energy density per unit volume, thereby facilitating logistics throughout the supply chain.

ROLE OF PROCESS TIME AND TEMPERATURE ON THE QUALITY OF AGRO-WASTE-DERIVED SOLID FUEL

The conversion of Walnut shell (WS) and pearl millet (PM) to solid biofuel was carried out by Abdullah et al. (2022). This was performed at a torrefaction temperature of (230-300)°C, times (30-90 min) and varying biomass composition. It is intended to upgrade their biochar properties to a equivalent comparable to coal. In this process, the highest biomass mass yield of 91% was achieved at (230°C, 30 min) and the lowest, which is 41% at (300°C, 90 min). It has a Gross calorific value (GCV) of 22 $MJ.kg^{-1}$ at the raw state and 27 $MJ.kg^{-1}$ at 300°C, accounting for a 22-59% HHV increase. At the optimal parameters of 260°C, 30 min, and a blend of (PM 70%, WS:30%), 80-88% yield was reported.

(Nigran Homdoung et al. (2019) examined the outcome of the torrefaction of wood chips and oil palm fronds under 200-400°C and a 20-60 min. It was clear that the energy properties and solid product were affected by the torrefaction time and temperature. No visible change in mass and energy yield was observed at 200C°C; 20-60 min for the mass yield of both oil palm fronds and wood chips. Hence, it was regarded as the optimum condition. The volatile% reduces as the torrefaction temperature moves from 200-400°C for both wood chips and oil palm fronds.

HHV of both biomass materials was improved to 17.65 $MJ.kg^{-1}$-24.86 for wood chips and 16.34 $MJ.kg^{-1}$-18.58 for oil palm fruit fronts, 20-30% higher than the original values. Oil palm fruit fronts have a higher ash content of up to 15% which was responsible for their lower HHV. The torrefaction process also helps achieve improved fixed carbon content of wood chips by 29-85% and oil palm front by 76-97%.

Yang et al. (2015) examined the fuel properties of wet torrefied biomass, namely the Humulus lupulus (HL), Plumeria alba and Calophyllum inophyllum L. (CIL) with varied component weights. The mass yield decreases as presented in Fig. 4a. 50.9% mass yield was obtained for CIL, 31.5% for PA and 26.5% for HL at 260°C. CIL has a greater mass yield due to its higher lignin content of higher decomposition temperature than hemicellulose and cellulose. It is hence considered more thermally stable than PA and HL. The HHV and energy yield increased from 17.5 $MJ.kg^{-1}$ to 25.3 $MJ.kg^{-1}$ for HL;17.7 $MJ.kg^{-1}$ to 25.7 $MJ.kg^{-1}$ for PA, and 18.4 $MJ.kg^{-1}$ to 23.6$MJ.kg^{-1}$ for CIL as the temperature advanced from 180 to 260°C. The HHV obtained at elevated torrefaction, which is 23.5 and 25.7 $MJ.kg^{-1}$ as shown in Fig.

4b, is comparable to that of some commercial coal. The H/C and O/C ratios of HL-260, PA-260, and CIL-260 were similar and closer to those of lignite, as shown in Fig. 4c**.**

Energy sorghum and sweet sorghum were torrefied under different temperatures (250, 275 & 300°C) for 30 min by Yue et al. (2017). Torrefied energy sorghum has 53.1-69.8% while torrefied sweet sorghum has 41.3- 64.7% solid yield at 250-300°C. The process of torrefaction helps to achieve an improvement in the HHV of energy sorghum from 17.33 MJ.kg^{-1} (raw) to 23.62MJ.kg^{-1} at 300°C, while

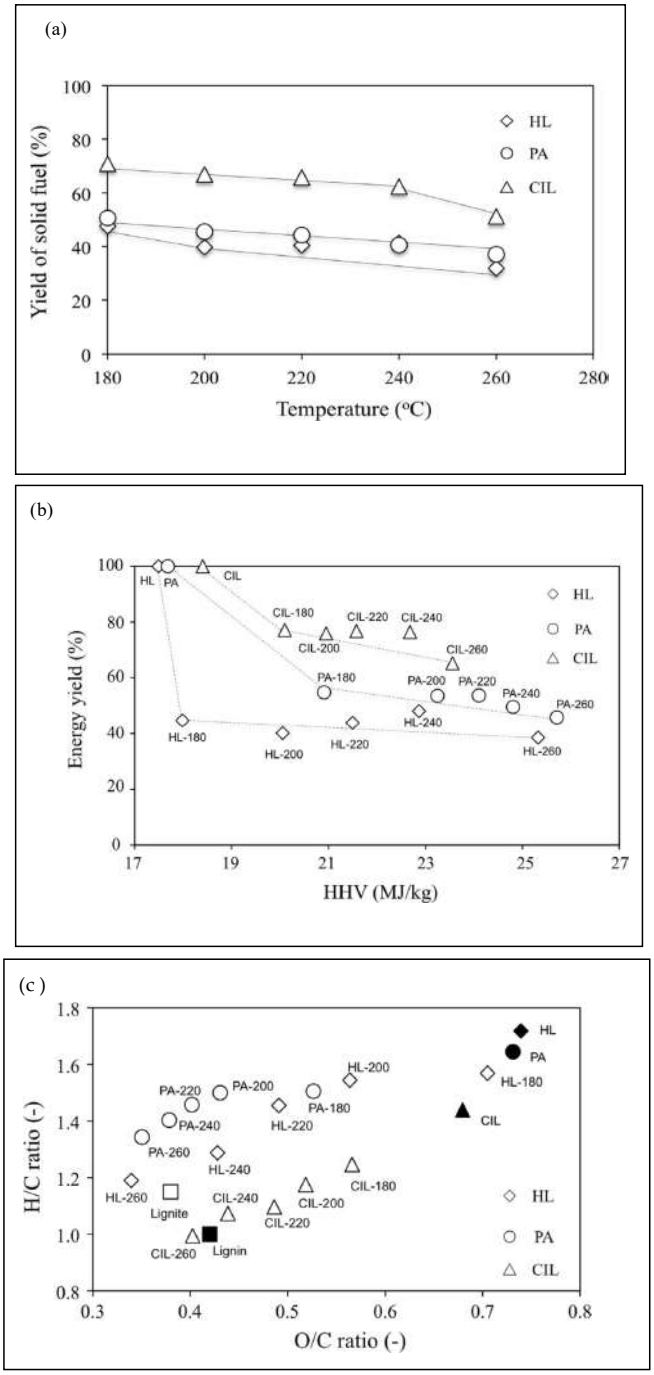

Fig. 4: (a) Mass yield of HL, PA, and CIL at various temperatures, (b) Energy yield against HHV for all the biomasses and their delivered solid fuel, (c) Van Krevelen diagram of the biomasses (Yang et al. 2015).

for sweet sorghum bagasse, 16.45 MJ.kg^{-1} (raw) to 26.88 MJ.kg^{-1} at 300°C.

Zhang et al. (2016a) carried out a wet torrefaction process on duckweed within 130–250°C to improve its fuel characteristics. The volatile content declined from 76.9%-60.0%, while the ash content improved from 7.65 to 19.9% within the range of 130-250°C. There was a notable decline in the mass yield from 64.8% -30.4% and the energy yield from 77.9%-40.1% across the torrefaction temperature range. Also, the energy density first shifted from 1.20 (D130) to -1.38 (D220)- 1.32(D250). Hence, 220°C is identified as the ideal reaction temperature for wet torrefaction of duckweed samples. The HHV improved from 14.34MJ.kg^{-1}, which is the raw sample, to 19.84 MJ.kg^{-1} at D220 and then declined to a more severe temperature of 250°C. Wet torrefaction of duckweed at 250°C gave a closer H/C and O/c atomic ratio closer to lignite, which is indicative of improved solid fuel properties. Other features are improved C-content from 34.5% to 48.3%, reduced nitrogen and sulfur content.

The torrefaction of corncob, cotton stalks, and sunflower agricultural residues was performed by Akhtar et al. (2021) at 200-320°C; 10-60 min. Corncob has a mass yield of 63% and optimum GCV of 5444 kcal.kg^{-1} at 290°C, 20 min. Torrefied cotton ball has optimal GCV of 4481 kcal.g^{-1} at 270°C, 30min. An optimum condition at 260°C, an energy value of 4370 kcal.kg^{-1}, and a decline in mass yield of 85-71% at 10-60 min residence time was obtained for the sunflower. The process of torrefaction produced a biochar of reduced hemicellulose content, and more lignin and cellulosic content. This process leads to a brittle, grindable, and less reactive biochar with a break in biomass interlocking blocks.

Corncob and khat stem biomass's energy content was explored via torrefaction, and optimization was enhanced by Jifara Daba and Mekuria Hailegiorgis (2023). Investigation performed at 200, 250, and 300°C; 15, 30, and 45 min. The volatile matter content reduced from 77.7-64% for the Khet stem and from 76.9-67% for the corn cob. Khat stems burn and ignite better due to their higher volatile content. The ash content of raw corn cob increased from 3.24-10% and khat from 7.4-15% respectively, as Khat contains more inorganic compounds than corn cob. A moderate improvement of about 6% was observed in the fixed carbon content of the khat stem and corncob across the temperature range, with similar carbon contents of 43.43% (corncob)and 42.18% (khat stem). Predicted corncob has higher energy content than khat steam. Khat stem has a mass yield (68.85%), energy yield (98.5%) and HHV (24.95 MJ.kg^{-1}), while corncob has a mass yield of 56.80%, energy yield (94.9%) and HHV (23.37 MJ.kg^{-1}).

The role of torrefaction on biomass stalk on fuel yield and properties by Chen et al. (2015) at 220,250, and 280°C temperatures was investigated. As temperature progresses, bio-char mass yield declines while the bio-oil yield advances significantly. HHV value improved from 16.53 MJ.kg^{-1} for dried cotton stalk (DCS) to 20.31 MJ.kg^{-1} for torrefied cotton stalk (TCS)at 280°C. The volatile content dropped from 75.38 (TCS-220) to 56.23wt% (TCS-280) with higher ash and fixed carbon content at higher temperatures. Torrefaction temperature significantly improves the% carbon (C) content and reduces the oxygen (O) content. The biomass stalk has poor thermal stability, leading to the decomposition of a large proportion.

Almond Shell (AS) and Olive pomace (OP) were torrefied at conditions of 280-320°C, 500°C, and at various times by Alcazar-Ruiz et al. (2022).In their study, OP was confirmed to be thermally unstable compared to AS. OP has the highest carboxylic acid yield at (280°C; 20s) while AS at (300°C; 20s). As torrefaction became severe, the phenolic compound was noticeable for OP. This was attributed to the elevated lignin content and natural metals present in Olive Pomace that enhance catalytic reactions during the process. The maximum yield (47.7%) was achieved at (320; 240 s).

Bach et al. (2013) compared the role of process parameters on Norway spruce (softwood) and birch (hardwood) local biomass in a wet torrefaction process of 175,200, 225°C and at 10,30 and 60 min. The energy yield was observed to decline as temperature and holding time increased. Hence, it has a significant influence on fuel properties and solid products. However, the lower yield of solid products was observed at smaller particle sizes. The analysis and predictions proved that greater heating values are obtained at lower temperatures and shorter times. The fixed carbon content of truce wood biochar products was enhanced from 13.3-27.1% and 10.3-27.5% as the temperature and holding time advanced. Torrefied spruce experienced an HHV rise from 1.9-12.5% while torrefied birch had a 1.3–15.0% increase in the range of 175-225°C. At 225°C, the HHV of torrefied birch wood is comparable to that of torrefied spruce wood.

Norway spruce stem wood, stump, and bark were torrefied in a tubular reactor (Wang et al. 2017). The mass yield of all the torrefied samples declined as torrefaction conditions became severe which is from 225°C, 30min to 300°C, 60min. Stump recorded a drop in mass of 44% and 54% at 300°C for 30 and 60min, while stem wood showed a 30% and 40% decline in mass loss at 30 and 60 min residence time under 300°C residence time.

A carbon-rich solid feedstock was produced from torrefied olive mill waste (TPOMW) in a study by Benavente and Fullana (2015) carried out at 150-300°C for 2h. The study showed that carbon content was enhanced from 56-68 wt%%

and HHV from 26.4-30.0 MJ.kg^{-1}, by increasing the process temperature, which upgraded the value of the TPOMW comparable to sub-bituminous coal. Optimal heating value and minimised energy loss were obtained at 200°C. The synergetic process of torrefaction and densification was observed to enhance the energy density of TPOMW to a maximum of approximately 242% at t-TPOMW-300 briquettes.

Cetinkaya et al. (2024) also attempted to optimize the temperature and holding time as process parameters on Rosa Damascena Mill solid waste (RP) and red pine sawdust (PS). They produced bio-pellets of different weight ratios. The average HHV of the RP sample shifted from 19.8 MJ.kg^{-1} for the raw PS sample to 21.2 MJ.kg^{-1} at (290°C; 60 min). The average HHVs of the RP raw samples increased from 18.3 MJ.kg^{-1} temperature to 21.3 MJ.kg^{-1} at 290°C;60 min. It is also worth noting that the mass yields declined at severe torrefaction conditions ($p < 0.05$). At (290°C; 60 min) mass yield of the RP (57%) and PS (63%), which are the lowest yields, was recorded.

The torrefaction of pomaces and nutshells in a muffle furnace was investigated by Chiou et al. (2015). Apple pomace has lower thermal stability, hence it was torrefied at 200, 230, and 260°C, while nutshell was torrefied at 230, 260, and 290°C; all at 20, 40, and 60 min. All the samples have high energy yield at 230°C, but declined rapidly at 260°C. Apple pomace greatly declined by 42.3-14.9% while grape pomace decreased the least, ranging from 92.3-59.7% with a residence time of 20-60 min residence. However, energy yield was steady at 290°C, recording the highest value (71.4%) with grape pomace at 20 min and 62.6% at 60 min. This could be connected to grape pomace having a high mass yield at these temperatures.

Solid fuel from the torrefaction of passion fruit peel waste (PF) and pineapple fruit waste (PA) was obtained by da Silva et al. (2022). It was carried out at 200, 250, and 300°C and (15 and 60 min)using the macro-TGA with GC-TCD/FID analysis. From the figure, it was obvious that the torrefaction process enhanced HHV with the highest value of 22.97 MJ.kg^{-1} and 20.78 MJ.kg^{-1}, fixed carbon content of 52.95 wt.% and 40.19 wt.% for PA and PF at (300°C;60 min), respectively. Solid yields of 56.21% for PA and 40.86% for PF at 300°C, 60 min were obtained. This is as presented in Fig. 5

Dhungana et al. (2011) compared non-lignocellulosic and lignocellulosic waste biomass in a torrefaction process. The non-lignocellulosic biomass was undigested sludge, chicken litter, digested sludge, and, while coffee husk, switchgrass, and wood pellet are the lignocellulosic biomass waste. The investigation was conducted within 250-280°C, and the residence time was 15-60 min. The energy density of the biomass was enhanced, and some of the biomass polymers decomposed, letting out oxygen through CO_2 and H_2O, and retaining some carbon in char. HHV increased from 19.18-24.20 MJ.kg^{-1} for non-lignocellulose biomass at (280°C; 30 min). Similar values are obtainable with lignocellulosic biomass. Results confirm that HHV increases steadily with higher temperature, as well as the residence time and a further increase in the energy density of this biomass.

The fuel properties of Olive pruning (OP) and vineyard pruning(VP) were improved by Duman et al. (2020) via

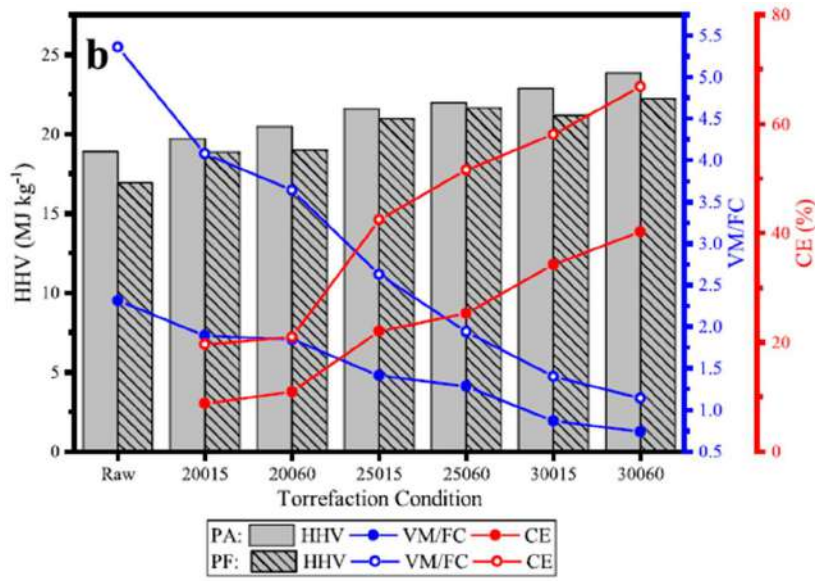

Fig. 5: Relationship between VM and FC (VM/FC), carbon enrichment (CE), and Higher heating value (HHV) (da Silva et al. 2022).

the torrefaction and hydrothermal route. Biochar has a mass yield of 82.1% (OP) and 81.0% (VP) at torrefying conditions (200°C; 60 min). Hydrochars have lower values of 58.2% for OP and 59.1% for VP. This difference in the mass yield can be attributed to the nature and amount of lignin in the biomass. In the HTC process, a lower mass yield was observed as the temperature increased, and a higher energy density of up to 1.45 times for hydrochar. Biochars have ignition temperatures at 270-346°C for OPB and 279- 353°C for VPB, and hydrochars between 268 and 409°C for OPH and 273 to 304°C for VPB. These temperatures exceed those of raw biomasses. However, the burnout temperature of biomasses was not affected by dry torrefaction, which is between 489 and 503°C for OP and approximately 490°C for VP, but the burnout temperature increased with HTC-treated biomass(from 494-561°C for OP and 487-534°C for VP). The ash content of biochar and hydrocarbon significantly differs and changes with biomass type.

The study investigating the combustion characteristics of torrefied almond hulls and shells, olive seeds, and corn stalks was conducted by Duranay et al. (2023). The torrefaction was carried out at 300±5ºC for 41 min. The torrefaction yield is dependent on the type of biomass. It was deduced that almond shells and olive kernels (hard woody waste) have higher solid product yields, which are 80.8% and 78.4%, respectively. Almond hull and corn stalks (flexible and fibrous waste) have lower solid product yields, which are 53.4% and 43.7% respectively. Harder agricultural wastes have a high amount of solid product based on their difficulty to thermally decompose, while more liquid and gaseous products were found during the thermal treatment of fibrous biomass. Torrefaction helps to improve the fixed carbon amount of almond and olive kernels by 30–55%. Volatile matter of corn stalk and almond hull declined by 42% and 32%, respectively, while the fixed carbon contents increased by 309% and 96%, respectively.

Cassava rhizome, sugarcane bagasse and straw briquette were torrefied at (250ºC; 90 min) by Granado et al. (2023). Cassava rhizome, sugarcane straw and sugar cane bagasse had relaxed densities of 1270 kg.$^{-3}$, 1240 kg.m^{3} and 1300 kg.m^{-3}, respectively. Torrefied cassava rhizome, sugarcane bagasse and sugarcane straw gave improved HHV of 19.2, 18.4 and 19.0 MJ.kg^{-1}, respectively.

Auricularia auricula-judae, commonly known as the wood ear, was torrefied by Zhang et al. (2016b). Torrefaction was carried out under 200-320 °C and residence time (120-15 min). The mass yield continuously declined from 92.23% (200°C, 15min) to 46.65% (320°C, 120min) and the energy yield from 92.20% (200°C, 15 min) to 57.28% (320°C, 120 min). The C-content improved from 51.73% to 64.94% (320 ^{o}C;120min) and also an enhancement in the HHV from 21.13 MJ.kg^{-1} -25.96 MJ.kg^{-1} from 200°C; 15 min to 320°C; 15min. A decline was noticed in the O/C and H/C ratio from 0.571-0.332 and 1.594-0.907, respectively, within the torrefaction condition.

Leucaena, a woody biomass feedstock, was microwave torrefied by Huang et al. (2017). As the power level increases, the temperature and heating rate also increase. Microwave power, as an operating parameter, was noticed to have a greater effect than time. The HHV of the biochar increases with increases in power level and time. However, the reverse was witnessed with the energy and mass yield of the product as it declined from 72.30 wt.% at (100 W; 15 min) to 17.25% at 250W; 30min. A similar trend was observed for energy yield. HHV of 30 MJ.kg^{-1} was reached at 250W power for 30 min processing time. The torrefied leucaena produced a fuel ratio of up to 3.7 at power levels of 200 and 250 W, which is greater than that of bituminous coal. As microwave power and time increased, the fixed carbon content rose while the volatile content decreased. This development suggests a potential alternative fuel source to substitute for coal or be used in co-firing.

In their 2017 study, Ianez-Rodriguez et al.(2017) optimized Greenhouse Crop Residue (GCR) torrefaction at various temperatures (200, 250, and 300°C) and times (15, 30, and 60 min). At 200°C, no significant impact on solid properties was observed. The most favorable conditions were noted at 263°C for 15 min. As the temperature increased to 300°C, the carbon content steadily rose from 34.02% to 43.78%, with a marked decrease in oxygen and a slight decline in hydrogen content. The resulting torrefied product had a high ash content (approximately 24%), making it more suitable for soil amendments than as a fuel source. Although the combination of 300°C and 15 min yielded the highest Higher Heating Value (HHV) of around 20.5, the low mass yield made it less desirable. Both mass yield and energy yield were inadequate at this temperature-time combination. The torrefaction process enhanced the sample's calorific value by increasing carbon content and reducing volatile matter. Hydrogen content remained nearly constant regardless of the torrefaction temperature.

The value of sugarcane bagasse (SBG) was upgraded for the production of quality fuel in a study by Jarunglumlert et al. (2022a). The torrefaction was both a dry and a wet process. The wet torrefaction process achieved a notable reduction in ash content, as it witnessed a less than 1% ash content above 180°C, making it a better fuel quality. The wet torrefaction process was also characterized by a higher yield than dry torrefied pellets. The heating value of both WT and DT ranged from 15.84-17.46 MJ.kg^{-1}; the raw baggase

was 7.53 MJ.kg^{-1} and the dry baggase was 15.04 MJ.kg^{-1}. Torrefaction was observed to enhance the calorific value by 5.0-17.9%. At high temperatures, the product heating values were enhanced, while mass yields were lower. Nevertheless, the specific energy demand of WTP production is almost double that of DTP.

Gaur & Pooniat (2024) improved the biochar quality of invasive weed (*Crotalaria burhia*) under the optimised condition of pyrolysis temperature:450°C, residence time of 1h. This process enhanced the carbon content of the waste biomass remarkably from 39.59-57.77% with a nose dive in hydrogen and oxygen content, resulting in a very low H/C and O/C ratio of 0.10 and 0.47, respectively. It also witnessed an astronomical increase in fixed carbon content from 19.09-81.24% and a major decline in volatile content from 70.26% to 8.48%. The process achieved a good biochar quality, which is suitable for enhancing soil fertility and carbon sequestration.

ROLE OF CARRIER GASES ON THE QUALITY OF AGRO-WASTE-DERIVED SOLID FUEL

N_2, CO_2, and a gas mixture of air and CO_2 were used as carrier gases to torrefy corn cob under 250°C and 300°C for 1h (Lu & Chen 2013). All carrier gases showed solid product characteristics near to those of coal. Both carbon dioxide and nitrogen carriers exhibited similar FC, HHV, mass, and solid yield at 300°C. As shown in Fig. 6 (a), all the carriers exhibited solid yield above 50 wt.% gas at 250°C torrefaction. However, air+co2 carrier gas shows a solid yield of about 45.4 wt.% at 250°C. At 300°C, declined below 50wt% (by air + CO_2, N_2 and CO_2). The effect of the torrefaction temperature on the solid yield was observed to be more than that of the carrier gas. Fig. 6(b) shows that across all the carrier gas choices, the HHV (average) of corncob waste progresses from about 23.3 MJ.kg^{-1} to -26.8 MJ.kg^{-1}, respectively, at 250°C and 300 °C; 1h as shown in Fig. 6 (c). These results confirm that CO_2 or air + CO_2 carrier gas can torrefy corncob waste. It was also observed that the kind of carrier gases also affects the amount of VM removal, and higher temperature with air+CO_2 taking the lead, however, with a subsequent reduction in FC generation.

Empty fruit bunches (EFB) were torrefied in a study by Uemura et al. (2017) under biomass combustion gas and nitrogen atmosphere at 473, 523, and 573 K. It was observed, as shown in Fig. 7, that the mass yield of torrefaction in the

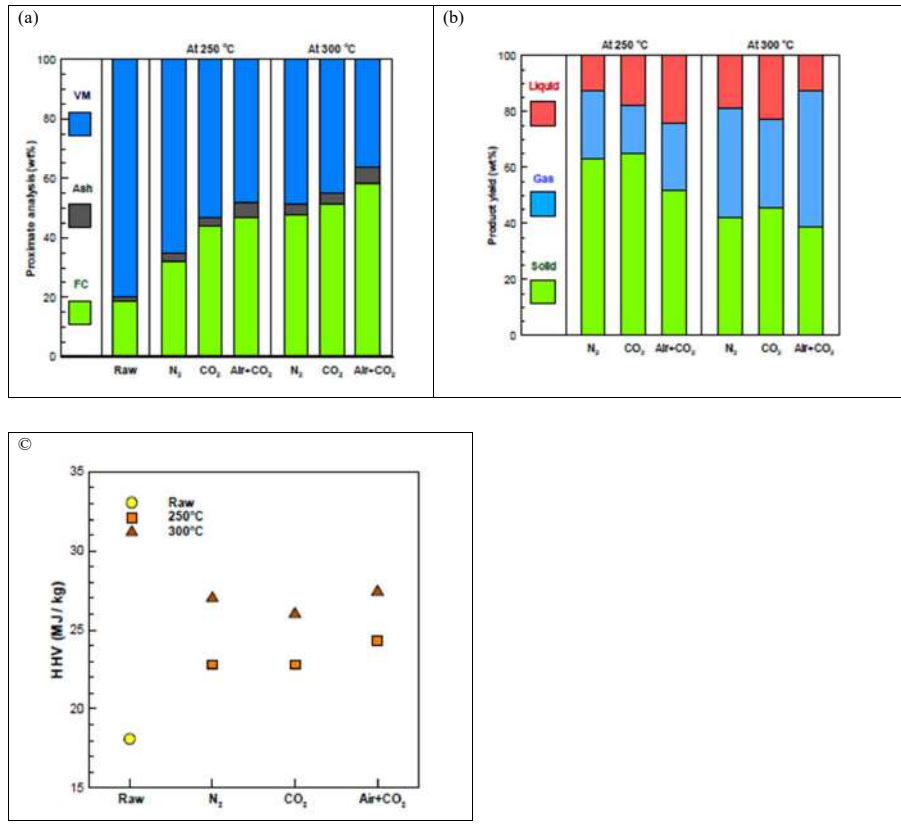

Fig. 6: (a)Proximate analyses of corncob waste, (b) Torrefaction product yields from corncob waste, (c) HHV profile of corncob waste (Lu and Chen, 2013).

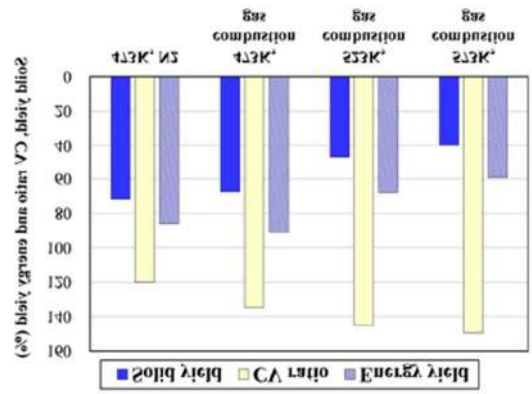

Fig. 7: Solid, calorific value and energy yields for torrefaction of EFB (Uemura et al. 2017).

nitrogen atmosphere is greater than that of combustion gas. O_2 and CO_2 decomposed more in the combustion gas. With a combustion gas atmosphere, the mass yield of torrefied EFB reduces with temperature increase. The torrefied EFB has a smaller mass yield of 67% in the combustion gas as temperature increases than that which was torrefied in N2 (72%), as O_2 and CO_2 enhanced decomposition in the combustion gas. This hence attests that combustion gas can help save energy.

Corncob was torrefied to charcoal by Li et al. (2018) under N_2 and CO_2 atmosphere at 200-300°C. Mass yields declined from 95.03-69.38% to 94.99-67.20% and increased HHV of 16.58-24.77 MJ.kg^{-1} and 16.68-24.10 MJ.kg^{-1} were obtained under N_2 and CO_2, respectively, within the torrefaction temperature. Hemicelluloses were not detected at a high temperature of 300°C. The C-concentration rises with increasing temperature from 200-300°C, while H and O concentrations decline. Corn cob torrefied at 260°C under CO_2 was observed as the most suitable condition. In the N_2 atmosphere, C-contents increased from 48.15-53.97%, accompanied by a decline in the H and O contents from 5.94-5.70% and from 45.91-40.33%, respectively. In the CO_2 atmosphere, the C-contents of the samples rose from 48.52-55.47%, while the H and O contents declined from 5.92-5.89% and 45.60-38.61%, respectively. The report clearly shows the greater role temperature plays than gas in the cellulose and hemicellulose decomposition.

Yard waste was valorised by Jaideep et al. (2021) to obtain solid fuel by torrefaction at 170, 200, 250, and 300°C; under different atmospheres of flue gas, CO_2 and N_2. As the temperature advances in the process, the mass yield declines. The highest mass yield was recorded in a flue gas atmosphere, while the lowest mass yield was with N_2. However, CO_2 carrier gas recorded the highest energy value (HHV) enhancement from 15.6- 22.2 MJ.kg^{-1} at 300°C, and 98.1% energy yield. No visible property changes were reported for flue gas at 250°C. At 300°C, hemicellulose was completely degraded while cellulose was partially degraded. N_2 and CO_2 degrade the biomass much better than flue gas, as confirmed in other analyses. The energy yield using fuel gas is relatively constant across the temperatures, which defies the common trend, which is a lowering mass yield and energy yield with temperature increase. Despite the improvement in HHV, energy yield for NO_2 and CO_2 declined as temperature increased. The C content of 40.58%, H content of 5.08% and N content of 1.22 wt.%, N of EFB samples with 15.15 MJ.kg^{-1} HHV were obtained. This shows that the combustion gas decomposes the EFB better than pure N_2.

Oil palm fiber pellets (OPFP) were torrefied in a study by Chen et al. (2016) under inert and oxidative atmospheres at(275-250)°C, O_2 concentration of 0-10 vol.% and duration of 30 min. The HHV of the biomass was enhanced significantly at 275°C in the oxidative environment, more than in the non-oxidative environment. However, at 300°C, regardless of the atmosphere, torrefied OPFP improved the fuel quality of the biomass. HHV of OPFP improved from 18.37- 20 MJ.kg^{-1}. At OPFP, torrefied in N2 attained HHV of 20.33 MJ.kg^{-1}. At 5% and 10% in the O_2 environment, the HHV of 22.22 and 22.59 MJ.kg^{-1} was reached. It was therefore established that an inert environment supports the possibility of increasing the HHV of OPFP by temperature increase. However, in oxidative torrefaction, higher temperatures do not enhance the HHV.

Empty fruit bunches (EFB), mesocarp fiber (MF), and kernel shell(KS) were torrefied as a solid fuel (Uemura et al. 2011). High energy yield values of 96% and 100% were achieved for MF and KS, respectively, while EFB shows a poor yield of 56%.HHV increased from 17.02 MJ.kg^{-1} to 20.41 MJ.kg^{-1} in the process. Also, 19.61 MJ.kg^{-1} for dried mesocarp fiber to 22.17 MJ.kg^{-1} at 300°C. Similarly, the Kernel shell has HHV of 19.78 MJ.kg^{-1} - 21.68 MJ.kg^{-1} at 300°C torrefaction. The decrease in H2 and O2 as

temperature rises due to dehydration and de-carbon dioxide from the biomass. The carbon content of the dried EFB moved from 45.53-49.56wt.%, and the H Content declined from 5.46-4.38 wt.%. The mesocarp fiber also has increased C-content from 46.93 to 48.68 wt.%, and an H content of 5.50 wt.% to 4.87%. The kernel shell C-content shifted from 45.87 wt.% to 54.21 wt.% while H content declined from 6.31-5.08 wt.%. A steady decline in mass yield as the temperature increases, with EFB as the highest decreasing ratio, and kernel shell has the lowest.

Pimchuai et al. (2010) torrefied some agricultural waste in N_2 at 250-300°C and 1-2 h. Maximum HHV of 25.68 $MJ.kg^{-1}$ was obtained at 300°C; 1.5 h for bagasse (comparable to HHV of lignite), least (21.02 $MJ.kg^{-1}$) at 250°C; 1 h. All the agricultural waste showed high HHV at 300°C;1.5h, rice husks (17.77 $MJ.kg^{-1}$), sawdust (23.94 $MJ.kg^{-1}$), peanut (19.1 $MJ.kg^{-1}$), water hyacinth (14.33 $MJ.kg^{-1}$). At severe torrefaction conditions, the moisture content and volatiles decreased. However, the fixed carbon content and ash content upwardly trend with higher temperature but decline massively as the residence time extends. No significant changes occur in volatile matters at 250°C, 1 h for all the residue. Mass and energy yields were reduced by about 41-78% and 55-98% of their initial values. The highest torrefaction temperature produces the lowest mass and energy yield.

The conditions of temperature and time in the torrefaction process were investigated on Norway spice stem wood, stump, and bark (Wang et al. 2017). The role of temperature was visible as the mass yield decreased across the temperature profile, which was significant and is associated with hemicellulose decomposition. At 300°C, stem wood lost was 30 and 42 wt%% with holding time of 30 and 60min, whereas the stump is 44 wt%.% and 54 wt%. A very slight reduction was witnessed with cellulose contents of the stem wood and the stump at 275°C. However, the cellulose content drastically reduced at 275 °C, with only a negligible remnant at 300°C torrefied biomass.

The effect of operating conditions on torrefied olive tree pruning was experimentally examined by Martín-Lara et al. (2017). At 300°C, 60 min, the fuel ratio shifted from 0.23-0.39, improving its fuel quality. O/C-1.02 (raw) reduced to 0.90 while H/C-0.17 (raw) to 0.15 at 300°C; 10 min. The decline in H/C attests to the moderate increase in the carbon content compared to other elements, and that of O/C is connected to the production of volatiles such as CO, CO_2, and H_2O. The elemental composition also confirms the shift of the native olive sample from that of lignocellulosic biomass to that of coal. The HHV of the biomass increased tangibly from 17.32 $MJ.kg^{-1}$ (native olive tree) to 20.50 $MJ.kg^{-1}$ at 200°C; 60 min torrefaction condition. However, the HHV drops at higher temperatures and longer residence times. The hemicellulose was strongly degraded in N2 atmosphere at high torrefaction conditions, and the thermal stability of cellulose was modified. The volatile content declined from 72.9% (200°C;10 min) to 69% (300°C; 60min), and the fixed carbon improved from 20.4% at 200°C; 10 min to 27.2% at 300°C; 60 min.

Martín-Pascual et al. (2020) numerically modelled olive tree waste biomass under torrefaction conditions of (200-300°C) and (0-120 min). Advancement in HHV of the torrefied sample was noticed within the 200-275°C range. However, the reverse was witnessed at 275-300°C, as the HHV declined. There were no remarkable differences in HHV with residence time at low temperatures, except for 120 min. It was generally inferred that the temperature shortens the time required to reach maximum HHV. It was then concluded that the optimum condition was 275°C, 30 min, with an optimum of 5830 $cal.g^{-1}$ HHV. A mass yield between 97.48-57.61% was achieved, which declines with the increasing residence time and temperature.

Oil palm agricultural residues, which are oil palm fronds (OPF)- non-woody biomass and *Leucaena leucocephala* (LL) -woody biomass, were also torrefied by Matali et al. (2016). The experiment was carried out within 200-300°C, and 60 min in an anoxic condition. At 300°C, O_2 and H_2 declined by 28% and 34% for torrefied OPF and LL, respectively, while C-content improved by about 37% for both torrefied samples. Fixed carbon was more than twice for all torrefied biomass, with OFP having the highest at 54 wt.%. H_2 and O_2 content with torrefaction was connected to the destruction of the (-OH) group in biomass samples, producing solid hydrophobic fuel. Mass yield experienced a 50% decline, with the raw biomass at 300°C for both OPL and LL. This was due to moisture removal and the release of volatiles such as hemicellulose and short-chain lignin compounds. Energy yield value reduced from 99.9 wt.% (OFP-200°C) to 71.2 (OFP-300°C); 29 wt.% and 40 wt.% for torrefied OPF and LL, respectively. HHV of torrefied OFP and LL at 300 °C; 60min improved from 18-25 $MJ.kg^{-1}$, for raw OFP and LL, comparable to sub-bituminous coal. Torrefied OPF was enhanced in energy densities by a factor of 1.42, while LL, respectively, by 1.39 at 300°C.

Wet torrefaction (WT) process was carried out on rice husk from 150-240°C for 60 min (Zhang et al. 2017). The mass yield decreased from 86.7-47.9% within the range of 150-240°C. Likewise, the energy yield during WT had a greater value than that of mass yield. Energy density was enhanced via torrefaction as it increased from 1.01 (150°C) to -1.12 (240°C). The HHV value was improved

from 16.2 MJ.kg^{-1} (RH) to 18.1 MJ.kg^{-1} at 240°C and a C-content enhanced from 40.8% (RH) to 45.8% (240°C). The atomic ratio O/C of RH declined from 0.74-0.54, and H/C shifted from 1.68-1.36, which was due to the dehydration, decarboxylation, and demethanation reaction.

High-energy-yield biocoal was obtained by torrefaction of rice straw within 250-400°C and an isothermal time of 30 min in a nitrogen atmosphere by Pandey et al. (2019). The optimization of the process parameter brought about a desirable HHV of the torrefied product (bio-coal), which was equivalent to that of bituminous coal in thermal power plants. The energy yield decreases steadily from 75% at 250°C to 62% at 400°C with temperature increases. GCV of raw rice straw powder was enhanced from 3640 kcal.kg^{-1} (untorrefied), 3762 kcal.kg^{-1} (250°C), 4342 (300°C), 5129 kcal.kg^{-1} (350°C) and 5339 kcal.kg^{-1} (400°C).

At 200-250 °C, structural deformation and decoloration occur from light brown to dark brown. Above 300°C, a destructive drying phase where exothermic reaction and gas production (CO and other hydrocarbons) increase takes place, transforming the product from dark brown to black due to carbonization and devolatilization. As shown in Fig. 8 (a,b), the DTG peaks around 290-327°C, thermal degradation of hemicellulose and cellulose results in the release of volatile matter. At torrefaction 350°C and 400°C, the peak of hemicellulose and cellulose is almost extinct, an indication of total degradation of both hemicellulose and cellulose, while lignin is partially retained. The peak between 440-480°C denotes the lignin.

Coffee residue, sawdust, and rice husk were also torrefied to examine their solid fuel properties in a study carried out by Chen et al. (2012) at 240°C and 270°C, 0.5 and 1 h. The

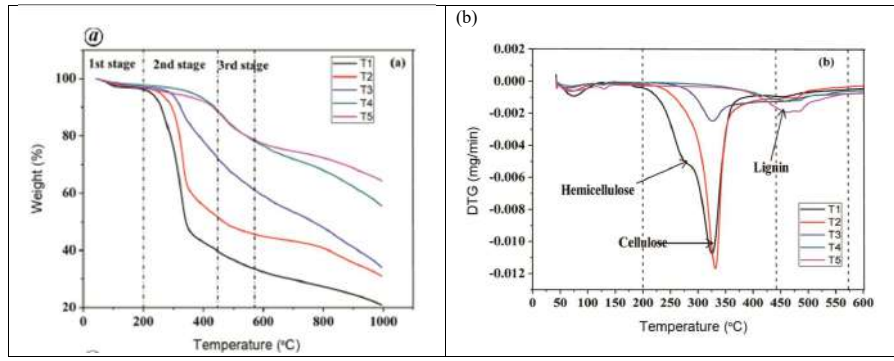

Fig. 8:(a) TGA and (b) DTG curve of raw rice straw and torrefied product. (Pandey et al. 2019).

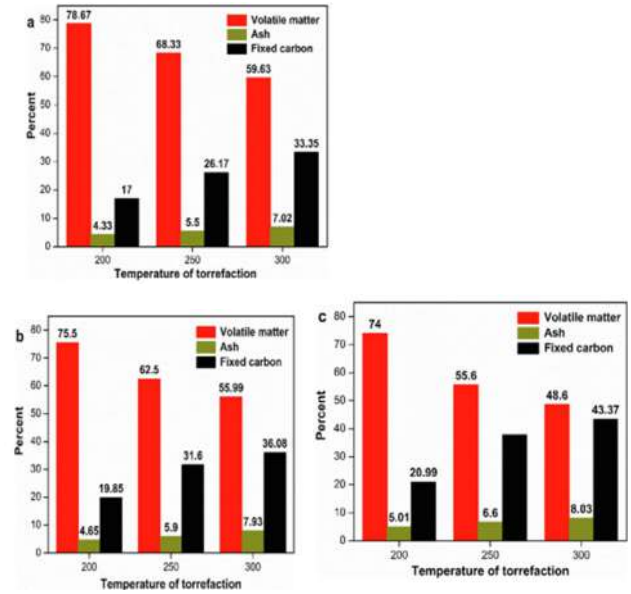

Fig. 9: Proximate analysis of torrefied MCR for: (a) 30 min; (b) 45 min; (c) 60 min RT at different torrefaction temperatures (Patidar & Vashishtha 2021).

result was compared with high-volatile bituminous and low-volatile coal. HHV of the coffee residue increased from 20.2 $MJ.kg^{-1}$ - 28 $MJ.kg^{-1}$ at 270°C, 60min. It was observed that coffee residue has more hemicellulose content, which makes it the most active biomass with improved HHV up to 38%. The properties of the torrefied biomasses were close to high-volatile coal at higher torrefied temperature and duration.

(Teh & Jamari 2016 torrefied rice husk and rice straw biomass at 220, 250, and 280 °C, 30 min and under the heating rate of 15 $°C.min^{-1}$. HHV of rice husk increased from 17.67 - 21.46 $MJ.kg^{-1}$ at 280°C. The HHV for rice straw was enhanced from 18.32 - 21.14 $MJ.kg^{-1}$ at the 280°C torrefied state. The energy yield of the torrefied rice husk was 93.47%,95.41% and 92.51% at 220,250 and 280°C. Rice straw has an average energy yield of 93.77%, 98.83% and 98.41% at 220, 250 and 280°C. An optimal temperature of 250°C gave the most valuable biofuel.

Mustard crop residue MCR was characterized and torrefied by Patidar & Vashishtha (2021) at 200, 250, 300°C and 30, 45, 60 min. The Highest mass yield was 95.54% at 200°C, 30 min; the lowest yield was 64.5% at 300°C,60 min. Also, the energy yield from 95.54% (200°C, 30min) to 65.23% (300°C, 60 min). The percentage of carbon also increases with the severity of torrefaction due to the release of volatiles. The HHV increases from 16.92 $MJ.kg^{-1}$ (MCR raw) - 21.94 $MJ.kg^{-1}$ for torrefied MCR (300; 60min). It experienced different stages of decolouration due to the thermo-degradation of the biopolymer and the oxidative reaction between the MCR and the atmosphere. As the temperature increased from 200-250°C, light volatiles were emitted, while hemicellulose and light aliphatic compounds were degraded. The effect of torrefaction conditions is presented in Fig. 9.

Sadaka and Negi (2009) enhanced the bioenergy properties of straws and wasted cotton gin feedstock by torrefaction at 260°C and varied time (0-60) minutes. In another phase of the experiment, wheat straw was torrefied at (200, 260, and 315 °C) and (60, 120, and 180 min). At 260°C, across all the residence time, there was no tangible decline in volatiles for wheat and rice straw. However, the HHV of wheat straw, rice straw, and cotton gin waste was enhanced by 15.3%, 16.9%, and 6.3% at 60 min. At 260°C, 60min, rice straw recorded the highest weight loss (30.7%) while cotton gin waste showed the lowest weight loss, due to its higher amount of lignin content than wheat and rice straw. Wheat straw showed a rise in HHV from 16.60- 22.75 $MJ.kg^{-1}$ at 315°C, 180 min. It was also obvious that the torrefied wheat biomass became very dark as the temperature and time advanced. It also experienced a decline in the mass yield at higher temperatures.

THE ROLE OF PRESSURE ON THE QUALITY OF AGRO-WASTE-DERIVED SOLID FUEL

Rice straw was also torrefied by Seithtanabutara et al. (2023), in a bid to know the role of pressure in enhancing its fuel properties. An initial investigation was carried out under (- -0.4, 0.4, 0.8 and 2 bar), 200°C, and 40 min. Although torrefied products are dark compared with the raw material, the product from (- -0.4 bar) is slightly darker and more brittle compared to the torrefied product of 0.8 bar and the 2 bar, respectively. It was attributed to negative pressure causing easier wall explosion than positive pressure, promoting better decomposition of the biomass structure. Torrefaction at -0.4 bar has a lower mass yield than higher-pressure torrefaction. However, there were no noticeable differences in SEC for negative and medium positive pressure. At 0.8 and 2.0 bar pressures, 0.8 and 2.0 bar pressures have similar mass yields. Moreover, the 2.0 bar torrefied sample has higher HHV and, consequently, a higher EDR. Hence, torrefaction at 2 bar pressure produces the highest energy yield of 94.95% and EC (25.43 $Wh.g^{-1}$), high. At -0.4 bar, the lowest energy yield of 92.93% and the lowest energy consumption of 24.79 $Wh.g^{-1}$ were obtained. This indicates that the performances of EY and SEC are dependent on torrefaction pressure.

INFLUENCE OF BULK ARRANGEMENTON THE PERFORMANCE OF TORREFIED AGRICULTURAL WASTE BIOMASS

Soponpongpipat & Sae-Ueng (2015) confirmed that biomass bulk arrangement affects the decomposition pathway of sugarcane trash in a torrefaction. The experiment was performed within a temperature range of 250-290°C and for 60 min. Untreated biomass has an HHV of 16.08 ± 0.23 $MJ.kg^{-1}$. However, as the temperature progresses, the HHV rises from 18.55 - 20.76 $MJ.kg^{-1}$ to 250-290°C in the hollow bulk arrangement. In the dense bulk configuration, the Higher Heating Value (HHV) of the torrefied biomass ranged from 20.18 to 23.87 $MJ.kg^{-1}$ as the temperature increased from 250 to 290°C. Comparatively, the dense bulk arrangement yielded higher HHV values than the hollow bulk setup across all temperature ranges. The dense bulk arrangement facilitates an autocatalytic decomposition pathway, leading to more extensive decomposition. In the hollow bulk configuration at 250°C, the rate of weight loss (Mt/Mo) decreased rapidly with increasing time until 400 seconds. Conversely, in the dense bulk density setup, weight loss declined quickly until reaching 700 seconds, after which it stabilized at a constant level.

The impact of the dry and wet torrefaction process with an additive was compared on the pyrolysis performance of

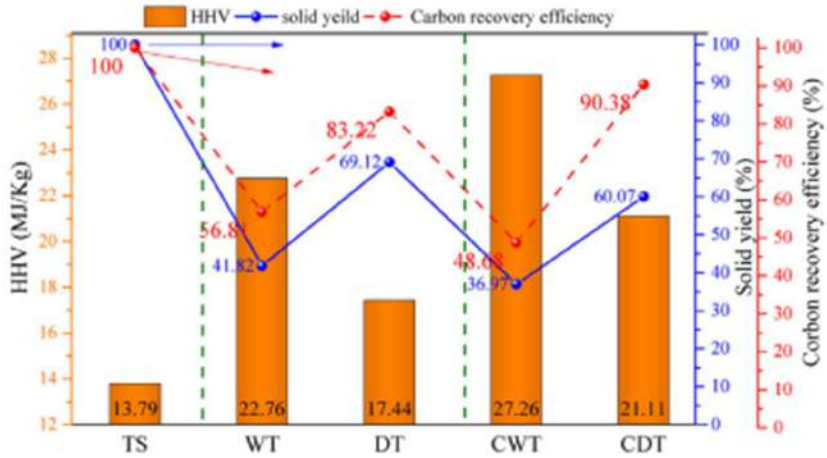

Fig. 10: HHV, solid yield and CRE of torrefied biochar (Sun et al. 2019).

tobacco stalk Sun et al. (2019). In this study, HHV improved after the wet/dry torrefaction process, with WT having 22.76 MJ.kg^{-1} HHV and DT having 17.44 MJ.kg^{-1}. It was also observed that additives enhanced HHV from 13.79-27.26 MJ.kg^{-1}.

As shown in Fig. 10, DT and CDT have a much higher solid product of 69.17% and 64.19% respectively, compared to wet torrefaction (WT)-41.78% and catalytic wet torrefaction (CWT)-36.48%. It was also observed that both dry and wet torrefaction decreased the H/C and O/C of the product, with the torrefied sample's aromaticity in the coal range. However, CWT showed a more significant decrease in H/C and O/C from 1.89-0.24 and 1.89-0.55, respectively. WT has more heat, mass transfer and contact area than DT.

In addition, catalytic torrefaction(wet/dry) produces a lower yield than non-catalytic, converting TS into bio-oil and non-condensable gases during torrefaction. There is a significant decline in the hemicellulosic content for both processes. However, it was converted to gas and liquid products after CWT or CDT. Hence, WT with additives effectively improved the energy value of torrefied biochar.

The torrefied lemongrass (Cymbopogon citrates) residue was examined by Tan et al. (2017) using microwave-induced torrefaction at 200 -300 ^{o}C in an anoxic atmosphere. This process helps to improve the HHV of raw biomass, which was 17.93 MJ.kg^{-1} - 19.37 MJ.kg^{-1} at 300 ^{o}C, with a 37.7% increase in fixed carbon of lemongrass residue. From raw lemon grass residue to (300 ^{o}C;30 min) H/C moves from 0.28-0.24, a 14.3% increase and O/C 0.80-0.32, a 60.0% increase, respectively, were observed. Also, the mass and energy yield of the torrefied lemongrass residue declined from 81.50%-61.20 and 83.85 to 66.11% respectively. The physical appearance of the untreated lemon grass visibly changed from medium brown to dark brown at 300°C. The decoluration due to exothermic reaction in this process led to the loss of moisture content, CO_2, large amounts of acetic acid and phenols. The C-content improved slightly from 47.18 wt.% (untreated) to 49.05% at 300°C,30min. However, the H content declined moderately from 13.12 wt%-11.95 wt% at 300°C,30min. Moreover, the O content massively declined from 37.93wt% to 15.63 wt%. Table 1 shows the calorific/energy value of common agricultural residues.

RELEVANT ADVANCES ON THE TECHNO-ECONOMIC IMPLICATION OF AGRO-WASTE TORREFACTION

Integrated Torrefaction, Scaling Up and Pelleting Process Approach for Economic Feasibility and Environmental Impact

The constraint on torrefaction of biomass and its preference over coal is its cost implication. However, the larger plant capacities with an integration system will serve to cut down cost, and achieving carbon credits can improve economic viability (Niu et al. 2019). This will help to reduce costs and improve market penetration (Kumar et al. 2017).

The torrefaction process also helps achieve a reduction in carbon emissions when carbon capture and utilization processes are incorporated into the facility. Instances are presented by Cutillo et al. (2024) on the integration of torrefaction with chemical looping combustion and methanation to achieve net-zero or negative carbon emissions. According to Pirraglia et al. (2013), carbon credit serves as a route to increase income on the internal rate of return (IRR) and net present value (NPV) per metric ton of a torrefied biomass. Batidzirai et al. (2013) also attested that technological scaling up can help achieve a 50% reduction in total cost and cut down production costs.

Table 1: Calorific/Energy value of Common Agricultural Residues.

References	Process conditions	Calorific value	Common agricultural waste residue/ feedstock	Mass Yield (M.Y)/ Energy yield (E.Y)
(Abdullah et al. 2022)	230-300)°C; (30-90)min;Muffle Furnance under N_2; Dry torrefaction	(22-25) MJ.kg^{-1} 27 MJ.kg^{-1}-300 C	Walnut shell (WS) 30%: Pearl Millet (PM) 70%	M.Y: 41-91%
(Yang et al. 2015)	(180-260)°C; stainless steel batch reactor; Wet torrefaction	17.5-25.3 MJ.kg^{-1} 17.7-25.7 MJ.kg^{-1} 18.4-23.6 MJ.kg^{-1}	Humulus Lupulus (HL) Plumena Alba (PA) Calophyllum Inophyllum (CIL)	M.Y: 26.5% (HL) M.Y: 31.5% (PA) M.Y; 50.9% (CIL)
(Yue et al. 2017)	(250-300)°C; steel batch torrefaction reactor, electric furnace; Wet torrefaction	17.33-23.62 MJ.kg^{-1} 16.45-26.88 MJ.kg^{-1}	Energy sorghum (ES) Sweet sorghum baggase(SSB)	M.Y: 43-65% (SSB) M.Y: 51-70% (ES)
(Zhang et al. 2016a)	(130-220)°C; autoclave reactor; Wet torrefaction	14.34-19.84 MJ.kg^{-1}	Duckweed	M.Y:30.4-64.8% E.Y: 40.1-77.9%
(Akhtar et al. 2021)	(200-320)°C 10-60min; Tube furnace, N_2, Dry torrefaction	3600-5444 kcal.kg^{-1} (290 °C; 20 min) 3696-4481 kcal.kg^{-1} (270°C;30 min) (3435-4370) kcal.kg^{-1} (260°C, 60 min)	Corncob (CC) cotton ball (CB) sunflower (SF)	M.Y: 45-54% (CC) M.Y; 71-84% (SF) M.Y; 44-88% (CB)
(Jifara Daba & Mekuria Hailegiorgis 2023)	(200-300)°C; 15-45 min; Muffle furnace CO_2 gas; Dry torrefaction	14.80-23.37 MJ.kg^{-1} 16.54-24.95 MJ.kg^{-1},	Khat stem Corncob	E.Y-98.5% (CC) E.Y-94.9% (KS)
(Chen et al. 2015)	(220-280)°C; Tubular furnace, N_2 flow	16.53 -20.31 MJ.kg^{-1}	Cotton stalk	----------
(Alcazar-Ruiz et al. 2022)	(280-320)°C, 500°C	22.56- 17.93-	Olive pomace (OP) Almond shell (AS)	----------
(Bach et al. 2013)	(175-225)°C; 10-60min; 15.54-250 bar; Wet torrefaction; Benchtop autoclave reactor	19.94-~20.42 MJ.kg^{-1}	Norway spruce (softwood) Birch (hardwood)	S.Y: 76.4-731.1(15.54-250 bar) S.Y:88.3-69.7(175-225 degC/spruce) S.Y:79.0-58.0(175-225 degC/spruce)
(Benavente & Fullana 2015)	(150-300) °C; 2h; oven model UFP500 from Memmert GmbH	26.4-30.0 MJ.kg^{-1}	Torrefied olive mill waste (TPOMW)	S.Y: 35-98%
(Cetinkaya et al. 2024)	(250-290)°C; 15-60min; Ash furnace (Nüve MF 5000).	19.8-21.2 MJ.kg^{-1} (290°C;60 min). 18.3 MJ.kg^{-1} -21.3 MJ.kg^{-1} (290 °C;60 min)	Rosa Damascena Mill solid waste (RP) Red pine sawdust (PS).	M.Y: 89-57% (RP) M.Y; 90-63% (PS)
(Chiou et al. 2015)	(200, 230, and 260)°C; 230, 260, and 290 °C An Isotemp muffle furnace under N_2		Apple (A) grape pomace (G), olive and tomato pomace (T)	-
(da Silva et al. 2022)	(200, 250, and 300°C); (15 and 60 min)	18.83-20.78 MJ.kg^{-1} 20.48-22.97 MJ.kg^{-1}	Passion fruit peel waste (PF) Pine-apple fruit waste (PA)	S.Y:77-81% (PF/200C);41-44% (300C) S.Y: 56-61% (PA/300C); 87-90%(200C)
(Dhungana et al. 2011)	250-280°C; 15-60 min Muffle furnace	19.18-24.20 MJ.kg^{-1} 19.89 MJ.kg^{-1} 20.16 MJ.kg^{-1}	non-lignocellulose biomass switch grass coffee husk	86.35- 96.60% (250C);62.80-89.85% (280C) 74.84-94.53% (250C); 56.84-88.88% (280C) 86.06-98.68% (250C);73.71-97.73% (280C)
(Duman et al. 2020)	200-350°C;120min 500 mL batch reactor.	23.4-26.5 MJ.kg^{-1} 20.9-28.4 MJ.kg^{-1}	Olive pruning (OP) Vineyards pruning(VP)	HY;42-60% (VP); 42-58% (OP)

Table Cont....

References	Process conditions	Calorific value	Common agricultural waste residue/ feedstock	Mass Yield (M.Y)/ Energy yield (E.Y)
(Duranay et al. 2023)	300°C+/-5C; 41min; cylindrical tube furnace	----------	almond hulls Almond shells, Olive seeds corn stalks	M.Y; 53.4% (AH) M.Y; 80.8% (AS) M.Y: 78.4% (OS) M.Y: 43.7% (CS)
(Granado et al. 2023)	(250ºC; 90 min)	17.8-19.2 MJ.kg^{-1} 16.8-18.4 MJ.kg^{-1} 16.9-19.0 MJ.kg^{-1}	Cassava rhizome, sugarcane bagasse sugar cane straw	77.7% (CR) 62.5% (SB) 58.0% (SC)
(Zhang et al. 2016a)	200-320°C; (15-120 min)	21.13 MJ.kg^{-1} -25.96 MJ.kg^{-1}	Auricularia auricula-judae (wood ear)	----------
(Huang et al. 2017)	(100 W; 250W) (15-30min);single-mode microwave oven	21.18-29.65 MJ.kg^{-1}	Leucaena, woody biomass	M.Y: 17.27-72.3% E.Y: 27.87-83.0%
(Ianez-Rodriguez et al. 2017)	(200, 250 & 300°C) (15-60 min).	17.75- 20.5 MJ.kg^{-1}	Greenhouse Crop Residue (GCR)	
(Jarunglumlert et al. 2022a)	240-300°C	15.84-17.46 MJ.kg^{-1}	sugarcane bagasse (SBG)	----------
(Lin et al. 2021)	(210- 300°C); (30 and 60 min; A steel batch torrefaction reactor	19.9 MJ.kg^{-1}-27.7 MJ.kg^{-1}; 19.1 MJ.kg^{-1}-23.3 MJ.kg^{-1}	Ananas comosus peel (ACP) Annona squamosa peel (ASP)	48-73.3% 48-87.3%
(Lu and Chen, 2013)	250°C; 60 min 300°C; 60 min; An electric furnace.	22.8-24.3 MJ.kg^{-1} 27.0-27.4 MJ.kg^{-1}	Corncob	>50% (250C) <50% (300C)
(Uemura et al. 2017)	473, 523 and 573 K, N_2, vertical tubular reactor	22.6 MJ.kg^{-1}	Empty fruit bunches (EFB)	E.Y: 91% (473k) S.Y: ~70% (473k)
(Li et al. 2018)	200-300°C; Horizontal tubular quartz tube reactor heated by a furnace	16.58–24.77 MJ.kg^{-1}-N2 atm 16.68–24.10 MJ.kg^{-1}CO2 atm	Corncob	M.Y: 69.36-95% (N2) M.Y: 67.2-95% (C02)
(Jaideep et al. 2021)	170, 200, 250, and 300°C	15.6- 22.2 MJ.kg^{-1}	Yard waste	M.Y: 60-87.6%; E.Y:81-95% (N2) M.Y: 61-91%;E.Y: 98-103% (CO2) M.Y: 73-89% ; 86.2-86.7%(flue gas)
(Chen et al. 2016)	(275-250)°C	18.37-20 MJ.kg^{-1}-N2 22.22-22.59 MJ.kg^{-1}-O2	Oil palm fiber pellets (OPFP	S.Y:43 -65 wt.%
(Uemura et al. 2011)	300°C; Horizontal tubular type reactor	17.02-20.41 MJ.kg^{-1}. 19.61- 22.17 MJ.kg^{-1}. 19.78 - 21.68 MJ.kg^{-1}	Empty fruit brunch (EFB) Mesocarp fiber (MF) Kernel fiber	M.Y:24.16-43.16% (EFB) M.Y: 52.46-63.08% (MF) M.Y: 71.27-77.44% (KF)
(Pimchuai et al. 2010)	250-300°C and 1-2 h; Muffle furnace, N_2	21.02-25.68 MJ.kg^{-1} 15.89-17.81 MJ.kg^{-1} 19.55-25.09 MJ.kg^{-1} 16.35-19.36 MJ.kg^{-1} 12.68-14.33 MJ.kg^{-1}	Baggase Rice husk Saw dust Pea nut Water hyacinth	M.Y: 41-78% E.Y; 55-98%
(Wang et al. 2017)	275°C and 300°C; bench-scale tubular reactor.		stem wood, stump bark woody biomass	M.Y: 60-90% (SW) M.Y; 46-90% (S) M.Y: 56-90%(W)
(Martín-Lara et al. 2017)	200-300°C; Electric muffle furnace	20.09-20.50 MJ.kg^{-1}	torrefied olive tree	M.Y: 57.0-87.4% E.Y: 64.0-101.5%
(Martín-Pascual et al. 2020)	(200-300°C); (0-120 min); Thermogravimetric analyser; N_2	4884-5893 Cal.g^{-1} HHV	olive tree waste	M.Y: 57.61-97.48%

Table Cont....

References	Process conditions	Calorific value	Common agricultural waste residue/ feedstock	Mass Yield (M.Y)/ Energy yield (E.Y)
(Matali et al. 2016)	200-300°C; 60 min; Horizontal furnace with 80mm-IDquartztube reactor,	18.58 -25.16 $MJ.kg^{-1}$ 18.31-24.92 $MJ.kg^{-1}$	oil palm frond (OPF), *Leucaena leucocephala* (LL) -woody biomass	M.Y: 43-92% ; E.Y: 60.1-93.9% (LL) M.Y:50-95%; E.Y:71.2-99.9% (OPF)
(Nam and Capareda, 2015)	(210, 250 and 290°C) (20, 40, and 60 min) bench-scale batch type Parr pressure reactor; N_2	20.3-28.6 $MJ.kg^{-1}$ 19.3-23.3 $MJ.kg^{-1}$	Rice straw (RS) cotton stalk (CS)	M.Y:76.61-91.3% (RS) M.Y: 68.45-99.4% (CS)
(Zhang et al. 2017)	150-240°C; 60 min; high-pressure batch reactor, Wet torrefaction, N_2	18.8- -18.4 $MJ.kg^{-1}$	rice husk (RH)	M.Y: 27.4-42.7% (RH)
(Pandey et al. 2019)	(250-400)°C ; 30 min; Stainless steel (SS) Reactor; N_2	3762 -5370 $kcal.kg^{-1}$	Rice straw (RS)	M.Y: 42.01- 72.6% (RS) E.Y; 62- 75%
(Chen et al. 2012)	(240 and 270°C); (30 and 60min), Reactor; N_2	20.2 -28 $MJ.kg^{-1}$ 17.8- 18.4 $MJ.kg^{-1}$ 16.2-18.2 $MJ.kg^{-1}$	Coffee residue, rice husk sawdust	E.Y: 88-99%
(Teh and Jamari, 2016)	(220, 250 and 280°C) ;30 min, Tubular reactor; N_2	18.44 -21.46 $MJ.kg^{-1}$ 18.78-21.14 $MJ.kg^{-1}$	Rice husk Rice straw	M.Y:78.98-91.2%;E.Y: 92.51-95.41% (RH) M.Y: 82.28-89.85%; E.Y: 93.77-98.83% (RS)
(Patidar & Vashishtha 2021)	(200, 250 and 300)°C ; 30, 45, 60 min; Tube Furnace; N_2	16.92- 21.94 $MJ.kg^{-1}$	Mustard crop residue MCR	M.Y; 53.47-95.54%; E.Y: 65.23-97.7%
(Sadaka & Negi 2009)	(260)°C; 0, 15, 30, 45, and 60 min (200, 260, and 315°C) (60, 120, and 180 min); Bench scale reactor; N_2	16.2-18 $MJ.kg^{-1}$ 14-15.6 $MJ.kg^{-1}$ 16 $MJ.kg^{-1}$ 16.60- 22.75 $MJ.kg^{-1}$	Wheat straw, Rice straw Cotton gin waste Wheat straws	----------
(Seithtanabutara et al. 2023)	positive and negative pressure (0.4, 0.8 and 2); bar) at temperature and time of 200-220°C and 30-50 min; stainless-steel tube reactor; N_2	17.29 $MJ.kg^{-1}$ (-0.4bar) 17.64 $MJ.kg^{-1}$ (2.0bar)	Rice straw	E.Y: 94.95% max. (2 bar); 92.93% min (0.4 bar)
(Soponpongpipat and Sae-Ueng, 2015)	250, 270, and 290°C;60 min; stainless steel cylinder reactor; N_2	18.55 - 20.76 $MJ.kg^{-1}$ 20.18- 23.87 $MJ.kg^{-1}$	sugarcane trash- hollow bulk arrangement compact bulk arrangement	----------
(Sun et al. 2019)	240°C,1hr-DT/WT; N_2 With additive Fixed bed reactor with the quart tube	22.76 $MJ.kg^{-1}$ -WT 17.44 $MJ.kg^{-1}$-DT 27.26 $MJ.kg^{-1}$-(WT+additive) 21.11 $MJ.kg^{-1}$-(DT+additive)	Tobacco stalk	WT:41.2% (S.Y) DT:69.12% (S.Y) CWT:36.97% (S.Y) DWT:60.07% (S.Y)
(Tan et al. 2017)	200 -300°C; modified bench top microwave oven; N_2	17.93 - 19.37 $MJ.kg^{-1}$	lemongrass (Cymbopogon citrates) residue	M.Y: 61.20- 81.50%

A study by Zhao et al. (2024) reveals that the energy efficiency of torrefaction can be improved by its integration with steam gasification up to 58.9% which is higher than the direct gasification. Biomass is made an efficient fuel source by torrefaction, with its enhanced energy density and combustion characteristics.

Goyal et al. (2023) proposed an integrated system of torrefaction and pelleting process for rice straw. It synergizes torrefaction and pelleting steps into a single process, also harnessing the inherent natural lignin in biomass as a binder without the need for external binders and reducing process complexity. The economic analysis of this process revealed a return on investment (ROI) of 30%, a payout time of 2.4 years, and a break-even point of 42% at a selling price of $73 per ton of briquettes, indicating significant profitability potential.

Bampenrat et al. (2023) upgraded waste sugarcane bagasse (SBG) and palm kernel shell(PKS) through torrefaction, under a temperature of (225–300 °C) and residence time of (30–90 min). Torrefaction temperature

has a stronger implication on mass yield and calorific value than residence time. The optimal conditions of SBG and PKS were attained at 275 °C for 90 min, having bio-coal values (approximately 23 $MJ.kg^{-1}$) and energy yields of 73.93-77.41%. This makes it fit to be co-fired with coal in thermal power plants. The energy yield and calorific value prove its economic viability.

Shah et al. (2012) In a bid to assess the techno-economic feasibility of a production-scale torrefaction, analysed its mass-energy balance was analyzed. In this study, the net external energy required for the torrefaction process increased while the energy efficiency decreased with increasing moisture content. However, both energy metrics show a decreasing trend as process temperatures increase. The unit torrefaction process cost decreases with decreasing initial moisture contents and decreasing torrefaction process temperatures. For the typical moisture content of 30% wet basis (wb), process temperature of 240°C, plant operating window of 6 mo yr^{-1} and initial capital investment of $7.5 million for the system with rated capacity of 25 Tton. hr–1, the unit torrefaction process cost was estimated to be 17.5 $\$\text{-Tton}^{-1}$. Additional system improvements through capital cost reduction and wider operating windows can yield a torrefaction product cost of ~12 $\$\text{-Tton}^{-1}$.

Jarunglumlert et al. (2022b) measured the impact of the torrefaction process on the ash content and overall quality of the pellets from sugar cane bagasse. Wet torrefaction was found to significantly reduce ash content to 1% at temperatures above 180°C, resulting in higher quality and more marketable fuel pellets compared to dry torrefaction. An economic feasibility analysis revealed that the production of wet torrefied fuel pellets yields greater net present value and profitability than dry torrefied pellets, indicating that both methods are economically viable for producing biomass fuel pellets, with wet torrefaction being the more advantageous option.

A study by Abelha (2019) carried out a preliminary economic analysis on high moisture content roadside grass and low moisture content (wheat straw and miscanthus). The analysis indicated that all tested materials could be technically upgraded to commodity fuels. The analysis revealed that the upgraded roadside grass could be offered at an attractive price of 4.7 E/GJ, leading to an internal rate of return (IRR) of 14%, which could increase to 18% with reduced sludge disposal costs. In contrast, upgrading wheat straw was found to be unprofitable unless a gate fee could be charged, with a competitive price of 6.2 E/GJ yielding an IRR of 7%.

Alherbawi et al. (2024) examined the economic implications of pyrolyzing cucumber, tomato, and carrot wastes as feedstocks in various blends and conditions (temperature and moisture content. It carries out other investigations, such as product yields and energy requirements. The bio-oil yields were observed to increase with higher temperatures and moisture content, while biochar yields declined, and syngas production occurred only at elevated temperatures. Economic analysis revealed a promising return on investment (ROI) of 29% for the single component at 5% moisture content and 300°C, with a payback period of 3.4 years.

The techno-economic and environmental feasibility of rice husks was assessed by Diemuodeke et al. (2021) as a fuel source for a combined heat and power plant in a cluster of rice mills in Abakaliki, Nigeria.

The application of organic Rankine cycle-based synergy for heat and power plants was able to sustainably meet the energy demand of the cluster's rice mills. The analysis shows the capacity of rice husk to generate daily electrical power of 20-30 MWh and thermal power of 4-91 MWh with an efficiency of 14.5-21%. The proposed energy system offers a significant cost advantage, with electricity production costs ranging from 0.12 to 0.159 /kWh, compared to 0.947/kWh for diesel generators, while also contributing to substantial CO_2 emissions reductions of 270-483 $kg.MWh^{-1}$, thereby supporting Nigeria's commitments to the Paris Agreement.

Sarker et al. (2023) harnessed the torrefaction and pelletization process route for pellet production, providing a conceptual design for torrefied fuel pellet production. This is an attempt to reduce over-reliance on wood or fossil fuel for the target application in the rural areas. The entire design contains a torrefaction unit, grinding, preparation of pellet formulation, pelletizing, and finally cooling of pellets. It compares the process of pelletisation with (category 1) or without additives (category 2). The lowest selling price of generated torrefied pellets was found to be $103.4 and $105.1 per tonne at the plant gate for the categories, respectively. Sensitivity analysis shows that, among all variable costs, labor cost has the strongest influence on both net present value (NPV) and minimum selling price (MSP) in making pellets for both scenarios. Furthermore, the internal rate of return was found to be 25% and 22% at a 10% discounted cash flow rate for scenarios 1 and 2, respectively. The framework that was created was found to lessen over-dependence on wood or fossil fuels and facilitate the promotion of bioenergy in rural areas.

Winjobi et al. (2016) compared a one-step pyrolysis process with a two-step process that includes a torrefaction step. The economic analysis reveals that incorporating a torrefaction step reduces the minimum selling price of bio-oil, with the lowest price of 1.04 per gallon achieved at a

torrefaction temperature of 330°C compared to 1.32 per gallon for the one-step process. However, there is a trade-off between the bio-oil quality and selling price, as a higher minimum selling price of 22.19 per GJ compared to 16.89 per GJ for the one-step process on an energy basis.

FUTURE DIRECTION

The abundance of agricultural waste in certain regions of the world has created market opportunities for investors and scientists to transition into alternative sources of energy, of renewable and sustainable sources. Future Market Insight (Outlook 2022), projects that the production of biomass pellets from agricultural residue will increase by 7.1% between 2022 and 2032. Hence, creating an avenue to achieve a carbon neutral, reduce greenhouse gas (GHG) emissions, and abate pressure on the increasing demand for woody biomass as bioenergy sources.

The holistic implementation and adoption of torrefied biomass residue and pellets for energy (heat and electricity) production over coal power generation may be a long-term projection. However, the torrefaction and valorisation of agro-waste can be appreciated progressively by co-firing it with coal at various percentages of the torrefied agro-waste. Co-firing with a certain percentage of torrefied pellets has been seen to bring a significant reduction in greenhouse gas emissions (GHG).

Also, countries with a sufficient supply of agro-waste should promote its use as a domestic and industrial raw material, and can be used as exported to nations with a shortfall. The technology of torrefaction and pelletisation should also be encouraged for efficient transportation and storage means of agro-wastes.

CONCLUSIONS

In pursuit of GHG net-zero emissions and to tackle the climate change crisis, energy decarbonization has become a global discourse. Several considerations and attempts to migrate or mitigate the overdependence on fossil fuels to renewable energy have been made across several technological frontiers. It is therefore concluded in this review that torrefied biomass provides a possibility to serve as a coal alternative if harnessed through sustainable routes such as torrefaction, as it also generates carbon-neutral energy. Torrefied biomass can be used directly or as a coal admixture for energy applications. The typical HHV of torrefied biomass is in the range of 18-30 $MJ.kg^{-1}$, as waste such as torrefied olive mill waste, corn cobs, and oil palm fruit, with the highest energy value. Catalytic torrefaction could also be explored to improve the properties and energy value of agricultural waste biomass. Process optimization of factors such as temperature, residence time, pressure and gas carrier is also essential in getting an improved calorific value with optimal mass and energy yield.

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Impact of Well Water Hardness and Fluoride on Chronic Kidney Disease of Unknown Aetiology in Sri Lanka– Zebrafish as an Animal Model

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ABSTRACT

Chronic kidney disease of unknown etiology (CKDu) is a significant health problem in Sri Lanka. Several hypotheses, such as soil geology, pesticide exposure, cyanotoxins, and prolonged dehydration, have been suggested as possible contributors to CKDu; however, the precise etiology remains unclear. This study aimed to investigate the potential impact of groundwater hardness and fluoride content on CKDu using zebrafish (*Danio rerio*) as an animal model. Sixty well water samples were randomly collected during the dry season from CKDu endemic regions in Sri Lanka, including Galnewa, Rajanganaya, and Medirigiriya in the North Central Province, Dehiattakandiya in the Eastern Province, and Agunukolapelassa (non-endemic area) in the Southern Province as a control. Water pH, conductivity, and dissolved oxygen were measured onsite and within the Sri Lankan Drinking Water Quality Standards (SLS 614: 2013). The highest mean water hardness (285.2 ppm) was recorded in Medirigiriya, and the highest mean fluoride (1.24 ppm) was recorded in Dehiattakandiya, both of which are identified as CKDu high-prevalence areas. The acute toxic effects of these parameters were assessed using zebrafish (*Danio rerio*) embryos. Ninety embryos were exposed up to 96 h post-fertilization (hpf) to water samples representing the highest and lowest water hardness and fluoride concentrations, as single and combined solutions. Embryonic mortality rate in combined exposure (highest hardness and fluoride) was 49.0±0.58%, while hardness alone was 16.7±0.58% and fluoride alone was 18.8±1.15%. Statistical analysis revealed a significant synergistic effect of hardness and fluoride combination ($p < 0.05$) on mortality compared to single exposure. Subsequently, combined exposure caused developmental delays (> 65%) and morphological abnormalities, such as bent body axis and yolk sac edema. Fluorescence images indicated that the damaged premature pronephros in the combined exposure group emitted bright green fluorescence compared to that in the single exposure group. Thus, the findings suggest that the synergistic nephrotoxic effect of high water hardness and fluoride can be a prominent cause of CKDu etiology in Sri Lanka.

INTRODUCTION

Chronic kidney disease (CKD) is a significant health concern worldwide, affecting more than 15% of the population (Cockwell & Fisher 2020). Asia, Central America, Africa, and the Middle Eastern communities have experienced a remarkable increase in CKD cases, especially during the last three decades. CKD of unknown etiology (CKDu) has emerged in farming communities in South Asia (Priyadarshani et al. 2023). CKDu is characterized by a gradual loss of kidney function over time, and the exact cause remains debatable. CKDu is considered multifactorial and is linked to various environmental, occupational, and lifestyle factors. In Sri Lanka, the highest prevalence of CKDu has been reported in the North Central Province, extending to the North Western, Uva, and Eastern provinces, particularly among

farmers (Rajapakse et al. 2016). Several hypotheses have been proposed to explain the origin of CKDu in Sri Lanka. Epidemiological data suggest that CKDu is associated with certain agricultural practices, especially rice farming. These farmers often work under hot and humid conditions, which can lead to heat stress and frequent dehydration. Repetitive heat stress and insufficient hydration may affect kidney function over time (Xu et al. 2021).

Additionally, prolonged exposure to agrochemicals, including pesticides and fertilizers used in agricultural practices, could potentially contribute to the origin and progression of CKDu (Priyadarshani et al. 2023). As most of the population in this region predominantly relies on well water for drinking purposes, poor water quality has been proposed as another contributor to CKDu in the dry zone due to the specific soil geology. Elevated water hardness, fluoride, and the presence of heavy metals act as possible triggering factors. Heavy metals, such as cadmium and arsenic, are known to be nephrotoxic (toxic to the kidneys) and can act as progression factors with long-term exposure, either through contaminated water or soil (Kim et al. 2015). Contaminated drinking water sources, either through natural pollutants or agricultural runoff containing agrochemical residues, heavy metals, and other toxins, have been postulated as other risk factors for CKDu (Pinto et al. 2020). Considering the distribution of patients around the country and the number of patients from specific regions, genetic predispositions may increase individual susceptibility to CKDu when combined with environmental stressors (Abeysiri et al. 2024, Friedman 2019). Some studies have discovered that the role of dietary factors, such as low intake of antioxidants and essential nutrients, increases the vulnerability to CKDu (Wimalawansa & Dissanayake 2019). Throughout prolonged exposure, a combination of these hypotheses, including environmental, occupational, and genetic factors, could contribute to the progression of CKDu (Campese 2022, Liyanage 2022).

Despite these numerous hypotheses, the exact cause of CKDu has not yet been established. This study focused on the synergistic effect of water hardness and fluoride as triggering factors for CKDu in Sri Lanka. Endemic CKDu regions overlap with hydrogeochemical zones that contain elevated fluoride levels and hard water. Available data on groundwater sources demonstrate elevated levels of fluoride and hard or very hard water (Dilrukshi et al. 2023a, Levin et al. 2016), which reflect the unique hydrogeochemistry of the region. Districts with high CKDu prevalence, such as Anuradhapura (18.9%) and Polonnaruwa (13.9%), which are in the North Central Province, have recorded high concentrations of both fluoride (Anuradhapura >2 $mg.L^{-1}$, Polonnaruwa >1 $mg.L^{-1}$) and hardness in Anuradhapura (241-300 $mg.L^{-1}$) and Polonnaruwa (181-240 $mg.L^{-1}$) (Gunawardena et et al. 2021, Indika et et al. 2022). Scientists have pointed out that the cytotoxicity effect of the Na^{2+}/Ca^{2+} ratio in water may influence F^- metabolism. The kidney accumulates more fluoride than all other soft tissues, except for the pineal gland (Inkielewicz & Krechniak 2003). Excess fluoride and hardness exposure may contribute to the development of kidney disease, as CaF2 deposits can promote tubular blockages. This study aimed to evaluate the well water hardness and fluoride content covering random sample points of the North Central Province and Eastern Province, and their possible effect on CKDu. There are high-prevalence (Medirigiriya and Dehiattakandiya) and low-prevalence areas (Galnewa and Rajanganaya) within the endemic region due to the mosaic distribution of cases. CKDu non–endemic area (Agunukolapelassa) in the Southern Province was selected to serve as the control region.

Zebrafish (*Danio rerio)* were used as the animal model to assess the acute toxic effects of water hardness and fluoride on early development and kidney function. *Danio rerio* is considered a well-established vertebrate model and has been successfully used in studies for nearly two decades. Zebrafish have been utilized in different research fields, including biology, toxicology, reproductive studies, genetics, and environmental studies (Hoo et al. 2016). They possess some favorable characteristics to be an ideal model organism, including their small size, rapid development with a short life cycle, optical transparency during early development, tractability in forward genetic screens, genetic similarity to humans (69%), and easy maintenance (Mahanayak 2024). Organogenesis occurs at a very early stage (48 hpf) of embryonic development. Owing to their transparency and genetic similarity to humans, zebrafish provide an effective platform for evaluating the effects of exposure components in human diseases. Therefore, zebrafish can be used as a model organism to understand the toxic effects of different water constituents on body organs, especially kidney development and function. In addition to acute toxicity observations, fluorescent dyes can be used to indicate cellular-level organ damage in zebrafish due to their optical clarity (Lee et al. 2019).

Acridine orange (AO) staining is widely used to detect apoptotic or damaged cells, including renal cells, in zebrafish. Acridine orange selectively binds to nucleic acids and emits different fluorescent signals depending on cell integrity (Hung et al. 2022). In cases of kidney damage, AO helps identify regions of cell death (apoptosis) or injury in developing nephrons. Under a fluorescence microscope, healthy cells emit green fluorescence. In contrast, apoptotic or damaged

cells emit bright green or even orange fluorescence due to changes in nucleic acid structure or increased permeability in damaged cells (Ishaque & Al-Rubeai 2004). This technique was applied in the study to identify the damaged pronephros in exposed zebrafish larvae to assess the potential synergistic effects on kidney development and the possible connection to CKDu.

MATERIALS AND METHODS

Environmental Sample Collection and Analysis

Environmental samples were collected from CKDu-endemic and non-endemic regions in Sri Lanka (Fig. 1). Thirty well water samples were collected using a random sampling method from CKDu high- and low-prevalence sampling sites in CKDu-endemic areas. Medirigiriya and Dehiattakandiya were selected as high-prevalence areas, and Galnewa and Rajanganaya were selected as low-prevalence areas. Agunukolapelassa was considered a non-endemic region (control). Water pH, temperature, conductivity, and dissolved oxygen were measured onsite, and 1 L of water from each sample site was transported to the laboratory in pre-cleaned high-density polypropylene bottles for further analysis. Water hardness was measured in the laboratory using the standard EDTA titrimetric method, and Fluoride, Nitrate, Nitrite, Ammonia, and Total phosphate were measured using spectrophotometric methods (APHA 2017).

Solution Preparation

Desired Hardness solutions (572.6, 40.0 ppm) were prepared using 600 ppm $CaCO_3$ and $MgCl_2$ stock solutions, and fluoride solutions (2.33, 0.37 ppm) were prepared using 100 ppm NaF stock solutions. Embryos were exposed to well water samples from the field and synthetically prepared hardness and fluoride solutions simultaneously in the combined exposure.

Zebrafish Breeding and Exposure

A total of 20 female and male zebrafish were kept separately in glass aquaria (length 80 cm, height 50 cm, width 46 cm) at the Animal House of the University of Sri Jayewardenepura, Sri Lanka. Fish were maintained at 27±1 ^{o}C temperature, and a natural dark/light cycle of 12 h was provided with continuous water exchange. The water pH was maintained within 7-8. The fish were conditioned for two weeks and fed twice daily with artificial fish feed and brine shrimp. Brood stock maintenance was performed according to the OECD (2013) guidelines. The spawning cage was made with a fixed wire mesh in the middle to prevent adults from cannibalizing the eggs. Fish were added to the spawning cage immediately before the onset of darkness, the day before the experiment, at a ratio of 2:1 (male to female) for breeding. Spawning and fertilization occurred within 1 h of light onset the following morning. After spawning, the adult fish and the wire mesh were removed, and the embryos were transferred to a Petri dish (60 mm) containing distilled water. Fertilized eggs were selected using an optical microscope in the presence of embryo cleavage after fertilization.

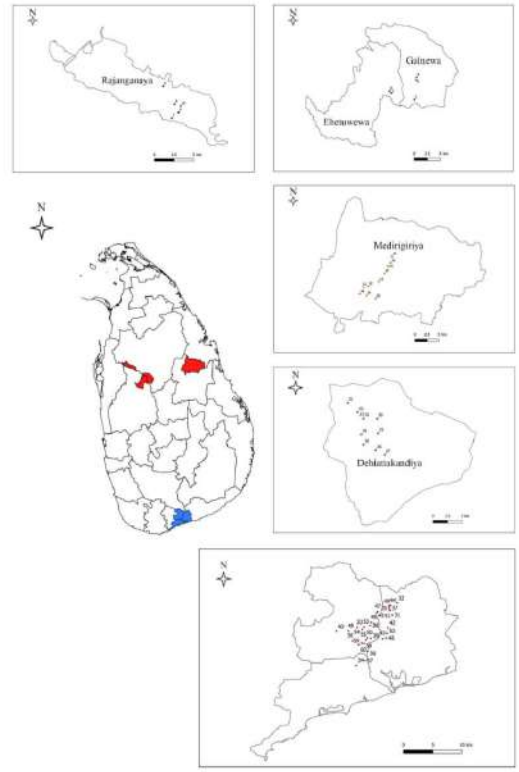
Exposure series of water hardness (572.6 ppm, 40.0 ppm) and fluoride (2.33 ppm, 0.37 ppm) were prepared according to the environmental sample analysis, and 30 embryos per concentration were exposed in 24-well plates within 2 h of post-fertilization (hpf). To obtain the acute toxic effects of water hardness and fluoride, experiments were conducted with single (hardness and fluoride alone) and combined (hardness + fluoride) exposures. Combined exposure experiments were performed using both environmental samples and synthetic solutions. Negative controls with distilled water and positive controls with 4 ppm 2,4-dichloroaniline were conducted simultaneously. The entire experiment was performed in triplicate using three different batches of embryos.

Observations and AO Staining

Mortality rates, hatching rates, heart rate, developmental delays, and morphological abnormalities were recorded every 24 h until 96 hpf using a light microscope (Optika). At 10 days post-fertilization, the larvae were stained with acridine orange to observe organ damage. Embryos were dipped in 10 mL of 100 $\mu g.mL^{-1}$ of Acridine Orange for 1 h, and after that, several times with E3 buffer media (14.61 g NaCl, 0.63 g KCl, 2.43 g $CaCl_2 \cdot 2H_2O$, and 1.99 g $MgSO_4$ in 1 L Milli-Q water) to remove excess dye (Kaufman et al. 2009). The larvae were then mounted on slides and observed under a fluorescence microscope (Zeiss Axio) to evaluate cell apoptosis.

Statistical Analysis

One-way ANOVA was used to evaluate the significant difference between the fluoride and hardness, single exposures and mortality rates of the zebrafish. Two-way ANOVA was used to evaluate the significant differences in fluoride, hardness, combined exposure, and mortality rates. If $p < 0.05$, the null hypothesis was rejected, which means that there was a statistically significant difference in embryonic mortality and different concentrations of the solutions, and a significant difference between single and combined exposures. Fluorescence intensity was measured to quantify the severity of cell apoptosis using the mean gray value of the fluorescent regions by ImageJ software.



(- CKDu endemic areas (High and low prevalence)

- CKDu non-endemic areas)

Fig.1: Map of the sampling locations.

Table 1: Hardness and Fluoride concentrations in well water samples in CKDu endemic areas (mean±SD).

Sample No.	Area	Hardness [mg.L^{-1}]	Fluoride [mg.L^{-1}]	Sample No.	Area	Hardness [mg.L^{-1}]	Fluoride [mg.L^{-1}]
1	Galnewa (CKDu low prevalence)	300.00±0.58	0.37±0.00	16	Medirigiriya (CKDu high prevalance)	164.00±0.58	1.33±0.00
2		160.00±0.58	0.53±0.00	17		248.00±0.58	1.32±0.00
3		92.00±0.58	0.86±0.00	18		220.00±0.00	1.21±0.00
4		124.00±0.00	0.72±0.00	19		572.60±0.58	0.45±0.00
5		268.00±0.00	0.54±0.00	20		296.00±0.58	0.52±0.00
6	Rajanganaya (CKDu low prevalence)	260.00±0.00	0.81±0.00	21	Dehiattakan-diya (CKDu high prevalence)	232.00±0.58	1.71±0.00
7		160.00±0.58	0.61±0.00	22		360.00±0.00	0.65±0.00
8		240.00±0.00	0.51±0.00	23		140.00±0.58	0.56±0.00
9		280.00±0.58	2.33±0.00	24		160.00±0.00	0.76±0.00
10		260.00±0.58	0.38±0.00	25		224.00±0.58	1.35±0.00
11	Medirigiriya (CKDu high prevalance)	164.00±0.58	1.04±0.00	26		460.00±0.00	1.17±0.00
12		368.00±0.00	0.88±0.00	27		140.00±0.58	2.06±0.00
13		280.00±0.00	1.97±0.00	28		272.00±0.00	1.57±0.00
14		440.00±0.58	1.80±0.00	29		240.00±0.58	0.70±0.00
15		100.00±0.58	1.26±0.00	30		181.33±0.00	1.87±0.00

RESULTS

Thirty well water samples were randomly collected from drinking water wells from CKDu endemic areas: Galnewa and Rajanganaya had a low prevalence, and Medirigiriya and Dehiattakandiya had a high prevalence. The Agunukolapelassa area was selected as the CKDu non-endemic area, and 30 water samples were collected. Fig. 1 shows the sampling location map.

The highest water hardness was measured as 572.60±0.58 ppm in Medirigiriya, and the lowest was 40.00±0.58 ppm in Dehiattakandiya (Table 1). The highest mean water hardness was recorded as 285.2 ppm in Medirigiriya, and the highest mean fluoride was recorded in Dehiattakandiya as 1.24 ppm, which are identified as CKDu high-prevalence areas.

Agunukolapelassa, where no CKDu patients are recorded, has low water hardness and fluoride compared to CKDu-endemic regions (high and low prevalence) (Table 2). The highest hardness was recorded in the Yakagala area (158.67 ± 0.06 ppm), while the lowest was in Barawakumbuka (16.00 ± 0.06 ppm). Fluoride levels were also lower and remained within the WHO guidelines. Sites with both high hardness and fluoride levels were not identified in this area.

Water pH, conductivity, and dissolved oxygen were measured on-site and were within the Sri Lankan Drinking Water Quality Standards (SLS 614:2013) (Table 3).

The results of the single exposure showed a mortality rate of less than 20% in each case (Table 4). Fluoride had a higher effect on embryonic mortality than hardness, as it caused 18.85% death out of 90 exposed embryos. The hatching rate was affected more by hardness than by fluoride, as it was reduced by more than 10%. A single exposure did not induce any morphological abnormalities or growth retardation.

Table 5 shows the results of the synergistic exposure to water hardness and fluoride on zebrafish embryos. Mortality rates increased with higher concentrations of hardness and fluoride compared to single exposures. Exposed embryos showed more than 50% mortality in both environmental and synthetic solutions at the highest hardness-fluoride combination. However, in the single exposure, it was less than 20%. Hatching rates were severely affected by high water hardness and fluoride, as it resulted in 0% larvae out of embryos after 54 hpf, while in the control, the hatching rate was 83.3%

Fig. 2 illustrates the embryonic developmental stages of zebrafish after exposure. Morphological abnormalities [2(e)], such as edema and bent body axis, were observed in approximately 15% of the larvae at the highest combined exposure. At the same time, they showed less swimming ability and body movements than the other exposures and the control. Low concentrations of combined exposure, single exposure, and control larvae showed no abnormalities after 96 hpf.

Mortality rates at every 24 h interval showed a significant difference between single and synergistic exposures. Mortality rates were higher after 48 hpf and 72 hpf than at 24 hpf and reduced at 96 hpf (Fig. 3). Most internal organs, including the pronephros, become functional at approximately 48 h post-fertilization (hpf). The combined effect of water hardness and fluoride was more fatal at

Table 2**:** Hardness and Fluoride concentrations in well water samples in the non-endemic area of Angunukolapelassa (mean±SD).

Sample No.	Hardness	Fluoride	Sample No.	Hardness	Fluoride
1	98.67±0.06	0.79±0.00	16	120.00±0.00	0.56±0.00
2	158.67±0.06	0.56±0.00	17	61.33±0.06	0.83±0.00
3	60.00±0.06	0.32±0.00	18	53.33±0.06	0.58±0.00
4	53.33±0.06	0.53±0.00	19	42.67±0.00	1.18±0.00
5	114.67±0.00	0.22±0.00	20	62.67±0.06	0.38±0.00
6	32.00±0.06	0.47±0.00	21	16.00±0.06	0.32±0.00
7	29.33±0.00	0.18±0.00	22	25.33±0.06	0.50±0.00
8	22.67±0.06	0.41±0.00	23	41.33±0.06	0.79±0.00
9	24.67±0.06	1.16±0.00	24	54.67±0.06	0.31±0.00
10	140.00±0.06	0.16±0.00	25	114.67±0.06	0.75±0.00
11	85.33±0.06	0.55±0.00	26	68.00±0.06	0.90±0.00
12	58.67±0.06	0.25±0.00	27	81.33±0.06	0.41±0.00
13	93.33±0.06	0.93±0.00	28	45.33±0.06	0.28±0.00
14	62.67±0.06	0.69±0.00	29	81.33±0.06	0.56±0.00
15	78.67±0.06	0.54±0.00	30	60.00±0.06	0.55±0.00

Table 3: Water quality parameters of well water samples from CKDu endemic areas (Galnewa, Rajanganaya, Medirigiriya, and Dehiattakandiya).

Sample No.	Water temperature (°C)	Water pH	Conductivity [µS. cm^{-1}]	DO [$mg.L^{-1}$]	Nitrate [$mg.L^{-1}$]	Nitrite [$mg.L^{-1}$]	Ammonia [$mg.L^{-1}$]	Total phosphate [$mg.L^{-1}$]
1	24.8	7.22	5520	4.87	4.45±0.01	ND	ND	4.41±0.01
2	25.9	7.53	743.9	8.49	4.27±0.02	ND	ND	6.12±0.01
3	28	7.05	721.3	4.18	4.11±0.01	ND	ND	5.73±0.01
4	24.3	7.67	1335	6.49	3.95±0.01	ND	ND	0.63±0.01
5	25.6	7.5	1371	1.17	2.63±0.01	0.016±0.00	ND	6.43±0.02
6	26.3	7.27	465.2	4.87	2.82±0.02	0.248±0.00	ND	6.64±0.01
7	27.5	7.09	829.1	3.55	2.51±0.01	0.014±0.00	ND	8.22±0.01
8	25.8	6.62	908.5	7.58	1.89±0.01	ND	ND	8.32±0.03
9	26.3	5.52	1067	7.2	1.98±0.01	0.013±0.00	ND	8.08±0.01
10	26.9	6.28	1420	7.43	1.53±0.01	0.012±0.00	ND	8.08±0.01
11	24.8	7.45	1703	4.37	1.79±0.01	0.018±0.00	ND	8.60±0.02
12	26.1	7.19	1279	2.68	2.12±0.01	ND	ND	8.60±0.01
13	26.4	6.95	395.4	2.26	0.00±0.00	ND	ND	8.46±0.01
14	26.2	7.18	675	1.62	1.08±0.01	0.031±0.00	ND	6.99±0.02
15	26.9	7.36	483.9	5.77	1.64±0.01	0.014±0.00	ND	3.29±0.01
16	24.9	7.26	939.6	5.99	1.93±0.01	0.019±0.00	ND	8.95±0.01
17	26.4	7.19	541	3.13	2.09±0.01	ND	ND	6.64±0.01
18	27.2	7.06	461	2.52	2.16±0.01	ND	ND	7.48±0.03
19	25.7	7.99	775.4	5.89	2.08±0.02	ND	ND	7.76±0.01
20	26.5	7.49	988	2.71	2.04±0.01	ND	ND	2.59±0.01
21	26.0	7.47	827.1	3.6	0.18±0.01	ND	ND	8.39±0.01
22	26.8	6.76	442.3	3.7	0.67±0.02	ND	ND	0.14±0.04
23	25.8	7.13	310.3	3.87	1.87±0.01	ND	ND	0.14±0.01
24	26.3	6.79	280.4	2.48	1.88±0.01	0.023±0.00	ND	0.29±0.01
25	25.6	7.29	311.3	2.1	0.78±0.01	0.024±0.00	ND	0.14±0.02
26	26.0	7.39	361.9	4.06	0.00±0.01	0.014±0.00	ND	2.59±0.01
27	26.6	7.11	376.9	3.04	2.04±0.01	ND	ND	6.99±0.02
28	25.7	7.21	245	4.21	1.53±0.01	ND	ND	8.22±0.01
29	26.4	6.88	289.7	4.75	3.95±0.01	ND	ND	5.73±0.01
30	27.1	7.05	780.3	2.45	2.09±0.01	ND	ND	2.59±0.01

Table 4: Embryonic acute toxicity observations of single exposure to water hardness and fluoride.

Sample	Mortality rate%	Hatching rate% [54 hpf]	Heart rate [bpm] [72hpf]
572.6ppm (H)	16.7±0.58	26.7	136±4
40.0ppm (H)	10.0±1.15	46.7	140±6
2.33 ppm (F)	18.8±1.15	40.0	150±8
0.37ppm (F)	13.3±1.15	66.7	152±4
Control	0	83.3	140±2

Table 5: Embryonic observations to combined exposure of water hardness and fluoride (ES- Environmental sample, SS- Synthetic solution, H- Water Hardness, F- Fluoride).

Solution [ppm]	Mortality rate%		Hatching rate% [54hpf]		Heart rate [bpm] [72 hpf]	
	ES	SS	ES	SS	ES	SS
572.6 ppm (H) + 2.33 ppm (F)	48.2±1.15	49.0±0.58	0	0	164±4	160±2
572.6 ppm (H) + 0.37 ppm (F)	32.4±0.58	36.7±1.15	0	0	165±6	158±2
40.0 ppm (H) + 2.33 ppm (F)	40.3±0.00	43.3±0.58	6.6	10.0	141±8	144±4
40.0 ppm (H) + 0.37 ppm (F)	19.8±1.15	23.3±1.15	16.6	23.3	127±4	115±2
Control	0		83.3		144±2	

2 (a) 2(b) 2(c) 2(d) 2 (e)

Fig. 2: Light microscope images of embryonic development after exposure 2(a) 2 hpf, 2(b) 24 hpf, 2(c) 48 hpf, 2(d) 72 hpf, 2(e) embryo with bent body axis and yolk sack edema (10×4).

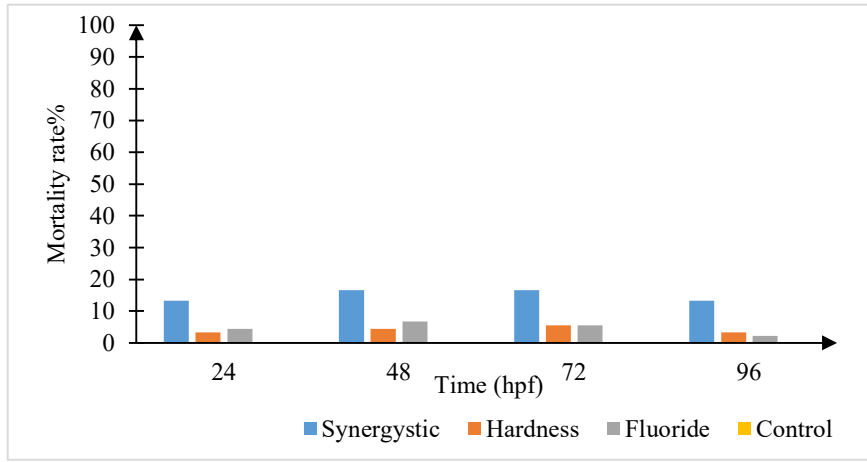
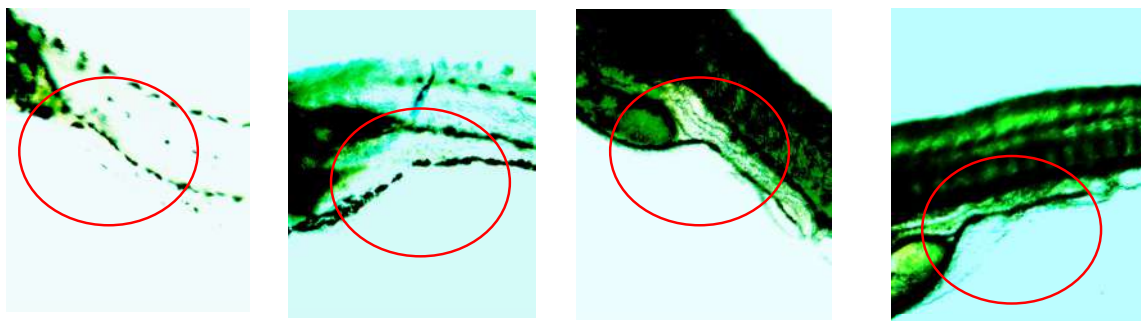


Fig. 3: Comparison of mortality rates of embryos exposed to single and synergistic exposures over time (hpf).

each stage, whereas fluoride exhibited a more lethal effect than water hardness alone. The control group exhibited no mortality throughout the experiment.

One-way ANOVA showed a significant effect ($p < 0.05$) of both single and combined exposure on embryonic mortality. Two-way ANOVA comparison indicated a significant difference ($p < 0.05$) between single and combined exposure, as combined treatments (hardness + fluoride) caused significantly higher mortality than individual treatments. Subsequently, Pearson's correlation showed a weak positive correlation ($r = 0.3$) for single exposures of hardness, fluoride, and mortality rates, while a moderate positive correlation ($r = 0.5$) for combined exposure and mortality rates.

The pronephros is a paired organ that filters blood, which is composed of lymphoid tissue, adrenocortical cells, and the pronephric duct. Fluorescence microscopic images



4(a) 4 (b) 4(c) 4(d)

Fig. 4: Fluorescence microscopic images of the AO staining larvae (a) Control, (b) Hardness single exposure, (c) Fluoride single exposure, (d) Combined hardness and fluoride exposure.

(Fig. 4) indicated kidney cell apoptosis after Acridine Orange staining. It filters blood circulation and is responsible for osmoregulation during larval life. After staining, the larvae in the control group showed no bright green emissions in the pronephros area. At the same time, single hardness [4(b)], single fluoride [4(c)], and combined exposure [4(d)] showed increasing bright green emissions in the pronephros located in the embryo's trunk region. Fluorescence intensity was measured as the mean gray value of the fluorescent regions, which were 146, 190, and 204, respectively. According to the degree and pattern of fluorescence, these emissions correlate with the severity of kidney injury.

DISCUSSION

CKDu is defined as chronic kidney damage or dysfunction without conventional risk factors such as diabetes, hypertension, or genetic conditions. The exact cause of CKDu remains unclear, but several factors are suspected to contribute to its etiology. According to recent research findings, high levels of calcium and magnesium in water (hard water) are potential risk factors (Wasana et al. 2016). Elevated fluoride levels in drinking water have been studied as potential contributors to CKDu, with concerns about fluoride accumulating in the kidneys over time (Liyanage et al. 2022). North Central Province in Sri Lanka is a hotspot for CKDu, where water hardness and fluoride levels exceed the WHO water quality guidelines in most drinking-water wells. According to Yang et al. (2022), local groundwater in a CKDu prevalent area of Sri Lanka could cause kidney damage, implying that high water hardness and fluorine might be the inducible environmental factors for the etiological cause of CKDu. Therefore, the synergistic effect of water hardness and fluoride may cause the progression of CKDu among communities using drinking-water wells. In this study, the fluoride level reached 2.33 ppm, which is the highest, and the mean value was 0.69 ppm, which is higher than that in other regions in Sri Lanka. According to the WHO drinking water guidelines, the maximum level of fluoride is 1.5 ppm, and the study area has recorded more than seven sites with fluoride levels exceeding this limit. According to the water quality parameters, the water hardness ranged at the very hard water level. These parameters have alarming values that can lead to CKDu in endemic areas, with their synergistic effect on kidney function. CKDu non-endemic area water samples did not exceed the WHO maximum value (<1.5 ppm) but recorded very hard water values. This also suggests that a synergistic effect could act as a triggering factor for CKDu in the country.

In this study, the synergistic effects of water hardness and fluoride were evaluated using zebrafish embryos and larvae. Animal models can be used to assess the acute toxic effects of drinking water and determine kidney damage at the pronephric level. Zebrafish share approximately 70% of their genes with humans, and their nephron structures (the basic functional unit of the kidney) are remarkably similar, making them a good model for studying renal diseases. Zebrafish embryos can be used to test various environmental substances to determine their nephrotoxic potential, aiding in the identification of potential CKDu triggers. The study data suggest a significant relationship ($p< 0.05$) between elevated water hardness levels and fluoride with kidney damage in the exposed embryos. Both environmental and synthetic solutions showed mortality in over 50% of the embryos and morphological abnormalities, such as yolk sac edema and curved body axis, which are expressed due to kidney damage. Single exposures to fluoride and hardness revealed less acute toxicity in embryos than combined exposures. Fluoride alone had a more lethal effect on embryonic development than hardness alone. The role of the kidneys can be explained, the body's primary excretory organs in regulating the metabolism of poisons and foreign toxins. According to studies, the kidneys filter and reabsorb 50–80% of the fluoride that the body consumes (Dharmaratne 2019), which causes abnormal development and cell death in the embryonic stages. Fluoride-induced kidney structural damage can inhibit renal cell proliferation when exposed to higher concentrations for extended periods (Wimalawansa 2020).

Hard water mainly consists of Ca^{2+} and Mg^{2+} ions. Ca^{2+} plays various roles in the composition of bones and teeth and can control nerve transmission and material release (Salama & Mohammed 2023). Many previous studies have found that F^- and Ca^{2+} antagonistically affect biology. Once F^- is absorbed by the body, it enters the blood to form insoluble CaF_2 precipitates and deposits in the kidney tissue while being excreted (Nóbrega et al. 2019). When a combination of hardness and fluoride is exposed to zebrafish embryos for a certain period, it accumulates CaF_2 in the kidney as precipitates, which can ultimately cause tubular damage and renal failure in adult zebrafish. This can be identified externally by the edema conditions of the larvae after hatching. Edema is a pathological condition characterized by abnormal fluid accumulation in the body (Wimalawansa, 2020). Zebrafish depend on their skin, kidneys, and gills to maintain proper internal water volume and osmotic pressure. Kidneys are essential for controlling ion and water levels for appropriate osmoregulation. As the kidney is responsible for waste elimination in fish, any dysfunction can lead to fluid retention (Wang et al. 2024). This expanded the larvae's body shape and exhibited edema under a light microscope.

The mortality of embryos is higher after 48 hpf, when larvae start to form vital organs in the body. The pronephros, the sole kidney of zebrafish larvae, develops during embryogenesis and is fully formed approximately 48 h after fertilization (Serluca & Fishman 2001). Accelerating the lethal effect at this stage can be due to the damage of vital organs by the exposed synergistic solutions. The pronephros is highly sensitive and accumulates more CaF_2 at this stage, which can cause tubular degeneration. Long-term exposure to well water with elevated hardness and fluoride can have a similar effect on human kidneys. The origin and progression of CKDu are triggered by well water from the CKDu prevalence area, which recorded higher water hardness and fluoride levels exceeding the WHO guidelines.

Pronephros damage was confirmed using acridine orange staining. Acridine orange (AO) staining is a common technique used to detect apoptotic or damaged cells, including kidney cells, in zebrafish embryos. Acridine orange selectively binds to nucleic acids and emits different fluorescent signals depending on cell integrity. In cases of kidney damage, AO helps identify regions of cell death (apoptosis) or injury in developing nephrons (Plemel et al. 2017). Under a microscope, damaged cells emit bright green fluorescence due to changes in nucleic acid structure or increased permeability in damaged cells (2017). The degree and pattern of fluorescence correlated with the severity of kidney injury and apoptosis. The highest emission was observed after combined hardness and fluoride solution exposure, as it recorded the highest mean gray value (204). A single exposure to fluoride (190) had a higher green emission intensity than a single exposure to hardness (146), suggesting that fluoride has a more significant effect on kidney damage than hard water. Hardness triggers the impact of fluoride on kidney tubules when combined, and induces prolonged renal damage (Dilrukshi et al. 2023), causing higher emissions of fluorescence due to cell apoptosis.

Most farming communities in the North Central Province depend on deep or shallow wells for drinking and other purposes. Due to the soil geology of the area, the water contains elevated levels of Ca^{2+}, $Mg^{2+,}$ and F^- ions. Continued exposure to very hard water and high fluoride levels may be the primary reason for the unknown etiology of Chronic Kidney Disease in Sri Lanka. Dissanayake (2005) suggested that the fluoride concentration and its interactions with other ions, such as Ca^{2+}, $Mg^{2+,}$ and $Na^{2+,}$ could trigger the onset of CKDu in Sri Lanka. According to Wickramarathna et al. (2017), hardness reached values as high as 516 $mg.L^{-1}$ in some groundwater in CKDu-affected regions. The mean hardness of groundwater in all samples collected during the pre-monsoon period was 181 $mg.L^{-1}$ Fig. 3, indicating very hard water.

In this study, the highest hardness was recorded as 572 ppm, and the mean hardness was 251 ppm, indicating very hard water according to the WHO guidelines. Fluoride was also found at alarming levels in most wells in the area, exceeding the safe drinking water levels. Individuals consume water with high hardness and fluoride, which can accumulate CaF_2 in kidney tissues with long-term exposure. Similar to zebrafish kidney damage, human kidneys are affected by continuous deposition, leading to oxidative stress, inflammation, and tubular damage. Patients with CKDu often show signs of proximal tubular injury, which aligns with the effects of fluoride toxicity. In particular, farming communities are exposed to high temperatures and reduced kidney perfusion, which exacerbates fluoride retention. This study proposes that the combination of water hardness and fluoride is one of the triggering factors behind Chronic Kidney Disease of unknown aetiology in Sri Lanka. To mitigate CKDu progression among communities, several actions can be taken, as this has become a severe health concern in the country. Routine water testing in high-risk areas, continuous monitoring, and regulation of groundwater wells in the North Central, Eastern, and Central Provinces must be implemented to overcome this issue. Most areas have reverse osmosis water systems and filter units at the household level. People should be encouraged to use treated water rather than well water by providing adequate awareness of the risk of CKDu. Agricultural practices and the use of agrochemicals must be monitored, and safety measures are needed. Most importantly, early detection of the disease and affordable treatments are essential as long-term prevention strategies.

CONCLUSIONS

The study data revealed a significant relationship between water hardness and fluoride levels in CKDu in Sri Lanka. Both parameters exceeded the WHO drinking water guidelines in most wells consumed by the affected community. Zebrafish were used as an animal model to express embryonic and cellular-level damage to the organs. Morphological abnormalities and mortality rates were positively correlated with exposure to water hardness and fluoride concentrations. Combined exposure to hardness and fluoride resulted in more apoptotic cells than other exposures, with a higher degree of emissions in the kidney area. Hence, prolonged exposure to the combination of hardness and fluoride and its synergistic effect can be one of the prominent causes of CKDu progression, as it causes renal cell damage.

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Impact of Different RPM on BOD, COD and Turbidity Reduction Using Natural and Synthetic Media in Dairy Wastewater Treatment

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ABSTRACT

This study lays the foundation for an integrated and adaptive method for treating dairy wastewater within a multi-stage Rotating Biological Contactor (RBC) system. Rotational speed optimization from stage to stage, dynamic control of hydraulic retention time (HRT), and better media performance evaluation are the ingredients drawn into the proposed model for the efficient removal of biochemical oxygen demand (BOD), chemical oxygen demand (COD), and turbidity. Each RBC stage works with pollutant-specific RPM levels, that is, at the maximum biofilm interaction and pollutant reduction. HRT adjustments in real-time work via feedback mechanisms, whereas biofilm attachment is optimized through shear stress and media characteristics. The aim of this study was to demonstrate through comparative experiments that both natural (coconut coir) and synthetic (polyethylene) media can result in high biofilm growth and pollutant degradation rates. Robustness tests under varying influent loads indicated the same steady performance of the system. The integrated model achieved BOD removal of 90.1%, COD reduction of 85.3%, and turbidity removal of 79.8%. The final effluent quality is stringent as it meets discharge limits, with BOD<10 mg.L^{-1}, COD<50 mg.L^{-1}, and turbidity<10 NTU. The proposed framework presents an efficient and scalable option for the treatment of high-strength dairy effluents and can be deployed in several additional industrial waste scenarios.

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INTRODUCTION

Dairy wastewater has relatively high organic loads, turbidity, and variable composition. Effluent with elevated BOD, COD, and suspended solids levels requires effective treatment before it can be released into natural water bodies. Traditional wastewater treatment systems (Katare et al. 2024, Katare et al. 2024, Ogedey et al. 2024) tend to reach their limitations in the management of the dynamic nature of dairy effluents, especially concerning the consistency in pollutant reduction levels across several parameters in the process. Although Conventional Rotating Biological Contactors have been established to be effective in industrial wastewater treatment, their performance under operation is significantly affected by critical variables such as rotational speed, hydraulic retention time (HRT), and biofilm attachment media. If not optimized, such factors will lead to biofilm instability, suboptimal reduction of pollutants, and inefficiencies in the treatment process. Although many studies have explored various configurations of RBCs, most existing systems are devoid of stage-wise optimization of operational parameters. Most approaches (Cargnin et al. 2024, Belkodia et al. 2024, Elkady et al. 2024) overlooked biofilm dynamics, the interdependence of shear stress, and pollutant-specific treatment efficiencies. The choice of medium, whether natural or synthetic, also heavily influences biofilm development and pollutant removal, although comparative studies are still in their infancy. The treatment technologies that advance possess a real need for solutions for both the improvement of removal of BOD, COD and turbidity, and for adaptation to changing wastewater compositions. Effective provision is also an integral component in achieving stringent discharge standards by providing effective residual pollutant-

removing post-treatment units. This study aims to fill such gaps by introducing a novel multifaceted approach for the treatment of dairy wastewater using multiple-stage RBCs. The proposed framework optimizes the rotational speed, media type, and HRT at each stage of the RBC for progressive pollutant reduction. Complementary methods, such as feedback-based HRT control, media performance evaluation, and shear stress analysis, were incorporated into the system for enhanced robustness and efficiency. Post-treatment units with sedimentation and filtration are added to improve the quality of the effluent. Combined, these strategies not only demonstrated improved pollutant reduction performance but also established a scalable model for industrial wastewater treatment.

Motivation and Contribution

This study was motivated by the urgent need to optimize the efficiency of dairy wastewater treatment systems. Currently, existing technologies are hampered by the lack of interaction between key operational parameters and their combined effects on pollutant removal. Rotating speed, HRT, and media type have been analyzed as separate factors with inevitable suboptimal designs and inconsistent performance. Dynamic dairy wastewater has fluctuating levels of BOD, COD, and turbidity, which requires adaptive treatment strategies that can be maintained under realistic conditions. These challenges necessitate the development of an integrated approach consisting of multiple optimization techniques, where system robustness must be maintained across variable influent compositions. The principal contribution of this study is the development of an integrated framework for dairy wastewater treatment using multistage RBCs. The rotational speeds were optimized at different stages to ensure that the targeted pollutants were removed at each RBC stage, depending on the requirements of BOD, COD, and turbidity. Introduction of HRT-Based Adaptive Biofilm Stimulation and RPM-Shear Correlation for Biofilm Stability Improves Operational Flexibility and Integrity: Critical Areas Missing in Standard RBC Designs. Media selection can be an informed choice by comparing the differences between natural and synthetic media, whereas post-treatment units will contribute to meeting effluent quality standards. Real-world variability testing verified the resilience and scalability of the system. These contributions, in aggregate, form a comprehensive model for the treatment of dairy wastewater by creating high efficiency in managing industrial effluents.

In-Depth Review of Models Used for Wastewater Analysis

A review of the latest research on wastewater treatment indicates crucial advances in technology and methodology for industrial, agricultural, and domestic uses. Taken together, the surveys reviewed provide a broad view of current practices, innovative processes, and challenges in the treatment of complex effluents. Each paper in its own turn contributes to an understanding of wastewater treatment, but together they mark trends, limitations, and possible future directions of the research. Katare et al. (2024) studied the electrocoagulation of oil-field-produced water containing large amounts of COD, BOD, and turbidity. These reductions may have a significant impact on both the oil and gas industries. Ikhlaq et al. (2024) researched the catalytic ozonation of pharmaceutical wastewater and attained considerable effectiveness in removing persistent compounds such as enrofloxacin. Ogedey & Oguz et al. (2024) applied electrocoagulation to landfill leachate and attributed its effectiveness in treating combined COD and ammonia-nitrogen levels.

Cargnin et al. (2024) developed an integrated biological and physicochemical approach for shrimp farm wastewater with thorough contaminant removal. The treatment of cheese wastewater by Belkodia et al. (2024) through the application of advanced oxidation processes highlights the utility of integrated methodologies in developing approaches toward reuse standards for the irrigation process. Elkady et al. (2024) used carbonized sawdust and textile filtration in the treatment process for slaughterhouse wastewater, thereby underlining material reusability. Among the various wastewater and effluent treatment alternatives, Ahsan et al. (2023) specifically challenged the removal of dyes from textile wastewater, while Gholami et al. (2024) highlighted microalgae biofilters as an alternative for sustainable urban effluent treatment. Vermifiltration techniques applied to domestic wastewater (Kumar & Khwairakpam 2024) showcased the ecological advantages of biological processes. Ettaloui et al. (2023) used a sequence batch reactor for the treatment of oil washing wastewater with high efficiency under controlled environmental conditions. Ashar et al. (2024) investigated catalytic thermolysis as an advanced hybrid treatment process for textile wastewater and provided simple, scalable views of the process. Baghizade et al. (2023) considered biological treatment after flocculation, and Vezar et al. (2024) emphasized the use of natural bio-coagulants, such as papaya seeds, in the wastewater treatment of laundry. Lory et al. (2024) proposed microbial fuel cells for wastewater treatment, achieving pollutant removal and energy generation simultaneously. Bhuvanendran et al. (2024a) considered dairy wastewater by incorporating natural coagulants and product recovery. Katare et al. (2023) reviewed activated sludge processes for pharmaceutical effluents, with an emphasis on adaptability to many contaminant profiles. Constructed wetlands using *Pistia stratiotes* for domestic wastewater were presented

by Ali et al. (2024), showing eco-friendly scalability. Mao et al. (2024) reviewed floating wetlands and highlighted the application of such systems in industrial wastewater treatment. (Das and Paul et al. 2023) have explored the scopes of treatment of dairy wastewater, with a discussion on the promising technique of peroxi-electrocoagulation and are studied further by Bhuvanendran et al. (2024b) and Dey and Pal et al. (2023) proposed the scope for the sustainable treatment potential of tannery effluents in a recent study on cyanobacteria.

Iteratively, as per Table 1, Radeef et al. (2024) integrated microbial fuel cells with biofilters for kitchen wastewater samples. Selvaraj and Arivazhagan et al. (2024) optimized textile treatments using electrocoagulation and adsorption with the help of advanced statistical tools for better results. The synthesis of nanoparticles for wastewater treatment, as demonstrated by Maloma et al. (2023), has antimicrobial benefits in addition to pollutant removal. Similarly, Thomas et al. (2023)applied neural networks to optimize laccase production and enhance dye removal. For instance, Elhadeuf et al. (2023) successfully optimized textile effluent treatment using electrocoagulation and microfiltration, while Fotoohi et al. (2024) attempted ultraviolet disinfection of biological systems. Cherni et al. (2024) investigated the treatment of rural wastewater using TiO□-based photo-Fenton processes. Bedane and Asfaw (2024) showed hybrid anaerobic reactors coupled with microalgae for slaughterhouse wastewater and focused significantly on synergistic systems. Ayhan et al. (2024) and Mohan et al. (2024) analyzed different industrial applications, ranging from petroleum refinery effluents to graywater. In contrast, Kumari et al. (2024) discussed in detail several techniques for dye removal and presented future prospects. Benbouzid et al. (2024) used natural porous media for domestic wastewater, just like the sustainable approaches by Nzeyimana & Mary's (2024) that used Moringa oleifera. Yadav et al. (2024) and Sandhya Rani et al. (2023) referred to an advanced trend of applying advanced filtration technologies for coke oven and rubber-processing wastewater. Photocatalytic processes for petrochemical wastewater, as studied by Aghazadeh et al. (2023), showed novel proxone techniques. Detho et al. (2023) applied zeolite-based adsorbents for ammonia and COD reductions in rubber effluents.

Circular economy-based valorization of boiler ash to produce coagulant (Dalmora et al. 2024, Monroy-Licht et al. 2024) emphasized the application of phytoremediation with water hyacinth, while Boraghi et al. (2023) applied sinusoidal electrocoagulation along with ceramic filters for complete removal of pollutants. Nano-adsorbents of chitosan have been used to treat dairy effluent (Dinesha et al. 2023), opening up new horizons in the adoption of methodologies during the adsorption process. This holistic review of recent articles outlines the richness, diversity, and innovation of wastewater treatment technologies. This reflects the

Table 1: Comparative analysis of existing methods.

Method	Application	Key Findings	References
Electrocoagulation	Oil-field produced water	Achieved substantial COD, BOD, and turbidity reduction; scalable and efficient for high-strength effluents.	Katare et al. (2024)
Catalytic Ozonation	Pharmaceutical wastewater	High degradation efficiency for recalcitrant compounds like enrofloxacin using molecular ozone reactions.	Ikhlaq et al. (2024)
Combined Biological and AOPs	Cheese wastewater	Integrated approach achieved reuse standards for irrigation; reduced phytotoxicity significantly.	Belkodia et al. (2024)
Microbial Fuel Cells (MFCs)	Domestic wastewater	Dual benefits of pollutant reduction and bioelectricity production; sustainable and scalable.	Lory et al. (2024)
Constructed Wetlands	Domestic and industrial wastewater	Eco-friendly scalability using Pistia stratiotes; effective for nutrient and pollutant removal.	Ali et al. (2024)
Peroxi-Electrocoagulation	Dairy wastewater	High pollutant reduction; demonstrated potential for pigment recovery from waste sludge.	Bhuvanendran et al. (2024)
Advanced Oxidation Processes (AOPs)	Petroleum refinery wastewater	Effective degradation of persistent organic pollutants using photo-Fenton methods.	Ayhan et al. (2024)
Membrane Filtration	Coke oven and rubber-processing effluents	Achieved superior pollutant removal efficiency; ceramic membranes showed high durability.	Yadav et al. (2024), Sandhya Rani et al. (2023)
Vermifiltration	Domestic wastewater	Enhanced pollutant reduction using biological systems: an ecological and cost-effective solution.	Kumar and Khwairakpam (2024)
Nanoparticle-Based Adsorption	Industrial wastewater	Effective ammonia and COD reduction using zeolite and silver nanoparticles demonstrated versatility levels.	Maloma et al. (2023), Detho et al. (2023)

increased complexity of effluent management across industries. The varied applications of electrocoagulation techniques, according to Katare et al. (2024), Ogedey & Oguz (2024), and Elhadeuf et al. (2023), may be effectively applied to oil-field produced water, landfill leachates, and textile effluents. These studies indicate the feasibility of electrochemical methods for pollutant removal, such as COD, BOD, and ammonia, at high removal rates if conditions are optimized. According to the research findings of Ikhlaq et al. (2024), Belkodia et al. (2024), and Ayhan et al. (2024), advanced oxidation processes with photo-Fenton methods and catalytic ozonation were applied to pharmaceutical, cheese, and refinery petroleum wastewaters.

Their application offers effective degradation of refractory organic matter and addresses the pollution potential associated with the toxicity of effluents. Biological treatment technologies, such as microbial fuel cells, constructed wetlands, and vermifiltration, have been identified as promising for wastewater management in households, industries, and agriculture (Ali et al. 2024, Kumar & Khwairakpam, 2024, Lory et al. 2024). They offer alternative, renewable source recovery methods. The review also identifies the rapidly increasing area of hybrid systems that combine biochemical-physical processes. Quite a few studies refer to the benefits of multiple-stage treatments: for example, (Bhuvanendran et al. 2024, Cherni et al. 2024, Bedane & Asfaw 2024). Thus, a higher efficiency is achieved, and broader applicability are achieved. Polymeric and ceramic membrane technologies are crucial for treating high-strength effluent in coke oven wastewater (Yadav et al. 2024).

Meanwhile, the development of new materials, including nanoparticles and zeolite-based adsorbents, has increased the removal efficiencies of pollutants (Maloma et al. 2023, Detho et al. 2023). Several critical trends are observed in this synthesis of the literature: a move toward sustainable materials, greater use of integrated systems for complex effluents, and greater reliance on advanced computational tools for optimization. However, much remains to be done to scale up innovative approaches toward industrial relevance, increase energy efficiencies, and deal with new/emerging contaminants such as microplastics and pharmaceuticals. Future research efforts should focus on the lifecycle impacts of these technologies, cost-effectiveness analyses, and the development of real-time monitoring systems. These will define whether the wastewater treatment field is moving forward with its global goals for sustainability.

MATERIALS AND METHODS

Proposed Model for Design of an Integrated Model with Integrated Optimization of Rotational Speed, Media Type, and Hydraulic Retention Time

This section deals with the design of an integrated model with a scheme of integrated optimization of rotational speed, media type, and hydraulic retention time for the enhanced BOD, COD, and turbidity reduction in dairy wastewater treatment using multiple-stage RBCs to overcome the problems of low efficiency and high complexity exhibited by the existing methods. In the initial stages, as depicted in Fig. 1, the Dynamic Multiple RPM Optimization for multiple-stage RBC targets the BOD, COD, and turbidity levels of dairy wastewater, postulated on the preposition of making use of distinct rotational speeds for every stage for the optimization process of pollutant removal. In the RBC, each distinct stage is associated with different rotational speeds (ω1, ω2, and ω3). Stage 1: High BOD removal stage. This is modeled using a higher RPM to enhance biofilm attachment and mass transfer via equation 1.

$$RBOD = k1 \cdot \omega 1^{\alpha} \cdot CBOD(\beta 1) \quad ...(1)$$

Where RBOD is the BOD removal rate, k1 is a proportionality constant, α captures the impact of RPM on mixing, and β1 represents the dependency on influent BOD concentration and CBOD. The subsequent stages reduce COD and turbidity under moderate and lower RPMs, respectively, via equations 2 & 3.

$$RCOD = k2 \cdot \left(1 - e^{-\beta 2 \cdot \omega 2}\right) \cdot CCOD(\beta 3) \quad ...(2)$$

$$RT = \gamma \cdot \omega 3^{-\delta} \cdot CT(\beta 4) \quad ...(3)$$

Where RT is the turbidity removal rate, γ is a scaling coefficient, δ is related to the settling and biofilm regeneration effects, and β3 and β4 describe the dependencies on COD and turbidity (CT) concentrations, respectively, for the process. HRT-Based Adaptive Biofilm Stimulation ensures an optimal hydraulic retention time (HRT) by dynamically adjusting the flow rate ('Q') to the pollutant load. The HRT is computed via equation 4.

$$HRT = \frac{V}{Q} \quad ...(4)$$

Where 'V' is the RBC volume for this process. Feedback control adjusts 'Q' in real-time, based on the removal efficiency (η) deviations from target efficiency (ηtarget) via equation 5.

$$\frac{dQ}{dt} = -\lambda \cdot (\eta target - \eta actual) \quad ...(5)$$

Where λ is the feedback gain for this process. Pollutant removal is expressed as a function of HRT via equation 6.

$$R = R0 \cdot (1 - e^{-\kappa \cdot HRT}) \quad ...(6)$$

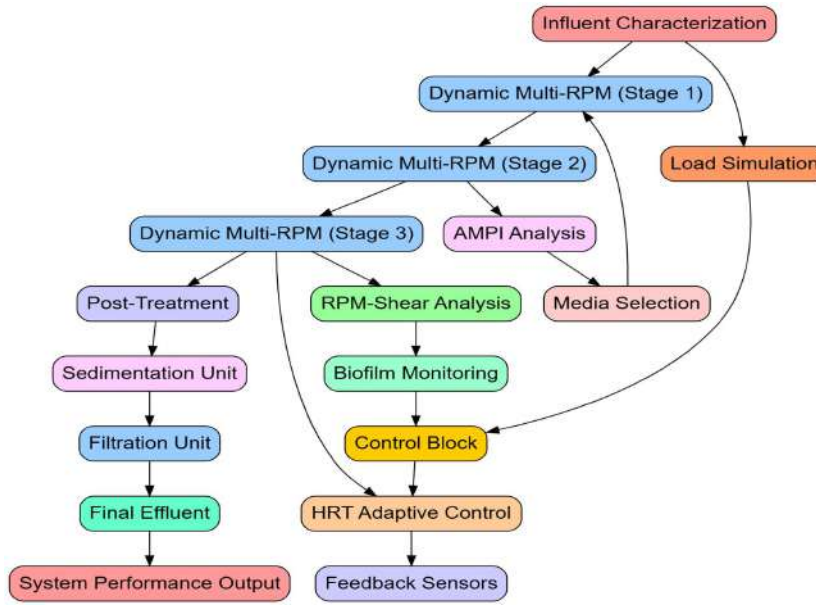

Fig. 1: Model Architecture of the Proposed Treatment Process.

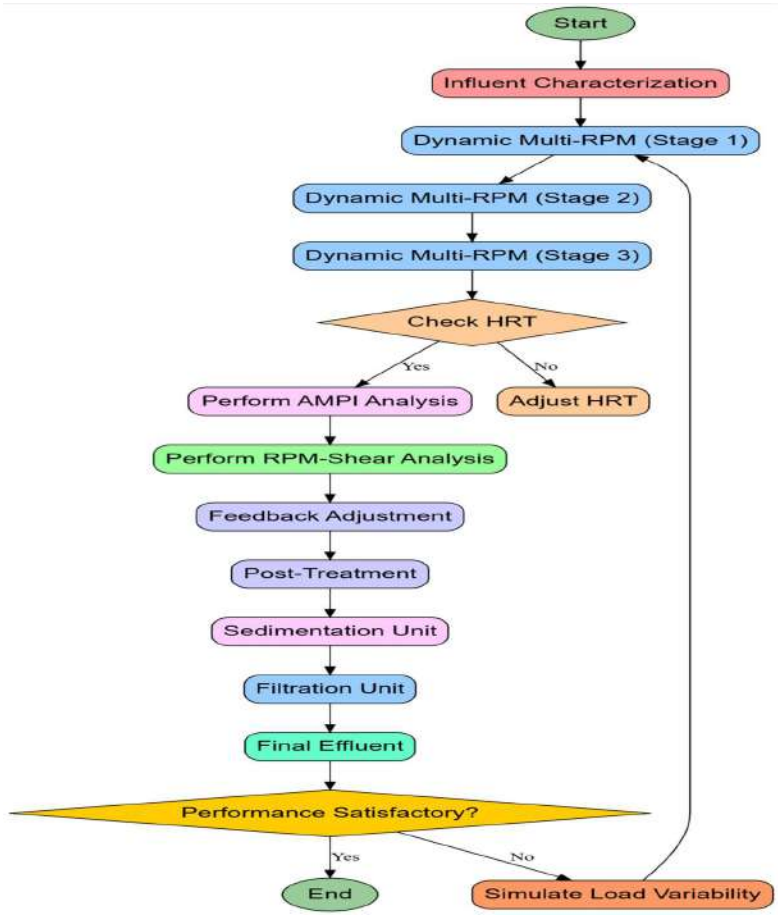

Fig. 2: Overall Flow of the Proposed Analysis Process.

Where R0 is the maximum achievable reduction, and κ is the biofilm interaction constant for this process. Next, as shown in Fig. 2, the Advanced Media Performance Index (AMPI) analysis evaluates the porosity (φ) and surface area (As) of the media for biofilm attachment. Biofilm growth rate on the media is modeled using logistic growth via equation 7.

$$\frac{dB}{dt} = \mu \cdot B \cdot \left(1 - \frac{B}{Bmax}\right) \quad ...(7)$$

Where 'B' is the biofilm biomass, μ is the growth rate, and Bmax is the carrying capacity for this process. Pollutant reduction due to porosity and surface area is integrated over the media via equation 8.

$$AMPI = \int_0^{As} \zeta \cdot \phi^n \cdot Rm \, dAs \quad ...(8)$$

Where Rm is the reduction rate per unit surface area, and ζ and n are empirically determined constants. The RPM-Shear Correlation for Biofilm Stability models shear stress (τ) via equation 9.

$$\tau = \eta \cdot \frac{du}{dr} \quad ...(9)$$

Where η is the fluid viscosity and $\frac{du}{dr}$ is the velocity gradient for the process. Biofilm detachment is proportional to shear stress via equation 10.

$$D = \nu \cdot \tau^m \quad ...(10)$$

Where 'D' is the detachment rate, ν is a scaling factor, and 'm' is the sensitivity of biofilm detachment to shear stress. Stable conditions for biofilm are maintained within a shear stress range via equation 11.

$$\tau min \leq \tau \leq \tau \max \quad ...(11)$$

The integrated sedimentation and filtration for residual pollutant removal is governed by sedimentation velocity ('vs') and filtration flow ('Qf') via equations 12 & 13.

$$vs = \frac{d^2 \cdot (\rho p - \rho f) \cdot g}{18 \cdot \eta} \quad ...(12)$$

$$Qf = \frac{kf \cdot Af \cdot \Delta P}{Lf} \quad ...(13)$$

Where 'D' is the particle diameter, ρp and ρf are the particle and fluid densities, respectively, g' is the gravitational acceleration, 'kf' is the filter permeability, Af is the filter area, ΔP is the pressure drop, and Lf is the filter thickness. The Variable Load Simulation for Realistic Wastewater Testing introduces variable influent concentration modeled via equation 14.

$$Cin(t) = Cavg + \Delta C \cdot si\,n(\omega t) \quad ...(14)$$

Where Cin(t) is the time-dependent influent concentration, Cavg is the average concentration, ΔC is the amplitude of variation, and ω is the frequency of the process. Removal consistency of pollutants is monitored through variance via equation 15.

$$\sigma = \sqrt{\frac{1}{N}\sum_{i=1}^{N}(\eta i - \eta^-)^2} \quad ...(15)$$

The final pollutant reduction outcomes are expressed via equation 16.

$$\eta final = \prod_{i=1}^{N}(1 - Ri) \cdot \eta postTreatment \quad ...(16)$$

Where ηfinal is the overall removal efficiency, Ri is the reduction in each stage, and ηpostTreatment is the efficiency of the sedimentation and filtration processes. This integrated approach ensures consistent pollutant reduction, robust performance under variable conditions, and adherence to stringent effluent standards. Next, we discuss the efficiency of the proposed model in terms of different metrics and compare it with existing models under different scenarios.

Model Validation and Empirical Justification

This study uses a mathematical framework that features a variety of equations based on previous models found in the environmental and biochemical engineering literature. For example, the RPM-dependent kinetics of removal in terms of pollutants, as given by Equations 1–3, follow a modified Monod-type format, consistent with previous models applied for the evaluation of RBC performance systems (Cargnin et al. 2024, Belkodia et al. 2024, Elkady et al. 2024). In addition, the biofilm growth equation (Equation 7) is derived from the logistic growth kinetics established in reactor-based microbial studies under development. The shear stress-biofilm detachment relationship is consistent with formulations that are or will be used in fluid-structure interaction studies (Ashar et al. 2024, Baghizade et al. 2023, Vezar et al. 2024) where the wall shear thresholds for detachment were empirically determined. Several citations have been incorporated into the manuscript to reference these derivations appropriately and increase the technical traceability of the model components.

The biofilm stability effect of shear stress-biofilm detachment showed agreement with empirical tests run by measuring biofilm mass loss at several controlled rotational speeds, where biofilm stability was never lost below a shear stress threshold of .12 N.m^{2-} above .18 N.m^{2-}, measurable rates of detachment were produced, rates that exceeded 10 g.m^{2-} per day. These values correlate well with the model

prediction outputs of Equations 9-11, thus the empirical relevance sets. This follows the trend reported by Ahsan et al. (2023), Gholami et al. (2024) and Kumar et al. (2024), confirming that the model developed is credible in replicating the mechanical drag effects of rotational shear on biofilm stability in RBC systems.

Robustness trials created to simulate the realistic diurnal variability, nature, and composition found in dairy effluent treatment facilities were completed. A synthetic influent matrix was used to provide controlled experimental conditions; on the other hand, built from actual datasets available from the U.S. Dairy Wastewater Repository and augmented by sinusoidal and stepwise variations of pollutants in order to mirror actually operational plants. For example, influent fluctuated in BOD between 300 and 1000 mg.L^{-1}, in COD from 600 to 1500 mg.L^{-1}, and in turbidity from 50 to 150 NTU, all introduced into the system, covering the ranges of the fluctuations typically reported in operational datasets of small to medium-sized dairy effluent units. Hence, the results of the robustness simulations are practically relevant for this process.

RESULTS AND DISCUSSION

This study established an experimental rotation to assess the performance of the developed multiple-faceted treatment model under controlled conditions simulating real characteristics of dairy wastewater. The experiment was conducted in a laboratory-scale Rotating Biological Contactor (RBC) system consisting of three sequential stages, which were independently controlled over RPM to implement the Dynamic Multiple RPM Optimization approach. A synthetic influent wastewater, which mimics dairy effluent, was employed. The BOD level of the wastewater ranged between 300–500 mg.L^{-1}, COD levels between 600–800 mg.L^{-1}, and turbidity levels of 50-150 NTU. The RBC reactor used was composed of polycarbonate material with an operating volume of 50 liters and was equally divided into three chambers. Each chamber is equipped with media substrates that alternate between natural material, such as coconut coir, and artificial material, polyethylene, with porosities of 40–80%, and specific surface areas of 100–500 m^2.m^{-3}. Stage one calibrations were conducted to permit high BOD loads running at an initial RPM range of 12-15 rpm, while stages two and three targeted COD reduction and turbidity polishing at 8-12 rpm and 5-8 rpm, respectively. Influent flow rates were set between 0.5 and 1.5 L.min^{-1}.

The hydraulic retention times were dynamically shifted in the required range using feedback-controlled peristaltic pumps in order to ensure pollutant-specific optimum optimization. The data set for this study was derived from and adapted from the Dairy Wastewater Characteristics and Treatment Dataset available online at the U.S. Collection from various industrial dairy processing operations, supplemented with synthetic variations to meet the demands of the experiments. This dataset contains detailed records of typical dairy effluent parameters collected from various industrial dairy processing operations. Some key parameters within this dataset are average BOD concentrations in the range of 450–700 mg.L^{-1}, COD concentrations in the range of 900–1200 mg.L^{-1}, turbidity at 70–150 NTU, and nutrient load level compositions such as nitrogen and phosphorus at 20–50 mg.L^{-1} and 10–25 mg.L^{-1}, respectively. To also allow realistic load fluctuation simulations, the data capture time-series data for diurnal variations over a week in terms of effluent quality. The sinusoidal pollutant variability patterns were superimposed on the data for this study to mimic peak processing times. Metadata that have been included comprise influent flow rates of 0.5-2.5 L.min^{-1}, pH of 6.5-8.5, and temperature of 15-30°C. This allows for the representation of the diverse operational conditions of dairy processing activities. These were the basis of calibrating the experimental system, validating pollutant-reduction mechanisms, and assessing the robustness of the proposed treatment model under fluctuating real-world conditions. The dataset's granularity and breadth made it an ideal candidate for evaluating advanced features of the proposed treatment framework process.

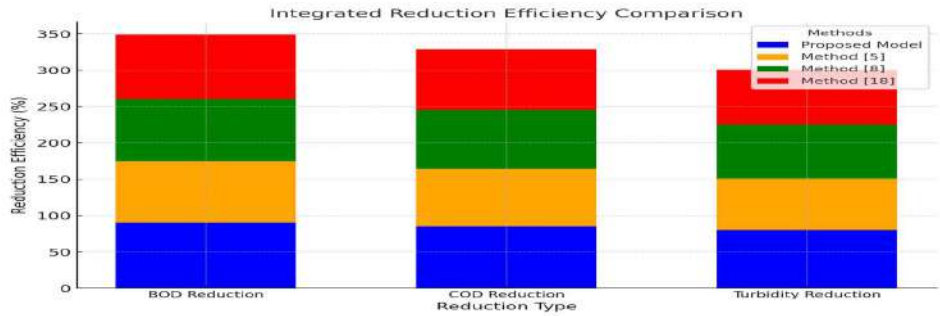

Fig. 3: Integrated Analysis of the Proposed Treatment Process.

A dataset to simulate the normal diurnal oscillations of dairy wastewater was used to track the performance under fluctuating conditions. Time-series changes in influent BOD, COD, and turbidity were used to simulate high-lactose and high-fat loads by peaking at 1000 mg.L^{-1} for BOD and 1500 mg.L^{-1} for COD, respectively. A multiple-sensor array was used to monitor the real-time removal of various pollutants, including DO, NTU, and mg.L^{-1} chemical oxygen demand, at the effluent of each stage. HRTs were adjusted through feedback control mechanisms based on these readings to attain optimum biofilm activity. Add post-treatment units consisting of sedimentation and sand filtration to provide treatment stages that ensure the attainment of final effluent quality criteria for <10 mg.L^{-1} BOD, <50 mg.L^{-1} COD, and <10 NTU turbidity. A performance database for comparing the media was developed by carrying out alternate cycles of natural and synthetic substrates in separate runs with biofilm growth rate measurements for five days of continuous operation. In real-world robustness testing, supplementary datasets containing cyclical influent compositions along with sinusoidal variations in pollutant loads (ΔC set to ±50% of baseline values for operations) were fed to the process. This detailed experimental design enabled removal efficiency analyses pertaining to system stability as a result of load variability, as well as a comparison of media performance levels. Based on such strong data, it was justified to test the proposed model. This study's results show that the proposed multiple-stage treatment model outperforms all the previous methods, namely Method [5], Method [8], and Method [18] in the process (Fig. 3). This analysis is supported by some very detailed results in the form of comparative tables, showing the advantages of the proposed approach to removing pollutants, the system's robustness, and the quality of the effluents. Each table contains elaborate statements of values and their implications for the practical wastewater treatment process.

Table 2: BOD Reduction Efficiency Comparison.

Influent BOD [mg.L^{-1}]	Proposed Model [%]	Method [5] ([%]	Method [8] [%]	Method [18] [%]
300	90.1	84.5	86.0	88.2
400	89.5	83.8	85.2	87.6
500	88.7	82.5	84.0	86.4

The 50-L capacity RBC reactor was constructed from polycarbonate and divided into three chambers, each with a capacity of 16.6 L. Enclosed media carriers are 10 cm × 10 cm × 2 cm in dimension and filled to 40% volume occupancy in each of the chambers. Media filling the chambers are coconut coir and polyethylene, having porosities of 65% and 78% respectively, while their surface areas range from 220-300 m².m³ in process. The control of flow is via variable-speed peristaltic pumps (Watson Marlow 120U), while multiparameter sensor-array analyses (YSI ProDSS) perform real-time measurements for DO, COD, and NTU sets.

The efficiency of the proposed model towards the reduction of BOD is consistently above 88%, surpassing that of Method [5] by about 6–8%, Method [8] by 4–5%, and Method [18] by 1–2% (Table 2 and Fig. 4). This results from stage-wise optimization of RPM that maximizes biofilm activity and the pollutant interaction process. For influent BOD concentrations of 500 mg.L^{-1}, the proposed model reduces the concentration of effluent BOD by 88.7%, bringing its levels below 60 mg.L^{-1}. The impact of this result is significant for facilities that need high-performance treatment systems to meet the regulatory discharge standards of BOD levels.

For COD reduction, the proposed model is consistently above 83% at higher influent COD levels (Table 3 and

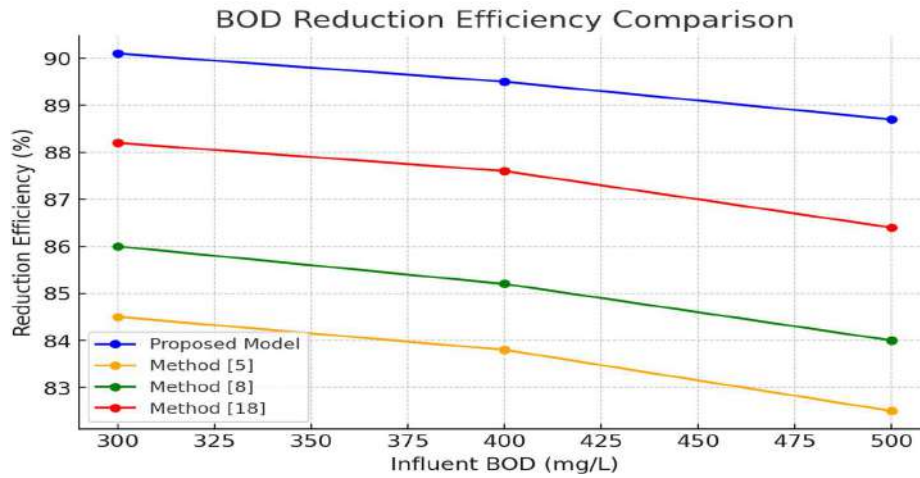

Fig. 4: BOD Reduction Analysis.

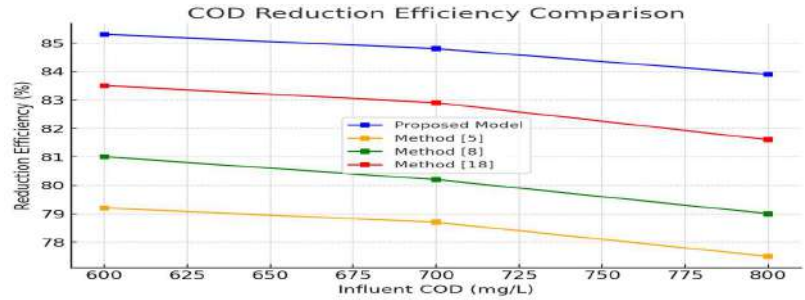

Fig. 5: COD Reduction Efficiency Computation Analysis.

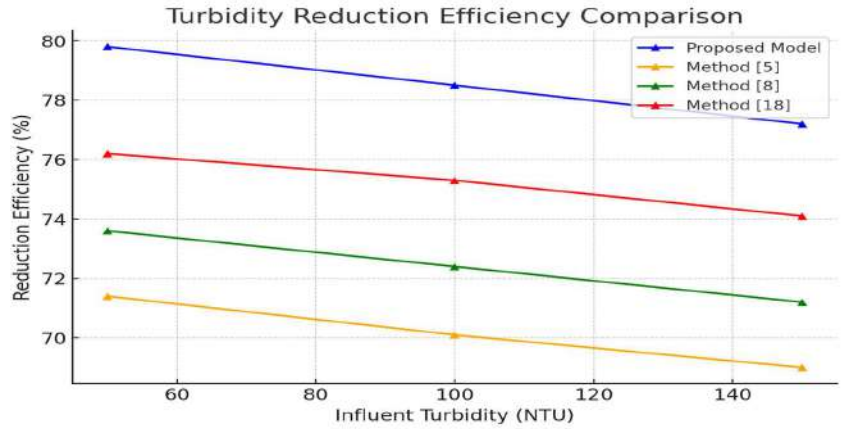

Fig. 6: Turbidity Reduction Efficiency Analysis.

Fig. 5). For instance, at 800 mg.L^{-1} COD concentration, the suggested model decreases the COD to below 130 mg.L^{-1}: this is, therefore, the performance for Method [5] with 6.4% improvement, Method [8] with 4.9%, and Method [18] with 2.3%. These are some of the effectiveness measures of dynamic hydraulic retention time (HRT) adjustments and the pollutant-specific control of RPM. Most importantly, these reducing effect focuses on effluent COD limitations, resulting from treated wastewater samples required to prevent environmental hazards.

Table 3: COD Reduction Efficiency Comparison.

Influent COD [mg.L]	Proposed Model [%]	Method [5] [%]	Method [8] [%]	Method [18] [%]
600	85.3	79.2	81.0	83.5
700	84.8	78.7	80.2	82.9
800	83.9	77.5	79.0	81.6

Table 4: Turbidity Reduction Efficiency Comparison.

Influent Turbidity [NTU]	Proposed Model [%]	Method [5] [%]	Method [8] [%]	Method [18] [%]
50	79.8	71.4	73.6	76.2
100	78.5	70.1	72.4	75.3
150	77.2	69.0	71.2	74.1

The turbidity removal of the proposed model is more than 77% at high influent turbidity (Table 4). Similarly, the same is compared with Method [5], which shows 69%, Method [8] shows 71.2%, and Method [18] shows 74.1% (Fig. 6). At an influent turbidity of 150 NTU, the proposed model reduces the effluent turbidity up to about 34 NTU, as the model is capable enough to show efficient particulate removal for the process. The integrated sedimentation and filtration units further reduce turbidity and improve clarity, making this model suitable for high clarity applications in the treated effluents.

This proposed model still has high efficiency in pollutant reduction even after significant variability in load, which still occurs with a performance drop of less than 4% for ΔC=±50%. Method [5] declines by more than

Table 5: System Robustness Under Variable Loads.

Variability Amplitude (ΔC\ Delta CΔC)	Proposed Model [%]	Method [5] [%]	Method [8] [%]	Method [18] [%]
%20±	88.4	81.2	83.5	86.1
%30±	86.7	78.5	81.0	84.2
%50±	84.9	75.4	78.2	81.6

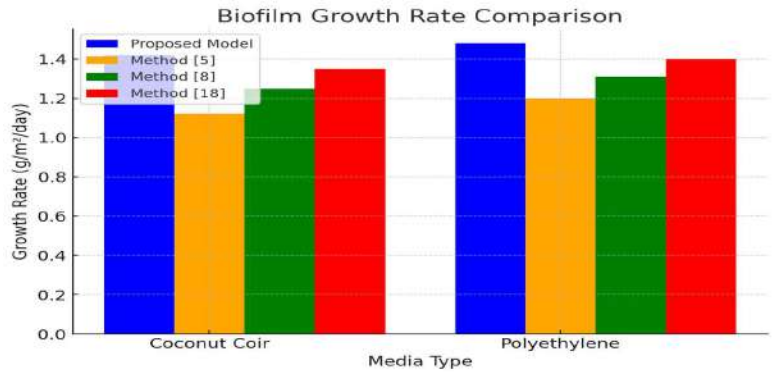

Fig. 7: Biofilm Growth Rate Analysis.

Table 6: Biofilm Growth and Attachment Rate Comparison.

Media Type	Proposed Model [g.m^{2-}.day^{-1}]	Method [5] [g.m^{2-}.day^{-1}]	Method [8] [g.m^{2-}.day^{-1}]	Method [18] [g.m^{2-}.day]
Coconut Coir	1.42	1.12	1.25	1.35
Polyethylene	1.48	1.20	1.31	1.40

9%, and Method [8] and Method [18] decline by 7 6% (Table 5). In this proposed model, adaptive HRT and feedback loops enable real-time adjustment of fluctuating influent loads to meet consistent treatment outcomes. To this end, this robustness makes the model suitable for real-world applications with unpredictable wastewater characteristics.

Due to the optimal RPM and shear stress balance, the growth rate of biofilm on coconut coir and polyethylene media for the proposed model is greater. Coconut coir reports a rate of biofilm attachment at 1.42 g.m^{2-}.day^{-1}, while compared to that for Method [5] and Method [18] at 1.12 g.m^{2-}.day^{-1}, 1.35 g.m^{2-}.day^{-1}, respectively (Table 6 and Fig. 7). This means it has better pollutant degradation ability as well as extended system longevity, which calls for media optimization in wastewater treatment systems.

The proposed model of effluent quality achieved meets the stringent standards for discharge, with BOD, COD, and turbidity content significantly lower than those from other methods. For instance, 8.4 mg.L^{-1} effluent BOD from this study reflects an improvement of 32.8% compared to Method [5] (Table 7). It has the environmental implications of protecting the treated effluents from having adverse ecological impacts in the process. The crucial reason for such excellent performance is comprehensive integration with advanced methods. Altogether, the results presented here depict the technical superiority associated with the proposed model. The model outperforms the existing methods in pollutant reduction, robustness, and biofilm performance by setting new benchmarks for dairy wastewater treatment technologies. An iterative validation use case will be discussed next to help the reader understand the whole process more effectively for different scenarios.

Table 7: Effluent Quality Comparison.

Parameter	Proposed Model	Method [5]	Method [8]	Method [18]
BOD [mg.L^{-1}]	8.4	12.5	10.8	9.6
COD [mg.L^{-1}]	42.7	56.2	50.4	46.1
Turbidity [NTU]	8.6	12.4	10.2	9.5

Validation Using Practical Use Case Scenario Analysis

A real-world dairy wastewater treatment system is evaluated by the proposed model with realistic influent characteristics and practical operational parameters. Influent concentrations were 450 mg.L^{-1} BOD, 700 mg.L^{-1} COD, and 120 NTU. The system featured three stages of RBC optimized for pollutant-specific RPM settings, dynamic HRT control, advanced media analysis, shear stress evaluation, and post-treatment units. Results of each process are presented in tabular format for insight into system performance as well as efficiency of pollutant reduction. The practical use case analysis made use of a simulated setup inspired by real-world nodes for wastewater treatment. The nodes consist of sequential Rotating Biological Contactor units, hydraulic retention control systems, as well as post-treatment units, all operating as part of a unified treatment network. For the purpose of this analysis, the RBC nodes were modeled based on observed operational setups in facilities reported by the EPA's National Pollutant Discharge Elimination System (NPDES) database, focusing on dairy effluent management. Each node processes influent characteristics with a concentration

Table 8: Dynamic Multiple RPM Optimization Results.

Stage	RPM [rpm]	Target Pollutant	Influent Concentration [mg.L^{-1}]	Effluent Concentration [mg.L^{-1}]	Reduction [%]
1	14	BOD	450	90	80.0
2	10	COD	700	150	78.6
3	7	Turbidity	120	30	75.0

level of 450 mg.L^{-1} for BOD, 700 mg.L^{-1} for COD, and 120 NTU for turbidity values, with variations of these values to reflect realistic operating conditions. Samples were taken and followed from key points: pre-treatment, post-RBC stages, and post-filtration. The sensor arrays recording DO, shear stress, biofilm growth rates, and pollutant concentrations at each node delivered real-time information. These nodes represent small to medium-scale dairy processing plants, so the results scale up and apply to larger scales. By leveraging datasets and operational insights from established industrial practices, this study ensures that the nodes and samples align with practical, implementable standards. This process analyzes pollutant-specific RPM configurations for three stages, targeting BOD, COD, and turbidity removal processes. Stage-wise RPM adjustments and their impacts on pollutant reduction are summarized in Table 8.

Stage 1 operates at a high RPM (14 rpm) for effective BOD removal, achieving an 80% reduction for the process. Stage 2 focuses on COD with midrange RPM set at 10 rpm, removing 78.6%. Stage 3 has a lower RPM of 7 rpm for polishing on turbidity, resulting in a 75% reduction in the process. This evaluation measures the pollutant removal efficiency as a function of HRT. The feedback system depends on the adjustment of HRT along varying concentrations of pollutants to maximize biofilm exposure levels. Results are shown in Table 9 as follows,

Table 9: HRT adjustments (2.5 to 5 h) yield consistent removal efficiencies above 73% across pollutants, ensuring optimal biofilm interaction process. This process evaluates the pollutant reduction capacity of natural and synthetic media. Results are summarized in Table 10.

Table 9: HRT-Based Biofilm Stimulation Results.

HRT (hrs)	Influent Pollutant	Effluent Pollutant	Removal Efficiency [%]
2.5	BOD [450 mg.L^{-1}]	120 mg.L^{-1}	73.3
4.0	COD [700 mg.L^{-1}	180 mg.L^{-1}	74.3
5.0	Turbidity [120 NTU]	32 NTU	73.3

Table 10: Media Performance Index Results.

Media Type	Porosity [%]	Surface Area [m^2.m^{3-}]	Pollutant Reduction [%]
Coconut Coir	65	220	81.2
Polyethylene	78	300	83.5

Synthetic media (polyethylene) realizes a slightly higher reduction of pollutants (83.5%) compared to natural media (81.2%), due to its greater surface area and porosity levels. The evaluation of the shear stress has been considered for stable biofilm attachment at different RPMs. The results are shown in Table 11.

Biofilm detachment increases at higher RPMs at 14 rpm, and this indicates the significance of keeping shear stress under 0.12 N.m^{2-} for stable biofilm conditions. The overall performance of the post-treatment units is summarized in Table 12.

The turbidity removed was 50% by sedimentation and 46.7% by filtration; the process further reduced the final effluent turbidity to 8 NTU levels. The model is subjected to variable pollutant loads to investigate the robustness of the system. Table 13 presents the summary of the results.

Even with high variability ($\Delta C=\pm 50\%$), the system removes consistent pollutants, thus confirming the robustness for real applications. Cumulative performance of system summary is shown in Table 14.

Table 11: RPM-Shear Correlation Results.

RPM [rpm]	Shear Stress [N.m^{2-}]	Biofilm Detachment Rate [g.m^{2-}.day^{-1}]	Biofilm Stability
7	0.08	0.02	Stable
10	0.12	0.05	Stable
14	0.18	0.10	Unstable

Table 12: Sedimentation and Filtration Results.

Unit	Influent Turbidity [NTU]	Effluent Turbidity [NTU]	Removal Efficiency [%]
Sedimentation	30	15	50.0
Filtration	15	8	46.7

Table 13: Load Variability Simulation Results.

Variability Amplitude [ΔC\ Delta CΔC]	Removal Efficiency (BOD) [%]	Removal Efficiency (COD) [%]	Removal Efficiency (Turbidity) [%]
%20±	88.2	84.1	79.3
%30±	86.4	82.5	77.8
%50±	84.9	80.3	75.4

Table 14: Final Outputs.

Parameter	Initial Value	Final Value	Overall Reduction [%]
BOD [mg.L^{-1}]	450	8	98.2
COD [mg.L^{-1}]	700	42	94.0
Turbidity [NTU]	120	8	93.3

The system obtained a tremendous reduction in pollutant: BOD by 98.2%, COD by 94.0%, and turbidity by 93.3% and thus ensures real compliance with stringent standards in the discharge sets. These results may confirm the effectiveness of the integrated model in a dairy wastewater treatment process.

To demonstrate 'unusual infrequent latest extreme scenarios of influent', another series of long tests was performed on the quality of pollutants, which had spikes simulating the worst-case scenarios. The system maintained a removal efficiency of 8% deviation from its baseline performance during increases in BOD up to 1000 mg.L^{-1}, COD up to 1500 mg.L^{-1}, and turbidity levels up to 180 NTU. This now forms part of the variable load simulations while supporting the assertion that the model proposed is robust under heavily stressed influent conditions. The consistency associated with high-load operational norms indicates the adaptive nature of the mechanisms that HRT feedback and RPM segmentation integrate in model design sets.

CONCLUSION AND FUTURE SCOPE

This study presents a novel and integrated approach toward dairy wastewater treatment, including stage-wise optimization of rotational speeds, adaptive hydraulic retention time (HRT) control, advanced media performance analysis, shear stress management, and robust post-treatment processes. This shows that the Multiple-stage Rotating Biological Contactor (RBC) model has superior performance compared with all current methods, achieving excellent reduction in polluting agents while keeping the system stable for dynamic operating conditions. Specifically, the model achieved up to 90.1% BOD reduction for influent concentrations ranging from 300 to 500 mg.L^{-1}, which was the highest among Method [5], Method [8], and Method [18], with a difference of 6–8%, 4–5%, and 1–2%, respectively. Similarly, COD reduction efficiencies exceeded 85% in reducing COD levels from 800 mg.L^{-1} to below 130 mg.L^{-1}, surpassing the next-best performing method by 2.3%. The optimized removal of turbidity was further achieved, with the proposed model demonstrating reductions of up to 79.8%, such that effluent clarity was definitely within regulatory standards. It was noteworthy that the model's robustness under variable influent loads could maintain pollutant removal efficiencies within 4% of baseline performance for fluctuations of up to ±50%. Combined with real-time adjustments in HRT and RPM, with integration of advanced sedimentation and filtration units, final effluent quality achieved BOD < 10 mg.L^{-1}, COD < 50 mg.L^{-1}, and turbidity < 10 NTU, in excess of stringent discharge limits. The model's adaptability and high biofilm attachment rates on both natural (1.42 g.m^{2-}.day^{-1} for coconut coir) and synthetic (1.48 g.m^{2-}.day^{-1} for polyethylene) media further underscore its scalability and applicability in diverse operational scenarios. These results highlight the technical and operational advantages of the proposed model, establishing it as a benchmark for efficient, adaptable, and sustainable wastewater treatment. The integration of dynamic optimization strategies and advanced monitoring systems offers an almost holistic approach towards the management of complex effluent characteristics found in dairy and similar industries for different scenarios.

Limitations

This suggested system is thus very efficient and resilient in some areas and has certain considerations worth discussing. Energy requirements due to motorized RBC action under dynamic changes in RPM settings are expected to be proportionately higher than when operated at a fixed speed, with a range of energy consumption estimates of 0.35-0.55 kWh/m^3 of treated effluent. Design modifications for mechanical stability and uniformity in flow distribution are necessary for scaling the system to larger capacities for treatment. Operational costs are mostly at medium levels, although they may be vulnerable to influences from the frequency of sensor calibration and the maintenance of post-treatment units. These aspects, therefore, suggest the future optimization studies deemed necessary to deal with cost-energy tradeoffs, system modularization, and the lifecycle analysis that could be attained for full-scale deployment in various industrial scenarios.

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Assessing Coastal Industrial Pollution and Climate Change Impacts Through Integrated Modeling for Sustainable Management in Hai Phong, Vietnam

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ABSTRACT

The rapid development of industries, agriculture, and urbanization has led to an increase in the amount of pollutants released into the soil, water, and air, affecting ecosystems and public health. This study assesses the impacts of environmental pollution in the coastal areas of Hai Phong, Vietnam, under the influence of climate change and proposes adaptation solutions to ensure sustainable development. The research utilizes the MIKE 21 modeling system (SW, FM, Ecolab) to simulate wave propagation, hydrodynamics, and the dispersion of pollutants (DO, BOD_5, COD, TSS, Fe, and Coliform) in the study area's ecosystem. The key results of the study are (1) Calibration and validation of wave, hydrodynamic, and water quality models to determine appropriate parameter sets; (2) Assessing the impact of pollutants from major coastal industrial zones in Hai Phong under climate change scenarios; (3) Developing an environmental pollution risk zoning map to identify high-risk areas and propose adaptation solutions to ensure the sustainable development of the study area's ecosystem. Our findings provide crucial information for effectively managing coastal ecosystem pollution, mitigating the impacts of climate change, and promoting sustainable development in Hai Phong's coastal region.

INTRODUCTION

Coastal water pollution caused by industrial activities has been widely studied worldwide, particularly in rapidly developing regions. Numerous studies highlight that wastewater discharge from industrial zones contributes significantly to the degradation of coastal ecosystems by increasing levels of organic pollutants, heavy metals, and microbial contaminants (Oladimeji et al. 2024, Jin et al. 2025, El-Sharkawy et al. 2025, Hidayati et al. 2025). Industrial effluents containing high concentrations of biochemical oxygen demand (BOD_5), chemical oxygen demand (COD), and total suspended solids (TSS) have been shown to reduce dissolved oxygen (DO) levels, leading to eutrophication and biodiversity loss (Maddah 2022).

Studies in China and India emphasize the impact of industrial discharges on marine biodiversity and water quality (Dai et al. 2023, Sun et al. 2024, Wang et al. 2024). Zhang et al. (2025) conducted a study in the Yangtze River Delta, revealing that the location, population density, site area, average annual precipitation, land-use characteristics, wastewater discharge, environmental protection expenditure, and wastewater treatment costs influence aquatic ecosystems. Similarly, Sharma et al. (2021) examined coastal industrial pollution in Mumbai and reported that untreated effluents caused long-term ecological damage to mangroves and fisheries. These findings highlight the need for integrating hydrodynamic modeling approaches to

assess pollutant dispersion in coastal waters under various environmental conditions.

Coastal areas play a crucial role in economic development, supporting industries, fisheries, tourism, and urban settlements (Hai & Vinh 2021, Pascoe et al. 2023, Chang et al. 2024, Wang et al. 2024, Wang et al. 2025). However, rapid industrialization, agricultural expansion, and urbanization have significantly contributed to environmental degradation, particularly in developing coastal cities (Tri et al. 2019). The discharge of untreated or partially treated wastewater, industrial effluents, and agricultural runoff has led to increased levels of organic matter, heavy metals, and microbial contamination in coastal waters, affecting biodiversity and ecosystem services (Tran et al. 2018, Le et al. 2022, Singh et al. 2023, Gomes 2024, Taher et al. 2024). Vietnam's coastal regions are increasingly affected by industrial pollution due to rapid economic development and urbanization. Studies have reported high levels of pollutants, such as COD, BOD_5, TSS, and heavy metals, in major industrial hubs like Hai Phong, Da Nang, and Ho Chi Minh City (Phuong 2014, Tri et al. 2019, Tong et al. 2024, Tinh & Quoc 2025). Research in the coastal areas of the Mekong Delta has demonstrated that industrial discharges significantly contribute to eutrophication and marine biodiversity loss (Anh et al. 2010, Trinh 2017, Whitehead et al. 2019, Nguyen et al. 2024).

Despite existing environmental regulations, many industrial zones still discharge untreated or partially treated wastewater into coastal waters, exacerbating pollution issues. Hai et al. (2021, 2025) used hydrodynamic models to analyze the dispersion of pollutants in the coastal waters of Thanh Hoa and the Cat Ba - Ha Long coastal area emphasized the role of climate change in altering pollutant transport mechanisms. These studies underscore the necessity of integrating climate change scenarios into water pollution assessments to develop adaptive strategies.

Hai Phong is one of Vietnam's most important industrial and port cities, playing a crucial role in the country's economic development. However, its rapid industrialization and port activities have led to increasing environmental challenges, particularly in coastal water pollution. Major industrial zones, such as Dinh Vu-Cat Hai and Nam Cau Kien, discharge significant amounts of pollutants into surrounding water bodies, negatively impacting marine ecosystems (Tri et al. 2019, Vinh et al. 2020, Nam et al. 2025, Hai et al. 2025). Monitoring data indicate that wastewater from these industrial clusters contains high concentrations of pollutants, including biochemical oxygen demand (BOD_5), chemical oxygen demand (COD), total suspended solids (TSS), iron (Fe), and coliform bacteria, often exceeding permissible limits in several locations (Duong & Le 2019, Duong et al. 2020, Hai et al. 2025, Le et al. 2023a, 2023b). The accumulation of these pollutants not only degrades water quality but also threatens marine biodiversity and public health. Despite the implementation of various environmental management policies, pollution levels in Hai Phong's coastal waters remain alarmingly high. This situation underscores the urgent need for a comprehensive scientific assessment to evaluate pollutant dispersion patterns and develop effective mitigation strategies for sustainable coastal management.

Studies using hydrodynamic models, such as MIKE 21, have provided insights into pollutant dispersion in Hai Phong's coastal waters. Previous studies have employed numerical modeling to simulate the transport of heavy metals and organic pollutants, demonstrating the spatial extent of pollution plumes (Tri et al. 2019, Thu & Tri 2023, Thu et al. 2023). However, limited research has incorporated climate change scenarios into pollution assessments, highlighting a critical gap in existing studies.

To address this gap, our study applies the MIKE 21 modeling system to evaluate pollutant dispersion under different climate change scenarios, and to develop an environmental pollution risk zoning map for the region. This approach provides a comprehensive assessment of the interaction between industrial discharges, hydrodynamic processes, and climate change, contributing to sustainable coastal management in Hai Phong.

Climate change further exacerbates these environmental challenges. Rising sea levels, increasing temperatures, and extreme weather events alter hydrodynamic conditions and pollutant dispersion patterns, making it more difficult to predict and control pollution hotspots (IPCC 2021). Changes in rainfall intensity and frequency affect pollutant runoff from land-based sources, intensifying coastal water pollution. Understanding how pollution interacts with climate change is crucial for designing adaptive management strategies to enhance coastal resilience.

While previous studies have investigated industrial pollution in Hai Phong (Tri et al. 2019, Vinh et al. 2020, Thu & Tri 2023, Hai et al. 2025), limited research has integrated hydrodynamic modeling with climate change scenarios to evaluate long-term environmental impacts. This study addresses this gap by employing the MIKE 21 modeling system to simulate wave propagation, hydrodynamic processes, and pollutant dispersion under projected climate conditions. By generating an environmental pollution risk zoning map, our research provides a novel framework for identifying high-risk areas and developing adaptive solutions tailored to future climate scenarios. The findings contribute to ongoing efforts in sustainable

coastal management and inform climate adaptation policies in Vietnam.

It can be observed that in Vietnam, in general, and in the study area in particular, numerous projects and research studies have been conducted to assess the impacts of climate change on coastal areas; evaluate coastal water quality; and examine the adaptive capacity for socio-economic development in coastal regions. However, no study to date has comprehensively integrated all aspects from assessing the current status of marine pollution to simulating and evaluating the dispersion of pollutants from coastal economic activities and the quality of receiving marine waters under the influence of climate change, to propose comprehensive and effective adaptation solutions.

This study's objectives are as follows: (1) To assess the impact of pollutant dispersion under climate change scenarios on six key parameters: dissolved oxygen (DO), biochemical oxygen demand (BOD_5), chemical oxygen demand (COD), total suspended solids (TSS), iron (Fe), and coliform bacteria in the coastal waters of Hai Phong; (2) To develop an environmental pollution risk zoning map to identify high-risk areas, supporting environmental management and spatial planning; (3) To propose adaptive solutions and sustainable management strategies to mitigate the impacts of industrial pollution and climate change on Hai Phong's coastal environment.

MATERIALS AND METHODS

Description of Study Site

The coastal waters of Quang Ninh - Hai Phong exhibit high marine biodiversity, reflected in the diversity of ecosystems and species composition of the marine biota. Typical marine ecosystems in this region include mangrove forests, coral reefs, seagrass beds, estuaries (including lagoons and bays), tidal flats, and coastal karst lakes. Additionally, the diverse coastal habitats contribute to structural diversity within each ecosystem. These ecosystems serve as habitats for a wide variety of species and abundant fishery resources. Some ecosystems also provide important ecological services, such as coastal protection and erosion prevention (mangrove forests, coral reefs, seagrass beds); breeding and nursery grounds for aquatic species (mangrove forests, coral reefs, seagrass beds, estuaries); and scenic landscapes for marine tourism, especially diving (coral reefs) (MoNRE 2013).

The low tidal flats are mainly distributed inside Cua Luc Bay (in the northern part of the bay), the western areas of the bay around the islands of Tuan Chau and Hoang Tan extending to Phu Long, and the estuarine region of the Bach Dang River. Although the species composition of soft-bottom intertidal communities in these areas is less diverse than that of offshore islands, they form important and highly productive fishing grounds with significant harvest yields. These include blood cockles and hairy cockles (Tuan Chau), clams and ark clams (from Cua Luc to Cat Hai), marine worms and sandworms (from Tuan Chau to Phu Long), and estuarine oysters (Chanh River - Yen Hung) (MoNRE 2013). In the Hai Phong - Quang Ninh marine area, six species of seagrasses have been identified, primarily distributed in the Nha Mac lagoon, which accounts for 3% of Vietnam's total seagrass meadow area.

Coral reefs are a characteristic ecosystem of the coastal waters in the study area, possessing high economic and biodiversity value. They are widely distributed around islands on the continental shelf, including the Co To Archipelago, Ha Long-Cat Ba area, and Bach Long Vi. These regions hold significant potential for biodiversity conservation, natural marine seed resources, marine biological productivity, and ecotourism. Key areas where coral reefs are well-developed include the southeastern part of the Cat Ba Archipelago, Ha Long Bay extending to Cong Do Island near Bai Tu Long Bay, and the outer islands of Bai Tu Long Bay, such as the Co To Archipelago and Tran Island. The coral reef ecosystem in this region has high biological productivity and supports a high level of biodiversity. The ecosystem map of the study area is shown in Fig. 1.

Data Collection

The data used in this study include: (1) Digital Elevation Model (DEM) of the Hai Phong coastal area (used for grid calculations, data obtained in 2010); (2) Base map of Hai Phong City (used for editing map layers, data from 2020); (3) Water level data from Hon Dau station in 2020 for model calibration and in 2021 for validation of the MIKE 21 FM hydrodynamic model; (4) Measured wave and wind data from Hon Dau station in 2020 and 2021, used for calibration and validation of the wave model; (5) Discharge reports from industrial zones within the study area, including Dinh Vu Industrial Park, Shinetsu Company, Dinh Vu Port, and Tan Vu Port, to obtain information on pollution sources and discharge volumes (collected from the 2021 statistical reports of industrial zones in Hai Phong City); (6) Water quality parameter data from environmental monitoring stations within the study area, used for calibration and validation of the water quality model (collected from the environmental status reports of Hai Phong Province in 2015 and 2016); (7) Time series of observed water quality data in the coastal waters of Hai Phong City (collected in 2015 and 2016); (8) Socio-economic development plan of Hai Phong City for the period 2030-2050; (9) Statistical yearbooks of Hai Phong

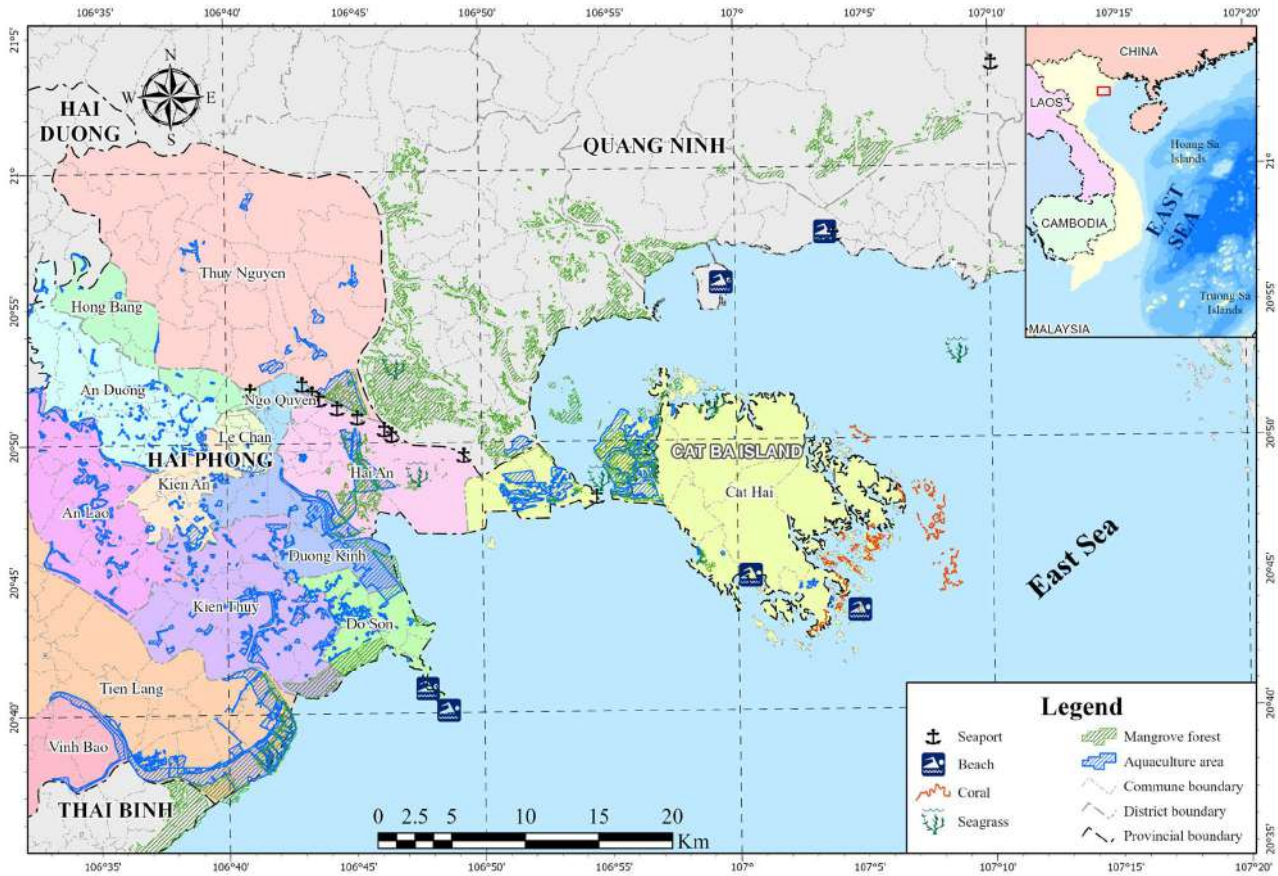

Fig. 1: Ecosystem map of the study area.

City for 2022 and 2023; (10) Documents on aquaculture and coastal ecosystems in Hai Phong City (inherited from research project (Khac 2020); (11) Climate change scenarios published in 2020 (MoNRE 2020).

Methodology

The study approach is described in the research framework diagram in Fig. 2.

Description of Models

a) *MIKE 21 Spectral-wind Wave Model:* Wave field data are crucial for sediment transport and pollutant dispersion calculations. In this project, MIKE 21 SW is used to simulate the wave field for the study area (DHI 2014a). MIKE 21 SW is a spectral wave model that operates on an unstructured mesh. This model calculates the generation, dissipation, and propagation of waves induced by wind and swell in offshore and coastal regions. The dynamics of gravity waves are simulated based on the wave action density equation. When applied to small regions, the fundamental equations are used in a Cartesian coordinate system, whereas for large areas, a spherical coordinate system is applied. The wave action density spectrum varies spatially and temporally as a function of two wave phase parameters. The fundamental equations are formulated in both Cartesian coordinates for small-scale applications and spherical coordinates for larger-scale applications. MIKE 21 SW incorporates the following physical phenomena: Wave growth due to wind forcing; Nonlinear wave-wave interactions; Wave dissipation due to whitecapping; Wave dissipation due to bottom friction; Wave dissipation due to wave breaking; Refraction and shallow water effects due to depth variations; Wave-current interactions; Influence of time-dependent depth changes (DHI 2014a).

b) *MIKE 21 FM Hydraulic Model:* The flow module is developed based on the finite element mesh method. It is derived from the numerical solution of the Navier-Stokes equations for incompressible fluids in two or three dimensions, combined with the Boussinesq approximation and the hydrostatic pressure assumption

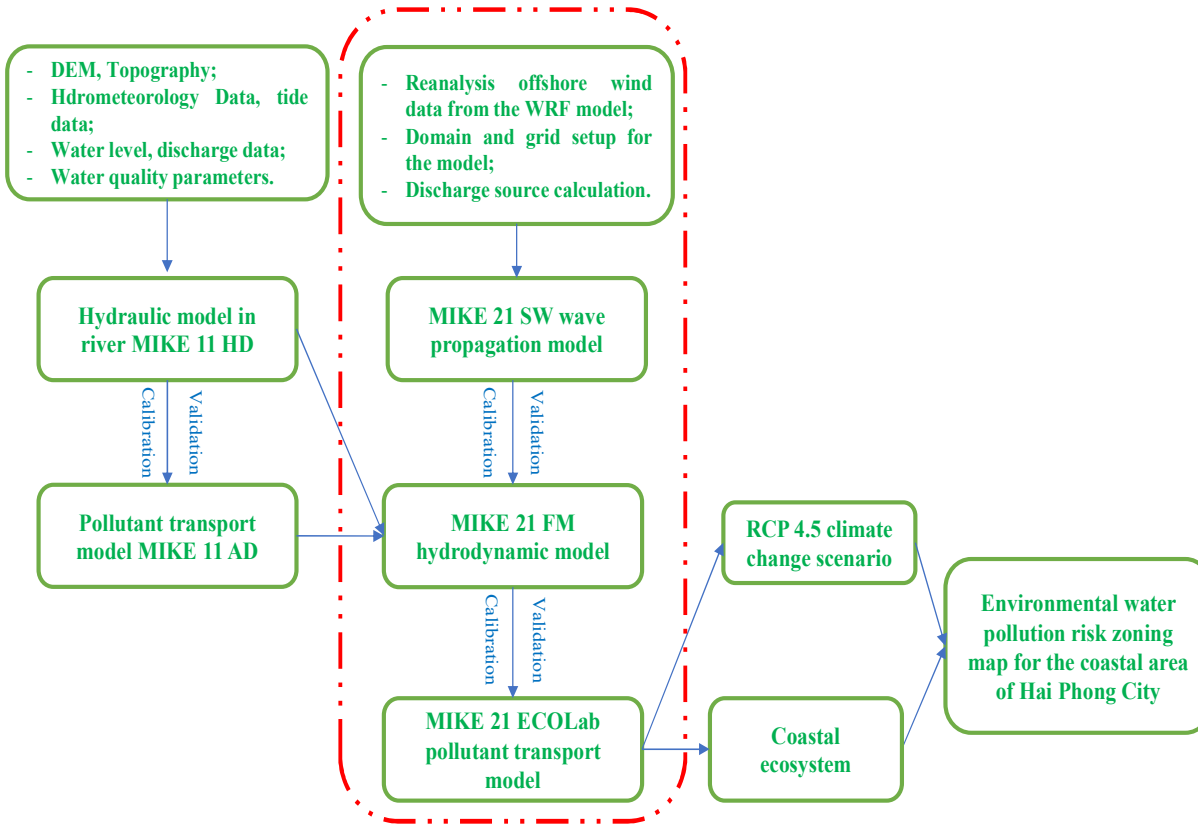

Fig. 2: The study framework flowchart.

(DHI 2014b-e). As a result, the module includes the following equations: continuity equation, momentum equation, temperature equation, salinity equation, and density equation, with closure achieved through a turbulence closure scheme. In the three-dimensional case, a sigma coordinate system is used. The spatial discretization of the fundamental equations is performed using the central finite volume method. The spatial domain is discretized by dividing the continuous domain into non-overlapping grid cells/elements. In the horizontal direction, an unstructured mesh is used, while in the vertical direction (for three-dimensional cases), a structured mesh is applied. In the two-dimensional case, elements can be triangular or quadrilateral. In the three-dimensional case, elements can be triangular or quadrilateral prisms, with surface elements taking the form of triangles or quadrilaterals.

c) *MIKE 21 EcoLab Water Quality Model:* The EcoLab model is integrated within MIKE 21, built on the HD hydraulic module, which calculates water levels and two-dimensional unsteady flows in a vertically homogeneous fluid layer (DHI 2014f-i). The mass and momentum conservation equations are vertically integrated to describe the variations in water levels and flow dynamics. The computational results from the hydraulic module enable the simulation of hydrodynamic physical processes, forming the basis and input for calculations in EcoLab. When combined with the advection-diffusion module, the equations are formulated for non-conservative substances. The numerical equations in EcoLab are solved using a time-dependent integration approach, incorporating both biochemical processes within EcoLab and transport-diffusion processes.

Establishing Models for the Study Area

a) *MIKE 21 SW Model:* The computational domain for this study is the coastal area of Hai Phong City, with coordinates ranging from 20°01'N to 21°31'N and 106°38'E to 107°14'E. The computational grid is constructed using a combination of unstructured and square grids, with a total of 10,512 grid cells and 7,047 grid nodes; the study area is simulated using an unstructured grid. Input data for the model boundary conditions include wave data, wind data, and water

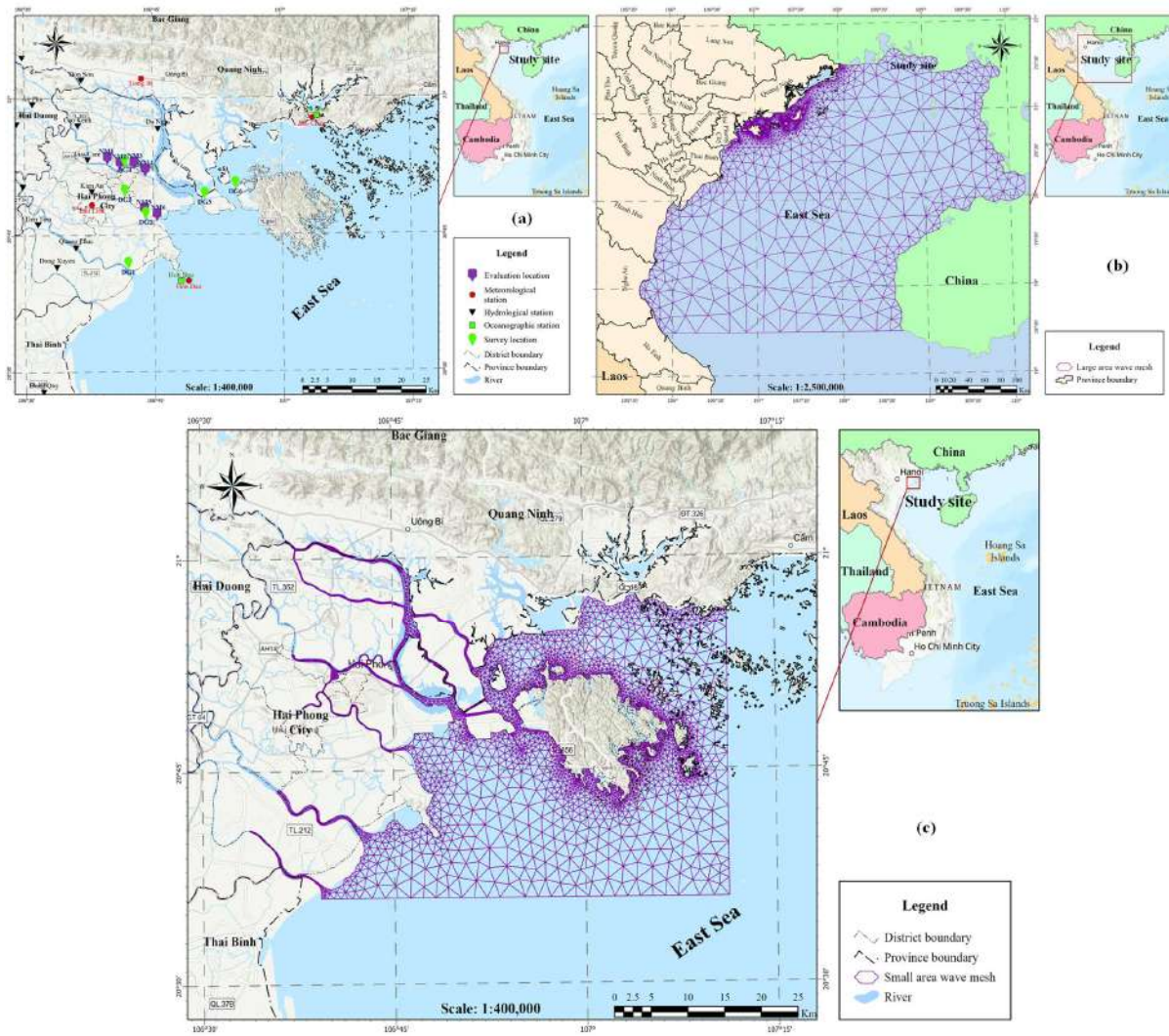

Fig. 3: (a) Meteorological, hydrological and ocenographical stations; (b) Estabilised mesh of tidal prediction; (c) Established mesh of wave propagation, hydraulic and water quality (Thu & Tri 2023).

levels collected from hydrographic stations within the study area. To simulate waves in the Hai Phong coastal region, the study first models waves for the larger surrounding sea area (large-scale waves) before transferring them to the shallow water coastal area of Hai Phong (small-scale waves). Fig. 3 presents the computational grids for the large-scale wave model and the study area wave model. The setup and configuration of the wave propagation domain for the study area were based on previous research findings (Thu & Tri 2023).

b) *Setup and Calibration of the MIKE 21 HD Hydraulic Model:* Input data used to set up and calibrate model in this study includes:

Study Area Topography: Obtained from nautical charts provided by the Vietnam People's Navy and survey data from previous projects.

River Discharge at River Mouths: Inherited from the calibrated and validated MIKE 11 model of the Red River - Thai Binh River system in the study by Dao (2023).

Offshore Boundary Water Levels: Computed using the tidal propagation tool in the MIKE 21 model.

Wind Data: Collected from the Hon Dau meteorological station.

Water Levels for Calibration and Validation: Measured at the Hon Dau Hydrographic Station.

Discharge from Industrial Zones: Includes wastewater discharge from factories, industrial zones, and industrial clusters within the study area.

Table 1: Information on some sources of waste from industrial parks/ industrial clusters.

No	Wastewater discharge sources	Discharge (m^3/s)
1	Dinh Vu Industrial Zone	0.11
2	Tan Vu Industrial Zone	0.02
3	Hai Phong Cement Plant	0.13
4	Bach Dang Shipyard	0.02

Marine Boundaries: Water levels at the eastern and southern boundaries of the computational domain, calculated using the tidal propagation tool in MIKE 21 based on tidal harmonic constants derived from observed data at the Hon Dau Hydrographic Station.

River Boundaries: Discharge at various river locations simulated using the MIKE 11 model for the entire Red River - Thai Binh River system, including the Da Bac River, Cam River, Lach Tray River, and Van Uc River.

The model was developed using an unstructured computational grid, with a total of 9,083 cells and 4,857 grid nodes in the computational domain, simulating the dry season period with a time step of 60 seconds.

c) *Setup and Calibration of the MIKE 21 EcoLab Pollutant Transport Model:* This study establishes a simulation model to assess water quality from several coastal industrial zones in Hai Phong City, focusing on six pollution parameters: Total suspended solids (TSS), Coliform, Biochemical Oxygen Demand (BOD_5), Dissolved Oxygen (DO), Chemical Oxygen Demand (COD), and heavy metal Fe. The Hai Phong coastal area hosts numerous large industrial parks and clusters. The study collected and compiled data on major industrial zones in the area and represented them as point sources in the model. While this study did not gather complete data on all wastewater sources from residential areas and small industrial zones discharging into the river system, it incorporated measured river water quality data during calibration and validation. This ensures a relatively accurate representation of water quality in the Hai Phong coastal and estuarine river system. Table 1 presents information on major wastewater discharge sources in the study area.

d) *Climate Change Scenarios and Analysis of Simulation Results:* To assess marine water pollution under the impact of future economic development activities on coastal aquaculture in Hai Phong, the study, after finding the appropriate set of model parameters, established an input data set corresponding to changes in rainfall and tidal water levels based on the climate change and sea level rise scenarios for Vietnam published by the Ministry of Natural Resources and Environment in 2020 (MoNRE 2020). The study selects the RCP4.5 scenario - a low-moderate greenhouse gas concentration scenario to simulate and calculate the potential impact of environmental pollution parameters on the coastal area of the study region. This is because it is a scenario with stable radiation forcing, where the total radiation forcing reaches approximately 4.5 W/m² by 2065 and remains stable until 2100, without any sudden increase over a long period (Tables 2 and 3).

The temperature and rainfall data were updated in the intermediate calculation step to determine the corresponding changes in river discharge based on the variation in rainfall, which were then used to update the boundary conditions of the MIKE 21 model.

Similarly, water level data under the climate change scenario were processed to generate tidal boundary conditions according to the scenario, which were then input into the MIKE 21 model.

With the above analysis, the study focuses on simulating and developing a zoning map of environmental conditions related to coastal aquaculture in the study area in the context of climate change under the scenario: Evaluation of marine water environment using rainfall, temperature, and sea level data according to the 2020 Climate Change Scenario RCP 4.5 for the period 2046-2065, with updated discharge sources based on expected future emissions according to the socio-economic development plan of Hai Phong City for the years 2030-2050.

Table 2: Changes in average temperature and rainfall in spring under the RCP 4.5 scenario (MoNRE 2020).

Temperature (^oC)		Precipitation (%)	
2046-2065	2080-2099	2046-2065	2080-2099
1.6 (1.0 ÷ 2.2)	2.1 (1.3 ÷ 3.0)	17.3 (3.2 ÷ 29.8)	32.3 (11.7 ÷ 51.2)

Table 3: Sea level rise according to the RCP 4.5 scenario (Unit: cm) (MoNRE 2020)

Region	Time milestones of the 21st century							
	2030	2040	2050	2060	2070	2080	2090	2100
Mong Cai - Hon Dau	12 (7÷17)	17 (10÷23)	22 (14÷30)	28 (17÷39)	34 (21÷47)	40 (25÷57)	46 (29÷66)	52 (33÷75)

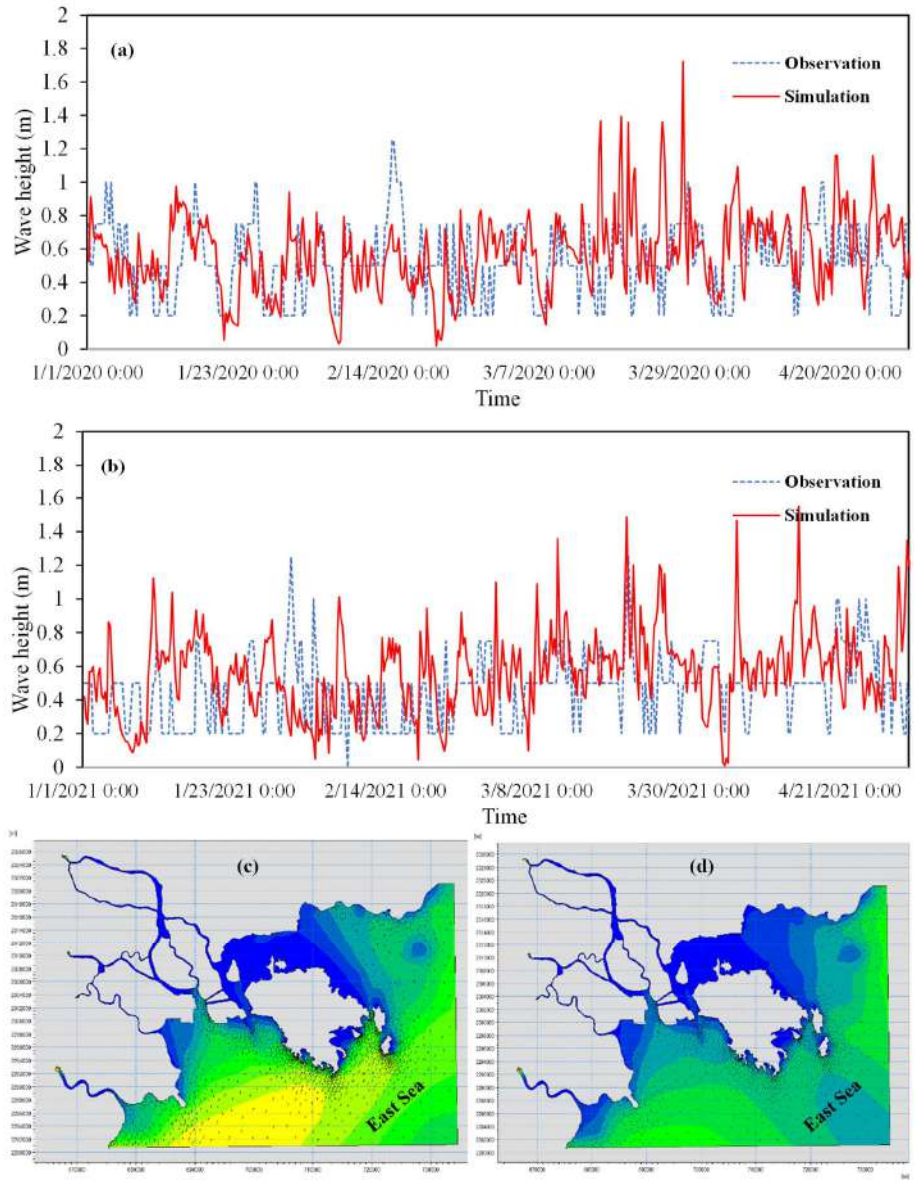

Fig. 4: Calibration and validation of the wave model: (a) Mean wave height in 2020; (b) Mean wave height in 2021; Wave simulation results for the study area: (c) 2020; (d) 2021 (Thu & Tri 2023).

RESULTS

Results of the MIKE 21 SW, MIKE 21 FM, and MIKE 21 EcoLab Models

a) *Calibration and Validation Results of the MIKE 21 SW Wave Model:* The wave model was developed for the study area, with simulation results calibrated using observed data from the Hon Dau station in 2020 and validated in 2021, focusing on the mean wave height parameter. The calibration and validation results of the wave model are shown in Figs. 4a-4b.

The calibration results for mean wave height in 2020 and 2021 indicate that the MIKE 21 SW wave model effectively simulates mean wave height. The calibrated and validated MIKE 21 SW model parameters are presented in Table 4. Key values include wave propagation directions, bottom friction, and whitecapping parameters (Cdis = 4.5 and Delta = 0.5), while depth-induced wave breaking is set at 0.73. The initial conditions are defined as deep-water spectra using the JONSWAP spectrum.

Boundary conditions include two open boundaries, allowing wave energy exchange between the interior and

exterior of the computational domain. After constructing the deep-water wave model for the study area, wave transmission from the large-scale wave model to the study region was performed. The simulation results for waves in the study area for 2020 and 2021 are shown in Figs. 4c-4d (Thu & Tri 2023).

b) *Calibration and Validation Results of the MIKE 21 FM Hydraulic Model:* The calibration and validation of the MIKE 21 FM hydraulic model for the study area were conducted for the periods from January to March 2020 for model calibration and from January to March 2021 for model validation. The results are presented in Figs. 5a-5b.

The accuracy of the calibration and validation process was evaluated using the Nash Sutcliffe efficiency coefficient and the correlation coefficient between the computed results and observed data at the Hon Dau station. The calibration and validation results indicate that the MIKE 21 FM model can simulate the hydrodynamics relatively accurately, with indices ranging from 0.85 to 0.90, are considered acceptable to good. The model captures the tidal regime in the area relatively well; however, it does not fully capture the tidal troughs and peaks accurately, with an error margin of approximately 0.1 m.

The optimal parameter set for the MIKE 21 FM model identified the following values: turbulent viscosity coefficient (0.28), Manning's roughness coefficient (57 $m^{1/3}/s$), wind friction coefficient (0.002), and computational time step (60 seconds). These values ensure reliability and can be applied to simulate water quality for the study area. With these results, the model is deemed reliable for simulating and assessing pollutant dispersion process from industrial discharge sources into the study area (Thu & Tri 2023).

c) *Calibration and Validation Results of the MIKE 21 EcoLab Water Quality Model:* The study collected pollutant parameter data at discharge sources and several locations within the study area, combined with water quality measurement data from 2015 and 2016. The calibration and validation results of the water quality model at various locations in the study area are shown in Table 4.

The calibration and validation processes were conducted for six key parameters: DO, BOD_5, COD, TSS, Fe, and Coliform at six monitoring and sampling locations (NM1-NM6). The 2015 calibration results indicate that the error between simulated and observed concentrations ranged from 5% to 19%. This suggests that the water quality

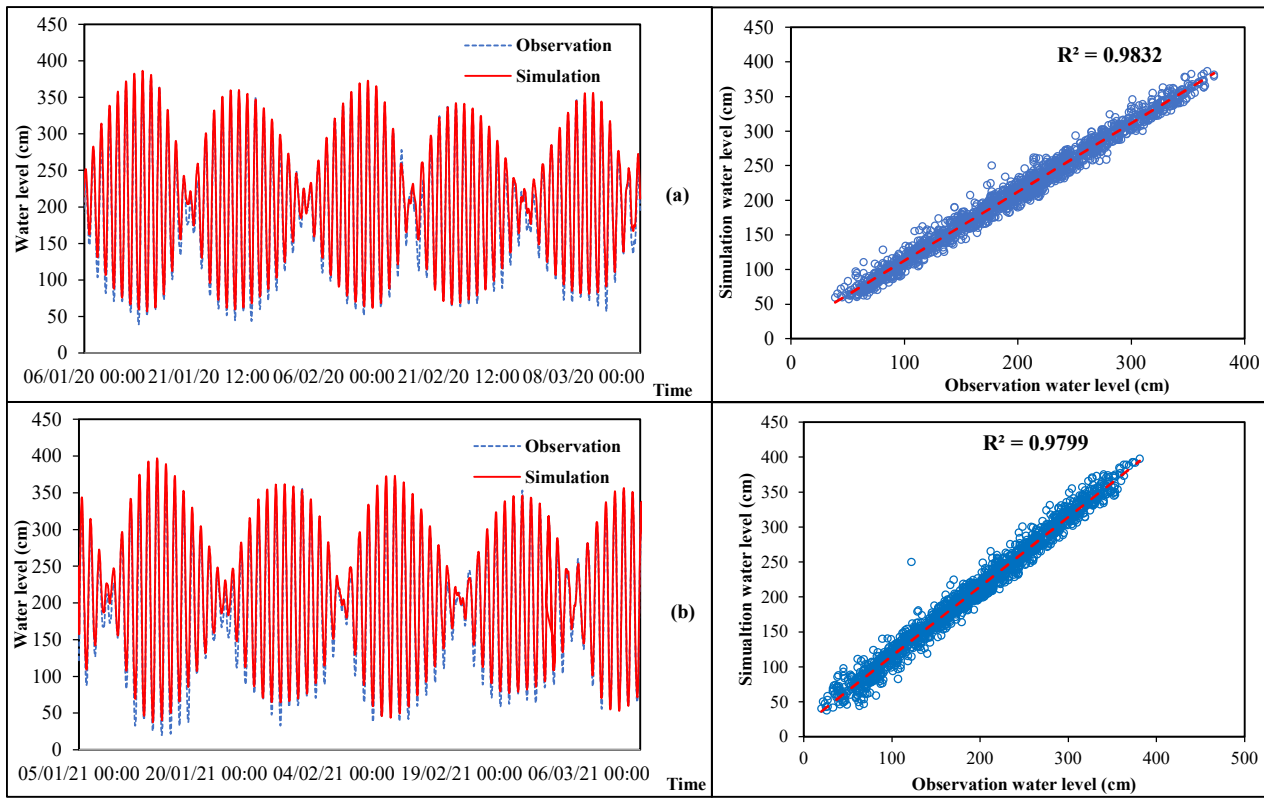

Fig. 5: The calibration (a) and validation (b) results of water level at Hon Dau station (Thu & Tri 2023).

Table 4: Evaluation of Calibration and Validation Errors of the Water Quality Model (Thu & Tri 2023).

No	Calibration (2015)						Validation (2016)					
	Coliform	BOD_5	COD	TSS	Fe	DO	Coliform	BOD_5	COD	TSS	Fe	DO
NM1	-10%	-8%	-10%	-8%	-18%		-8%	-13%	-17%	6%	-19%	
NM2	-8%	-10%	8%	12%	-22%		-7%	-7%	-19%	9%	-21%	
NM3	-5%	-5%	-11%	-16%	12%		-7%	-9%	-15%	-24%	-13%	-16%
NM4	-9%	-8%	-15%	-15%	14%		-7%	-7%	-14%	-21%	-11%	-15%
NM5	-7%	14%	-11%	-19%		-8%	-10%	-11%	9%	-19%		15%
NM6	-11%	12%	-12%	13%		-14%	-15%	-10%	6%	13%		8%

Table 5: Parameters of MIKE 21 model.

No	Parameters	Selection value
1	Turbulent viscosity coefficient - Smagorinsky	0.28 (m^2/s)
2	Bottom friction coefficient - Manning's coefficient	57 ($m^{1/3}/s$)
3	Wind friction coefficient	0.0027
4	Time step	60 (s)

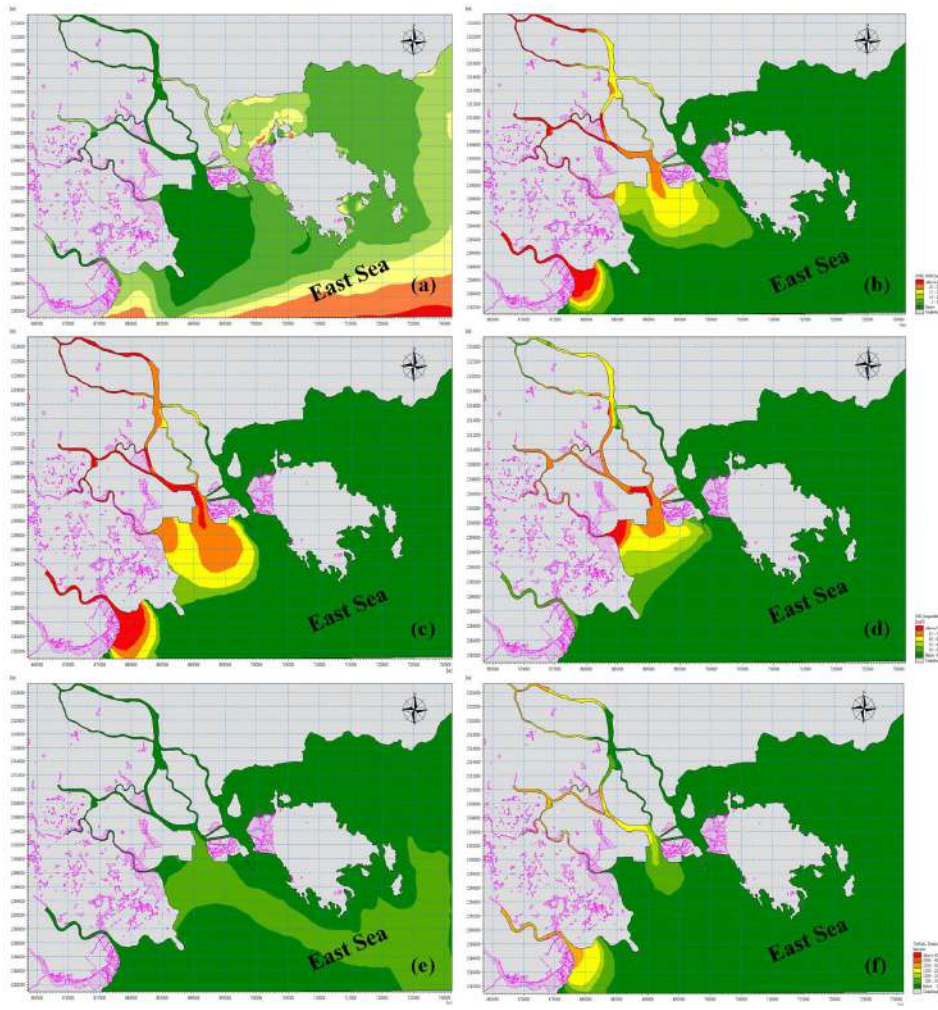

Fig. 6: Results of climate change scenario simulations: (a) DO; (b) COD; (c) BOD_5; (d) TSS; (e) Fe; (f) Coliform (Thu et al. 2023).

model parameters are relatively well-suited for the study area. However, to ensure a more accurate and objective assessment, the model was validated for 2016 using the same set of water quality parameters (Thu & Tri 2023).

The validation results of the water quality model for 2016 show that the error between the simulated and observed pollutant concentrations ranged from 6% to 24%. This indicates that the water quality model parameters are relatively well-suited for the study area. The model parameters mentioned in Table 5, were used to simulate the climate change scenarios established above.

Simulation Results of Marine Pollution Scenarios Considering Climate Change

Based on the selected climate change scenarios, the study utilized MIKE 21 Ecolab to simulate the dispersion of DO, BOD_5, COD, TSS, Fe, and Coliform in the study area. The simulation results indicate a decreasing trend in DO concentration, while the concentrations of BOD_5, COD, TSS, Fe, and coliform show an increasing trend. The scenario was simulated with input boundaries including wastewater discharge sources from industrial parks (KCN) and industrial clusters (CCN) planned for future development (with significant increases expected), along with rising sea levels and changing rainfall patterns according to the climate change scenario. Figs. 6a-6f illustrate the variations in the dispersion of key pollution parameters in the coastal waters of Hai Phong City.

Assessment of Pollutant Impacts on the Coastal Ecosystem

a) *Evaluation of DO Concentration Effects on the Ecosystem:* Any change in DO (dissolved oxygen) levels in water can significantly impact the normal development of aquatic organisms. When DO concentrations become too low, aquatic species experience difficulty in respiration, reduced activity, and overall adverse effects on the ecosystem. DO not only serves as an essential oxygen source for farmed aquatic organisms but also promotes the growth of aerobic microorganisms, enhances the decomposition of organic matter, and helps mitigate harmful substances. Maintaining appropriate

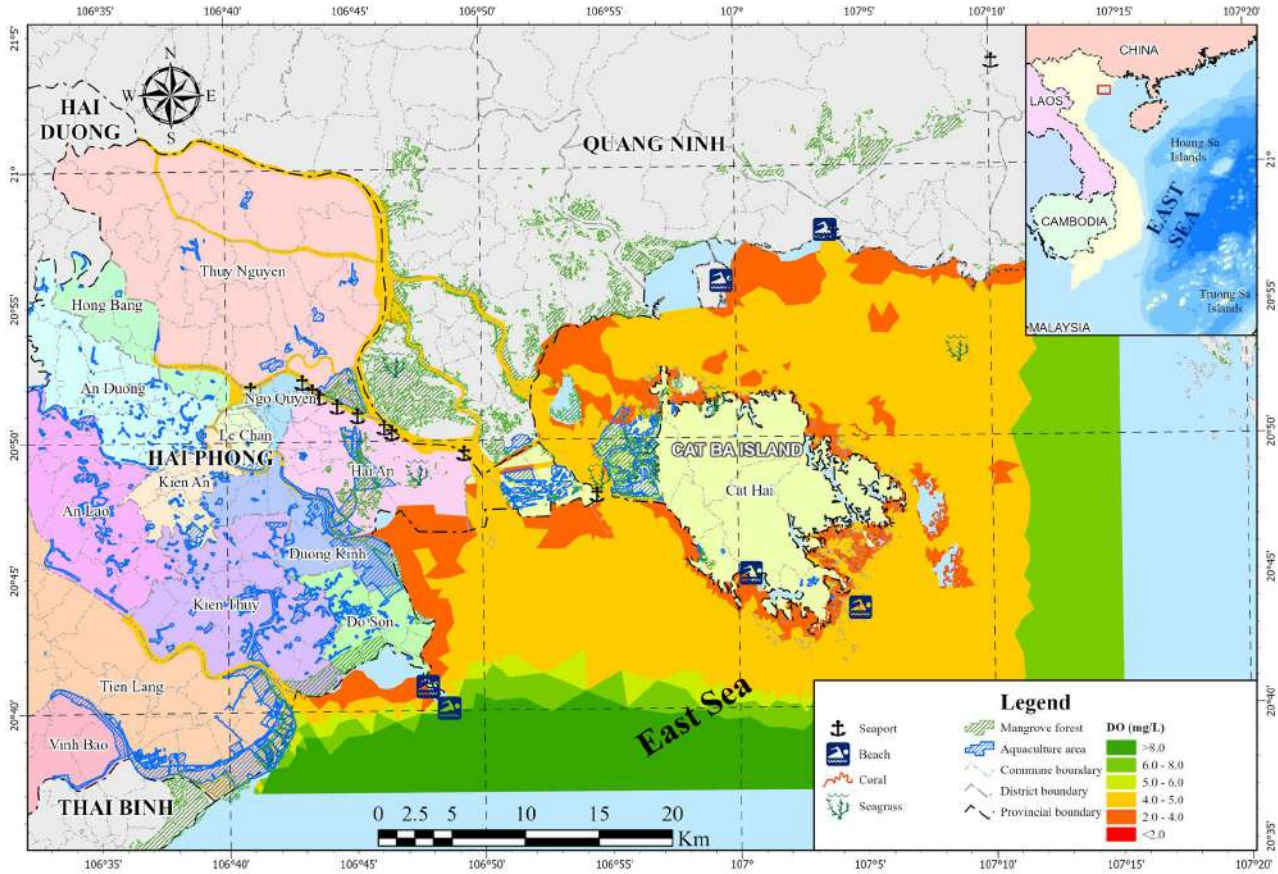

Fig. 7: The impact level of DO on the ecosystem in the study area.

DO levels also inhibits anaerobic microorganisms and boosts the immune systems of aquatic species.

The simulation results of DO dispersion in the coastal waters of the study area indicate reduced DO concentrations, which negatively affect the Hai Phong coastal ecosystem, including seagrass beds and various aquatic species in the region (Fig. 7).

b) *Assessment of the impact of COD concentration on the ecosystem:* When COD increases, the decomposition of organic matter in wastewater consumes a large amount of dissolved oxygen. This causes a significant decrease in oxygen levels in the water, leading to suffocation and mass death of aquatic species. The underwater ecosystem is disrupted, affecting the food chain and biodiversity.

The simulation results of COD dispersion from industrial and coastal wastewater sources in Hai Phong show that the COD concentration in the coastal area remains relatively low, and generally within the permissible limits for water used in agriculture and maritime traffic. However, the concentration is higher in river sections near the estuaries, impacting the mangrove ecosystem along the river, particularly in the Nha Mac lagoon area, where the seagrass ecosystem is concentrated and where the COD concentration is notably high (Fig. 8).

c) *Assessment of the impact of BOD_5 concentration on the ecosystem:* The BOD_5 concentration follows the same trend as the COD concentration in the study area. High BOD_5 indicates the presence of significant amounts of organic matter in the wastewater, which requires oxygen for decomposition. If BOD_5 is high, the wastewater will consume more oxygen, leading to a decrease in oxygen levels in the water environment and negatively affecting aquatic organisms.

As shown in Fig. 9, BOD_5 concentrations are at alarming levels in the Bach Dang River, Cam River, and Van Uc River areas. Among them, the lower reaches of the Bach Dang River, which have relatively rich seagrass and mangrove ecosystems, are particularly vulnerable to the elevated BOD_5 concentrations.

d) *Assessment of the impact of TSS concentration on the ecosystem:* TSS affects water clarity, so the higher the

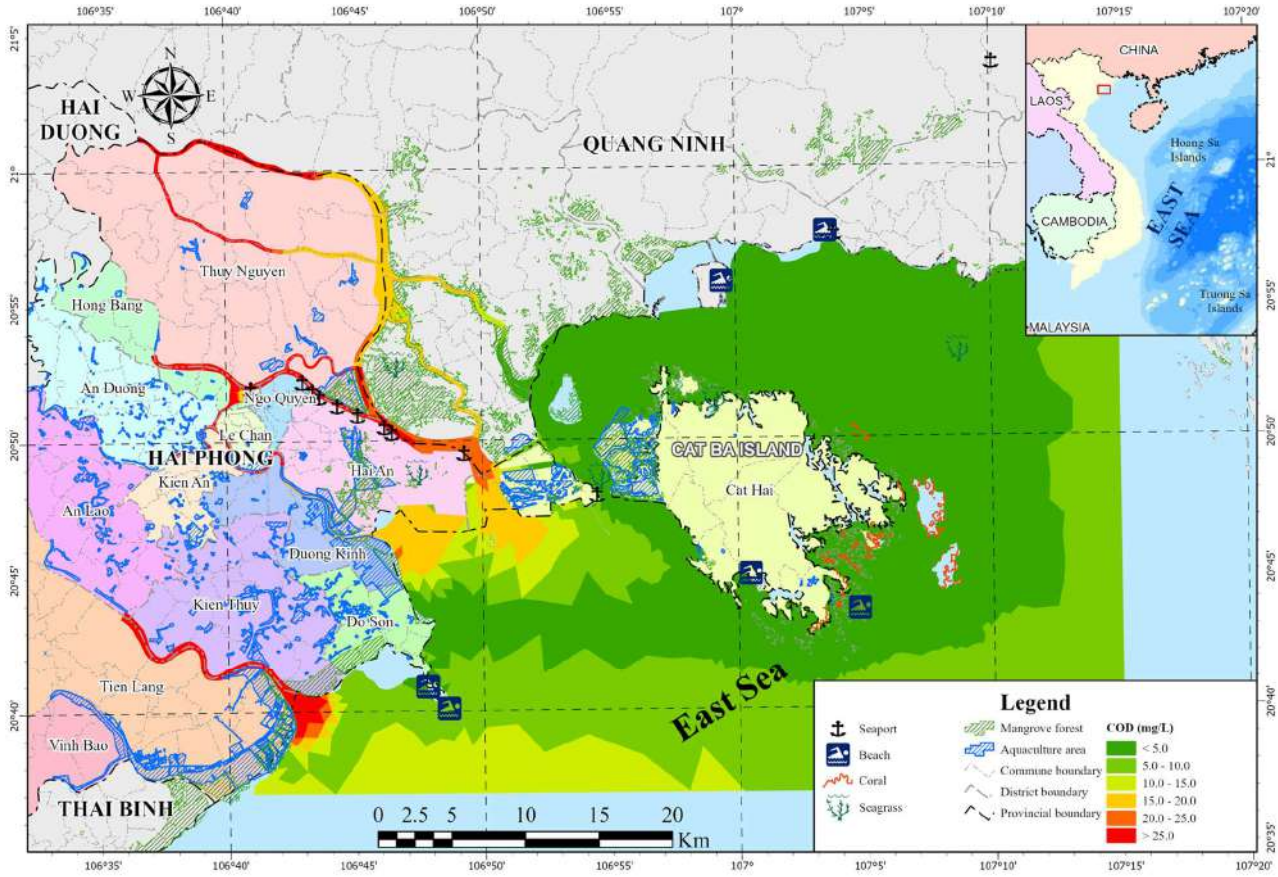

Fig. 8: The impact level of COD on the ecosystem in the study area.

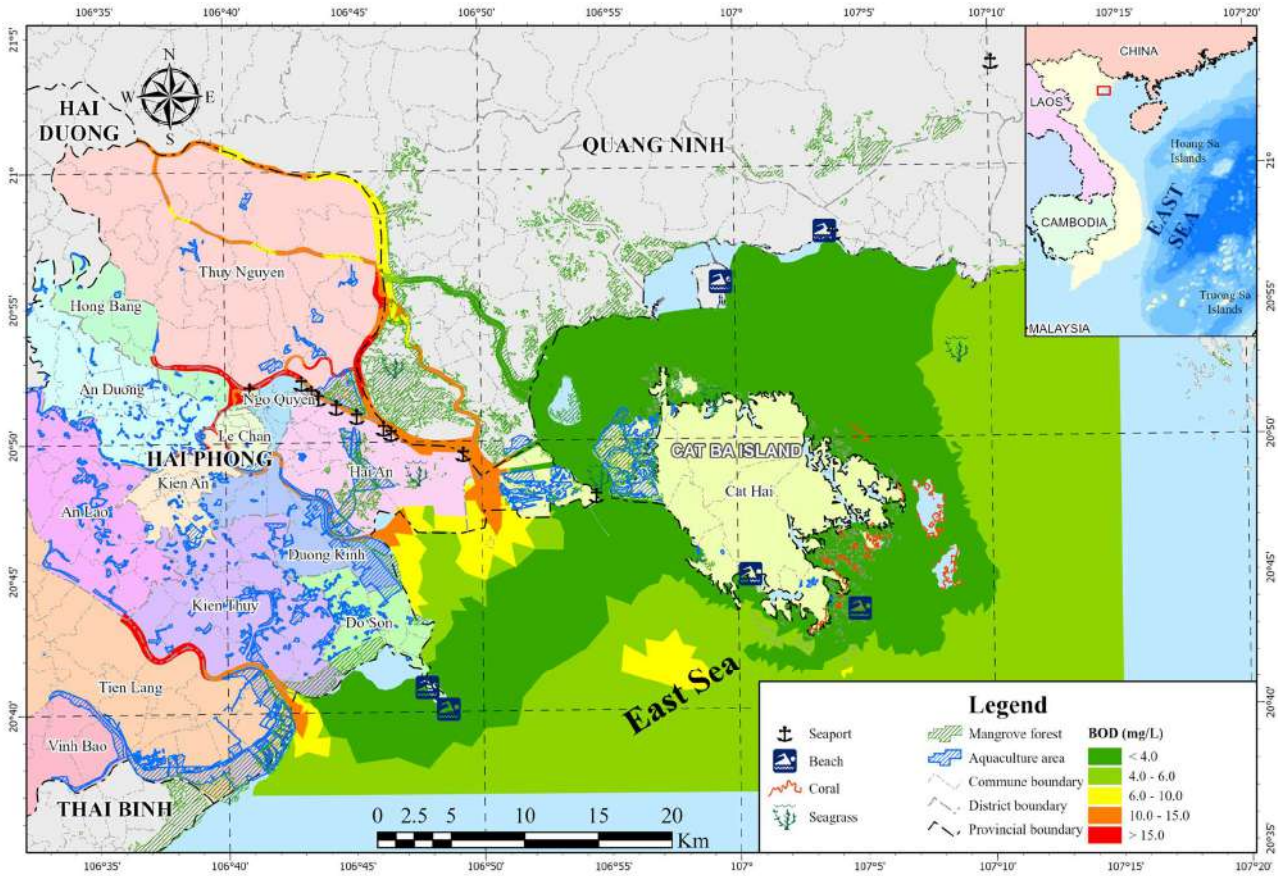

Fig. 9: The impact level of BOD_5 on the ecosystem in the study area.

TSS content in the water source, the lower the water's clarity. High TSS can reduce the natural dissolved oxygen levels in the water and increase the water temperature. This can hinder aquatic organisms, such as small fish, from surviving. TSS can also block sunlight, potentially halting the photosynthesis process, reducing plant life, and further decreasing oxygen concentrations in the water.

Simulation results show that the TSS concentration in the study area is relatively high, with an average concentration exceeding 20 mg/L. It can be observed that in the future, measures will be needed to control the TSS concentration around areas with concentrated ecosystems in the study area (Fig. 10).

e) *Assessment of the impact of Fe concentration on the ecosystem:* Dissolved metals in water are easily absorbed by aquatic organisms. The metal concentration in the bodies of aquatic species can accumulate over time, surpassing the concentration present in the water. Toxic metals have adverse effects on the survival, activity, development, circulation, and reproduction of organisms. Metals can cause poisoning, harming organisms without directly causing death (Mitra et al. 2022). Simulation results of heavy metal Fe dispersion in the coastal water environment show that the study area is not currently affected by elevated Fe concentrations (Fig. 11).

f) *Assessment of the impact of Coliform concentration on the ecosystem:* Coliform is a type of bacteria that naturally occurs and is present in all human and animal waste. Coliform bacteria in wastewater can indicate the presence of harmful, disease-causing organisms. These organisms are called pathogens and can include viruses, protozoa, or bacteria. The results show that the Coliform concentration ranges from 1000 to 4000 MPN/100mL (Fig. 12). This concentration is within the acceptable limits of Class A to Class B according to QCVN 08:2023.

Development of a Pollution Risk Zoning Map for the Study Area

To assess the comprehensive impact of coastal pollution on

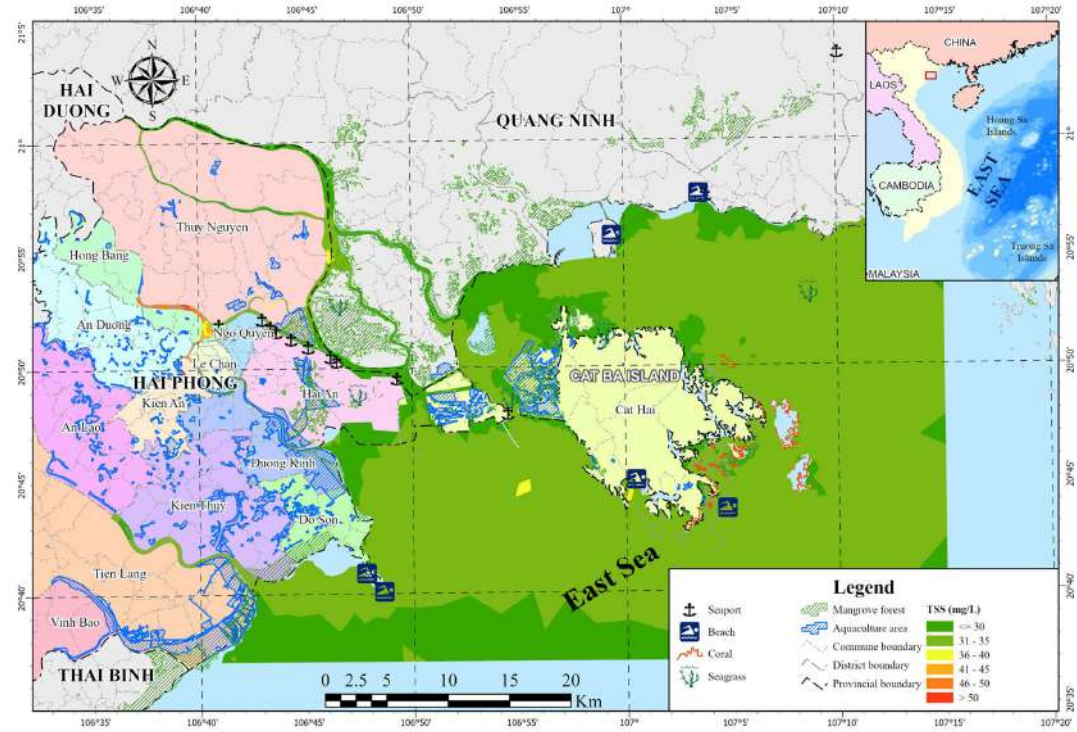

Fig. 10: The impact level of TSS on the ecosystem in the study area.

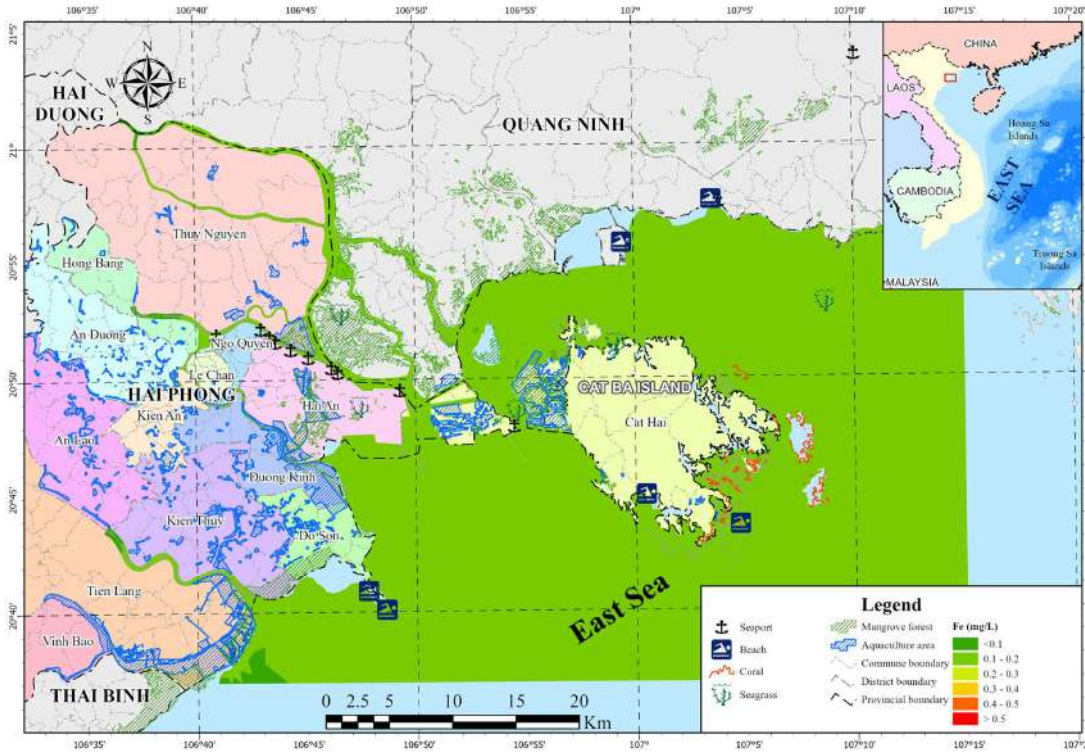

Fig. 11: The impact level of Fe on the ecosystem in the study area.

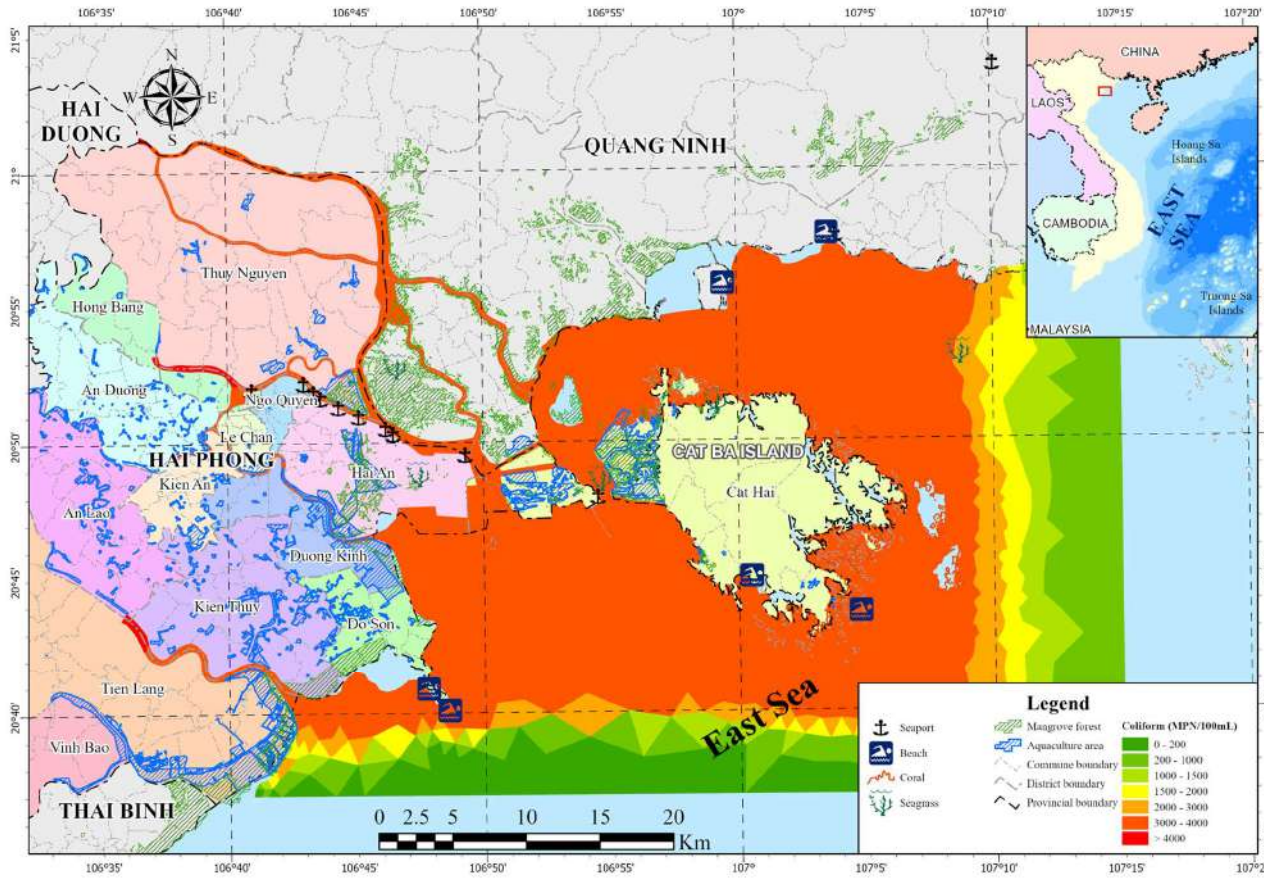

Fig. 12: The impact level of coliform on the ecosystem in the study area.

the marine ecosystem as well as on aquaculture areas, the report developed a pollution risk zoning map. The coastal waters of Hai Phong are divided into areas of water pollution risk, ranging from low to very high environmental pollution risk, according to the environmental risk classification scale (RQ) and are represented by different color scales. The method for assessing the risk of marine water pollution follows Circular No. 26/2016/TT-BTNMT (MoNRE 2016), and the formula for calculating the RQ index is as follows:

$$RQ = \frac{\sum_{j=1}^{m} W_j \left(\frac{MEC}{PNEC} \right)_j}{\sum_{j=1}^{m} W_j} \quad \ldots(1)$$

where MEC is the concentration of pollutant parameter j in the environment, calculated from monitoring data and measurements that comply with the current Vietnamese standards and national technical regulations for assessing environmental quality components and types of pollution; PNEC is the limit concentration of pollutant parameter j in the environment according to current standards (National Technical Regulation on the quality of coastal, nearshore, and offshore waters - QCVN 10-MT:2023/BTNMT); m is the total number of pollutant parameters being considered and evaluated; W_j is the weight factor used to calculate the risk coefficient for pollutant j, as specified in Clause 4, Article 11 of Circular No. 26/2016/TT-BTNMT (MoNRE 2016) in Table 5.

The pollution level index or the risk of marine and island environmental pollution is based on the average environmental risk index (RQ) of the zones, as specified in

Table 5: The weight factors of the pollution parameters (MoNRE 2016).

No	Pollutant parameters	Weight factor
1	Toxic substances	2.0
	Heavy metals (Fe)	2.0
2	Substances or factors that deplete oxygen in seawater	1.5
	BOD_5, COD, DO	1.5
3	Total coliform	1.3
4	TSS	1.0

Table 6: Criteria for assessing the Environmental Risk Index.

No	Criteria	Environmental Risk Index (RQ)
1	Very high	RQ > 1,5
2	High	1,25 < RQ ≤ 1,5
3	Medium	1 < RQ ≤ 1,25
4	Low	RQ ≤ 1

Clause 2, Article 11 of Circular No. 26/2016/TT-BTNMT (MoNRE 2016) (Table 6).

Based on the simulation results of pollutant parameter dispersion in the study area using the MIKE 21 model, the study calculated the RQ values for the coastal area of Hai Phong and developed a water pollution risk zoning map, as shown in Fig. 13.

The results show that the coastal area has a higher risk of pollution compared to the outer marine area, which is reasonable as the economic and social activities in the coastal area have intensified, increasing the risk of marine water pollution in this region. The coastal marine area of Hai Phong is significantly influenced by pollution from river water, with the most notable source being the discharge from the Bach Dang River. According to recent statistics (Mitra et al. 2022), the Bach Dang River estuary receives a considerable amount of waste each year: about 45,000 tons of COD; 8,100 tons of BOD_5; more than 13,700 tons of total nitrogen (of which about 3,700 tons is dissolved nitrogen); 3,200 tons of total phosphorus (1,200 tons in the form of PO_4^{3-}), and about 106,000 tons of TSS. Among these sources, domestic wastewater accounts for the highest percentage (nearly 41.31%), followed by livestock farming (30.27%), industrial sources (13.5%), and the lowest from aquaculture (2.27%).

The risk zoning map visually represents the potential risk levels for the coastal area of Hai Phong, using color scales to indicate different degrees of risk. This allows users to quickly identify high-risk areas and propose appropriate solutions for each period, aligned with socio-economic development orientations.

Proposed Solutions for the Study Area

Develop and Improve Policies, Laws, and Organizational Structure

(i) Review and propose improvements to policies for controlling marine pollution (for industrial zones, craft villages, aquaculture areas, environmentally sensitive regions, and specific local areas);

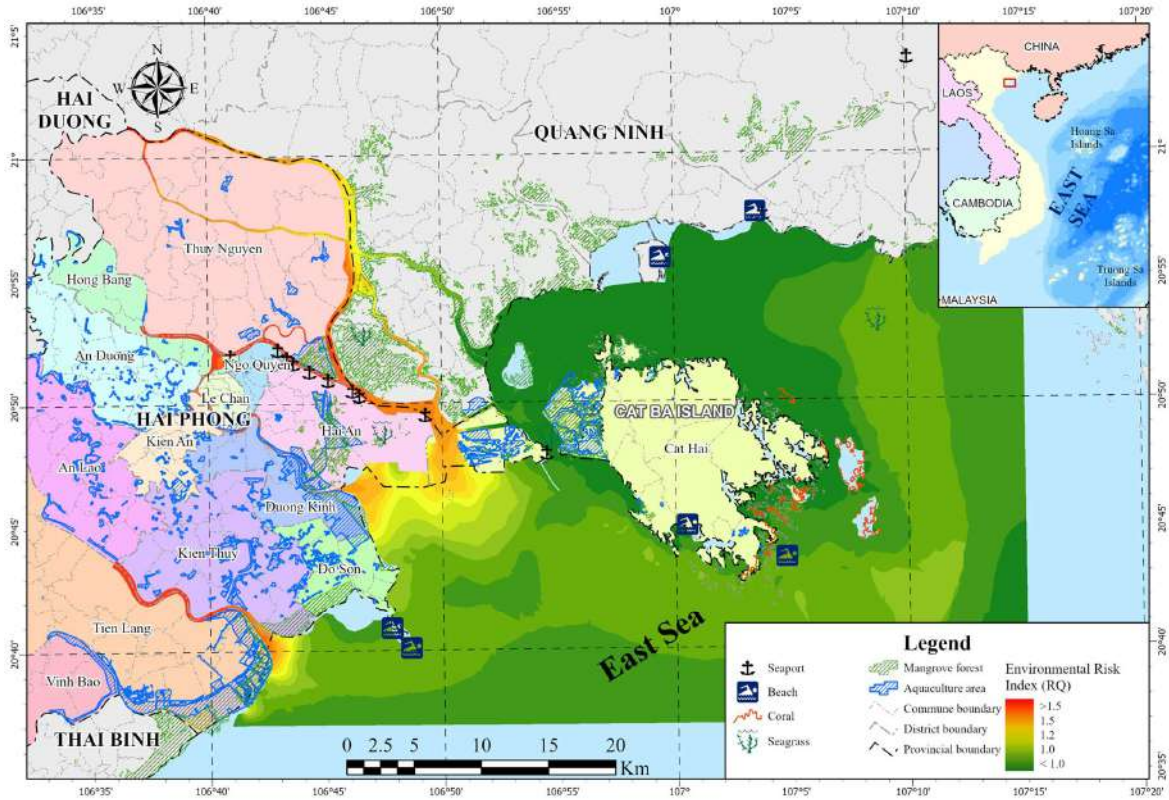

Fig. 13: The risk zoning map for water pollution in the coastal marine area of Hai Phong.

(ii) Develop and issue guidelines for applying marine environmental protection standards (KSONB) locally;

(iii) Create directives and procedures for monitoring and assessing marine pollution;

(iv) Develop technical guidelines (investigation, monitoring, incident response, environmental remediation);

(v) Develop an integrated environmental-communication plan to promote participation in marine-environment protection.

The lead agency for this plan is the Department of Agriculture and Environment of Hai Phong, with participating agencies including the People's Committee and all relevant departments in Hai Phong, and the Department of Marine and Island Affairs.

Strengthen Environmental Investigations and Build a Comprehensive Marine-Environment Information System

(i) Assess marine-environmental risks in the area: identify causes, risk levels, assess data reliability, identify information gaps, and propose improvements to the monitoring program;

(ii) Strengthen equipment at environmental-monitoring stations in Hai Phong;

(iii) Develop a comprehensive marine-environment database (including an updated baseline database and a specialized ATLAS map set);

(iv) Apply digital technologies to create a web platform for marine-environment pollution control.

The lead agency for this plan is the Department of Agriculture and Environment of Hai Phong, with participation from all relevant departments and local People's Committees.

Prevent and Reduce Marine Pollution from Socio-Economic Activities

(i) Review projects and development plans in coastal districts and integrate marine-environmental protection measures;

(ii) Review programs requiring Environmental Impact Assessment (EIA) and Strategic Environmental Assessment (SEA), assess compliance, and list facilities lacking EIA documents or causing serious pollution;

(iii) Strengthen inspections of discharge sources (industrial parks, factories, urban areas, mining zones, aquaculture areas);

(iv) Propose measures for improved monitoring, enforcement, and penalties for non-compliant facilities.

The lead agency for this plan is the Department of Agriculture and Environment of Hai Phong, with participation from all relevant departments and local People's Committees.

Review Pollution Hotspots in Coastal Areas and River Mouths

Identify severely polluted zones and prioritize wastewater collection-and-treatment projects for sources discharging into hotspot areas. Prepare pre-feasibility and feasibility studies and conduct cost-benefit analyses for each project. The lead agency for this plan is the Department of Agriculture and Environment of Hai Phong, with participation from all relevant departments and local People's Committees.

To Control the Risk of Marine Pollution

Strict control of on-site waste by collecting and treating waste at source with particular focus on diffuse emissions such as domestic and agricultural waste entering river and coastal-lake systems. Direct discharges into coastal rivers and lakes, including effluents from industrial zones, craft villages, and seafood-processing facilities, must meet technical standards before release into the environment. Table 7 summarizes the pollutant concentrations used in the mitigation scenario.

After simulating the above scenario, the report the environmental risk index (RQ) and developed a marine-pollution risk map (Fig. 13). The results indicate that if the emission sources are treated as per the simulation scenario, the water quality in the coastal area will improve significantly, and it is noticeable that the area of very high and high environmental pollution risk zones has decreased substantially. Therefore, to ensure sustainable economic development in the coastal city area, the proposed solution is to improve the quality of wastewater treatment plants, reducing the concentration of waste from industrial sources after treatment and discharge into the environment. However, to implement this solution, a balance must be made between the investment in wastewater treatment technology and the

Table 7: Concentration of parameters in the simulation scenario for mitigation.

No	Parameters	Column A concentration (mg/L) (QCVN 40:2011/ BTNMT)	Simulation scenario concentration (mg/L)
1	DO	-	10
2	BOD_5	30	15
3	COD	75	40
4	TSS	50	25
5	Fe	1	0.5
6	Coliform	3000	1500

potential profit from industrial zones and clusters, to establish the most suitable permissible standard thresholds, ensuring the reduction of marine pollution risks while promoting socio-economic development in the research area.

One solution that has gained attention in recent years globally is the reuse of wastewater to promote circular economy development. Wastewater from industrial zones and clusters, after being discharged, is partially treated with simple, low-cost treatment technology and then reused for appropriate purposes within those industrial zones and clusters. The remaining part is treated with advanced technology to meet standards for other uses, while ensuring the maintenance of the current load of receiving water bodies, without putting additional pressure on those receiving sources.

DISCUSSION

The simulation results using the MIKE 21 model reveal significant pollutant dispersion patterns in Hai Phong's coastal waters, with notable concentrations of DO, BOD_5, COD, TSS, Fe, and coliform exceeding permissible limits at multiple sampling locations. Estuarine waters near Dinh Vu - Cat Hai and the Nam Cau Kien industrial zones exhibit the highest pollutant levels, particularly for BOD_5 and COD, indicating strong organic pollution loads from industrial wastewater discharge. These findings align with previous studies (Tri et al. 2019, Vinh et al. 2020, Thu & Tri 2023, Le et al. 2023), which also reported severe water quality degradation in these industrialized coastal areas.

Our results further show that DO levels decrease significantly in pollution hotspots, confirming the impact of high organic loads on oxygen depletion, a key factor contributing to coastal eutrophication (Vinh et al. 2020). Similarly, TSS and Fe concentrations are notably high in areas influenced by industrial runoff and port activities, consistent with findings by Vinh et al. (2020), who highlighted sediment resuspension as a major contributor to metal contamination in Hai Phong's coastal waters.

The environmental pollution risk zoning map, developed based on pollutant dispersion simulations, identifies three high-risk zones where pollution levels are consistently above national water quality standards. These zones correspond to areas with dense industrial activity and limited water circulation, making them more vulnerable to pollution accumulation. This result supports previous assessments by Tran et al. (2018), who emphasized the role of hydrodynamic conditions in determining pollutant retention in coastal ecosystems.

Under projected climate change scenarios, our simulations indicate a shift in pollutant dispersion patterns due to rising sea levels, increased rainfall intensity, and altered hydrodynamic conditions. Higher precipitation rates intensify runoff from industrial areas, leading to increased concentrations of COD, TSS, and coliform bacteria, a trend also observed in studies on urban-industrial coastal regions (Müller et al. 2020). Moreover, extreme weather events, such as storm surges and typhoons, exacerbate pollutant transport, further spreading contaminants into sensitive marine habitats (IPCC 2021).

Interestingly, sea-level rise appears to enhance water circulation in some areas, potentially diluting pollutant concentrations in open waters. However, semi-enclosed coastal zones continue to exhibit high pollutant retention, increasing environmental risks for nearby aquaculture and marine biodiversity (Le et al. 2022, Phu et al. 2022). These findings underscore the necessity of integrating climate adaptation measures into coastal pollution management strategies.

While previous research has primarily focused on monitoring industrial pollution in Hai Phong's coastal areas (Tri et al. 2019, Vinh et al. 2020), limited studies have integrated hydrodynamic modeling with climate change scenarios to assess long-term environmental impacts. Our study fills this gap by applying the MIKE 21 modeling system to simulate wave propagation, hydrodynamics, and pollutant dispersion under various climate projections. Vinh et al. (2020) reported severe water quality degradation near industrial zones but did not assess future climate impacts. Our study extends this by modeling pollutant dispersion under multiple climate change scenarios, providing a more comprehensive risk assessment. Studies focused on water quality trends in industrial coastal areas but did not develop a pollution risk zoning map (Le et al. 2023, Thu & Tri 2023, Nam et al. 2025, Tinh & Quoc 2025). Our research enhances environmental planning by identifying high-risk areas for targeted pollution control measures. Singaraja et al. (2018) examined hydrodynamic influences on pollutant transport but did not incorporate long-term climate projections. Our study addresses this gap by analyzing future climate-induced changes in pollution dispersion patterns. By bridging these knowledge gaps, our findings provide a novel framework for adaptive coastal management, supporting policy development for sustainable environmental protection in Hai Phong and similar industrial coastal regions.

CONCLUSIONS

This study provides an integrated assessment of wave dynamics, hydrodynamic, and water quality in the coastal region of Hai Phong, Vietnam, under the influence of industrial pollution and climate change. The key findings and contributions are outlined as follows:

(1) Model Calibration and Validation: The research successfully calibrated and validated the wave, hydrodynamic, and water quality models using observed field data. This rigorous calibration process ensured the reliability of parameter sets, enabling the models to accurately replicate real-world conditions within the study area. The validated models offer a robust platform for simulating environmental changes and assessing future scenarios. Their credibility lays a strong foundation for analyzing the dispersion of pollutants and evaluating the impacts of human activities and climate variability.

(2) Pollution Impact Assessment under Climate Change Scenarios: The study systematically evaluated the influence of pollutant discharge from key coastal industrial zones in Hai Phong. It explored how pollutants disperse and accumulate in marine environments under various climate change scenarios, including sea level rise, altered river discharge, and increased storm frequency. This analysis underscores the heightened vulnerability of the coastal zone to compounded stressors, offering critical insights into potential shifts in pollution patterns and environmental risks over time. These results provide valuable guidance for anticipating and mitigating adverse ecological impacts due to both anthropogenic and climatic pressures.

(3) Development of Environmental Risk Zoning and Adaptation Strategies: Leveraging simulation results, the study developed an environmental pollution risk zoning map, which identified areas of high, medium, and low risk. This zoning tool is instrumental for environmental planners and decision-makers, offering spatial guidance for targeted monitoring, regulation, and intervention. Additionally, the study proposed adaptive strategies for sustainable ecosystem management, including regulatory frameworks, pollution control technologies, and ecological restoration measures aimed at enhancing the resilience of coastal ecosystems.

Despite its contributions, the study has some limitations that suggest directions for future research:

(1) Incorporation of Long-Term Climate Projections: While the study considered climate change scenarios, it lacked a comprehensive integration of long-term climate projections. Future research should extend simulations to mid- and late-century timeframes (e.g., 2050–2100) using high-resolution downscaled climate models to enhance the predictive capacity and support long-term planning.

(2) Assessment of Socioeconomic Impacts: The current focus is predominantly environmental. However, the socioeconomic dimensions of pollution such as impacts on fisheries, aquaculture, public health, and local livelihoods remain underexplored. Including these aspects would offer a more holistic understanding of risks and support the formulation of integrated adaptation strategies.

(3) Marine Biodiversity and Ecosystem Resilience: Further studies should examine the resilience and adaptability of marine species and ecosystems in polluted environments. Identifying ecological thresholds for degradation would be critical for developing biodiversity conservation plans and setting regulatory limits for pollutant discharge.

By addressing these gaps, future research can build a more comprehensive framework for coastal environmental management, balancing ecological sustainability with economic development under changing climatic conditions.

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Evaluating the Efficacy of Natural Zeolite in Treating Saline Seawater for Multiuse in Southern Jordan

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ABSTRACT

This study investigates the use of various types of natural zeolite stones to treat seawater in the Gulf of Aqaba in Southern Jordan. The research aims to examine the physical and chemical impacts of these stones on the permanent hardness of seawater and their potential for reducing this hardness to the lowest possible level while preserving the water's essential properties. The treated water is intended for diverse practical uses, including agricultural, industrial, and domestic applications. The study's main objective is to minimize the dissolved salts and hardness of highly saline water while maintaining the physical and chemical composition. This research focused on two categories of analysis: (1) the physical effects, which are focused on measuring the percentage of dissolved salts, electrical conductivity, turbidity, color, odor, and pH of water; (2) chemical effects, which cover water components such as carbonates, bicarbonates, sulfates, chlorides, calcium, magnesium, potassium, sodium, nitrates, nitrites, and heavy metals before, during, and after treatment. Field testing instruments were employed alongside laboratory-based sampling and analysis to ensure precise and reliable documentation throughout the study. The extent of lead removal from water using zeolite was implemented in Jordan by a researcher from Zarqa University, who achieved good results with water discharged from a battery manufacturing plant that processes lead. The results were as follows: The zeolite used was one ton; daily factory water usage was 20 m^2; working days were 100; and discharged water volume was 2000 m^3. Finally, permissible pollution limits were safe and compliant with international specifications. The experimental results in this study showed that initial TDS values exceeding 38,000 ppm were reduced by more than 50% in several cases.

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INTRODUCTION

The zeolite group includes several hydrous silicate minerals with similar chemical compositions and natural occurrences. These minerals are primarily aluminum, sodium, and calcium silicates and typically contain a high percentage of water. Zeolite minerals have a hardness ranging from 3.5 to 5.5 and a specific gravity between 2.0 and 2.4. Many zeolite minerals melt rapidly, often accompanied by swelling and foaming. They are classified as secondary minerals and are commonly found in volcanic rocks' voids, gaps, and veins. Zeolite minerals are lightweight and fragile, and they occur in various colors, such as orange, yellowish green, light green, and colorless or white, with fine crystal sizes measured in fractions of a millimeter. Water hardness is a scientific term used to describe the condition in which water contains high concentrations of dissolved salts, particularly calcium (Ca^{2+}) and magnesium (Mg^{2+}) ions. This condition affects the water's physical and chemical properties, often leading to reduced lathering with soap, a salty or bitter taste, and challenges in domestic and industrial applications. The World Health Organization (WHO) and the Food and Agriculture Organization (FAO) have established guidelines defining acceptable salt concentrations for water used for human, animal, and agricultural use.

The major contributors to water hardness include calcium (Ca), magnesium (Mg), bicarbonates (HCO_3^-), sulfates (SO_4^{2-}), calcium carbonate ($CaCO_3$), and calcium sulfate ($CaSO_4$). High levels of these dissolved salts, particularly bicarbonates, sulfates, chlorides, and carbonates, are often responsible for water hardness. In addition, there are two types of water hardness: temporary hardness, which is primarily caused by bicarbonate salts, and permanent hardness, which results from calcium and magnesium salts in the form of sulfates and chlorides. These salts do not precipitate upon heating and, therefore, require chemical treatment for removal.

The negative impacts of using hard water include the deposition of magnesium and calcium salts, which form scale layers on the interior surfaces of vessels and pipes, reducing their thermal conductivity. This issue leads to the formation of insulating layers, which increase energy consumption, whether electric or fuel-based, both in intensity and duration. Also, the accumulation of deposits increases the likelihood of pipe blockages and internal corrosion due to chemical interactions between the salts and the metallic components of the thermal systems. There are some restrictions related to zeolite-based nanocomposites because the process of breaking bonds destroys and disintegrates the crystalline structure of the zeolite material and causes it to lose one of its most important properties, which are the structural voids that are considered the storehouse of the outputs of the ion exchange process that zeolite possesses.

REVIEW OF LITERATURE

Jordan has severe water scarcity and highly salinized water sources, particularly in areas like the Gulf of Aqaba. Jordan's dedication to large-scale ecological wastewater management is exemplified by Al-Samra Wastewater Treatment Plants (2024), which uses life cycle assessment (LCA) techniques to maximize environmental and performance results. Several studies have also been conducted to study the effect of using zeolite in water treatment. Kumar and Singh (2024) provide a general overview of zeolite uses for eliminating various contaminants from wastewater, such as dyes, phenolic compounds, and heavy metals. They examine how synthesis methods, adsorption processes, and operational factors affect removal effectiveness. Li et al. (2024) explore the field of modified zeolites and critically assess their improved performance in heavy metal removal, corroborating their findings.

Al-Farajat & Al-Khashman (2024) approve using natural zeolites in aquatic environments. Their experimental investigation evaluated the effects of zeolite filtration on biological parameters and water quality in the aquaculture of European seabass. A thorough analysis of natural zeolites as dual-purpose sorbents that can remove carbon dioxide and heavy metals from wastewater is conducted by Mambetova et al. (2024). Their research highlights the zeolites' adsorption-facilitating structural properties and their promise as environmentally friendly materials for combined air and water purification systems. An overview of next-generation desalination methods, such as forward osmosis, membrane distillation, electrodialysis, and solar desalination, is given by Genesis Water Technologies (2024).

Despite their high technological cost, these new approaches could work with systems based on natural materials, such as zeolites, to provide hybrid solutions in areas with limited water resources. Indeed, Jordan's strategic efforts to secure a long-term water supply using Red Sea water are demonstrated by large-scale projects like the Aqaba-Amman Water Desalination and Conveyance Project (European Investment Bank, 2023) and the deep subsea desalination partnership between Waterise and Jordan Phosphate Mines Company (2024).

The literature evaluation confirms natural zeolites' effectiveness, adaptability, and environmental sustainability in water treatment, especially when eliminating heavy metals, ammonia, and other impurities. Because of its water problems, Jordan is a good place to install zeolite-based systems with cutting-edge desalination equipment. A realistic way to achieve sustainable water security is to combine natural resources with contemporary technologies, particularly in arid and semi-arid areas like Jordan.

Margeta et al. (2013) and Barloková (2008) have examined the effectiveness of using natural zeolite in water treatment. A review of modified zeolite as a material for wastewater purification was provided by Widiastuti et al. (2008), Hamed et al. (2022), and Shoumkova (2024). Nizam et al. (2021) investigated the application of natural zeolite to treat greywater, focusing on removing ammonia and heavy metals to improve water quality in fish ponds. Similarly, Onyutha et al. (2024) demonstrated using zeolite in household water treatment to remove arsenic, lead, and fluoride, following turbidity reduction through slow sand filtration. Using natural zeolite as a cost-effective adsorbent for removing ammonium from drinking water was explored by Eberle et al. (2022).

In another study, Mnyango et al. 2010, used clinoptilolite functionalized with the cationic surfactant hexadecyl trimethyl ammonium (HDTMA) to control nitrate concentrations in drinking water. Salman et al. (2017) examined the static absorption of heavy metals V^{+5}, Ni^{+2}, Zn^{+2}, and Pb^{+2} using natural zeolite sourced from southern Syria. Their research focused on ascertaining the adsorption capability of

inexpensive zeolite used to extract heavy metals from crude oil desalter effluent, which poses significant risks to human and ecological health if discharged untreated. Margeta et al. (2013) also discussed the removal of arsenic from water using both natural and modified clinoptilolite.

MATERIALS AND METHODS

Scientific literature and theoretical studies confirm the positive effects of the zeolite mineral in reducing water hardness and removing various impurities and chemical compounds that are foreign to water's natural composition (H_2O). These impurities are typically introduced through external environmental factors encountered during the water's natural cycle.

The methodology of this work is structured as follows:

1. **Treatment Objective:** Seawater from the Gulf of Aqaba was treated using natural zeolite stones to reduce the **concentration** of dissolved salts, which contribute to water hardness, to the lowest possible level.
2. **Application Scope:** The study aimed to evaluate the feasibility and effectiveness of the treated water for various societal uses, including human consumption, animal use, agriculture, and industry.
3. **Location:** Gulf of Aqaba
4. **Duration:** The experimental period was extended from November 5, 2022, to March 5, 2023, to ensure scientifically reliable results supported by laboratory test reports from accredited laboratories in Jordan.
5. **Zeolite Samples:** Three types of natural zeolite stones were used, identified as Z1, Z2, and Z3. These were sourced from different officially licensed extraction sites in Jordan.
6. **Water Samples:** After studying the conditions of the sea (the Gulf of Aqaba) in terms of tides, and noting that two high tides and two low tides occur every 24 hours, sampling was synchronized with these tidal variations across several days (Table 1). Geographic variability was incorporated by selecting sampling points at different depths and distances from the shoreline.
7. **Experimental Procedure:** A structured setup was implemented for the treatment and testing processes.
8. **Data Verification and Analysis:** Data collected from different samples were analyzed and validated to ensure reliability and accuracy.

Chemicals and Toxic Compounds: Toxic substances and chemicals must be absent from water intended for use. If present, their concentrations must remain within acceptable limits, as defined by international standards. Table 2 outlines the permissible concentration limits (in mg/L) for various toxic and chemical substances in water.

Table 1: Seawater sampling information and timings.

Sample name	Sample collection time	Sea condition	Sample quantity (L)	Sample collection date
A	9.30 am	After the first high tide	100	6/11/2022
B	2.00 pm	Near the first low tide	100	6/11/2022
C	8.20 pm	At the peak of the second high tide	100	8/11/2022
D	2.30 am	At the second low tide	100	9/11/2022

Table 2: Permissible limits of toxic and chemical substances in water.

Material	Ratio mg/L	Material	Ratio mg/L
Lead	1.0	Copper	5.1
Arsenic	50	Manganese	5
Zinc	15	Iron	1
Phenol	200	Nitrates	Max 45
Sulfates	400	Fluorides	Max 8
Chlorides	600	Mercury	0-100
Magnesium	150	Cadmium	10
Calcium	200	Cyanide	50
pH	6.5- 9.2	Lead	1.0

Specifications of Potable Water According to the WHO

The WHO has established 62 criteria for safe drinking water, including chemical, physical, microbiological, and sensory criteria that people may detect with their senses.

- Color: Acceptable and not exceeding 50 units on the scale of platinum cobalt.
- Taste: It should be pleasant and palatable.
- Odor: Should be odorless.
- Turbidity: Should be clear, with a maximum of 5 Jackson units for treated water and up to 52 units for groundwater.
- Dissolved Oxygen (O_2): 5–8 mg/L at 25°C.
- Dissolved Carbon Dioxide (CO_2): 2–3 mg/L at 25°C.
- Electrical Conductivity**:** 0.0004 µmho/cm² at 18°C.
- Thermal Conductivity**:** 1.555 W/m·K at 40.8°C.
- Refractive Index: 1.33 at 20°C.
- Vapor Pressure: 17.62 mm Hg at 20°C.
- Specific Heat: 0.99 kJ/kg·°C at 20°C.
- Density: 1.00 g/cm³ at 4°C and 0.99823 g/cm³ at 20°C.

Table 3: Results of Sample A Treated with Zeolite (Z1, Z2, Z3).

Sample name	Sample type	Color	TDS	pH
A	Filtered seawater	clear	35,000	7.8
AZ1-1	Sample treated after 1 hour	clear	33,020	6.8
AZ1-2	Sample treated after 2 hours	clear	31,150	5.6
AZ1-3	Sample treated after 3 hours	clear	29,820	4.5
AZ1-6	Sample treated after 6 hours	clear	22,320	4.8
AZ1-12	Sample treated after 12 hours	clear	20,120	5.2
AZ1-24	Sample treated after 24 hours	clear	18,500	7.7
AZ2-1	Sample treated after 1 hour	clear	32,800	6.9
AZ2-2	Sample treated after 2 hours	clear	30,110	6.1
AZ2-3	Sample treated after 3 hours	clear	28,150	5.1
AZ2-6	Sample treated after 6 hours	clear	21,000	5.8
AZ2-12	Sample treated after 12 hours	clear	19,550	6.4
AZ2-24	Sample treated after 24 hours	clear	17,420	7.6
AZ3-1	Sample treated after 1 hour	clear	33,500	7.2
AZ3-2	Sample treated after 2 hours	clear	31,100	6.6
AZ3-3	Sample treated after 3 hours	clear	28,850	5.2
AZ3-6	Sample treated after 6 hours	clear	20,440	6.1
AZ3-12	Sample treated after 12 hours	clear	17,820	6.8
AZ3-24	Sample treated after 24 hours	clear	15,250	7.9

- Freezing Point: 0°C
- Boiling Point: 100°C
- Latent Heat of Vaporization: 584.9 cal/g at 20°C
- Surface Tension: 72.75 dynes/cm at 20°C

RESULTS AND DISCUSSION

The tidal times in the Gulf of Aqaba were studied over 30 days to determine tidal levels. The sampling time was set to the time of highest tide (because the seawater was at its lowest salinity after testing), and the sampling time was set to the time of lowest ebb tide (when the water was at its highest salinity after testing) because there are no freshwater outlets in the Gulf. It is considered one of the coastal areas with the highest percentage of dissolved salts because it is relatively enclosed, except where it meets the Red Sea.

The experimental procedure began with physical testing of raw seawater, particularly assessing Total Dissolved Solids (TDS), color, and pH. Then, the water was passed through natural zeolite mineral stones using a controlled approach to determine the effectiveness of zeolite in reducing hardness and improving water quality. Three different types of natural zeolite (Z1, Z2, and Z3) were tested simultaneously. After each treatment interval, the same tests were conducted to measure changes in water characteristics. The performance of each zeolite type was monitored over varying durations of 1, 2, 3, 6, 12, and 24 hours. Further adjustments, such as pH balancing using chemical additives, were made to align the treated water's quality with recognized natural water standards, particularly for taste and usability.

Sample "A" Measurements

Table 3 presents the results of treating different Sample "A" using the three zeolite types over various time intervals.

Table 3 shows how zeolite affects the reduction of Total Dissolved Solids (TDS). The initial TDS value of 35,000 mg/L was reduced significantly after 24 hours of treatment:

- AZ1-24: 18,500 mg/L (~47% reduction)
- AZ2-24: 17,420 mg/L (~50% reduction)
- AZ3-24: 15,250 mg/L (~56% reduction)

These results indicate a consistent and effective performance of natural zeolite in reducing salinity and dissolved solids. Notably, the Z3 sample exhibited the highest efficiency, suggesting better adsorptive properties.

It is important to note that the experiment was conducted under static laboratory conditions, where the water remained stationary during contact with the zeolite. In real-world (dynamic) applications, where water is continuously moving, the interaction and mixing rates are higher, potentially improving the efficiency of ion exchange and enhancing the overall purification process.

Fig. 1 illustrates how sample A's pH changed over time. At the initial time point (0 hours), the pH of the filtered seawater was recorded at 7.8 across all three samples. As treatment progressed, a noticeable decrease in pH was observed, particularly within the first 3 to 6 hours. This decline indicates the acidic influence of natural zeolite on the treated water. After 3 hours, the pH values dropped to 4.5, 5.1, and 5.2 for AZ1, AZ2, and AZ3, respectively, suggesting the zeolite samples possess mildly acidic characteristics that lower the water's pH. To ensure the treated water met acceptable acidity standards, minor chemical adjustments were made, raising the pH to approximately 7.0–7.5, aligning the treated water with international potable water guidelines for usability.

Sample "B" Measurements

The results for the different Sample "B," collected near the first low tide, are presented in Table 4. As with Sample A, seawater was treated using three types of natural zeolite (BZ1, BZ2, and BZ3), and measurements were taken over different exposure times ranging from 1 to 24 hours.

Table 4 demonstrates how zeolite affects the reduction

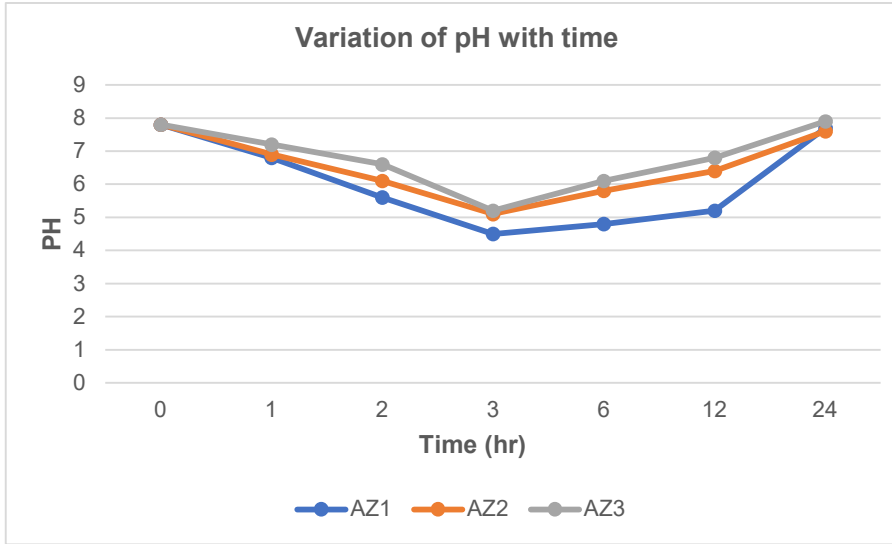

Fig. 1: Variation of pH with time for sample A.

Table 4: Data obtained from Sample B.

Sample name	Sample type	Color	TDS	pH
B	Filtered seawater	clear	38,100	8
BZ1-1	Sample treated after 1 hour	clear	35,200	7
BZ1-2	Sample treated after 2 hours	clear	33,050	6.5
BZ1-3	Sample treated after 3 hours	clear	31,300	6.1
BZ1-6	Sample treated after 6 hours	clear	26,350	5.9
BZ1-12	Sample treated after 12 hours	clear	21,825	6.8
BZ1-24	Sample treated after 24 hours	clear	19,850	7.9
BZ2-1	Sample treated after 1 hour	clear	36,050	7.2
BZ2-2	Sample treated after 2 hours	clear	32,800	6.6
BZ2-3	Sample treated after 3 hours	clear	29,150	5.8
BZ2-6	Sample treated after 6 hours	clear	24,250	6.1
BZ2-12	Sample treated after 12 hours	clear	21,500	6.7
BZ2-24	Sample treated after 24 hours	clear	18,560	7.8
BZ3-1	Sample treated after 1 hour	clear	34,550	7.3
BZ3-2	Sample treated after 2 hours	clear	31,325	6.9
BZ3-3	Sample treated after 3 hours	clear	29,100	5.9
BZ3-6	Sample treated after 6 hours	clear	25,400	6.3
BZ3-12	Sample treated after 12 hours	clear	21,125	7.7
BZ3-24	Sample treated after 24 hours	clear	16,800	8.1

of Total Dissolved Solids (TDS) across all three zeolite types over time. The initial TDS of the filtered seawater was 38,100 mg/L. After 24 hours of treatment, TDS values were reduced to 19,850 mg/L for BZ1-24, 18,560 mg/L for BZ2-24, and 16,800 mg/L for BZ3-24. These reductions represent an approximate 44–52% decrease, indicating a significant purification effect through the zeolite filtration process.

Fig. 2 illustrates the changes in pH levels of Sample B over time during zeolite treatment using the three different types. At 0 hours, all samples showed a consistent pH value of 8.0. The pH declined throughout the treatment, particularly within the 3–6 hour range. This decrease is attributed to the mildly acidic nature of the zeolite materials. After 3 hours of treatment, the pH values dropped to 6.1, 5.8, and 5.9 for BZ1, BZ2, and BZ3, respectively. Although this trend reflects the same behavior observed in Sample A, the reduction in pH for Sample B was slightly less steep. Nonetheless, minor chemical adjustments were applied to raise the pH to near-neutral levels (7.0–7.5) to ensure compliance with water quality standards and make the treated water suitable for practical uses. These results reaffirm zeolite's efficacy in reducing TDS and altering pH and highlight the importance of post-treatment pH correction when considering real-world applications.

Sample "C" Measurements

The results obtained from different "C" samples are presented in Table 5**.** As with previous samples, seawater was treated using three zeolite types (CZ1, CZ2, and CZ3), and measurements were taken at intervals up to 24 hours.

Table 5 demonstrates a gradual and consistent reduction in Total Dissolved Solids (TDS) over time across all zeolite samples. The initial TDS value of the filtered seawater was 33,280 mg/L. After 24 hours of treatment, TDS values were reduced to 15,500 mg/L for CZ1-24, 15,025 mg/L for CZ2-24, and 13,320 mg/L for CZ3-24. These values

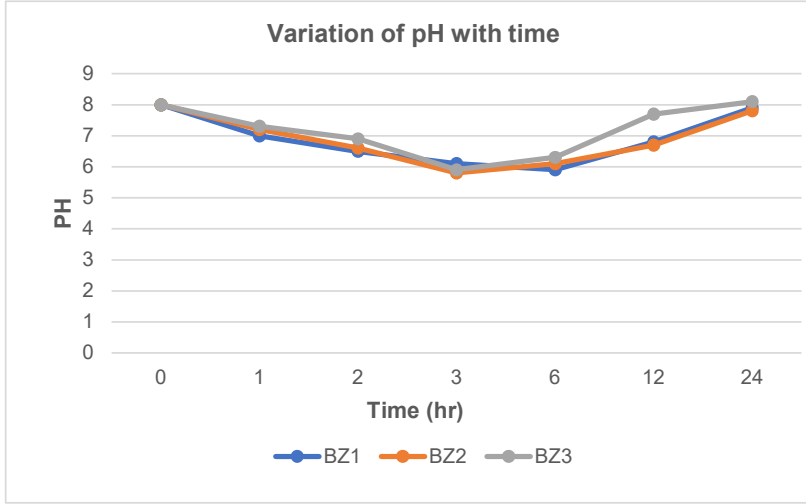

Fig. 2: Variation of pH with time for sample B.

Table 5: The data obtained from different "C" samples.

Sample name	Sample type	Color	TDS	pH
C	Filtered seawater	clear	33,280	7.9
CZ1-1	Sample treated after 1 hour	clear	32,050	7.7
CZ1-2	Sample treated after 2 hours	clear	30,100	7.2
CZ1-3	Sample treated after 3 hours	clear	27,500	6.8
CZ1-6	Sample treated after 6 hours	clear	23,875	6.1
CZ1-12	Sample treated after 12 hours	clear	21,000	6.8
CZ1-24	Sample treated after 24 hours	clear	15,500	7.8
CZ2-1	Sample treated after 1 hour	clear	31,950	7.6
CZ2-2	Sample treated after 2 hours	clear	29,750	7.1
CZ2-3	Sample treated after 3 hours	clear	25,330	6.5
CZ2-6	Sample treated after 6 hours	clear	20,410	5.9
CZ2-12	Sample treated after 12 hours	clear	14,850	6.8
CZ2-24	Sample treated after 24 hours	clear	15,025	7.9
CZ3-1	Sample treated after 1 hour	clear	31,100	7.5
CZ3-2	Sample treated after 2 hours	clear	28,450	7.1
CZ3-3	Sample treated after 3 hours	clear	25,330	6.3
CZ3-6	Sample treated after 6 hours	clear	21,650	6.9
CZ3-12	Sample treated after 12 hours	clear	18,825	7.1
CZ3-24	Sample treated after 24 hours	clear	13,320	7.8

reflect a decrease of approximately 40–46%, confirming the continued effectiveness of zeolite in reducing dissolved salts in seawater.

Fig. 3 shows that at the initial time (0 hr), all samples had the same pH value of 7.9. The pH values decreased over time due to the effect of zeolite, reaching lower values after several hours of treatment. This indicates the acidic influence of the zeolite used.

Sample "D" Measurements

The data obtained from different "D" samples are shown in Table 6.

Table 6, like the previous ones, demonstrates the reduction of Total Dissolved Solids (TDS) by zeolite. The TDS values for DZ1-24, DZ2-24, and DZ3-24 were significantly reduced to 17,500, 18,050, and 16,150 ppm,

Table 6: The data obtained from different "D" samples.

Sample name	Sample type	Color	TDS	pH
D	Filtered seawater	clear	37,900	8
DZ1-1	Sample treated after 1 hour	clear	35,010	7.2
DZ1-2	Sample treated after 2 hours	clear	31,990	6.7
DZ1-3	Sample treated after 3 hours	clear	27,830	6.1
DZ1-6	Sample treated after 6 hours	clear	23,000	5.6
DZ1-12	Sample treated after 12 hours	clear	20,580	6.4
DZ1-24	Sample treated after 24 hours	clear	17,500	7.9
DZ2-1	Sample treated after 1 hour	clear	35,980	7.2
DZ2-2	Sample treated after 2 hours	clear	33,025	6.8
DZ2-3	Sample treated after 3 hours	clear	30,950	5.8
DZ2-6	Sample treated after 6 hours	clear	27,860	6.1
DZ2-12	Sample treated after 12 hours	clear	21,045	6.9
DZ2-24	Sample treated after 24 hours	clear	18,050	7.8
DZ3-1	Sample treated after 1 hour	clear	35,650	7.4
DZ3-2	Sample treated after 2 hours	clear	31,870	6.2
DZ3-3	Sample treated after 3 hours	clear	27,540	5.7
DZ3-6	Sample treated after 6 hours	clear	22,450	6.1
DZ3-12	Sample treated after 12 hours	clear	19,230	6.6
DZ3-24	Sample treated after 24 hours	clear	16,150	7.9

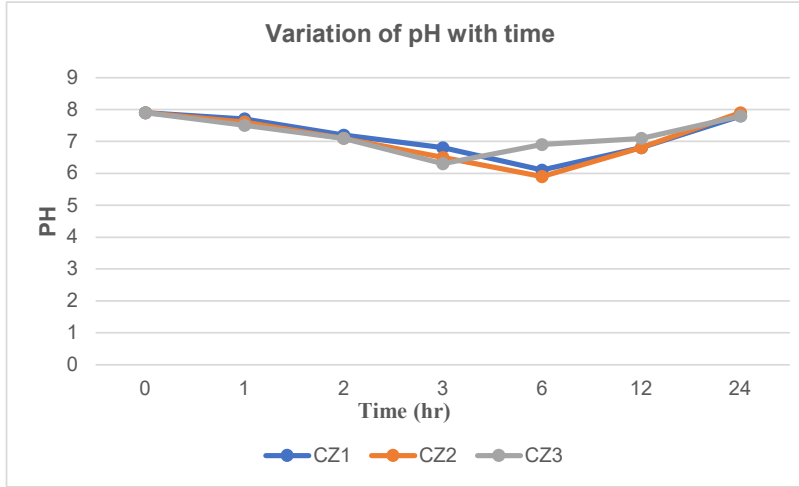

Fig. 3: Variation of pH with time for sample C.

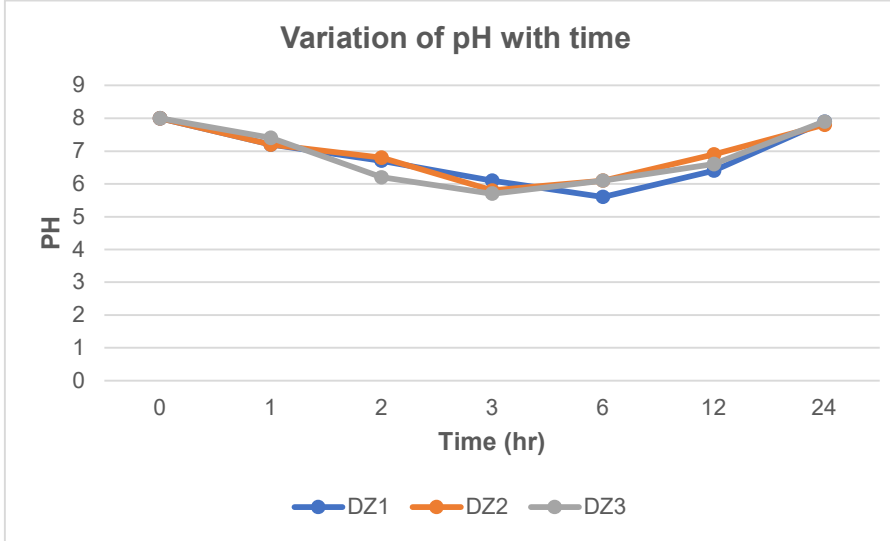

Fig. 4: Variation of pH with time for sample D.

respectively, representing a 42–46% reduction from the initial value of 37,900 ppm.

Fig. 4 shows that at the initial time (0 h), all samples had a pH of 8. Over time, the pH decreased due to the effect of zeolite, indicating its acidic nature. From the experimental data across all sample groups, the results confirm the hypothesis that natural zeolite effectively reduces the hardness and salinity of seawawater. The highest initial TDS values ranged between 33,280 and 38,100 ppm, and zeolite treatment reduced these by approximately 40–50%. The remaining dissolved salts were primarily sodium-based, representing temporary hardness, which can be relatively easily removed using modern treatment methods to make the water suitable for various applications.

Additionally, the decrease in pH values after treatment supports the acidic characteristics of the zeolite used. After treatment, a minor chemical adjustment was applied to restore the pH to a more neutral range (7.0–7.5). It is important to note that these tests were conducted under static conditions in the laboratory. In a dynamic setting, where water is flowing or circulating, the efficiency of zeolite treatment is expected to improve further due to enhanced ionic exchange between water and zeolite particles.

This research was conducted to find solutions and alternatives to the chemicals used in treating hard saline water, which cost high amounts of money and have a short operational life. In addition, zeolite raw materials are natural materials with a sustainable life and are considered

environmentally friendly. Their physical value is acceptable, and they can be recycled for agricultural uses when saturated in the water treatment field and do not require destruction. In the field of applications, they have been used in some countries around the world, the most important of which is water treatment after the well-known Chornobyl reactor accident, as well as for greywater treatment in several countries (Germany, Sweden, and Austria).

It should be noted that the work in this study depends on seawater loaded with high levels of salts, which cause permanent hardness, and on the presence of lead (Pb^{2+}), which is often very weak in open water. This is the reason for not considering interactions between Pb^{2+} and functional groups in zeolite.

CONCLUSIONS

The experimental results support the hypothesis that natural zeolite reduces seawater's hardness. Initial TDS values exceeding 38,000 ppm were reduced by more than 50% in several cases. Laboratory tests confirmed that the remaining dissolved salts are primarily sodium-based, contributing to temporary hardness, and can be readily removed using modern treatment technologies. This indicates that natural zeolite can significantly improve seawater quality, making it more suitable for various practical uses.

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Leveraging the Green Petroleum Hydrocarbon Remediation Potential of a Biosurfactant Producing Indigenous Oleophilic Bacterium Isolated from Hydrocarbon Soiled Environment

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ABSTRACT

The present investigation focused on the physicochemical characterization and bioprospecting of an indigenous oleophilic bacterium (OB) and its biosurfactant (BS) for bioremediation. Within 14 days of culture at 30°C with 2% (v/v) n-hexadecane, the OB could reduce the surface tension of the culture medium by up to 34.4 mNm^{-1}. Standard screening tests verified that the isolated OB produced BS and identified it as *Pseudomonas aeruginosa*. BS production was 434.7 $mg.L^{-1}$, with a CMC of 195.6 $mg.L^{-1}$, and was purified and characterized using standard chromatographic and spectroscopic techniques. FTIR analysis confirmed the glycolipid nature of BS. TLC of the partially purified BS revealed two homologues of rhamnolipid (RL), which were subsequently confirmed by NMR. Seven distinct RL congeners were identified using LC-MS, of which di-RLs constituted a notably large proportion. The surface and emulsification activities of BS demonstrated significant stability against various pH levels, temperatures, salinities, and metal ions. Furthermore, OB was able to utilize crude oil within 60 days, as confirmed by GC-MS. In the soil washing experiment, BS separated ≥80% of the crude oil from the contaminated sand at the CMC. The results suggest that the RLs and their producer isolated from automobile workshops in Mokokchung are not only the first report from Nagaland, India, but are also promising for various applications in the bioremediation of extreme and complex environments, including addressing regional environmental issues in Nagaland.

INTRODUCTION

There is mounting global concern over the degradation of the environment by various anthropogenic chemical contaminants that are continuously released into our surroundings, causing severe environmental degradation. Petroleum hydrocarbons are one of the most common contaminants that affect different components of the environment. These pollutants are released into the environment mainly through oil spills during extraction, transportation, and refinement leaks, raising serious issues for the environment, ecological systems, and public health, leading to both short- and long-term environmental harm (Chandankere et al. 2014, Gote et al. 2023). Heavy metals such As, Cd, Cu, Zn and Pb are another group of hazardous, non-biodegradable, and persistent environmental pollutants that cause prolonged ecological and environmental damage as they tend to pass and magnify from one food chain to another, obstructing biological pathways, disrupting cellular functions, and causing diseases and damage to various organs (Das et al. 2017). In the environment, these heavy metal pollutants are found in association with sub-surface soil sediments, which often results in their release into groundwater, leading to contamination (Yang et al. 2020).

Existing conventional physical and biological technologies used for the remediation of these environmental pollutants are often associated with several

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drawbacks, such as high costs, low efficiencies, and the production of secondary pollutants. Therefore, cleaner, eco-friendly, cost-effective, and efficient technologies, such as biologically derived treatment strategies, are needed to tackle these hazardous pollutants (Usman et al. 2016). One such environmentally benign approach is the use of BSs derived from microbes, such as bacteria, fungi, and yeast, for bioremediation (Rodrigues et al. 2006). The presence of BSs can potentially enhance the mineralization and biodegradation of hydrocarbon pollutants by solubilizing and emulsifying the contaminants and serving as an important mediator by increasing the bioavailability of hydrophobic contaminants for microorganisms (Parthasarathi et al. 2011, Ławniczak et al. 2013). BSs are also known to form non-ionic complexes with heavy metals in the soil, thereby reducing the interfacial tension between the soil and metal ions, resulting in the removal of heavy metals from contaminated environments (Santos et al. 2016).

BSs are amphiphilic compounds produced by microorganisms that adhere to cell surfaces or are secreted extracellularly into the surrounding medium (Chioma et al. 2013). They are amphiphilic organic compounds with hydrophobic (tails) and hydrophilic (heads) groups. The hydrophobic (non-polar) end of the BS is insoluble in water. It can contain long chains of fatty acids, hydroxyl fatty acids, or α-alkyl-β-hydroxy fatty acids. In contrast, the hydrophilic (polar) end may contain a phosphate, carboxylic acid, cyclic peptide, carbohydrate, amino acid, or alcohol (Chioma et al. 2013). Microorganisms produce a diverse range of BSs with different structural profiles, including polymeric, particulate, neutral lipids, glycolipids, lipopeptides, and lipoproteins (Wang et al. 2011).

Among the known BSs, rhamnolipids (RLs) have received considerable attention compared to chemically synthesized surfactants because of their unique physicochemical properties, low toxicity, biodegradability, and ecologically benign nature, which makes them potential candidates in the field of environmental remediation processes (Banat et al. 2010, Md Fakruddin 2012, Chandankere et al. 2014). RL belongs to the class of glycolipid BSs and is reported to have a significantly higher yield than other classes of BSs (Arutchelvi et al. 2010). RL consists of two molecules: lipid, sometimes referred to as the aglycon part, and rhamnose, commonly known as the glycon. A hydrophilic compound, the rhamnose moiety, is composed of mono- or di-(L)-rhamnose molecules connected by α-1, 2-glycosidic bonds (Thakur et al. 2021). The properties of RLs are influenced by the congener composition, which varies depending on the type of bacterial strain, growth conditions, and media composition (Costa et al. 2010).

The potential uses of RLs in the fields of environmental protection (Abdel-Mawgoud et al. 2008), petroleum extraction (Makkar & Cameotra 2002), synthesis of specific compounds (Das et al. 2013), agriculture (Sorhie et al. 2022), and the manufacturing of pharmaceuticals (Md Fakruddin 2012) and food (Cameotra & Makkar 1998) have sparked interest in them. Currently, there is growing interest in the market for RLs, but they cannot effectively compete with their chemical counterparts because of various factors, specifically their higher cost of production (Chrzanowski et al. 2012, Md Fakruddin 2012, Thakur et al. 2021). In this regard, screening for more efficient BS-producing native microbial strains can be an effective strategy to increase BS yield and prospects (Chandankere et al. 2014). Native BS-producing strains can be readily isolated from the natural environment, which might prove more efficient and cost-effective than mass culturing, maintaining, and effectiveness of an exotic microbial species in a foreign environment. Moreover, the use of native bacterial strains can address issues related to legitimate and biosafety concerns.

In the backdrop of the aforementioned information, the current research work was embarked upon with the prime objective to isolate a potential RL-producing bacterial strain from Mokokchung Town, Nagaland, India, for the first time, as per our information, with desirable physico-chemical properties to address the issues of soil contaminated with hydrocarbon and heavy metal pollutants.

MATERIALS AND METHODS

Isolation of Oleophilic Bacteria from Environmental Samples

Several bacteria were isolated from oil-contaminated soil samples collected from different automobile workshops in Mokokchung Town, Nagaland, India. Bacterial isolation was performed using the enrichment culture technique, where 5 g of each soil sample was inoculated into a 250 mL Erlenmeyer flask containing 100 mL of Bushnell-Haas medium with 2% (v/v) n-hexadecane as the sole carbon source. For the first cycle of incubation, the cultures were shaken in an orbital shaker for 14 days at 140 rpm at 30°C ±1. An aliquot of 1mL from each of the original cultures was transferred to 250 mL flasks containing fresh media with n-hexadecane and incubated for another 14 days for the second cycle. This process was repeated up to the fifth cycle. After the fifth cycle, the viable bacterial populations were isolated from the culture broth using the serial dilution technique in Bushnell Haas agar plates supplemented with n-hexadecane (2% v/v).

Culturing and Screening of BS-Producing Oleophilic Bacteria

The bacterial isolates obtained after five cycles of enrichment culture were later grown in mineral salt medium (MSM), and the pH of MSM was adjusted to 6.8 using 6 N HCl (Bharali et al. 2011). The culture was supplemented with 2% (v/v) n-hexadecane as the sole carbon source, and the temperature and pH were adjusted and maintained at 6.8 and 30°C, respectively, in an orbital shaker with an agitation rate of 140 rpm for 14 days (Bharali et al. 2022). The cell-free culture supernatant (CFCS) of bacterial cultures was used for the screening of BS production through various standard methods, such as surface tension (ST) measurement (Bodour & Miller-Maier 1998), oil displacement assay (Thavasi et al. 2011), drop collapse test (Bodour & Miller-Maier 1998), penetration assay (Kumar et al. 2017), CTAB agar test (Aparna et al. 2012), and hemolysis test (Mulligan et al. 1984, Youssef et al. 2004). The isolate that demonstrated the best results in the screening assays was selected for further characterization of its surface activity.

Characterization of Selected Bacterial Isolate

The morphological features of the selected bacterial isolate were examined using Bergey's Manual of Determinative Bacteriology (Bergey 1994). Standard biochemical testing was conducted as outlined by Cappuccino & Sherman (2013). Molecular characterization of the selected bacterial isolate was performed using a standard 16S rRNA gene sequencing approach. Based on the BLAST results, the evolutionary distance of the isolate with its most closely related strains was computed, and the sequence obtained was deposited in the NCBI GenBank database. Evolutionary analyses were conducted using MEGA 11 software with the maximum likelihood approach and 1000 bootstrap replicates (Tamura et al. 2021).

Growth Kinetics and BS Production

The chosen bacterial strain was cultivated in MSM enriched with 2% (v/v) n-hexadecane at 30°C in an orbital shaker operating at 140 rpm for 22 days (Bharali & Konwar 2011). Using a UV-Vis spectrometer (PerkinElmer UV/VIS Lambda 365), the optical density (OD) of the selected bacterial culture was recorded at 600 nm wavelength after every 24 h of incubation. For biomass quantification, the increase in dry bacterial biomass after every 24 h of incubation was determined gravimetrically. BS production in the culture medium was tracked every 24 h using the standard orcinol method (Rahman et al. 2010).

Isolation of BS

Bacterial cells were separated from the culture supernatant by centrifugation at 10000 rpm for 10 min. The CFCS was precipitated by acidifying it to pH 2 using 6 N HCl and stored overnight at 4°C. Acid-precipitated BSs were centrifuged for approximately 15 min at 10000 rpm and washed twice with phosphate-buffered saline (PBS). After washing, the recovered pellets were dried in a hot-air oven to remove moisture. The dried BS was then measured gravimetrically using a laboratory balance.

For the partial purification of the BS, the acidified precipitated CFCS was extracted three times using ethyl acetate at room temperature. The solvent was extracted from the organic phase and concentrated using a rotary evaporator with a round-bottom flask. A sticky, honey-colored BS was obtained at the end of the process (Bharali et al. 2011).

Purification of Partially Purified BS

Thin-layer chromatography (TLC) was used to identify and purify the BS. Chloroform: methanol: water (65:15:2) (v/v/v) was used as the mobile phase system (Bharali & Konwar 2011). Developing agents such as anthrone and iodine fumes were used to detect the presence of carbohydrate and lipid moieties of glycolipid-type BSs, respectively (George & Jayachandran 2013).

Approximately 5 g of a partially purified BS was dissolved in 10 mL of chloroform and loaded onto a glass column (26 cm × 3.3 cm), which was previously packed with 50 g of column-grade silica gel, followed by cleaning the column with chloroform to remove the neutral lipids. Subsequently, fractions were collected at a flow rate of 1 mL min^{-1}, with 20 mL portions obtained using chloroform: methanol mobile phases in a sequence of 50:3 v/v (1000 mL), 50:5 v/v (200 mL), and 50:50 v/v (100 mL). A final rinse with a 1:1 mixture of chloroform and methanol was used to remove residual RLs from the column. The collected fractions were evaporated to dryness under vacuum using a rotary evaporator (Mishra et al. 2019).

Physical Characterization of Isolated BS

Determination of Critical Micelle Concentration (CMC)

Various concentrations of BS solutions were prepared in water from the stock solution of the BS. The ST of the dilutions was measured at room temperature to determine the CMC. CMC was determined by plotting the ST as a function of the BS concentration. Three replicas of these studies were conducted (Chandankere et al. 2014).

Emulsification Index

The emulsification index (E_{24}) was determined by adding 2 mL of CFCS and an equal amount of different hydrocarbon oils, including diesel, kerosene, petrol, vegetable oil, crude oil, and waste vegetable oil, in different test tubes and vortexed for 2 min. The mixture was incubated for 24 h at room temperature (Sarubbo et al. 2006, Câmara et al. 2019). The percentage of emulsification after 24 h, E_{24} (%), was calculated using the following formulae,

E_{24} (%) = (Height of emulsion layer)/(Total height of the solution) × 100

Chemical Characterization of Isolated BS

Fourier-Transform Infrared Spectroscopy (FT-IR)

FT-IR analysis of the partially purified BS was performed in the 400–4000 cm^{-1} range. A translucent disc was prepared using a mixture of 10 mg of the extracted BS and 100 mg of spectral purity KBr at 25 Mpa for 30 second (Zhao et al. 2019). The resultant KBr discs were analyzed using a Perkin Elmer FTIR spectrophotometer (Perkin Elmer-Spectrum Two).

Liquid Chromatography and Mass Spectroscopy

The RLs were characterized using an MS Q-TOF Mass Spectrometer (model G6550B). The separation was based on reverse-phase chromatography using a Zorbax Eclipse XDB-C18 column (3.0 × 150 mm, 3.5 µm) at a temperature of 40°C. The flow rate was 0.2 mL min^{-1}. The mobile phase gradient consisted of 10 mM aqueous ammonium formate buffer with 0.05% formic acid (A) and acetonitrile (B). The gradient started at 20% B from 0 to 2 min, increased to 50% B from 2 to 10 min, and reached 100% B from 10 to 15 min. It remained constant at 100% for 5 min, then decreased to 20% over 8 to10 min, and remained constant until equilibration was achieved. The total run time was 30 min, and the injection volume of sample was 2 µL (Behrens et al. 2016).

Nuclear Magnetic Resonance

The samples were prepared by dissolving approximately 3 mg of BS in a 100% $CDCl_3$ solution and analyzed using an NMR spectrometer (Advance DPX 500 & 300 MHz FT NMR Spectrometer, Bruker). Proton and carbon NMR chemical shifts were measured in parts per million (ppm) with respect to the solvent shift, which served as the chemical standard. Using previously published data, the peaks were predicted and compared (Sharma et al. 2015).

Stability Studies

Effect of Environmental Variables on E_{24}%

The effect of time duration on the E_{24}% of the CFCS in diesel was calculated after 24 h, 15 days, and 30 days to determine the stability of the emulsification layer over time. The CFCS was exposed to 121, 100, 80, 40, 25, and 4°C for 60 min and emulsified with diesel to determine the effect of temperature on the E_{24}% of the BS. The pH of the CFCS was adjusted to 3, 5, 7, and 10 and emulsified with diesel oil to determine the effect of pH on the E_{24}% of the BS. Different concentrations of NaCl salt, 2%, 5%, 8%, and 10% (w/v), were added to CFCS to check the variation in the E_{24}% against diesel. Different heavy metallic salt, *viz*. $NaAsO_2$, $CdCl_2$, and K_2CrO_4 were added to the CFCS to determine the variation in the E_{24}% of the BS. For temperature, pH, NaCl, and heavy metal salts, the emulsion mixture was incubated for 24 h at room temperature. E_{24} (%) was calculated using the formula as described above.

Effect of Environmental Variables on Surface Activity

The reduction in the ST of the CFCS was checked after exposing it to a wide range of temperatures, namely, 121°C, 100°C, 80°C, 40°C, 25°C, and 4°C for 60 min to determine the effect of temperature on surface activity. To determine the effect of pH on surface activity, the pH of the CFCS was adjusted to 3, 5, 7, and 10. The effect of salinity on surface activity was evaluated by adding different concentrations of NaCl *i.e*., 2%, 5%, 8%, and 10% (w/v), to the CFCS. Different heavy metallic salts, *viz*. $NaAsO_2$, K_2CrO_4, and $CdCl_2$ were added at different concentrations (100 ppm, 300 ppm, and 500 ppm) to the CFCS to determine the variation in surface activity. The reduction in ST was measured using a surface tensiometer based on the *du Noüy* ring method.

Bioremediation Application

Soil washing experiment: 10% (w/w) of low viscosity crude oil was added to the acid-washed sand and allowed to sit at room temperature for seven days. Then, 5 g of sand samples were added to 100 mL of CFCS containing different concentrations of BS (at CMC, below CMC, and above CMC) in a 250 mL conical flask and rotated in an orbital shaker at 200 rpm for 24 h at 30°C. The aqueous solutions were decanted, and the sand was dried for 24 h at 50°C to evaporate the remaining solvent. The amount of oil that remained was measured gravimetrically, and the following equation was used to obtain the percentage of oil removed:

Crude oil removed (%) = (Oi – Or)/Oi × 100%

where Oi is the initial oil in the sand sample before washing and Or is the oil remaining in the sand sample after washing (Costa et al. 2010).

Crude oil degradation: 1 mL of the freshly grown culture supernatant of the bacterial strain was inoculated into 250 mL containing 2 mL of crude oil and 100 mL of MSM. The cultures were incubated at 30°C in an orbital shaker for 15, 30, 45, and 60 days. After each incubation period, the

cultures were centrifuged at 4500 rpm for 10 min to separate the bacterial biomass, and the residual hydrocarbon oil fractions in the CFCSs were extracted twice using dimethyl chloromethane (DCM) in a ratio 1:1. The contents of the residual crude oil extracted over 15, 30, 45, and 60 days were analyzed using Gas Chromatography-Mass Spectroscopy (Singh et al. 2016). Before being elevated to 280°C for crude oil analysis, the column temperature was maintained at 50°C for five min. A split ratio of 20:1 was used in all the studies. Helium was used as the carrier gas, and the flow rate was 0.8 mL min^{-1}. The injector was adjusted to a temperature of 250°C (Bharali et al. 2022).

RESULTS

Isolation of Oleophilic Bacteria from Environmental Samples

Thirty different numbers of soil samples were collected from ten hydrocarbon-contaminated sampling sites in the Mokokchung Town area of Nagaland, India (Fig. 1).

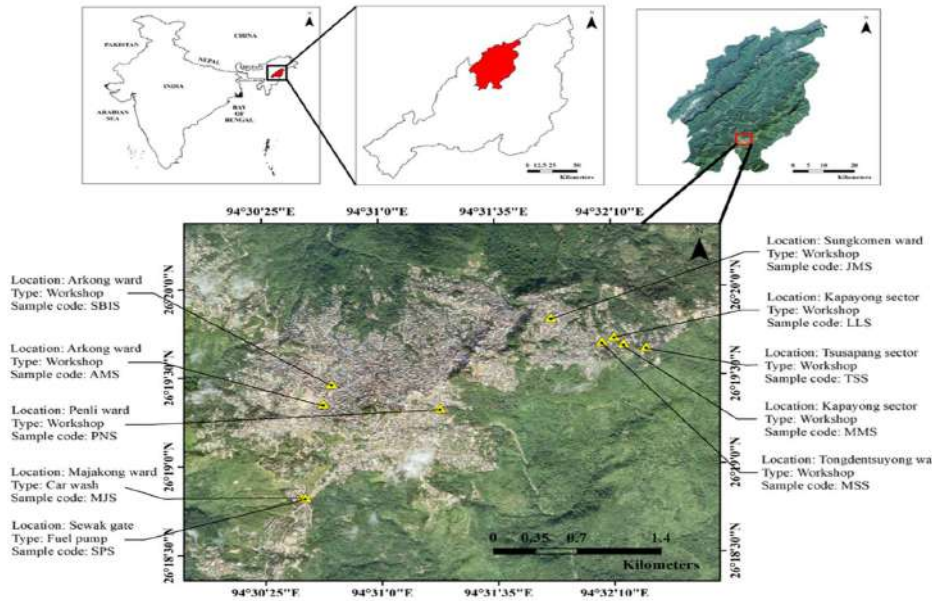

Fig. 1: Map illustrating the geographical position of the research region within Mokokchung town and the spatial layout of the sampling locations.

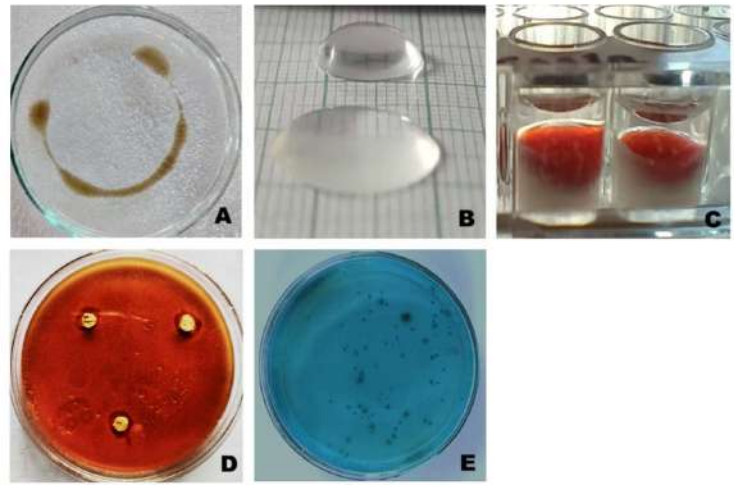

Fig. 2: Screening and confirmation of BS production by the selected isolate (A) oil displacement test, (B) drop-collapse test, (C) penetration assay, (D) hemolysis, and (E) CTAB agar assay.

From the environmental samples, approximately 60 bacterial isolates were obtained after the 5th cycle of enrichment culture of the soil samples using the serial dilution technique. The morphological characteristics of the obtained bacterial isolates were then recorded.

Culturing and Screening of BS-Producing Oleophilic Bacteria

The obtained bacterial isolates were further grown in MSM supplemented with 2% (v/v) n-hexadecane for screening and characterization. The reduction in the ST of the CFCS of the obtained bacterial isolates was determined. Isolates showing the best surface activity in terms of ST reduction measurement of the culture medium were selected for the second level of screening. Screening assays, such as oil displacement, drop collapse, and penetration assays, were performed to screen for BS production. The results of the screening assays of the selected bacterial isolate are presented in Fig. 2A, B, and C.

Potential strain was further screened using hemolysis and CTAB agar assays. The results are shown in Fig. 2D and E. The clear zone around the filter paper disc in the hemolysis assay indicated BS production. The anionic nature of the BS was confirmed using the CTAB agar test, which showed dark blue halos around the bacterial colonies.

Characterization of Selected Bacterial Isolate

The selected bacterial isolate was morphologically characterized using standard methods. The shape, size, and surface texture of the selected bacterial isolate were determined, and pure culture plates were prepared (Fig. 3A).

Table 1: Standard biochemical tests of the selected hydrocarbonoclastic bacterial isolate.

Biochemical Tests	Result
Gram staining	Negative rods
Spore staining	Negative
Catalase test	Positive
Oxidase test	Positive
Motility test (SIM)	Positive
Urea test	Negative
Indole test	Negative
MR test	Negative
VP test	Negative
Citrate test	Positive
TSI test	Alkaline
H_2S Test (SIM)	Negative
Nitrate test	Positive with Gas
Carbohydrate fermentation after 24 hrs	
Glucose	Positive
Sucrose	Negative
Lactose	Negative
Mannitol	Negative

The results of the standard biochemical characterization of the selected bacterial isolate are presented in Table 1.

The selected bacterium was identified as *Pseudomonas aeruginosa* (AMS1a) through 16S ribosomal RNA gene

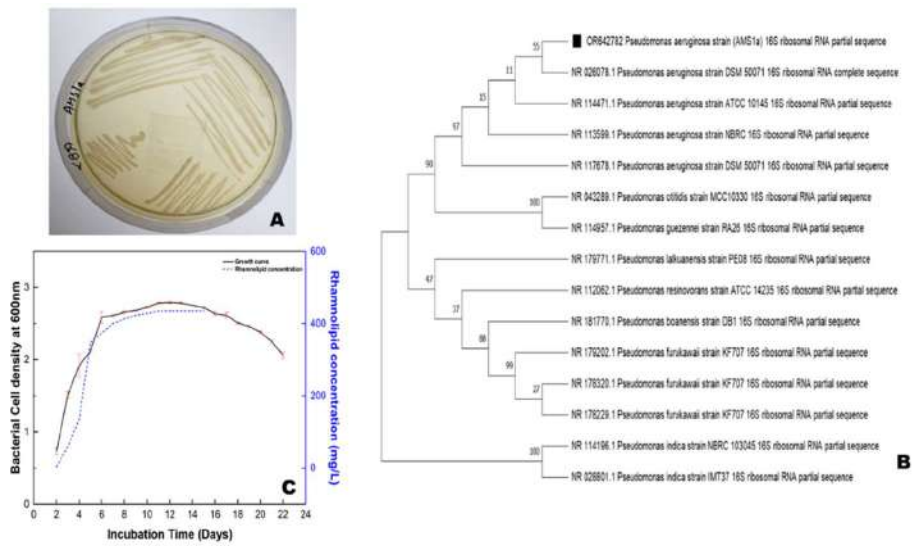

Fig. 3: (A) Colony morphology of pure culture on nutrient agar plate, (B) Phylogenetic tree of the selected bacterial isolate illustrating evolutionary relationships with closely related species, and (C) Growth and BS production curve of the selected bacterial isolate on MSM with n-hexadecane. Results are presented as the mean ± S.D. of three individual experiments.

sequencing, and the obtained sequence was submitted to NCBI with the accession number OR642782. In the phylogenetic tree analysis, the topology with the highest log-likelihood value was chosen. Fourteen nucleotide sequences were identified in this study (Fig. 3B).

Growth Kinetics and BS Production

The growth curve of the bacterial strain *P. aeruginosa* (AMS1a) was determined under constant temperature (30°C) and agitation (140 rpm) for 22 days. Fig. 3C shows the typical growth curve of the selected bacterial strain. It is evident from the growth curve that the bacteria were in the lag phase until the 3rd day and went into the exponential phase from the 4th day, indicating a rapid increase in bacterial population, which was continuous up to the 11th day. The time duration between the 2nd-3rd, 4th-8th and 9th-12th days represents the early, mid and late stationary phases, respectively. The stationary phase was sustained until the 15th day. Following the 15th day of incubation, the density of bacterial cells in the culture declined at a steady rate until the 22nd day, indicating the death phase.

It was found that the RL production increased with the increase in bacterial growth in the culture medium and accelerated in the exponential phase. Fig. 3C shows the pattern of BS production by the selected bacterial strain over 15 days. The RL concentration remained steady during the stationary phase. The final yield of RL reached a concentration of approximately 434.7 µg mL^{-1} after two weeks of incubation.

Isolation of BS

The CFCS containing the BS was acidified to pH 2 for approximately 24 h at 4°C, which caused the BS in the supernatant to protonate, resulting in the precipitation of the BS in the solution. The precipitated solution was then extracted using ethyl acetate as the solvent to separate the BS from the aqueous phase. The same is shown in Fig. 4A, 4B and 4C.

Purification of Partially Purified BS

TLC was used to determine the purity of the partially purified BS using a chloroform-methanol-water (65:35:2, v/v/v) solvent system. The separated compounds were observed under UV light (Fig. 4i). Two major spots and one minor spot were developed after exposure to iodine fumes (Fig. 4ii) and spraying with acidified anthrone reagent (Fig. 4iii) separately. The spots appeared at R_f values of 0.3, 0.5, and 0.7, as shown in Fig. 4iii.

The neutral lipids present in the partially purified BS were eluted from the column using chloroform. A solvent system comprising chloroform: methanol in a ratio of 50:3 (v/v) was used to separate the first fraction of the BS with a R_f value of 0.7. This was followed by a mobile solvent system comprising chloroform: methanol in a ratio of 50:5 (v/v) to separate the BS fraction with an R_f value of 0.5. Finally, a mobile system consisting of a 1:1 v/v ratio of chloroform: methanol was used to separate the final BS fraction, with an R_f value of 0.3. All the fractions of BS were confirmed by TLC before the introduction of the subsequent

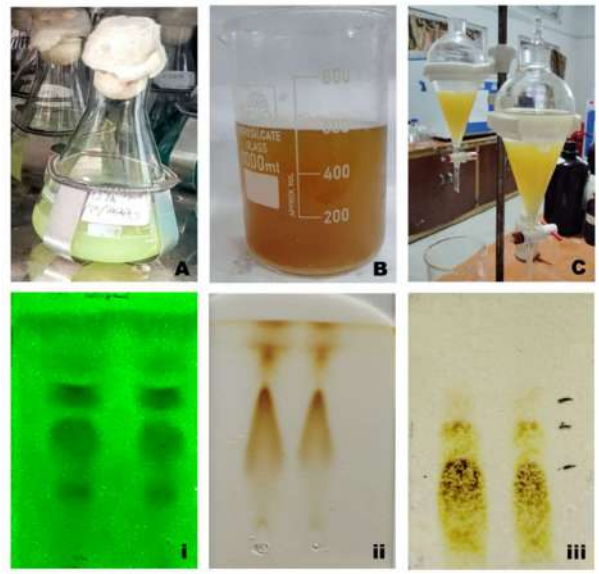

Fig. 4: (A) Matured MSM culture supplemented with n-hexadecane, (B) Acid ppt of crude BS from CFCS, (C) Solvent extraction of BS. TLC chromatogram of partially purified BSs after exposure to (i) UV lamp, (ii) iodine fumes, and (iii) anthrone reagent.

mobile system. The solvent system present in the separated BS fractions was evaporated and recovered using a rotary evaporator.

Physical Characterization of Isolated BS

Determination of CMC: The CMC of the BS produced by *P. aeruginosa* isolated from oil-contaminated sites of Mokokchung town, Nagaland, was found to be 195.6 mg L^{-1}, corresponding to a reduction in the ST of distilled water from ~72 mNm^{-1} to 34.4 mNm^{-1}. Fig. 5A shows the CMC of the isolated BS.

Emulsification activity: The emulsification indices of the tested BS against six different hydrophobic substrates were determined. The BS formed a stable emulsion with all the hydrophobic substrates used in the experiment, as evident from the height of the emulsion, as shown in Fig. 5B.

Chemical Characterization of Isolated BS

FTIR

The functional groups of the BS were further examined using FTIR spectroscopy. The results are presented in Fig. 6.

The presence of an aliphatic long fatty acid chain was indicated by the presence of distinctive peaks at 878, 1402, 2856, and 2931 cm^{-1}. The distinctive groups found in the BSs were the significant functional groups, namely the carbonyl group (C–O) (1260 cm^{-1}), alkene (C = C) (1644 cm^{-1}), and OH bond (3410 cm^{-1}). The vibration at 1124 cm^{-1} was observed to correlate with C–O stretching, which predicted the existence of the sugar moiety, while the presence of a more significant band at 1735 cm^{-1} implied the presence of a carboxyl group, which showed the linking group between the sugar and fatty acid. The FTIR results indicated that the isolated BS may belong to the glycolipid family.

Nuclear Magnetic Resonance

As shown in Fig. 7A and 7B, the presence of a chemical shift value in ^{1}H NMR at 1.24 ppm indicates the methyl group ($-CH_3$) corresponding to the sugar moiety.

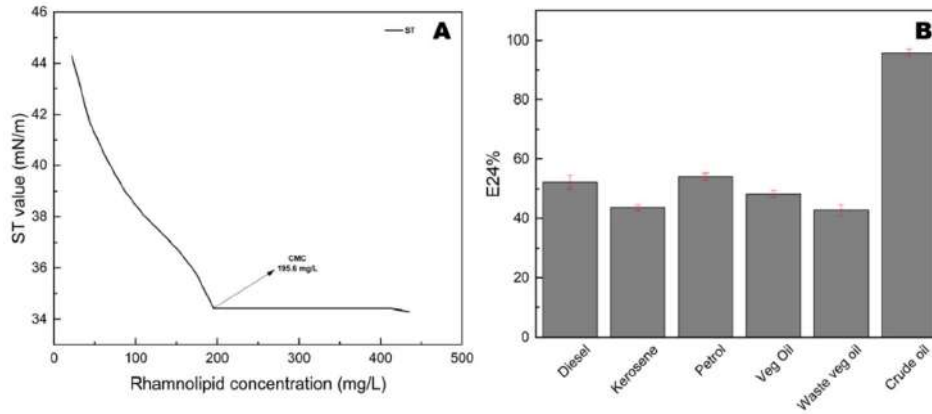

Fig. 5: (A) Determination of the CMC of the isolated BS and (B) emulsification activity (E_{24}%) of the BS toward selected hydrophobic substrates. Results represent mean ± S.D. of three individual experiments.

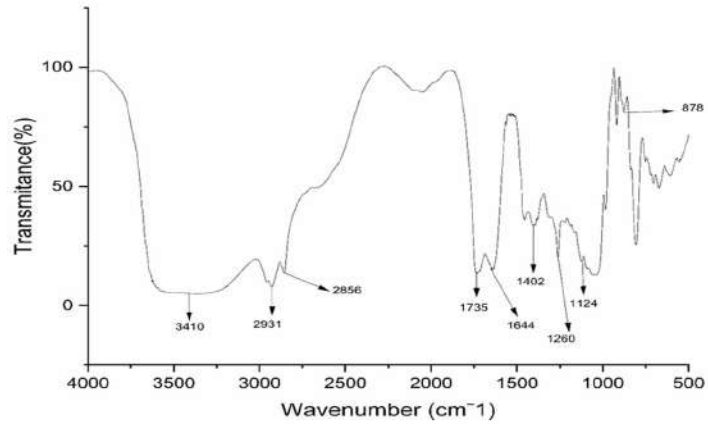

Fig. 6: FTIR spectrum of the isolated BS.

The peak at 0.86 ppm corresponds to the (–CH_3) group of the long-chain aliphatic group. The presence of the sugar moiety was confirmed by the presence of peaks at 4.93 (-1'-H), 3.63 (-5'-H), and 4.14-4.29 ppm (-2,' 3,' 4'-H). The sharp peaks at 1.24 ppm correspond to the long-chain hydrocarbon of the lipid group. Other characteristic peaks were also observed for -COO-CH- (5.81 ppm), -O-CH- (4.30 ppm), and -CH_2-COO- (2.32, 2.8 ppm), which confirms the presence of mono-RL with two long-chain fatty acids. Similarly, characteristic peaks at 0.82 (-CH_3), 1.24-1.21 ppm {-$(CH_2)_n$ and -CH_3 (ring)}, 5.34 (-COO-CH-), 4.16 ppm (-O-CH-), 2.35, 2.47 ppm (-CH_2-COO-) confirm the structure of di-RL.

In Fig. 7C and 8D, 170.85 and 171.61 ppm show the ester group (C=O) of mono and di-RL, respectively. The

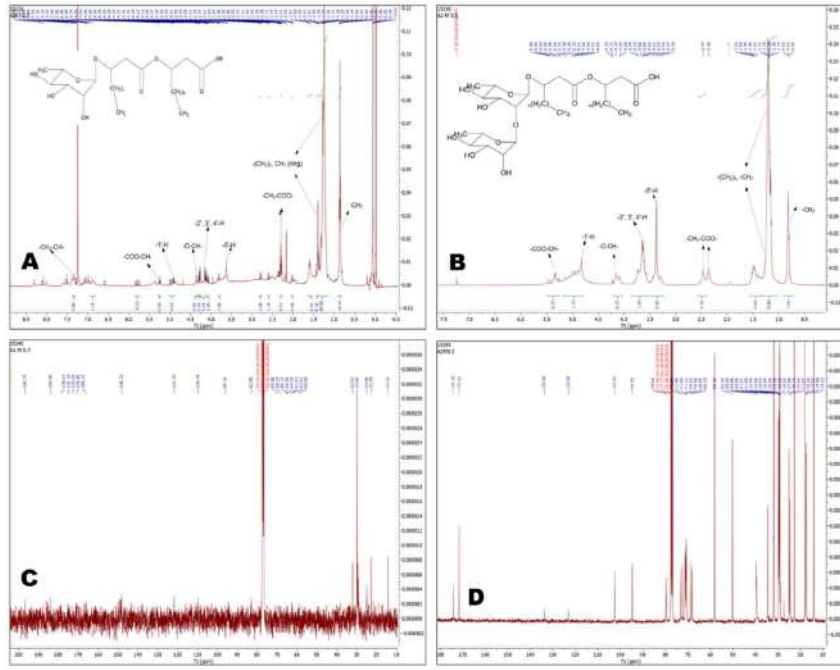

Fig. 7: (A and B) ^{1}H spectra of isolated mono-RL and di-RL, and (C and D) ^{13}C NMR spectra of isolated mono-RL and di-RL.

Table 2: The different RL congeners present in the isolated BS and their peak assignments.

Sl. No.	RT	m.z^{-1} $[M-H]^-$	Rhamnolipid/ HAA	Characteristic fragments								
				Rha-Rha-FA1	Rha-Rha-FA2	Rha-FA1	Rha-FA2	FA1-FA2	FA2-FA1	FA1	FA2	Others
1	22.06	621.37	Rha-Rha-C_8-C_{10}, Rha-Rha-C_{10}-C_8	-	479.27	-	-	-		141.1	169.13	204.9, 205.08, 247.1
2	23.05	649.4	Rha-Rha-C_{10}-C_{10}	479.27	-	-	-	--		169.2	-	163.07, 205.08, 247.10
3	23.5	675.423	Rha-Rha-C_{10}-$C_{12:1}$	479.27	-	-	-	-		169.2	195.1	247.1, 205
4	24.24	531.379	Rha-C_{10}-C_{12}	-	-	333.21		-		169.13	197.17	163.07, 205.08
5	24.5	703.45	Rha-Rha-C_{10}-$C_{14:1}$	479.27	-	333.21	-	-		169.2	197.17	163.07, 204.98, 248.97
6	24.9	705.4701	Rha-Rha-C_{10}-C_{14}, Rha-Rha-C_{14}-C_{10}	479.27	535.33	-	-	-		169.2	225.20	205.08, 163.07, 247.10
7	24.9	705.4701	Rha-Rha-C_{12}-C_{12}	507.3058	-	-	-	-		197.17	-	205.08, 163.07, 247.10

Note: RT - Retention time, Rha - Mono-RL, Rha-Rha - Di-RL, FA1-Fatty acid short chain, FA2-Fatty acid long chain.

presence of a carboxylic group was confirmed by the peaks at 173 ppm for mono-RL and 174.20 ppm for Di-RL. Peaks at 62.52-82.95 ppm and 68.13-73.36 ppm correspond to the rhamnose rings in both mono and di-rhamnolipid, respectively.

Liquid Chromatography and Mass Spectroscopy

LC-MS/MS was performed using the purified BS. The total ion chromatogram (TIC) obtained from LC-MS showed seven dominant peaks with retention times ranging from 22.059 to 24.907 min (Table 2).

These peaks detected in the TIC corresponded to a mixture of mono- and di-RLs. The peak detected at m/z 531 corresponded to Rha-C_{10}-C_{12}, while the rest of the signals were found to be di-RL, showing the m/z [M-H]- at 621.387 for Rha-Rha-C_8-C_{10}/ Rha-Rha-C_{10}-C_8, Rha-Rha-C_{10}-$C_{12:1}$ (m/z 675.423), Rha-Rha-C_{10}-C_{10} (m/z 649.4), Rha-Rha-C_{10}-$C_{14:1}$ (m/z 703.45), Rha-Rha-C_{10}-C_{14}/Rha-Rha-C_{14}-C_{10} (m/z 705.4701), and Rha-Rha-C_{12}-C_{12} (m/z 705.4701). The different assignments for the pseudo-molecular ion peaks and other characteristic peaks shown by the cleavage of the rhamnose moieties are presented in Table 2. In the case of

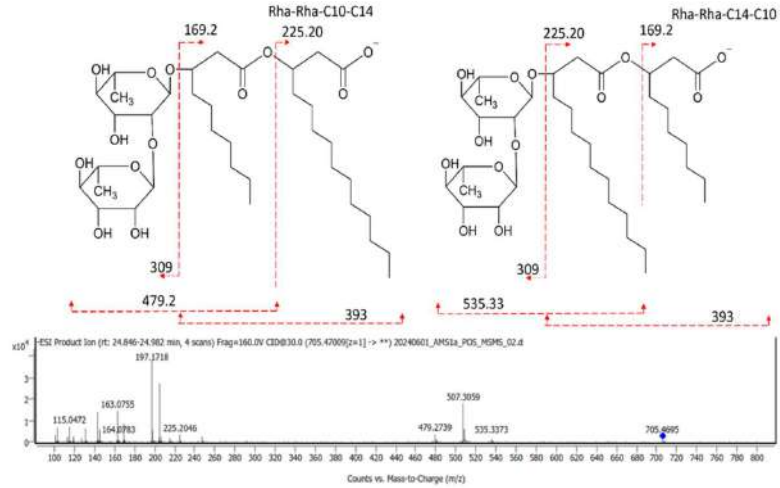

Fig. 8: ESI product ion of abundantly present Rha-Rha-C_{10}-C_{14} and Rha-Rha-C_{14}-C_{10} molecules.

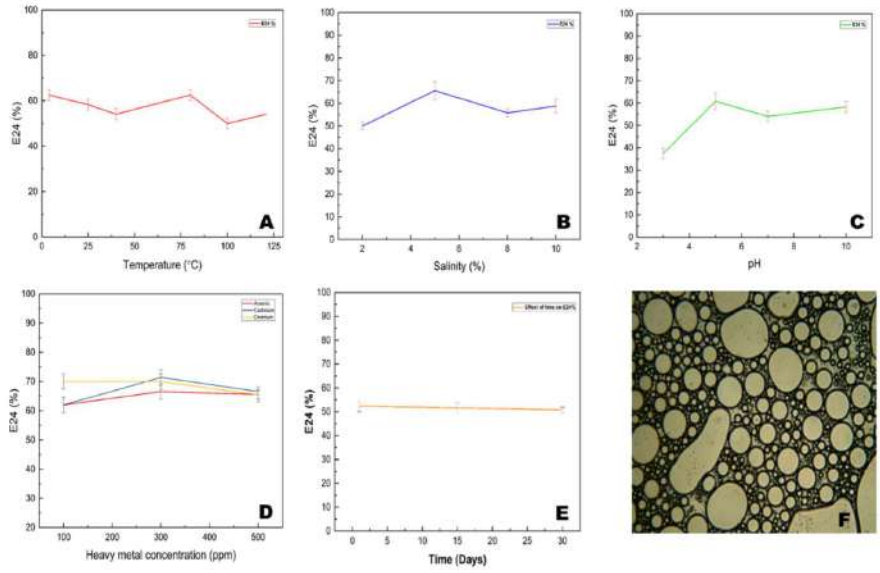

Fig. 9: Effect of varying (A) temperature (4-121ºC), (B) salinity (2-10%), (C) pH (3-10), (D) heavy metals (As, Cd, and Cr) at varying concentration (100-500 ppm), (E) time period (0-30 days) on the emulsifying activity of isolated BS, and (F) 10X magnification view of 30th day stable emulsion. Results represent mean ± S.D. of three individual experiments.

di-RLs such as Rha-Rha-C_{10}-C_{14}/Rha-Rha-C_{14}-C_{10} with m/z 705.4694, the fragmentation of the two fatty acid chains led to two peaks at m/z 225 and m/z 169.2. Another cleavage at the ether linkage of rhamnose and fatty acid chains gives two characteristic peaks at m/z 309 for the di-rhamnose moiety and m/z 393 for the two fatty acid chains, as shown in Fig. 8.

Stability Studies

Effect of environmental variables on E_{24}%: The BS exhibited and retained a stable E_{24}% within 54.1% to 62.5 at temperatures ranging from 4ºC to 121ºC, demonstrating the absence of any significant effect of temperature on the emulsification activity, and the same is presented in Fig. 9A.

At varying salinities (2%-10%), the BS was able to maintain a stable E_{24}% of 50% to 65.6%, highlighting the stable nature of the BS and the same is presented Fig. 9B. The BS was able to maintain a stable E_{24}% of 54% to 60% at the pH range of 5 to 10 and the same is presented in Fig. 9C. However, at an acidic pH of 3, the emulsification activity decreased to 37.5%. The BS showed a stable emulsification activity with an E_{24}% value of 62% to 71.4 with all the three types of heavy metal used (As, Cd, and Cr) at varying concentration (100-500 ppm), as shown in Fig. 9D. Over 30 days, no significant change in emulsification activity was observed (Fig. 9E). The BS maintained an average emulsification index value of approximately 51.6% ± 0.07 for 30 days of incubation. A compound microscopic image of the stable emulsion at 10 X magnification is presented in Fig. 9F.

Effect of environmental variables on surface activity: The BS was able to maintain a consistent reduction of ST of distilled water at an average of approximately 35 mNm^{-1} ± 0.05 at a temperature range of 25ºC to 121ºC. However, a slight increase in the ST value was observed, 36.1 ± 0.15 mNm^{-1} at 4°C, as shown in Fig. 10A.

As shown in Fig. 10B, the BS was able to maintain the reduction of ST value within the value of 34.2 ± 0.15 to 35.6 mNm^{-1} ± 0.05 at the salinity range of 2% to 10%. A small but steady reduction in ST values was observed with an increase in salinity. At pH values ranging from 3 to 10, the ST of the BS increased marginally and consistently with increasing pH. The maximum reduction in ST of 31.5 mNm^{-1} ± 0.1 was observed at pH 3, which steadily increased to 36.6 mNm^{-1} ± 0.05 at pH 10, as shown in Fig. 10C. The BS maintained a stable reduction in ST within the range of 34.4 mNm^{-1} ± 0.05 to 35.8 mNm^{-1} ± 0.11 with all the three types of heavy metals used (As, Cd, and Cr) at varying concentration (100-500 ppm), as shown in Fig. 10D.

Bioremediation Application

Soil washing experiment: In the crude oil washing experiment, BS performed much more consistently and efficiently after 24 h of washing (Fig. 11A). The average removal rates of the natural surfactants at CMC, below CMC,

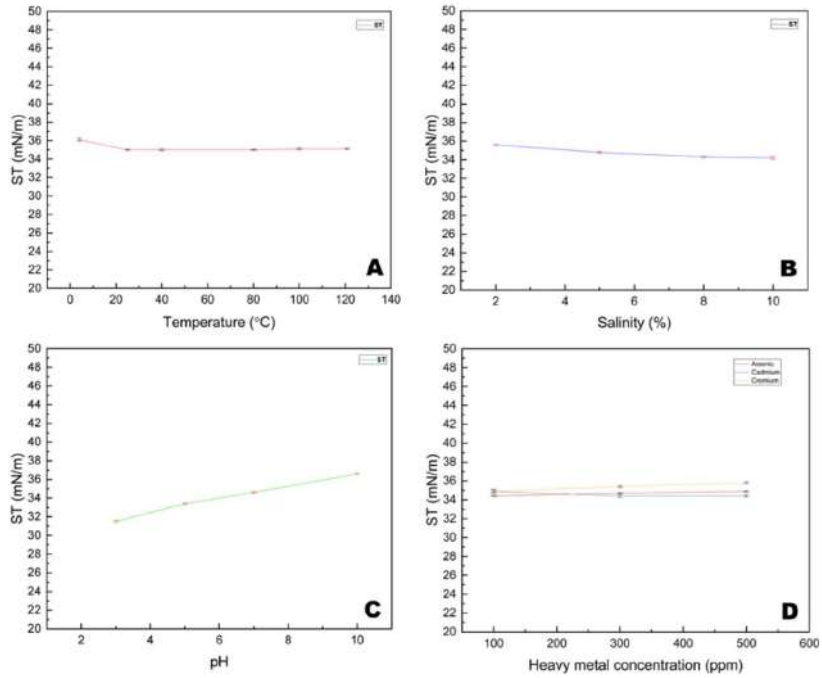

Fig. 10: Effect of varying (A) temperature (4-121ºC), (B) salinity (2-10%), (C) pH (3-10), and (D) heavy metals (As, Cd, and Cr) at varying concentration (100-500 ppm) on the surface activity of the isolated BS. Results represent mean ± S.D. of three individual experiments.

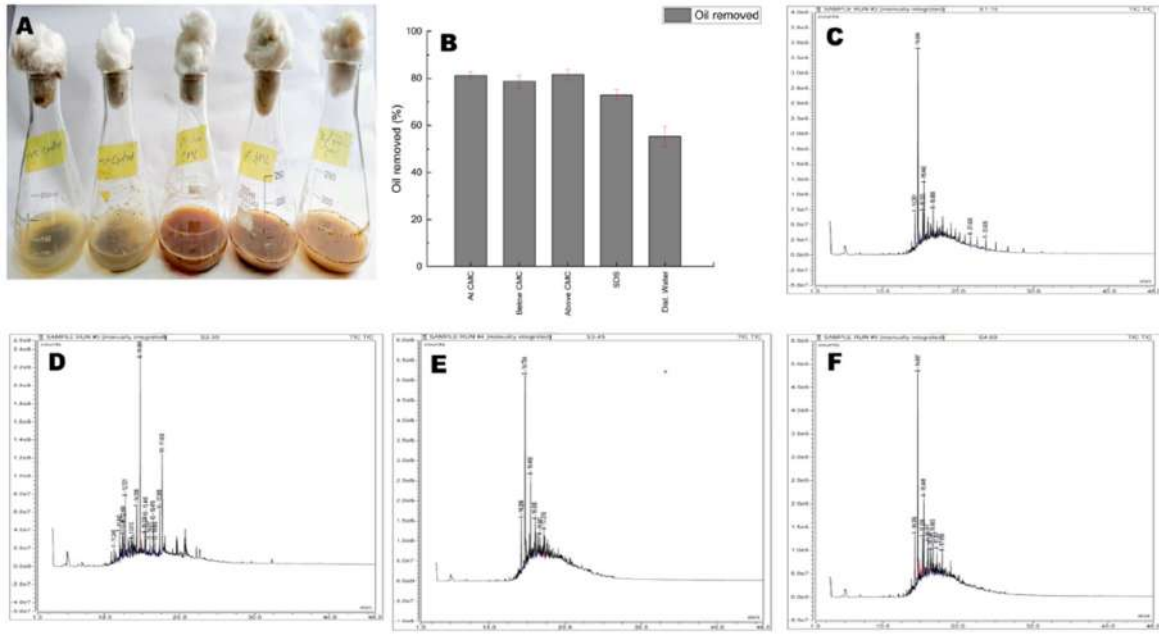

Fig. 11: (A) Soil washing experiment, (B) Effect of BS concentration, SDS, and water on soil washing. Results represent mean ± S.D. of three individual experiments and degradation pattern of crude oil by the selected bacterial strain after (C) 15 days, (D) 30 days, (E) 45 days, (F) 60 days.

and above CMC were 81.2% ± 1.5, 78.6% ± 2.7, and 81.6% ± 2.2, respectively. This is illustrated in Fig. 11B. The synthetic surfactant, SDS (0.1% w/v), was able to remove an average of 72.9% ± 2.3 of the crude oil, and the control, distilled water, was able to remove approximately 55.4% ± 4.2 of the oil.

Crude oil degradation: The ability of the selected bacterial strain to degrade crude oil was validated using GC-MS. Fig. 11C, D, E, and F display the chromatograms for 15, 30, 45, and 60 days, respectively. The results demonstrated that only specific medium to long-chain hydrocarbons were degraded into simpler and shorter molecules throughout the degradation process. The use of a complex hydrocarbon mixture as the exclusive carbon source resulted in the formation and accumulation of by-products during the 60-day degradation process, leading to the emergence of multiple additional compounds.

DISCUSSION

Isolation of Oleophilic Bacteria from Environmental Samples

In the present study, 30 different hydrocarbon-contaminated soil samples were collected from ten different automobile servicing stations within Mokokchung town of Nagaland, India. A total of sixty hydrocarbonoclastic bacterial isolates were obtained from the oil-contaminated soil samples at the end of the enrichment culture method. In accordance with the present investigation, Bekele et al. (2022) isolated two potent diesel-degrading strains *of P. aeruginosa* (AAUW23 and AAUG11) and one strain of *B. subtilis* (AAUG36) using the enrichment culture technique. Using an enrichment culture technique and burnt motor oil and diesel as the only sources of carbon, Hossain et al. (2022) used an oil sample collected from a petrol pump disposal site. They identified three distinct bacterial isolates closely linked to *Enterobacter* sp., *Pseudomonas* sp., and *Acinetobacter* sp. During isolation and screening, the enrichment culture approach promotes the proliferation of specific microorganisms with the characteristics of interest and increases the number of such target species. The selection of species unique to the metabolism of specific compounds using a certain carbon and/or energy source is one of the simplest methods for isolating novel species (Bhatt et al. 2023). Rahman et al. (2002a) documented a crude oil-metabolizing *Pseudomonas* sp. DS10-129 was isolated from soil samples obtained from gasoline and diesel stations. Rahman et al. (2002b) successfully obtained 130 bacterial isolates capable of degrading oil using the enrichment culture approach, out of which two *Pseudomonas* sp. were identified as proficient in using crude oil and producing BS. Fleck et al. (2000) and Bharathi & Vasudevan (2001) used the enrichment culture approach to isolate several bacterial strains that have the ability to produce BSs and metabolize crude oil.

Culturing and Screening of BS-Producing Oleophilic Bacteria

The OB obtained from the enrichment culture approach was

further inoculated into MSM supplemented with n-hexadecane as the sole carbon source. This approach confirmed the hydrocarbonoclastic nature of the OB isolates. The same strategy was previously used by Bordoloi & Konwar (2008) and Bharali et al. (2022) to isolate potential hydrocarbon-utilizing bacterial strains. The selected hydrocarbonoclastic OB were screened for their BS production capacity using the *du Noüy* ring method, crude oil displacement test, drop collapse test, penetration assay, and CTAB agar assay. The du-Noüy-Ring technique is widely employed for screening BS-producing bacteria because of its simplicity and reliability. It measures the force required to detach a platinum ring from the interface or surface of a liquid (Walter et al. 2010). Cooper (1986) stated that a culture is considered potential if it can decrease the ST of a liquid medium to 40 mNm^{-1} or below. The extent of the clear zone due to oil displacement upon the addition of CFCS was directly related to the activity of the BS. Morikawa et al. (2000), Cheng et al. (2017), and El-Housseiny et al. (2020) have established that the oil spreading approach is a dependable method for quantifying the surface activity of BSs. Furthermore, they demonstrated that this methodology is highly sensitive and capable of detecting even small quantities of BSs.

When a surfactant is present, the liquid drop expands or even collapses owing to the reduction in the force or interfacial tension between the liquid drop and the hydrophobic surface (Jain et al. 1991). The integrity of the drops is contingent upon the quantity of surfactants and is correlated with the degree of tension on the surface and interface (Walter et al. 2010). The penetration assay is appropriate for high-throughput screening and is based on the interaction between two insoluble phases, leading to a color change (Maczek et al. 2007, Walter et al. 2010). BSs can induce rupture of red blood cells (Walter et al. 2010). The selected bacterial isolate exhibited β-hemolysis and was confirmed to be positive for BS production. Mulligan and Cooper (1984) suggested using the blood agar assay as an initial assessment technique, which should be supplemented by additional techniques that rely on the measurement of surface activity. The CTAB agar plate method is a convenient screening technique used to identify the presence of exogenous glycolipids and other anionic surfactants (Siegmund & Wagner 1991). When bacteria in the culture medium produce anionic surfactants, they react with the cationic surfactant CTAB and the methylene blue dye to produce a dark blue, insoluble ion pair. BS-producing bacterial colonies were encircled by dark blue halos (Mulligan et al. 1984, Tuleva et al. 2002, Walter et al. 2010).

Characterization of a Selected Bacterial Isolate

Clustal Omega alignment tests showed that the isolate AMS1a sequence was 100% similar to various 16S rRNA sequences across *Pseudomonas* spp., particularly *P. aeruginosa*. The results of this analysis indicated that all 16S rRNA sequences obtained from NCBI were closely related. Specifically, the AMS1a sequence exhibited considerable homology (> 97%) compared to other Pseudomonas sequences. Phylogenetic analysis revealed that the AMS1a sequence was more closely related to the *P. aeruginosa* DSM 50071 and ATCC 10145 sequences. *P aeruginosa* ATCC 10145 was selected as the root of the phylogenetic tree because it was found to be closely related to AMS1a in the alignment test conducted using Clustal Omega. *P. aeruginosa* is a prevalent Gram-negative bacterial species among hydrocarbonoclastic oleophilic prokaryotes. It is recognized for its versatile metabolism and capacity to inhabit numerous environments (Mielko et al. 2019). Various species of Pseudomonas, capable of generating BSs, have been reported from diverse polluted environments such as petroleum-hydrocarbon contaminated fields (Barathi & Vasudevan 2001, Bordoloi & Konwar 2008, Saravanan & Vijayakumar 2012), metal-contaminated sites (Santos et al. 2024), pesticide-affected regions (Wang et al. 2022), and industrial effluent-laden habitats (Al-Ansari et al. 2021, Wang et al. 2023).

Growth Kinetics and BS Production

The current study showed a roughly sigmoidal growth curve over a 22-day incubation period, which closely aligns with the previously documented growth curves of other *Pseudomonas* species (Janek et al. 2013, Goswami et al. 2015, Ramírez et al. 2015). In comparison to earlier published studies, the selected bacterial strain exhibited optimal growth at 30˚C. This may be attributable to the abiotic factors at the contaminated sampling site from which the bacterial strain was isolated. According to records, the temperature in the Mokokchung township region does not exceed 32ºC during summer, with an average summer temperature of 27ºC (Central Ground Water Board 2013). Santos et al. (2024) isolated a strain of *P. aeruginosa* BM02 from acidic soil in a Brazilian municipality with temperatures ranging from 28 ± 6ºC, which is very near the ideal temperature for BS synthesis. Wei et al. (2005) and Chen et al. (2007) reported optimum RL-production by *P. aeruginosa* J4 at 30-37ºC, which decreased with further increase in temperature. Temperatures between 28-40ºC have been reported for the production of RLs by various strains of *P. aeruginosa* (Henkel et al. 2012, Müller et al. 2012). Such discrepancies in the reported optimum temperature for *P. aeruginosa* strains indicate their apparent physiological variations.

The bacterial strain exhibited progressive development from 1st to 14th day, persisted throughout the fermentation process, and entered the death phase after 14th day. It has

been proposed that cells enter the death phase due to the consumption of available nutrients in the culture medium, resulting in the accumulation of toxic substances and a restricted supply of dissolved oxygen, which may impede development (Lan et al. 2015). A spike in BS biosynthesis was observed even after the bacterial population entered the stationary phase. This could be attributed to the biosynthesis of BSs as secondary metabolites (Rahman et al. 2002a). The growth of hydrocarbonoclastic oleophilic can be accompanied by the production of BSs, which can aid in the attachment of cells to hydrophobic substrate molecules and facilitate their metabolism (Barathi et al. 2001). ST measurements in our experiments showed a decrease, indicating the presence of surface-active compounds such as RLs. Multiple studies have demonstrated that some bacteria can decrease the ST of their culture media by producing BSs during the shift from the exponential to the stationary growth phase (Yin et al. 2009, Safari et al. 2023).

Purification of Partially Purified BS

Previous studies have reported that different strains produce RLs with different compositions (Abdel-Mawgoud et al. 2010). The spot with a higher R_f value contains the mono-RLs, whereas the lower R_f value contains the di-RLs (Lotfabad et al. 2010). Moreover, in this study, a minor lower spot with an R_f value of 0.3 was also observed. A similar observation was reported by Lotfabad et al. (2010), where a lower R_f value of 0.31 referred to the di-RL structure, while the higher spot for mono-RLs had an R_f value of 0.76. Cheng et al. (2017) characterized the BS produced by *P. aeruginosa* ZS1 isolated from petroleum-sludge in Zhoushan islands, China, through the TLC technique. They were able to separate mono-RL, Rhamnose-C_{10}-C_{10}, di-RL, and Rhamnose-Rhamnose-C_{10}-C_{10} homologues from an RL mixture. Present findings are consistent with those of other studies by Abdel-Mawgoud et al. (2008), George and Jayachandran (2009), and Lotfabad et al. (2009). Abdel-Mawgood et al. (2008) obtained a lower spot (di-RL) and a higher spot (mono-RL) with respective R_f values of 0.4 and 0.68. El Housseiny et al. (2020) characterized the BS from *P. aeruginosa* P6 using TLC analysis and showed two spots, one major spot with a R_f value of 0.56 and another minor spot with a R_f value of 0.71, which corresponded to mono-RLs and di-RLs, respectively. Both mono-RLs and di-RLs are the two main types of RLs produced by most *P. aeruginosa* species (Lang et al. 1999, Maier & Soberon-Chavez 2000). Column chromatography was performed to separate the individual RL homologs using a chloroform-methanol solvent system at different ratios. Many compounds can be efficiently purified, and their structural and functional analyses can be performed using this chromatography technology. Perfect outcomes are obtained in the identification and isolation of RL congers using this approach (Sim et al. 1997). Column chromatography uses a cheap, disposable stationary phase that can be disposed of after use to avoid deterioration and cross-contamination. Additionally, recovering the mobile phase for later use is much simpler.

Physical Characterization of Isolated BS

Determination of CMC: According to the findings, the CMC of the extracted BS was measured to be 195.6 mgL^{-1}, which is equivalent to a ST of 34.4 mNm^{-1}. The CMC of RLs is significantly lower than that of synthetic surfactants, such as SDS, which has a CMC of 2200 mgL^{-1} (Khademolhosseini et al. 2019). The CMC values of mono- and di-RLs, as well as their combinations, produced by different techniques, have been reported to vary between 1 and 400 mgL^{-1} (Kopalle et al. 2022, Arkhipov et al. 2023). Higher CMC values indicate that a greater quantity of surfactant is required to reduce the ST. Therefore, a smaller quantity of BSs is required to achieve maximal ST reduction. This is an important aspect that contributes to the greater utility of BSs compared to chemical surfactants. According to Manivasgan et al. (2014), a fine-quality BS can decrease the ST of water from 72.75 mNm^{-1} to 35 mNm^{-1}. According to Zhang and Miller (1992), effective BSs can reduce the ST of water to less than 40 mNm^{-1}.

Moreover, variations in the purity and content of the BSs may have contributed to the disparate CMC values. For RLs derived from different microbial sources, CMC values ranging from 10 to 230 mgL^{-1} have been reported (Zhang & Miller 1992, Nitschke et al. 2005, Abdel-Mawgoud et al. 2010). It is well known that the distribution of homologs affects the characteristics of RLs. In general, the CMC tends to decrease as the length of the surfactant chain increases and the di-RL content increases (Perfumo et al. 2006). However, this trend was not observed in the current study. A possible explanation for this phenomenon is that the BSs examined in this study are not composed of one primary congener but rather contain an assortment of congeners with varying chain lengths in addition to the predicted congeners with a certain chain length. Additionally, the LC-MS results for the extracted BS indicated the presence of a combination of di-RL and mono-RL congeners, which may potentially impact its CMC values.

Emulsification activity: Emulsification activity is regarded as a critical parameter among the physicochemical qualities used to assess the commercial usability of BSs (Mendes et al. 2015). This is regarded as an indirect approach to evaluate BS production. The findings demonstrated a varied emulsifying capacity against a wide range of hydrocarbon

oils, reflecting the capability of the selected bacterial strain to produce BSs that improve the interaction of hydrophobic substances with water. According to Patel & Desai (1997) and Lovaglio et al. (2011), RLs have a great ability to emulsify a wide range of n-alkanes, aromatic hydrocarbons, petroleum-derived compounds, crude oil, and vegetable oils. According to an investigation conducted by Sun et al. (2018), *Pseudomonas* sp. CQ2 from the Changqing oil field, China, produces a BS with an $E_{24\%}$ of up to 61.5%. As reported by Kumari et al. (2012), the BS generated by *Pseudomonas* sp. BP10 exhibited a high emulsification activity of up to 75%. Hydrocarbons are pseudo-solubilized or emulsified at varying rates by the majority of microbial surfactants, which are substrate-specific (Etoumi et al. 2008). Several studies have found emulsifying indices ranging from 50% to 75% for various RL mixtures (Wei et al. 2005, Abdel-Mawgoud et al. 2008, Benincasa & Accorsini 2008, George et al. 2009, Lotfabad et al. 2009, Bharali et al. 2011, Abbasi et al. 2012, Aparna et al. 2012). The resulting water-oil (WO) emulsions were dense and persisted for over four weeks at room temperature, indicating the potential use of BS in bioremediation to improve the bioavailability of intractable hydrocarbons. In addition, as noted by Maier & Soberon-Chavez (2000) and Abdel-Mawgoud et al. (2008), the ability of BS to emulsify fossil fuel products, particularly kerosene, n-hexadecane, octadecane, diesel, and lubricating oil, may facilitate their microbial assimilation.

Chemical Characterization of Isolated BS

FTIR

The recovered BS was analyzed using FTIR to determine its functional groups. The FTIR investigation identified significant absorption bands in the tested BS at certain wavenumbers *i.e*. 3410, 2931, 2856, 1735, 1644, 1402, and 1206 cm^{-1}. The results provide compelling evidence that the detected wavenumbers align with the various functional categories of RL, as reported in the literature (Cortés-Sánchez et al. 2013, Araujo et al. 2020). Lan et al. (2015) and Ibrahim (2018) reported that the aforementioned primary chemical structure groups align with the structural features of RL, as determined by Fourier transform infrared (FTIR) analysis. Wavenumbers below 1200 cm^{-1} encompass many types of C–H, C–O, and $–CH_3$ vibrations that cannot be further classified (Zhao et al. 2013). It is also crucial to note that slight differences were observed in the infrared spectra compared to the previously published spectra. This variation may be ascribed to the specific constitution of RL mixtures obtained from various strains of *Pseudomonas* sp. and differences in the culture and purification conditions employed.

NMR

The signals identified in the ^{1}H and ^{13}C NMR spectra corresponded to the functional groups of RL, as described in the literature (Cortés-Sánchez et al. 2013, Araujo et al. 2020). Therefore, according to the explanations obtained through NMR studies, it can be concluded that BS is composed of RL. The inadequate clarity of the NMR spectra suggests that the substance being studied may include a combination of several RL congeners. The current results are consistent with those of Monteiro et al. (2007) and Wei et al. (2005), who used ^{1}H and ^{13}C NMR analyses to determine the chemical composition of a BS.

LC-MS/MS

The mass spectrometry data indicated the presence of both mono- and di-rhamnose groups, as well as mono- and di-lipid groups. There was a significant variation in the length and composition (saturated or unsaturated) of the lipid chains, with a particular focus on the presence of saturated C_{10} and C_{12} fatty acids in mono-RL and saturated C_8, C_{10}, C_{12}, and C_{14} fatty acids in di-RL. Previous investigations have reported that the most prevalent *P. aeruginosa* RL comprises fatty acids ranging from C_8 to C_{14}, with C_{10} being the most common. In their study, Christova et al. (2011) identified mono- and di-RL congeners as anions with m/z values of 503 and 649, respectively. These values corresponded to the deprotonated molecules of Rha-C_{10}-C_{10} (mono-RL) and Rha_2-C_{10}-C_{10} (di-RL), respectively. Déziel et al. (1999) and (2000) observed that the ions with the greatest abundance were found at m/z 649.9 and 503.6, respectively. These ions were identified as (Rha-C_{10}-C_{10}) and (Rha-C_{10}-C_{10}) congeners, respectively. It is important to note that there was no estimation of each congener; thus, it is possible that some of them, particularly the less frequent ones, might also be present in minimal proportions. The NMR investigation did not reveal the existence of unsaturated chain congeners, regardless of the weak signals in the 7.0–8.2 ppm region. However, these congeners were identified using mass spectrometry.

Stability Studies

Effect of Environmental Variables on E_{24}%

The efficacy of BS relies on its capacity to maintain stability in diverse environments, accounting for fluctuations in pH, temperature, and salinity (Gudina et al. 2010). The current study assessed the strength of the obtained RL by exposing the CFCS to severe environmental variables such as pH, temperature, salt, and heavy metals. BS demonstrated stability throughout all evaluated variables, with no precipitate formation or significant decline in surface-active behavior. The findings indicated that the BS being tested

exhibited robustness, as it was able to continue operating and retain its active property within a wide temperature range of 4–121˚C. Moreover, there was minimal variation in the $E_{24\%}$ when subjected to varying temperatures, with overall values ranging between 50% and 65.6%. In a similar study, Santos et al. (2024) examined the effects of RL isolated from *P. aeruginosa* BM02 on its ability to endure a wide range of temperatures. The results showed that RL can thrive even at extreme temperatures, ranging from 40 to 120˚C, with minimal variations in its $E_{24\%}$ value, which remained between 65% and 71%. RLs undergo precipitation at low temperatures (4˚C), which adversely affects their surface activity (El-Housseiny et al. 2020). Following autoclaving, there was a minimal reduction in the surface activity. We also examined the impact of elevated temperatures on the emulsifying activity of CFS and found that autoclaving led to a marginal decrease in the emulsification index (El-Housseiny et al. 2020). The proficient temperature stability and strong emulsifying activity of the investigated RLs render them suitable for implementing Microbial Enhanced Oil Recovery (MEOR) and bioremediation in oil-contaminated areas characterized by elevated temperatures (Zhou et al. 2019).

RL in its crude form (CFS) eliminates the need for expensive extraction procedures. Furthermore, employing unrefined RLs is advantageous as it will reduce expenses typically given away to the steps of separation and purification processes (El-Housseiny et al. 2020).

The optimal surface activity was achieved within a pH range of 6–8. However, a substantial decrease in surface activity was observed under extremely acidic (pH 3) or strongly alkaline (pH ≥ 8) conditions. This phenomenon arises due to the precipitation of RLs in highly acidic conditions, resulting in structural distortion and a diminished capacity to decrease ST. Under high acidic settings, the groups with negative charges at the polar ends of RL molecules undergo protonation (El-Housseiny et al. 2020).

The impact of ionic concentration on the surface-active properties was also examined, revealing that the surface activity was markedly diminished at NaCl concentrations exceeding 9% w/v. Reddy et al. (2016) conducted a study to examine the impact of different levels of salinity (2, 6, 10, 15, and 20% w/v) on the stability of RL BSs generated by *P. aeruginosa* DR1, utilizing mango kernel oil. The ST exhibited a progressive increase from 30 to 38 mNm^{-1} when the salt content increased from 6 to 20%. However, salt concentrations of 2-3% render commercial surfactants ineffective (Abdel-Mawgoud et al. 2008). Thus, the obtained RLs have demonstrated their superiority as a preferred option for bioremediation processes in high-salinity areas, such as polluted coastal environments.

Consistent with the present investigation, Santos et al. (2024) found that BS derived from *P. aeruginosa* BM02 remained stable under all tested conditions. The lowest ST value (~27 mNm^{-1}) was observed at pH 3.0, 2% NaCl concentration, and 80˚C. Joshi et al. (2008) found that the stability of the BS was maintained at a temperature of 80˚C, and within pH and NaCl ranges of 6 to 12 and 1 to 7%, respectively. However, the surface activity of BS declined as the NaCl concentration increased to 10 percent and the pH level decreased to the acidic range, resulting in the formation of precipitates. Khademolhosseini et al. (2019) conducted a study on a RL BS produced by *P. aeruginosa* HAK01. They revealed that the BS possessed excellent stability under various conditions, including temperatures ranging from 40 to 121˚C, pH values between 3 and 10, and salt levels of up to 10% (w/v) NaCl. Based on their findings, they proposed that BSs might be utilized in Microbial Enhanced Oil Recovery (MEOR) applications. Multiple researchers have reported similar findings, demonstrating that the activity of RLs remains consistent throughout a broad spectrum of temperature, salinity, and pH levels (Abdel-Mawgoud et al. 2008, El-Housseiny et al. 2020, Zhou et al. 2019, Zhao et al. 2018).

Effect of Environmental Variables on Emulsification Activity

The excellent emulsification activity demonstrated by RL against different hydrocarbon oils credits it as a powerful emulsifying agent that can be potentially used in environmental applications such as MEOR and oil mobilizing agents (El-Housseiny et al. 2020). Furthermore, the bioremediation of petroleum-contaminated settings may benefit from the capacity of BSs to emulsify various hydrocarbon oils, as this may facilitate the assimilation of such oils by BS-producing microorganisms (Maier & Soberon-Chavez 2000, Abdel-Mawgoud et al. 2008). The stability and ability of the BS to retain its properties can be determined from emulsification and surface activity stability tests against varying environmental factors, which underpin their potential bioremediation applications (Chandankere et al. 2014, Abdel-Mawgoud et al. 2008, Datta et al. 2018). It was noteworthy that it performed remarkably well even after autoclaving at 121ºC for 10 min (Abdel-Mawgoud et al. 2008). The CFCS exhibited stable and consistent emulsification activity with diesel oil for 30 days. A stable oil-water emulsion is typically used as a surface activity indicator (Hao et al. 2008). The stability of the emulsification activity of the CFCS containing BSs was also reviewed against varying temperatures, pH, and salinity. The results of the stability tests underscored the ability and efficacy of the BS under study to form stable emulsions with numerous oils over varying temperatures (5-80ºC), pH

(3-10), salinity (2-10%), and time (1-30 days). The stable water-oil emulsions also indicate the potential use of RL in the bioremediation process to improve the accessibility of resistant hydrocarbons.

Bioremediation Application

Soil washing experiment: The percentage of crude oil removed from the sand by the CFCS was relatively higher than that removed by SDS. Similar findings regarding the efficient crude oil removal capacity of RLs produced by *P. aeruginosa* have been reported in a previous study by Aparna et al. (2012). This shows that the BS from *P. aeruginosa* is highly effective for crude oil removal and can be an efficient alternative for the bioremediation of hydrocarbon oil contamination. The removal of oil from soil using BSs can be explained by two proposed mechanisms: mobilization and solubilization. Mobilization occurs when the surfactant concentration is below the CMC. In this phase, the capillary forces, surface and interfacial tension, contact angle, and wettability were reduced. Below the CMC, surfactants lower the surface and interfacial tensions between water, oil, air, and soil systems. When surfactants interact with the oil-soil system, they increase the contact angle and reduce the capillary forces that hold the oil to the soil by decreasing interfacial tension. The mobilization step also depends on the ionic charge of the surfactant, as surfactants are adsorbed onto the soil. This adsorption can lead to a reduction in surfactant concentration, making it less effective or even ineffective in soil treatment. When the concentration exceeds the CMC, the oil solubility increases significantly because of the formation of surfactant micelles. These micelles have hydrophobic ends that cluster inside and hydrophilic ends that face water. This structure provides a suitable environment for hydrophobic organic molecules, a process referred to as solubilization (Urum et al. 2004). Thus, it can be concluded that the main mechanism involved in the removal of crude oil by RL in the current study was greatly attributed to the mobilization phenomenon, as there was no substantial increase in the oil removal capacity at concentrations above the CMC. Gaur et al. (2021) demonstrated the excellent oil recovery capacity of BS produced by *P. aeruginosa* from soil contaminated with engine oil through centrifugation at 6000 rpm for 20 min. Bharali et al. (2022) also conducted a study on the efficiency of BSs produced by different strains of *P. aeruginosa* to recover residual crude oil from petroleum sludge and reported that the BSs were able to recover about 73.5–63.4% of residual oil from the sludge.

Crude oil degradation: The oil degradation potential of the selected bacterium was confirmed by GC-MS analysis. Selected complex and long-chain hydrocarbons are broken down into simpler and shorter compounds during degradation. As a result of the utilization of the complex mixture of hydrocarbons as the sole carbon source, the generation of bacterial by-products was observed during the entire degradation process of 60 days, leading to the formation of new major compounds such as erucic acid, cis-10-Nonadecenoic acid, 5,8,11,14-Eicosatetraynoic acid, methyl ester, 9-Hexadecenoic acid, and eicosyl ester. Additionally, the degradation of large hydrocarbon molecules leads to their breakage and the generation of several smaller molecules, which further serve as precursors for the formation of other complex and stable compounds. It is also evident from the GC-MS data that the bacterium was unable to breakdown the complex and highly branched hydrocarbons such as Pentadecane, 2,6,10-trimethyl, Heptadecane, 2,6,10,14-tetramethyl, Tetradecane, 2,6,10-trimethyl-, Heptadecane, 2,6,10,15-tetramethyl, Dodecane, 2,6,10-trimethyl, Octadecane, 3-ethyl-5-(2-ethylbutyl), Heptadecane, 9-hexyl, Tricyclo[7.4.1.1(2,7)] pentadeca-2,4,6,9,11,13-hexaene-8-ol, 10-Acetoxy-2-hydroxy- 1,2,6a,6b,9,9,12a-heptamethyl-1,3,4,5,6 etc. even after a period of 60 days. Previous studies have established that every bacterial strain has its limitations, and it can degrade only a selected number of hydrocarbons (Rehman et al. 2021). This restriction is the result of several reasons. Primarily, crude oil is composed of a wide variety of hydrocarbons with unique chemical structures and traits, making its composition extremely complicated. Second, nutrient availability and environmental variables can influence the efficacy of bacterial decomposition. For instance, some hydrocarbons may be more susceptible to bacterial enzymes, whereas others may exhibit greater resistance to breakdown owing to their chemical characteristics or spatial configuration within the oil matrix. Moreover, toxic chemicals in crude oil, including heavy metals and aromatic and polyaromatic compounds, can impede bacterial growth and catalytic activity, thereby restricting their capacity to completely break down the oil (Chuah et al. 2023). Hence, for the effective degradation of crude oil components, it is preferred to use a consortium of bacteria with different degradation potentials (Primeia et al. 2020).

Limitations

The constraint arises from the multitude of factors and their interconnectedness that define the production of microbial surfactants. The inability to identify the most economical conditions for producing BS in bioreactor systems for BS commercialization is one of the main limitations of the present study. In addition to the microbial producer, the fermentation and purification processes impact the physicochemical properties of the BS. Comprehending these attributes is necessary for accurately determining

their industrial use. Further industrial applications of this strain, such as the synthesis of enzymes and other bioactive compounds, may be expanded by thorough molecular investigation.

Additionally, this study did not assess the specific uses of the isolated BS other than bioremediation. To determine the precise biodegradation capacity of the chosen strain, a comprehensive molecular examination of its hydrocarbonoclastic characteristics is essential. Additionally, the strain was initially cultured in crude oil to verify its ability to utilize the different components of crude oil as the sole source of carbon and energy. However, studies on its innate ability to use and break down certain components of crude oil, such as aliphatic, aromatic, polyaromatic, and NSO-containing compounds, have yet to be conducted. All of these flaws would be seen as opportunities for further research on the topic in the future.

CONCLUSIONS

The current study revealed the isolation, characterization, and identification of *P. aeruginosa* AMS1a strain, a potential hydrocarbonoclastic, oleophilic, and BS-producing strain from Mokokchung, Nagaland, India. The strain was discovered outside an automobile repair shop in polluted soil and demonstrated a great capacity to degrade crude oil. When the BS production capability was assessed, the strain was found to produce BS that significantly reduced ST and exhibited effective emulsifying properties. Structural characterization using TLC, FTIR, ^{1}H and ^{13}C NMR, and LC-MS/MS verified that the BS generated by the selected strain comprised a combination of mono- and di-RLs. BS demonstrates significant surface-active properties and stability throughout a broad spectrum of temperature, pH, salinity, and heavy metals, rendering them appropriate for many commercial applications, including the pharmaceutical, cosmetics, food, and remediation technology sectors. Furthermore, the strain has the potential to be employed in bioremediation processes, which involve the biodegradation of petroleum-based pollutants and the removal of crude oil from contaminated soils by washing. However, given the costs associated with extraction and purification, the yield is rather low, which essentially prevents its industrial application. Further research and development are required to improve the culture conditions and increase the quality, yield, and efficiency of BS, which will increase the number of potential applications. To increase BS production on a wide scale, cutting-edge extraction techniques that use affordable, renewable carbon feedstocks must be investigated. Through further investigation into the optimization of growth parameters and exploration of novel genetically engineered microbes, researchers can fully realize the potential of BS, which will open up novel applications in disciplines such as biomedicine, environmentally benign remediation technology, organic compound synthesis, and nanotechnology. Additionally, to promote sustainability, various government organizations should implement policies to promote and encourage the use of greener and more environmentally friendly natural products, such as BS, to achieve the Sustainable Development Goals set by the UN.

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The Evaluation of Properties of Chars Produced from Wastes of Different Origins

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ABSTRACT

This paper presents a screening of primary char samples produced through the hydrothermal liquefaction of biomass. Currently, numerous studies demonstrate the potential of hydrothermal conversion char residues as sorbents. The relevance of this work lies in utilizing materials that are rarely used in other areas of production, such as aquatic vegetation, organic waste, algae, bark and wood waste, food waste, and waste from the agro-industrial complex. This study aims to evaluate the sorption capacity (for iodine, phenol, and methylene blue) of char sorbents obtained from various raw materials via hydrothermal liquefaction and to identify the most suitable raw materials. To assess the charcoals, their elemental composition and sorption capacities for methylene blue, iodine, and phenol were analyzed. The average yield of charcoal was 35%. The results indicate that the obtained chars demonstrated the highest sorption capacity for methylene blue (up to 239 $mg.g^{-1}$), while phenol sorption was the lowest (not exceeding 19 $mg.g^{-1}$). These findings suggest that the produced chars are promising raw materials for the production of sorbents.

INTRODUCTION

Pollution is a longstanding global environmental challenge, primarily driven by anthropogenic activities. This study explores one of the methods to mitigate anthropogenic impacts – wastewater treatment with sorbents.

In recent years, sorbents have gained increasing popularity for wastewater treatment due to their ease of use and regenerability (Fouda-Mbanga et al. 2024). Samodolova et al. (2024) employed a charcoal sorbent obtained by pyrolysis of walnut shells to treat wastewater contaminated with heavy metal ions. The authors investigated the dynamic sorption of aluminum, chromium, copper, iron, magnesium, and zinc ions. Their results demonstrated that the sorbents effectively removed chromium and copper ions from the solution, regardless of the process temperature.

Kaewtrakulchai et al. (2024) used carbon sorbents obtained through the hydrothermal conversion of shrimp processing waste to treat wastewater contaminated with antibiotics (oxytetracycline). Their experimental data demonstrated a 100% purification efficiency of the antibiotic solution when a sorbent concentration of 2 $g.L^{-1}$ was applied for a sorption period of 10 min (with an initial antibiotic concentration of 300 $mg.L^{-1}$).

Sorbents are also widely used in soil remediation. Wyszkowska et al. (2023) conducted soil remediation experiments on diesel fuel and petroleum ether contamination using biochar, halloysite, and alginite. All samples demonstrated potential for soil remediation, with biochar showing the highest efficiency.

Although sorbents are effective in wastewater and soil treatment, they remain an expensive product. Andriyko et al. (2024) calculated the cost of bentonite-carbon

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sorbent, which was produced using lignite, bentonite and a binder. The cost of raw materials alone amounted to $78 per ton of sorbent. Even without considering the cost of the equipment, it is evident that sorbents are costly.

Efficient and relatively inexpensive sorbents can be obtained through the pyrolysis of dry biomass. Chen et al. (2024) conducted the pyrolysis of dried coffee grounds at 550°C for 1.5 h. Structure analysis using SEM revealed high porosity. The authors tested the sorption of heavy metal ions (Pb^{2+}, Ni^{2+}), achieving sorption capacities of 67.037 $mg.g^{-1}$ for lead and 10.265 $mg.g^{-1}$ for nickel.

Amosa (2015) carried out the pyrolysis of palm oil production waste at a temperature of 900 °C for 15 min. The resulting product exhibited a surface area of 886.2 $m^2.g^{-1}$, as measured by BET, and high porosity, confirmed by SEM. The sorbent demonstrated purification efficiencies of 90% for hydrogen sulfide and 95% for manganese ions.

Irfan et al. (2023) performed pyrolysis of 2 types of raw materials: wheat straw and excess sludge, as well as their mixture in the ratio of 1:1. The raw materials were dried, crushed, and sieved before undergoing pyrolysis at 550 °C for 3 h. At a sorbent dosage of 3 $g.L^{-1}$ and above, the phosphate removal rate reached 100%. The used sorbents were used as fertilizer for mustard plant cultivation.

Unfortunately, the use of pyrolysis for waste treatment is not always effective. In most studies, the wet raw material requires prolonged drying (several days) before pyrolysis. Sylwan & Thorin (2023) used pyrolysis char derived from excess sludge to treat wastewater contaminated with heavy metals; however, the char demonstrated poor performance.

There are several methods for producing char sorbents. Güleç et al. (2022) compared three potential methods of obtaining char sorbents: hydrothermal conversion, torrefaction, and pyrolysis. Hydrothermal conversion is conducted at temperatures ranging from 100 to 320°C, with char yields ranging from 0 to 100%. Torrefaction is performed at 200-300°C with 20-95% char yield, while pyrolysis is carried out at 300-570°C, producing a 15-65% char yield.

Hydrothermal liquefaction is a process of thermochemical conversion of biomass to produce synthetic bio-oil as well as biochar. This process takes place in an aqueous environment and can be enhanced with the use of catalysts. One advantage of this method is the ability to control process parameters, making it more versatile (Sharma et al. 2024).

The use of char residue from hydrothermal liquefaction as a sorbent is currently gaining significant attention. Lu et al. (2021) performed hydrothermal liquefaction of algae, demonstrating the potential of this method for biomass conversion. Aktas et al. (2024) produced coal from excess sludge through hydrothermal conversion. After activation and pelleting, the surface area increased to 791 $m^2.g^{-1}$, enhancing its sorption capacity.

Char sorbents produced by hydrothermal conversion have found widespread application in wastewater treatment. Yu et al. (2021) investigated the sorption of lead ions using a char sorbent derived from algae. The chars obtained were low-porous. FTIR showed the presence of active groups on the sorbent surface: C=O, C=C, and SiO. The degree of purification of the solution ranged from 20% to 100%, depending on the lead concentration. Marx & van der Merwe (2021) performed phenol sorption experiments, reporting extraction efficiencies ranging from 5% to 14%, depending on the sorbent dosage. Unactivated chars were characterized by poorly developed BET surface area: mesopores 1.54 $m^2.g^{-1}$, micropores 24.78 $m^2.g^{-1}$.

Kulikova et al. (2024) compared the life cycle impacts of algae processing using pyrolysis and hydrothermal liquefaction methods. A comparison of the impact of the methods on climate change showed that using wind power for hydrothermal conversion will reduce the environmental load compared to pyrolysis, highlighting the ecological benefits of hydrothermal processing.

Summarising the studied material, we came to some conclusions:

1) Most often, cellulose-containing raw materials or other structural polysaccharides are used for the production of chars, regardless of the method of their production. Less often, a mixture of organic wastes with cellulose-containing raw materials is used. This is due to the prospects of such raw materials for obtaining char sorbents, with high carbon content, and porosity of the initial material.

2) Chars obtained by pyrolysis are characterized by high porosity. Both SEM and BET analyses show this. For example, Amosa (2015) obtained chars that exhibited a surface area of 886.2 $m^2.g^{-1}$, as measured by BET. At the same time, coals obtained by HTL exhibit low porosity. Marx and van der Merwe (2021) obtained chars characterized by a low BET surface area: mesopores 1.54 $m^2.g^{-1}$, micropores 24.78 $m^2.g^{-1}$. The surface of such chars is characterized by the presence of active centers (C=O, C=C, SiO groups). This proves chemisorption as the main sorption mechanism of such coals.

3) Hydrothermal conversion is a cheap, ecologically favourable and promising method of producing coal sorbents from various types of raw materials.

Many studies have demonstrated that hydrothermal conversion is a universal method for producing char sorbents, making it suitable for various raw materials, including wet biomass. However, the question remains as to which raw materials are most effective for the production of sorbents by hydrothermal conversion.

This study aims to evaluate the sorption capacity (for iodine, phenol, and methylene blue) of char sorbents obtained from various raw materials by hydrothermal liquefaction and to identify the most suitable raw materials.

MATERIALS AND METHODS

The experiment was performed 3 times. The error of the experiment should not exceed 10%. For each sample, the chars were produced three times. A series of experiments was performed in each of the 3 chars. This allowed levelling out the influence of uncontrollable factors that could occur in the process of char production.

Materials

Dichloromethane, phenol, iodine, methylene blue, potassium bromate, potassium bromide, hydrochloric acid, potassium iodide, and sodium thiosulfate were of ACS purity grade and purchased from Diaem (Moscow, Russia). Water was distilled on an evaporative distiller immediately before the experiments.

Objects of Research

The objects of research include raw materials that are not commonly utilized in traditional methods. All materials were categorized into the following groups:

1. Plant biomass: *Phragmites australis, Scirpus lacustris, Typha angustifolia,* and *Miscanthus*
2. Organic waste: Excess sludge
3. Baltic Sea algae: *Furcellaria*
4. Bark wood waste: Pine bark
5. Food waste: Apple waste and coffee waste
6. Agro-industrial complex waste: Cow manure and wheat straw

The plant biomass used in the study is widely distributed in the Kaliningrad region. Aquatic vegetation contributes to the purification of nutrients in water bodies, provided that it is collected in a timely manner (Kulikova et al. 2023). The organic composition of aquatic vegetation is rich in cellulose, hemicellulose, and pectin (Beyzi et al. 2023). The plant samples used in this study were collected from the Curonian Bay area in the Kaliningrad region, Russia. Fig. 1 presents the appearance of the plants used.

Countries near the Baltic Sea produce approximately 4.0 million tons of sewage sludge per year (dry matter) (Kacprzak et al. 2017). Most of this sludge is disposed of in landfills, reducing the amount of usable land (Xu et al. 2021). Excess sludge primarily consists of minerals (Mulopo 2024). In this study, sludge samples were collected from the water treatment plant in Sovetsk, Kaliningrad region, Russia. The sludge appeared as a black, wet substance (Fig. 1). For the experiment, the sludge was dried for 24 h at 80°C.

Furcellaria is a red algae found in the Baltic Sea. From late fall to early spring, biomass is deposited onto the shore, creating an unfavorable appearance on the beaches. Along with the algae, oil and plastic contaminants that accumulate in the algae thickets are also washed ashore (Gorbunova & Esyukova 2020). *Furcellaria* contains alginates, fucoidan, laminarin, and other compounds (Olsson et al. 2020). In this study, the algae were collected from the shores of the Baltic Sea (Kaliningrad region, Russia). *Furcellaria* has a branched structure and is black in color (Fig. 1).

Bark waste is a by-product of wood product manufacturing and is most frequently burned for energy production (Cesprini et al. 2020). Bark waste is rich in lignin, hemicellulose, and cellulose (Alonso-Esteban et al. 2022). In this study, common pine bark collected from the Kaliningrad region of Russia was used. The bark samples ranged in size from 2 cm^2 to 10 cm^2 and were brown in color (Fig. 1).

Food waste, a by-product of the food industry, was also used in this study. Coffee waste was obtained from the operation of a coffee machine, while apple waste was obtained in the production of apple juice. The main components of apple waste are fiber and pectin (Li et al. 2024). Local apples from the Kaliningrad region were used. The food waste samples were dried before the experiments (Fig. 1). The food waste was dried in a drying oven at 70-80 degrees centigrade for several hours until free moisture was removed.

Among agro-industrial wastes, cow manure (classified as IV type of hazard) and wheat straw (classified as V type of hazard) were used in this study. Wheat straw primarily contains cellulose and lignin (Yuan et al. 2024). Both straw and manure samples were collected from farms in the Kaliningrad region. Fig. 1 demonstrates the appearance of the raw materials.

Determination of Raw Material and Char Compositions

Ash content was determined according to GOST 26226-95: "Fodder, mixed fodder, mixed fodder raw materials. Methods of determination of crude ash" and GOST R 55661-2013: "Solid mineral fuel, Ash content determination".

Water content was determined according to GOST R 57059-2016 "Fodder, mixed fodder, mixed

Fig. 1: Objects of the research.

fodder raw materials. Express method of moisture determination".

Carbon content was determined using a Vario EL Cube elemental analyzer (Elementar Analysensysteme GmbH, Langenselbold, Germany). The CHNS analysis was based on the area of the chromatographic peaks of N_2, CO_2, H_2O, and SO_2.

Char Production

To produce chars, the raw materials were dried to adjust the hydromodulus. Plant-based raw materials were additionally cut into 3-5 cm long sticks.

The prepared raw materials were loaded into a hydrothermal liquefaction reactor, with adjustable parameters for temperature and speed of rotation of the magnetic stirrer (Fig. 2). An anchor and distilled water were added. The hydromodule was 1:10. The process of hydrothermal liquefaction was conducted under the following conditions:

- Temperature**:** 260°C

Fig. 2: Reactor for hydrothermal liquefaction.

- Pressure**:** 5–6 MPa
- Duration**:** 20 min

After the hydrothermal liquefaction process, the reactor was cooled. The contents were filtered, and the reactor vessel and solid residue were washed with dichloromethane to ensure complete extraction of the oil. The char was then dried at 104°C for 2 h.

Evaluation of Sorption Capacity

Sorption capacity was determined using methylene blue, following GOST 4453-74: "*Active Absorbing Powder Charcoal*. *Specifications*". The iodine sorption capacity was measured by calculating the difference in iodine concentration in the solution before and after sorption (30 min) (Evsina 2012). The phenol sorption capacity was evaluated by measuring the difference in phenol concentration after 5 h of sorption (Tukhvatullina et al. 2015).

Sorption of the above compounds is a standard method for evaluating sorbent properties. Sorption of iodine shows the adsorption capacity of the char. Sorption of methylene blue is characterised by the presence of mesopores on the surface of the char. Sorption of these compounds is an indirect indicator of porosity. Phenol sorption is used to evaluate the efficacy against organic compounds.

RESULTS AND DISCUSSION

Elemental Composition of Raw Materials

For the initial evaluation of the selected material, the elemental composition was analyzed. Since the target product of this study is char sorbents, the key element of interest

Table 1: Elemental composition of raw materials (dried).

Sample	Ash, %	Carbon, %	Hydrogen, %	Nitrogen, %	Sulphur, %	Oxygen, %	Moisture content, %
Phragmites australis	4.68±0.40	44.75±0.10	6.897±0.080	0.57±0.02	0.100±0.006	38.79±1.10	4.21±0.10
Scirpus lacustris L.	5.45±0.50	42.89±0.07	6.896±0.097	0.73±0.00	0.357±0.000	37.08±1.40	6.60±0.10
Typha angustifolia L.	5.18±0.50	43.93±0.06	6.757±0.085	0.80±0.03	0.221±0.007	37.21±2.10	5.90±0.10
Miscanthus	8.87±0.30	43.27±0.22	6.620±0.017	0.42±0.01	0.070±0.000	36.85±0.90	3.90±0.10
Sewage sludge	29.51±0.07	31.97±0.20	6.340±0.011	5.60±0.07	0.954±0.002	12.13±2.30	13.50±0.10
Furcellaria	19.51±0.01	33.90±0.16	6.277±0.000	4.77±0.01	3.631±0.001	24.81±0.60	7.10±0.10
Pine bark	2.43±0.20	52.08±0.09	7.133±0.011	0.33±0.07	0.073±0.008	30.65±0.70	7.30±0.10
Coffee waste	1.66±0.10	50.06±0.05	8.509±0.007	2.22±0.05	0.195±0.008	30.86±1.50	6.50±0.10
Apple waste	1.43±0.01	40.78±0.09	7.921±00.46	0.33±0.00	0.051±0.000	43.89±1.60	5.60±0.10
Cow manure	12.1±0.16	43.51±0.05	6.459±0.080	1.66±0.04	0.211±0.007	31.36±2.00	4.70±0.10
Wheat straw	8.52±0.10	44.2±0.33	6.112±0.070	0.79±0.07	0.019±0.016	34.76±±0.69	5.60±0.10

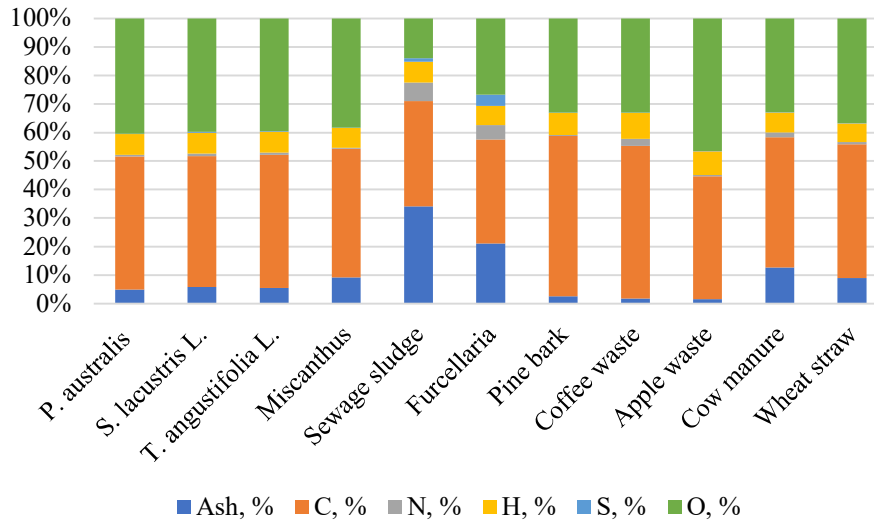

Fig. 3: Elemental composition of raw materials.

Table 2: Hydrochar yield.

Raw materials	Yield, %
Phragmites australis	39±2
Scirpus lacustris L.	31±1
Typha angustifolia L.	42±3
Miscanthus	44±2
Sewage sludge	32±2
Furcellaria	35±3
Pine bark	59±3
Coffee waste	36±2
Apple waste	31±1
Cow manure	44±2
Wheat straw	32±4

for the study is carbon. At high ash content, char has poor sorption properties (Nature Technology 2022); therefore, the ash content of the selected materials should be low ash content. Table 1 summarizes the elemental composition of the dried raw materials.

For visual clarity, the elemental composition is presented in Fig. 3. Elemental composition of raw materials showed that the most promising raw materials for obtaining char sorbents are food waste, bark waste and aquatic vegetation. The highest carbon content was observed in pine bark (52%), making it the most suitable raw material for sorbent production.

Characterization of Produced Chars

An important economic parameter in char production is the product yield. Table 2 shows the yield of chars obtained from different raw materials through hydrothermal liquefaction.

The average char yield across all raw materials was approximately 38%. The highest char yield was obtained

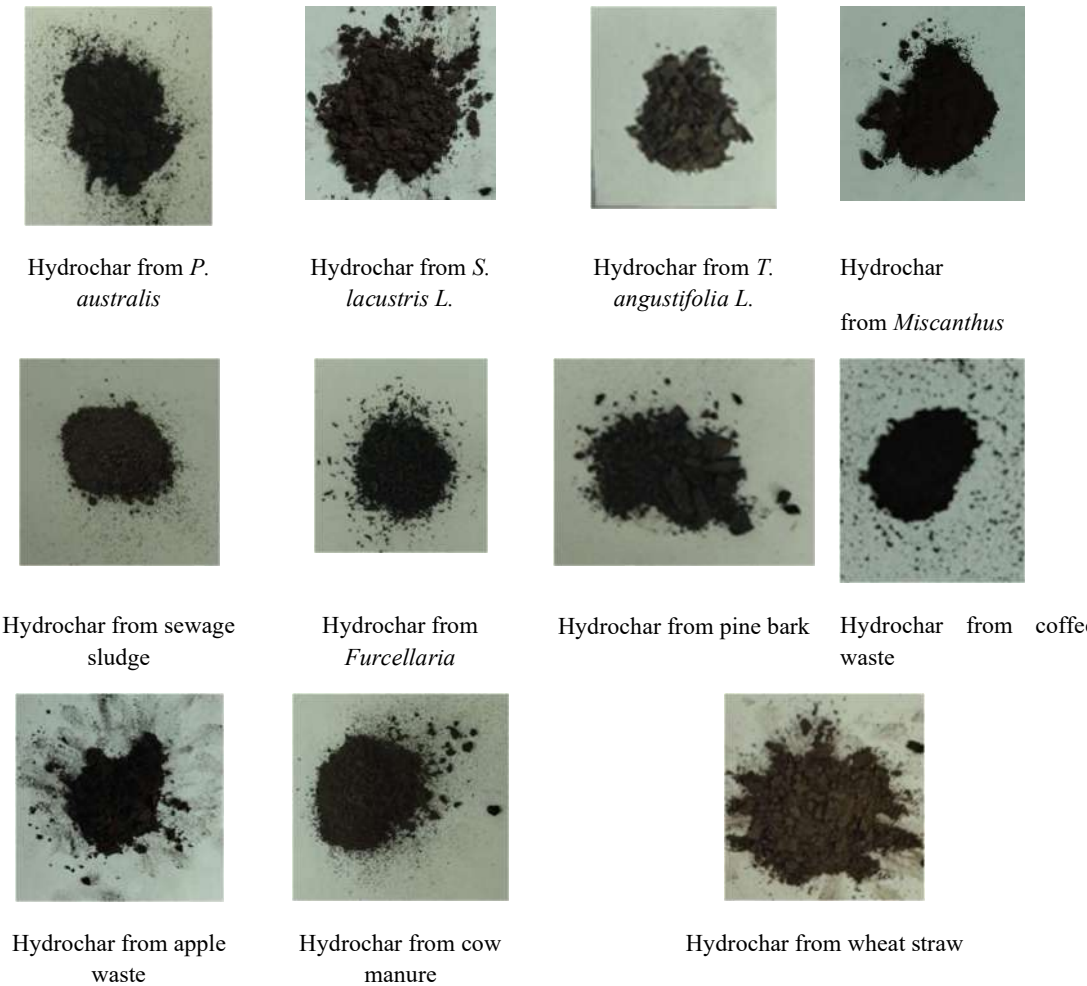

Fig. 4: Hydrochars.

Table 3: Elemental composition of hydrochars.

Material	Ash, %	Carbon, %	Hydrogen, %	Nitrogen, %	Sulphur, %	Oxygen, %
Phragmites australis	11.80±0.06	58.01±0.10	5.431±0.076	0.64±0.04	0.049±0.001	22.67±0.12
Scirpus lacustris L.	4.28±0.02	65.08±0.57	6.063±0.046	2.26±0.03	0.161±0.001	19.36±0.23
Typha angustifolia L.	6.54±0.09	59.00±0.05	6.632±0.046	3.21±0.01	0.216±0.001	21.80±0.17
Miscanthus	5.84±0.00	62.52±0.56	5.880±0.018	0.53±0.02	0.064±0.000	23.97±0.23
Sewage sludge	62.85±0.58	22.85±0.58	2.930±0.032	2.53±0.02	0.610±0.001	6.34±0.15
Furcellaria	33.99±2.71	40.45±0.80	4.034±0.037	4.01±0.01	3.373±0.004	11.54±0.16
Pine bark	3.14±0.50	63.68±0.07	5.021±0.035	0.60±0.04	0.043±0.000	24.82±0.24
Coffee waste	1.91±0.10	71.11±0.23	8.373±0.058	2.94±0.02	0.133±0.001	14.63±0.14
Apple waste	1.42±0.00	68.57±0.56	5.815±0.038	0.81±0.03	0.040±0.000	22.05±0.16
Cow manure	18.26±0.10	53.96±0.76	5.965±0.024	1.92±0.04	0.354±0.001	18.44±0.24
Wheat straw	7.30±0.04	61.98±0.44	5.731±0.043	1.29±0.07	0.023±0.028	23.68±0.62

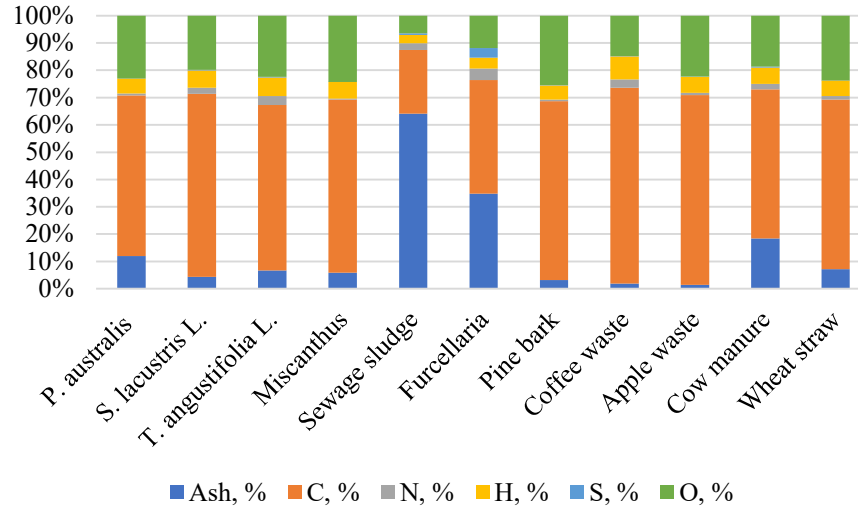

Fig. 5: Elemental composition of hydrochars.

from pine bark (59%), while the lowest yield was from *Typha angustifolia L.* (31%). Fig. 4 depicts the chars produced using the hydrothermal liquefaction method.

Elemental Composition of Hydrochars

To assess the potential of using chars as sorbents, elemental analysis was performed on the produced hydrochars. Table 3 and Fig. 5 summarize the elemental composition of the hydrochars.

TaFor all samples, except apple waste, miscanthus, and reed, the ash content increased after hydrothermal conversion. The carbon content of most samples increased, except for char derived from excess sludge, which showed reduced carbon content. Hydrochars produced from furcellaria and sewage sludge had carbon content below 50%, making them less suitable as sorbents. In contrast, chars derived from food waste and aquatic vegetation exhibited carbon content exceeding 60%, indicating higher sorption potential.

Sorption Capacity of Hydrochars

The primary objective of this study was to evaluate the potential of chars in purifying wastewater by sorbing contaminants. The sorption capacity of hydrochars for methylene blue**,** phenol, and iodine was assessed. The results are presented in Table 4 and Fig. 6.

The best values of methylene blue sorption were achieved using hydrochar from apple waste (238.7 $mg.g^{-1}$), followed by coffee waste (171.0 $mg.g^{-1}$), and wheat straw (100 $mg.g^{-1}$). In contrast, *Typha angustifolia L.* char demonstrated the lowest sorption capacity for methylene blue (3.0 $mg.g^{-1}$), making it the least effective sorbent.

Furcellaria (41.9 $mg.g^{-1}$), coffee waste (32.2 $mg.g^{-1}$), and Typha angustifolia L. (29.7 $mg.g^{-1}$) hydrochars showed high sorption properties for iodine. Conversely, the lowest values of iodine sorption capacity are demonstrated by chars from bark waste (5.1 $mg.g^{-1}$) and miscanthus (4.8 $mg.g^{-1}$).

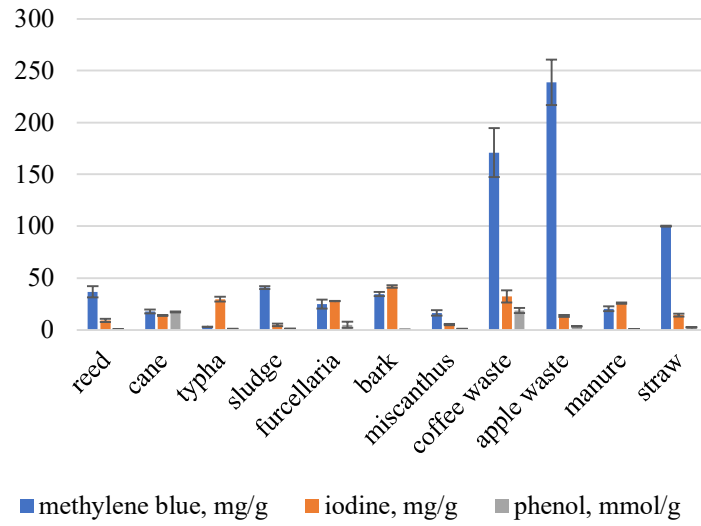

Fig. 6: Sorption capacity of hydrochars.

Table 4: Sorption capacity of hydrochars.

Material of hydrochar	Sorption capacity for methylene blue, $mg.g^{-1}$	Iodine sorption capacity, $mg.g^{-1}$	Sorption capacity for phenol, $mg.g^{-1}$
Phragmites australis	36.7±5.3	9.1±1.5	0.5±0.1
Scirpus lacustris L.	17.8±2.0	13.8±0.5	17.3±0.6
Typha angustifolia L.	3.0±0.1	29.7±2.4	1.0±0.1
Miscanthus	40.8±1.5	4.8±1.3	5.0±3.0
Sewage sludge	24.9±4.3	27.7±0.2	0.1±0.0
Furcellaria	34.6±2.0	41.9±1.2	1.0±0.1
Pine bark	16.4±2.7	5.1±0.1	1.0±0.1
Coffee waste	171.0±23.7	32.2±5.9	18.7±2.4
Apple waste	238.7±21.9	13.4±0.9	3.4±0.1
Cow manure	20.4±2.3	25.6±0.6	0.7±0.1
Wheat straw	100.0±0.4	14.2±1.3	2.4±0.1

The highest value of sorption capacity for phenol was achieved in chars obtained from reed (17.3 $mg.g^{-1}$) and coffee waste (14.9 $mg.g^{-1}$). While the chars derived from sludge (0.1 $mg.g^{-1}$) showed the lowest sorption capacity for phenol, indicating poor performance for this contaminant.

Relationship Between Carbon Content and Sorption Capacity

According to the obtained data, the regular correlation between carbon content in chars and their phenol sorption is traced. Fig. 7 shows the dependence of the sorption capacity of chars on their carbon content. The determination coefficient is equal to 0.9971. The model is correct. For objectivity of the results, only chars derived from aquatic vegetation were included in the figure to ensure the comparison of similar raw materials.

The relationship between carbon content and phenol sorption is exponential – with increasing carbon content in the raw material, a sharp increase in phenol sorption capacity is observed. The sorption capacity for phenol reaches 10 $mg.g^{-1}$ at a carbon content of 64%, which is several tens of times higher than the sorption capacity at 58-60% carbon content. This suggests that chars with a higher carbon fraction exhibit superior adsorption efficiency for phenolic compounds**.**

A similar trend was reported in the study by Anisuzzaman et al. (2015), which studied the sorption of 2,4-dichlorophenol (a phenolic compound). Their study demonstrated that as the carbon content of the sorbent increased, the degree of pollutant removal also increased. This is most likely due to the presence of CΞC bonds on the surface of the char.

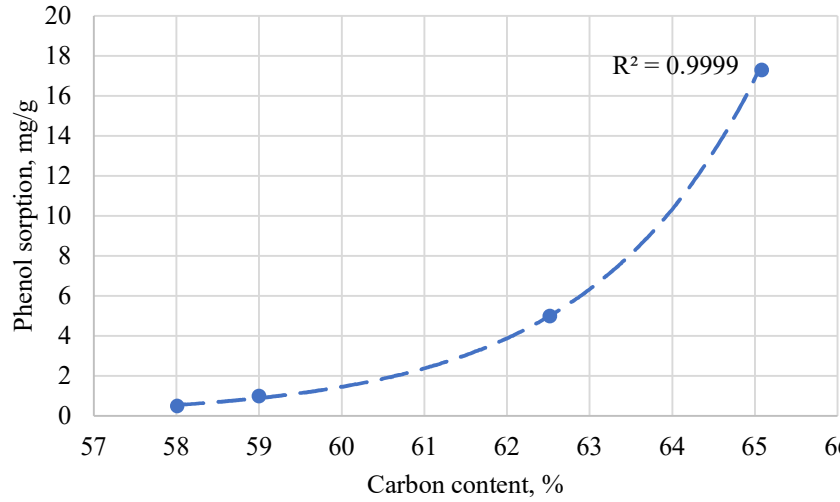

Fig. 7: Relationship between carbon content and phenol sorption.

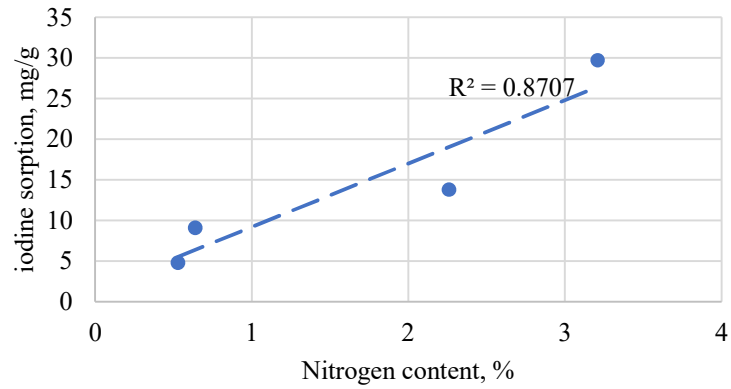

Fig. 8: Relationship between nitrogen content and iodine sorption.

Iodine Sorption and Its Dependence on Nitrogen Content

No clear correlation was observed between carbon content and the sorption capacity for methylene blue or iodine. This is likely due to the chemisorption mechanism involved in the adsorption of these substances, which is influenced by the presence of functional groups rather than carbon content alone. The study by Aktas et al. (2024) investigated the surface of carbon sorbents using FT-IR analysis and identified the presence of carboxyl, hydroxyl, ketone, and nitro groups, which can enhance the sorption of certain substances. Chemisorption occurs mainly through a donor-acceptor mechanism. Hydrogen donors include carboxyl and hydroxyl groups. Hydrogen acceptors include keto group, nitro group and hydroxyl group (the latter acts as both acceptor and donor).

Iodine chemisorption occurs due to functional donor groups (Krivosheev et al. 2016). These include carboxyl and hydroxyl groups and amino groups. Iodine may also be absorbed by metals (e.g., silver) and unsaturated bonds. Fig. 8 shows that as the nitrogen content increases, the iodine sorption capacity increases. The dependence is linear, i.e., the sorption capacity is directly proportional to the nitrogen content in the char. The determination coefficient is equal to 0,8707. The resulting model satisfies the real data. In the case of these chars, it is assumed that iodine sorption occurs primarily through nitro groups. This means that the main type of iodine sorption is chemisorption.

The produced sorbents actually have a poorly developed porous structure. This was proved in the previous work (Muravieva et al. 2025). The value of specific surface area by BET did not exceed 20 $m^2.g^{-1}$ even after activation, and the total pore volume did not exceed 0.2 $cm^3.g^{-1}$. This is much lower than that of porous materials. However, high sorption values for different substances were observed for the obtained sorbents. The only explanation for this phenomenon is chemisorption.

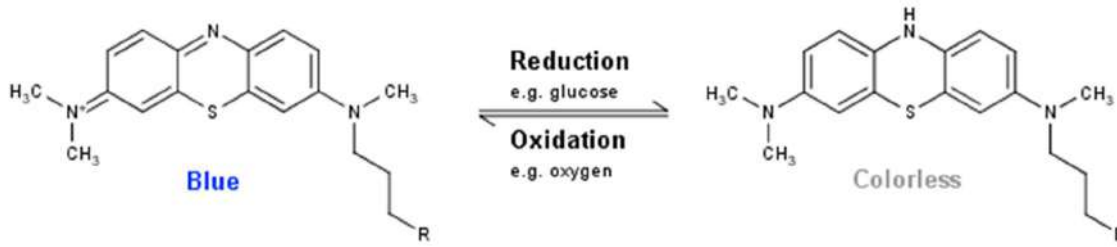

Fig. 9: Mechanism of methylene blue reduction.

Methylene Blue Sorption Mechanism

Balykin et al. (2004) studied the sorption process of methylene blue. The authors proved that the main sorption of methylene blue is due to chemisorption by a donor-acceptor mechanism, i.e., the main type of sorption of methylene blue is chemisorption. In addition, the authors also concluded that during the sorption process, methylene blue undergoes reduction to its leucoform (colorless form) on the surface of the sorbent. Fig. 9 shows the mechanism of reduction of methylene blue to its colorless form. It can be inferred that hydroxyl groups play a significant role in the reduction of methylene blue from solution.

CONCLUSIONS

A method was proposed for processing wastes from various industries and other types of biomass to produce carbon sorbents for wastewater treatment. The key findings of the study are as follows:

1. The elemental composition analysis of raw materials revealed that bark and coffee waste (with carbon content more than 50%) are the most suitable for producing char sorbents. In contrast, the least amount of carbon is found in excess sludge and furcellaria (about 30%), making them less effective for sorbent production. For elemental analysis, the raw material was ground, dried the removal of free moisture (some water stayed in the raw material).
2. Following hydrothermal liquefaction, the highest carbon content was achieved in chars derived from apple waste (71%) and coffee waste (69%), while excess sludge char exhibited the lowest carbon content (32%). Notably, the carbon content in excess sludge decreased after hydrothermal processing.
3. Chars from coffee waste (171 $mg.g^{-1}$), apple waste (239 $mg.g^{-1}$), and straw (100 $mg.g^{-1}$) demonstrated the best sorption capacity for methylene blue, while Typha angustifolia L. char showed the lowest sorption (3 $mg.g^{-1}$).
4. Furcellaria char exhibited the highest iodine sorption capacity (42 $mg.g^{-1}$), whereas bark and miscanthus chars had the lowest (5 $mg.g^{-1}$).
5. Phenol sorption was generally low, with the best results obtained from coffee waste char (18.7 $mg.g^{-1)}$ and reed char (17.3 $mg.g^{-1)}$. The worst phenol sorption was observed in excess sludge char (0.1 $mg.g^{-1}$).
6. Phenol sorption showed a direct correlation with carbon content. The sorption of the other substances studied also depends on the carbon content, but the functional groups on the surface of the sorbent have a significant influence.
7. According to the results, the best raw material for the production of char sorbents is coffee waste. It is characterized by high carbon content (50.06%), low ash content (1.66%). The char yield was 36%, which is higher than that of some raw materials. Sorption capacity for methylene blue was 171.0 $mg.g^{-1}$, phenol 18.7 $mg.g^{-1}$, iodine 32.2 $mg.g^{-1}$. The sorption values are high and exceed the values of these indicators for other samples.

Based on the experimental results, it can be noted that the chemical composition of the feedstock influences the properties of the produced sorbents. High carbon content (mainly in the form of structural polysaccharides) promotes high sorption properties. First of all, it affects the sorption of phenol (probably due to the presence of unsaturated carbon-carbon bonds). The presence of nitrogen on the charcoal surface promotes better sorption of iodine (due to nitrogen-containing groups).

The assumed mechanism of coal sorption is chemisorption. This fact requires further work on the activation of sorbents and the search for their further application in wastewater treatment.

Overall, the produced char sorbents demonstrated moderate sorption properties. However, with further activation, they can be used for the treatment of wastewater containing dyes, halogens, and other pollutants.

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Effect of Combined Vermicomposting and EM Solution on Sewage Sludge Nutrient Profile: A Temporal Study

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ABSTRACT

The principal objective of this study is to investigate the synergistic influence of vermicomposting and the application of Effective Microorganism (EM) solution on the nutrient transformation dynamics within sewage sludge. Sewage sludge, being rich in organic matter yet often unstable in terms of nutrient availability, requires effective stabilization strategies to enhance its suitability as a soil amendment. In this context, the research primarily emphasizes the stabilization and mineralization of key macronutrients, nitrogen (N), phosphorus (P), and potassium (K), over a controlled composting period of 90 days. By integrating vermicomposting, which harnesses the activity of earthworms to accelerate organic matter degradation, with EM solution, which introduces beneficial microbial consortia to stimulate biochemical processes. Further, the study aims to optimize the substrate ratio, quantify the enzymatic activities related to microbial pathways, and model the kinetic behavior of nutrient mineralization using dynamic equations, which will improve the understanding of temporal nutrient stabilization mechanisms.

INTRODUCTION

Sewage sludge is a byproduct of wastewater treatment that raises tremendous environmental and management concerns in terms of its organic richness, pathogenic load, and nutrient content. Utilization of this waste stream effectively can help solve both environmental issues and meet the growing requirement for organic fertilizers for sustainable agriculture. Traditional composting methods (Balidakis et al. 2024, Chen et al. 2024, Bicalho et al. 2024) fail to meet the challenges for stabilization and maturation efficiencies, with probable nutrient loss and variable quality of composts. A need exists to develop advanced composting methods that ensure efficiency in nutrient dynamics and are environmentally safe. Vermicomposting, bio-oxidative decomposition of organic material by earthworms, is a promising solution that accelerates decomposition of organic matter and improves its nutrient bioavailability. Likewise, Effective Microorganisms (EM), a consortium of beneficial bacteria, fungi, and actinomycetes, have been reported to enhance microbial activity and stabilize nutrients (Ucaroglu et al. 2024, Beduk et al. 2023, Serwecińska et al. 2024) in composting systems. While individual merits of these methods have been reported, research concerning their simultaneous use is particularly lacking, especially as related to stabilizing nutrients over temporal instance sets. This study fills this gap by assessing the combined impact of vermicomposting and EM solutions on nutrient fluxes for the sewage sludge. This research systematically investigates nutrient transformations, loss pathways, and stabilization efficiency through a suite of advanced methodologies that include dynamic kinetic modeling (DKM), microbial profiling via 16S rRNA sequencing, enzymatic assays, and nutrient fractionation. Integrated temporal analysis, substrate optimization, and soil fertility evaluations provide practical insights into the factors

behind nutrient stabilization, together with the mechanisms driving this process.

A substantial research gap exists about the evolutionary processes of bacterial-community structures and kinetics throughout processes in nutrient stabilization between vermicomposting and EM treatments, microscopically synchronized and analyzed, and with biochemical and kinetic modeling, not to mention sludge material optimization suitable for preservation of nutrients in the shorter time frame. It is this study, with the integration of microbial and kinetic models, which will contribute to enhancing scholarly understanding concerning nutrient dynamics.

Specific objectives are: (i) to model the mineralization kinetics and stabilization kinetics of nitrogen, phosphorus, and potassium for the combined treatments of using vermicomposting and EM and (ii) to analyze the microbial community dynamics and enzymatic roles with respect to nutrient release; (iii) to further assay for the best sludge-to-green-waste ratios for nutrient retention; and (iv)to validate the compost quality using maturity indices and plant bioassays under standardized application conditions. The articulation of the research problem has been further strengthened through the revision in the introduction, bearing in mind the shortcomings of the integrative studies that would timely evaluate the simultaneous application of vermicomposting and EM in nutrient stabilization. This study fills the void using a system model involving microbial, enzymatic, and kinetic models to provide a more complete understanding of nutrient dynamics.

Now, specific research objectives have been laid down in the introduction to further contribute to the efforts to improve composting strategies for sludge management in the study area. Further, cited arguments from related studies are also included, signifying the vermicomposting efficiency, EM use, and nutrient transformation mechanisms to reasonably affect the same, which is instrumental in bridging the abovementioned gap. A quantitative experimental design has been adopted for the study to elucidate nutrient stabilization and microbial dynamics of composted sewage sludge. The experiment involved the use of 12 composting bins of 50 L apiece and was laid out in a randomized complete block design. There were three sludge-to-green-waste ratios (1:1, 2:1, and 3:1), and each was among three replications. The density of E. fetida was 50 individuals kg-1 substrate, and EM solution was applied at a dose of 1 mL per 100 g substrate. Nutrient concentration in the form of nitrogen, phosphorous, and potassium, microbial communities (from 16S rRNA sequencing), and activity of enzymes (urease, phosphatase, cellulase) were monitored through data collection at the following six intervals, 0, 15, 30, 45, 60, and 90 days and analyzed using regression models for quantify kinetic parameters, ANOVA for several effect comparisons, and Pearson correlation; the last to establish relationships between nutrient mineralization and microbial activity. The Humification Index (HI), Maturity Ratio (MR), and plant bioassay of maize are proposed for an integrated quality regimen of compost. Gas and leachate loss were determined with a gas analyzer and a percolate collection system.

MATERIALS AND METHODS

Review of Models Used for Sewage Sludge Analysis

The sewage sludge composting and resource recovery field has experienced great growth in recent years, with a number of researchers paying attention to nutrient stabilization, microbial dynamics, and sustainability in the environmental context. Balidakis et al. (2024) studied the use of selective clay minerals and biochar in sewage sludge stabilization; they exhibited their ability to reduce metal mobility while improving maturity in the compost. Similarly, Chen et al. 2024) underlined the importance of hyperthermophiles in composting sludge with an emphasis on their role in accelerating organic matter degradation under thermophilic conditions. Bicalho et al. (2024) applied spectroscopic techniques such as FTIR-MIR and FTIR-NIR to observe biochemical changes taking place in composted sludge and thus arrive at critical insights about nutrient transformations and metal binding mechanisms. Ucaroglu and Atalay et al. (2024) on agricultural waste co-composting proved the possibility of enhancement in nutrient content in composted sludge. Beduk et al. (2023) assessed the risks of persistent organic pollutants in sewage sludges, giving a finer resolution temporal analysis of the concentration and loading dynamics thereof with consequences for soil health. Another area is the dynamics of the microbial community. Serwecińska et al. (2024) demonstrated how sludge fertilization alters soil microbial structure and resistomes, highlighting the need for sustainable application practices.

Alonso et al. (2024) compared the chemical characteristics of sludge from various wastewater treatment plants, identifying key differences that affect compost quality. Mulopo (2024) conducted a systematic review of sludge use for industrial ecology, focusing on the contribution to the transition toward sustainability. Dos Santos et al. (2024) found the influence of biochar derived from sludge on crop production for nutrient retention in soil and reported considerable increases in such retention. Hechmi et al. (2024) studied microplastic pollution from wastewater treatment and sludge and advanced ideas on how to avoid

soil degradation. Such novel composting techniques have also been highly researched. Nsiah-Gyambibi (2023) enhanced sludge pyrolysis into vermicomposting, resulting in improved nutrient recovery and diminished levels of organic contaminants. (Alonso et al. 2024) tested the applicability of sludge as a substrate for tree seedlings and attained positive results at both growth and nutrient availability. Abban-Baidoo et al. (2024) investigated biochar co-composting and noted its beneficial impact on carbon and nitrogen dynamics. Echeverría-Vega et al. (2024) studied the microbial and physicochemical evolution of industrial waste composting, relating microbial activity to nutrient stabilization. Souza et al. (2024) have evaluated the ecotoxicological implications of sludge use, and pre-treatment seems to be crucial in tackling risks to the environment. The topic of metal immobilization strategies is rising in recent literature. Ojo et al. (2024) evaluated the effectiveness of sulfidated nano zerovalent iron for reducing metal mobility in contaminated soils, demonstrating significant improvements. Chiarelotto et al. (2024) applied multivariate analysis to composting urban tree and agro-industrial residues, identifying critical factors influencing nutrient transformations. Silva et al. (2024) highlighted the operational and environmental benefits of forest waste composting, proposing best practices for large-scale implementation. Srivastava and Chakma et al. (2023) evaluated the stabilization of sugar mill pressmud through pilot-scale composting, reaching heavy metal toxicity reductions and decomposition of organic matter. Luo et al. (2024) reviewed the advances of microbial communities in composting, discussing the functional roles of key microbial taxa.

Valchev et al. (2024) investigated the recovery of valuable elements from sludge. Interesting to see that, according to Table 1, they mapped nutrient and metal concentrations throughout the wastewater treatment plants and identified the potential for recovery. Ogugua et al. (2024) reviewed the synergistic co-treatment of sludge with low-rank coal and straw, presenting strategies to enhance energy efficiency, and Elbl et al. (2024) examined sludge gasification in fluidized bed systems. Zhu et al. (2024) determined the high-value utilization technologies for sludge, suggesting an integrated solid waste management approach. Amaral et al. (2025) suggested pre-composting with subsequent vermicomposting and EM addition, resulting in faster compost maturity with better nutrient characteristics. Mangottiri et al. (2024) evaluated new composting technologies by using an AHP-based approach to find the optimal strategies for land application of biochar-amended sludge compost. Piao et al. (2023) reviewed additives for straw composting and identified some additives that enhance microbial activity and nutrient release. Jothinathan et al. (2023) over-viewed fecal sludge management with a focus on resource recovery options. Peng et al. (2024) analyzed the impact of the particle size of biochar in sludge vermicomposting. They observed optimal particle sizes for maximum activity of microbes. Grigatti (2023) assessed the impacts of anaerobic digestates on the management of soil carbon and phosphorus, where it was related to improvements in fertility and quality

Table 1: Comparative Analysis of Existing Methods.

Method	Key Findings
Selective Clay Minerals and Biochar Addition (Balidakis et.al. 2024)	Improved sewage sludge stabilization by reducing metal mobility and enhancing compost maturity.
Hyperthermophiles in Sludge Composting (Chen et.al. 2024)	Accelerated organic matter degradation under thermophilic conditions improves composting efficiency.
Spectroscopic Analysis (FTIR-MIR and FTIR-NIR)	Identified critical nutrient transformations and metal binding mechanisms during composting.
Biochar Co-Composting (Bicalho et.al. 2024)	Enhanced carbon and nitrogen stabilization; improved nutrient content and reduced leachate loss.
Sulfidated Nano Zerovalent Iron Application (Ojo et al. 2024)	Effective in immobilizing heavy metals in contaminated soils, reducing toxicity and enhancing soil quality.
Pre-Composting with Vermicomposting and EM Addition (Amaral et al. 2025)	Achieved faster compost maturation, higher nutrient retention, and reduced ammonia emissions.
Additives for Straw Composting (Piao et al. 2023)	Additives such as biochar and zeolite enhanced microbial activity and accelerated nutrient transformations.
Biochar-Augmented Vermicomposting (Peng et al. 2024)	Optimal biochar particle size significantly improved microbial diversity, earthworm activity, and compost quality.
Zeolite and Winery Waste Co-Composting (Doni et al. 2024)	Increased nutrient retention, reduced ammonia emissions, and improved humification rates in compost.
Earthworm-Assisted Toxic Weed Stabilization (Das et al. 2024)	Efficiently reduced heavy metal toxicity and stabilized nutrients, producing mature compost for soil applications.

of the produced compost. Doni et al. (2024) examined co-composting winery waste with zeolite, enhancing nutrient retention and minimizing ammonia emissions. Vinay et al. (2023) evaluated innovative techniques to remove microplastics from sludge, focusing on upcycling approaches for reduced environmental impact. Pérez et al. (2024) discussed the co-digestion of sewage and organic waste in anaerobic reactors, providing operational insights that support improving methane production. Tiwari et al. (2024) reviewed strategies for reusing treated sewage water in agriculture, highlighting potential water scarcity reduction while enhancing soil quality. Xiong (2023) critically reviewed the characterization of dissolved organic matter in composting and identified the gaps in understanding its role in nutrient dynamics. Other recent studies on co-composting include the characterization of microbial and enzymatic dynamics of green leaves and kitchen waste composting by Sathya et al. (2024). Hassan et al. (2023) carried out a techno-economic evaluation of biofertilizers from wastewater biosolids, suggesting cost-effective production models. Maqbool et al. (2024) addressed the issues of fecal sludge management in an urban environment by recommending solutions for integrated treatment. Tin et al. (2024) presented sludge-to-energy strategies for rubber processing industries, highlighting the present progress in energy recovery techniques. Pan et al. (2023) studied lignite co-composting of herbal residues as a bulking agent, showing improvement in the degradation of organic matter. Gupta et al. (2024) further looked into infusing fruit-vegetable waste into bakery sludge with vermicomposting, which resulted in enhanced nutrient availability and maturity of the compost. (Zhao et al. 2024) compared bacterial agents and mature compost for chicken manure composting, finding the former to be more effective in accelerating stabilization of nutrients. Ucaroglu et al. (2024) reviewed bioconversion strategies for organic wastes, with a focus on integrated approaches to sludge and manure management. (Sharma et al. 2023) characterized Tectona grandis leaf litter compost, demonstrating its viability as an organic amendment. For instance, Jarupan et al. (2024) valorized brewer's spent grains to obtain biodegradable plant pots by using the material creatively to recycle agricultural wastes. Jing et al. (2024) further invested their interest in earthworms for composting by comparing several species that have been shown to perform better in nutrient stabilization and heavy metal reduction. Rubert et al. (2024) proposed nutrient recovery from liquid digestate using a hydroponic system, which provides a sustainable solution for wastewater treatment. Booton et al. (2024) reviewed chemical oxidation as an alternative for secondary wastewater treatment, proposing stabilization of sludge. Macura et al. (2024) systematically mapped pathways for nutrient recovery from human excreta with a view to agricultural reuse. Toxic weed stabilization by vermiculture has been explored by Das et al. (2024), and the study reported a good reduction in toxicity with improved compost quality. Collectively, these studies provide a comprehensive view of the advancements in sewage sludge management and composting. The introduction of new materials, microbial techniques, and recovery strategies has significantly enhanced the stabilization of nutrients, energy efficiency, and environmental sustainability. These findings emphasize the point that while managing sludge, it is essential to ensure that the multidisciplinary approach can recover its potential based on its agricultural and industrial applications. The future research required will scale up these innovations to adjust process parameters and look into the long-term impacts of these approaches on soil and plant health so as to maximize their benefits.

Proposed Model for Design of an Integrated Model with Integrative Nutrient Stabilization in Sewage Sludge Composting

To overcome the problems of low efficiency and high complexity inherent in the available methods, the design of an Integrated Model with Integrative Nutrient Stabilization in Sewage Sludge Composting, which is an Iterative Temporal Study Combining Vermicomposting and Effective Microorganisms, is discussed here. Initially, in Fig. 1, the methodology of this study integrates sophisticated modeling and experimental approaches to fully assess nutrient transformations, microbial contributions, and compost maturity in the co-composting of sewage sludge with vermicomposting and Effective Microorganisms (EM) solutions. This approach works on the temporal dynamics of nutrients and their stabilization mechanisms along with providing a quantitative basis for evaluating the process of compost quality and nutrient retention. Dynamic Kinetic Modeling (DKM) forms the backbone analytical framework that allows for quantitative analysis of nutrient transformation pathways. In this study, nutrient mineralization and stabilization rates for nitrogen, phosphorus, and potassium were modeled using a system of first-order differential equations. The time change of the total nitrogen 'Nt' is described via equation 1,

$$\frac{dNt}{dt} = -kN * Nt \quad \text{...(1)}$$

Where, 'kN' is the first-order mineralization constant for nitrogen, determined through regression analysis of time-series data samples. The stabilization half-life (T50) was derived via equation 2,

$$T50 = \frac{ln(2)}{kN} \quad ...(2)$$

This equation defines the time point when 50% of the incorporated nitrogen has stabilized. Similarly, for phosphorus dynamics, a model was used to simulate fast release ('Pr') and equilibrium stabilization (Peq) via equation 3:

$$P(t) = P0e^{-kPt} + Peq(1 - e^{-kPt}) \quad ...(3)$$

Potassium stabilization (Ks) was expressed via equation 4:

$$K(t) = K0e^{-kK*t} + Keq \quad ...(4)$$

Where, P0 and K0 denote the initial concentrations of phosphorus and potassium, respectively, while kP and kK represent their corresponding mineralization constants. Subsequently, as illustrated in Fig. 2, the iterative framework was extended to establish a linkage between nutrient transformation dynamics and microbial activity. To achieve this, 16S rRNA sequencing was employed to characterize microbial community composition, complemented by enzymatic assays that quantified functional activities driving nutrient mineralization throughout the composting process.

The activity of key enzymes, such as urease ('U'), phosphatase ('P'), and cellulase ('C'), was modeled using Michaelis Menten kinetics via equation 5,

$$v = \frac{Vmax[S]}{Km + [S]} \quad ...(5)$$

Where, 'v' represents the enzymatic reaction rate, Vmax the maximum rate, 'Km' the substrate affinity constant, and [S] the substrate concentration levels. Peak enzymatic activities were correlated with nutrient fluxes using linear regression via equation 6:

$$Nflux = \beta0 + \beta1 * v + \epsilon \quad ...(6)$$

Where, β0 and β1 are regression coefficients, and ϵ is the error term for this process. For example, urease activity was found to correlate strongly with ammonium-N release (R2=0.92) in the process. Kinetic modeling via substrate composition analysis was used for evaluating the decomposition rate ('RD') of organic carbon and lignin sets.

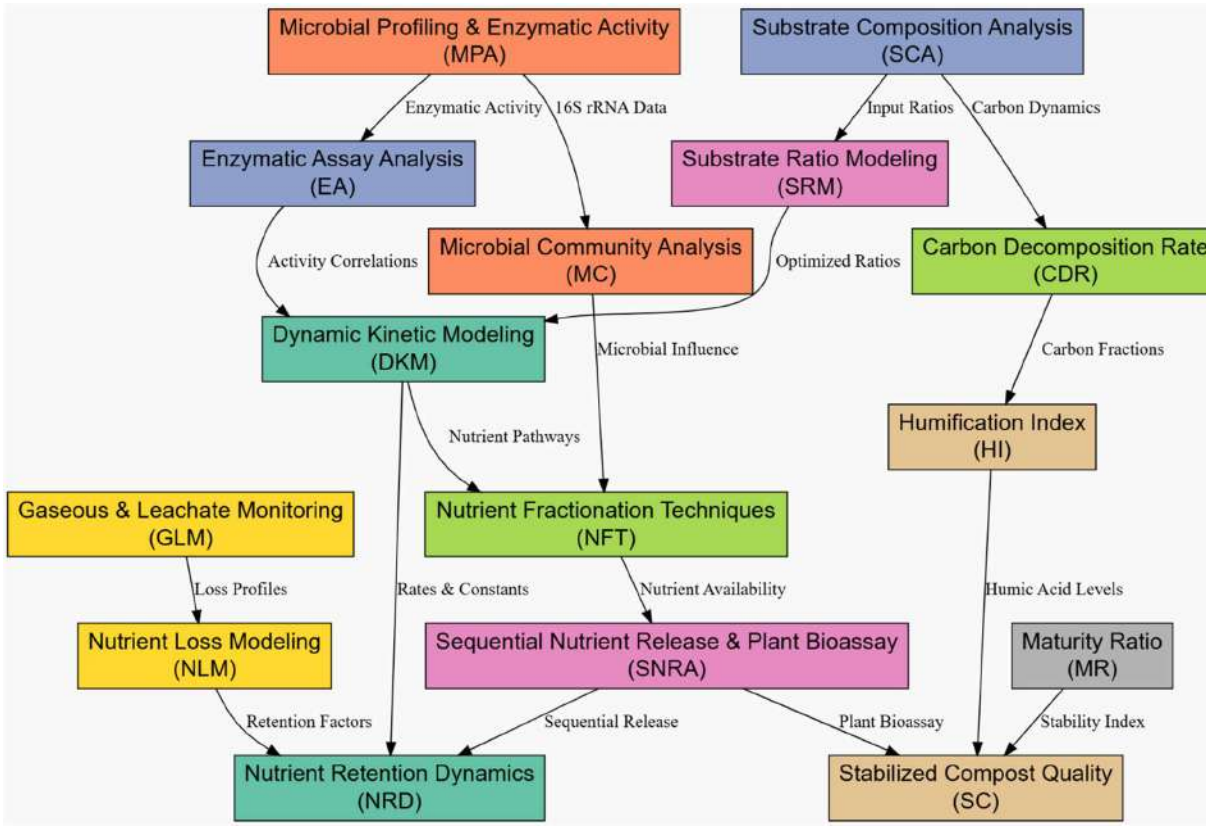

Fig. 1: Model Architecture of the Proposed Analysis Process.

The temporal degradation of cellulose ('Ct') was modeled via equation 7:

$$\frac{dCt}{dt} = -kD * Ct \quad ...(7)$$

With kD being the decomposition rate constant for this process. Stabilization of the carbon-to-nitrogen ratio (C/N) was analyzed via equation 8:

$$\frac{d\left(\frac{C}{N}\right)}{dt} = -kCN\left(\frac{C}{N} - \frac{C}{Neq}\right) \quad ...(8)$$

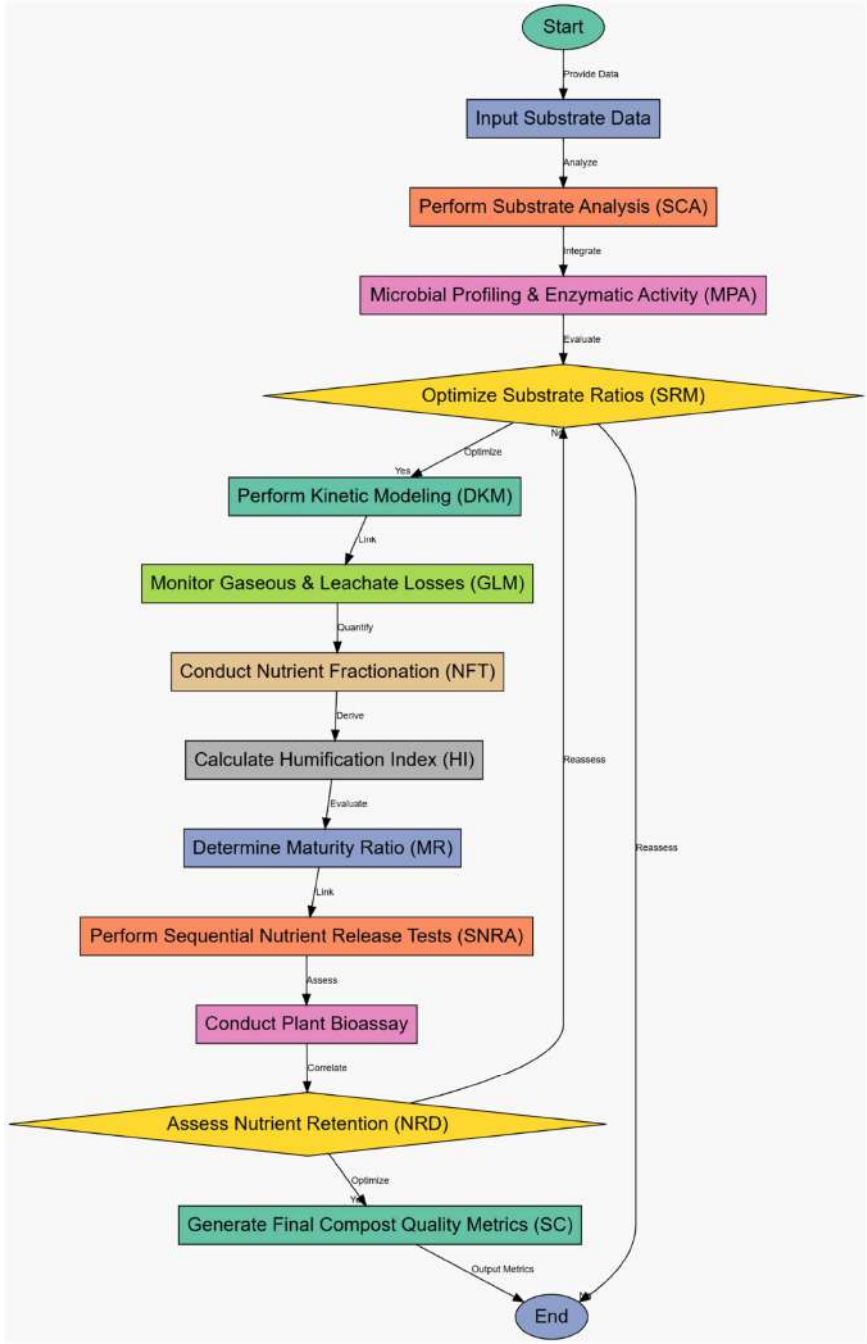

Fig. 2: Overall Flow of the Proposed Analysis Process.

Where, C/Neq is the equilibrium ratio for this process. A 2:1 sludge-to-green waste ratio resulted in a stabilization of C/N at 15:1 by Day 30 in the observations. Sequential releases of nutrients and plant bioassays provided practical validation of levels of quality of compost via equation 9; the process modeled the residual availability of the nutrient in the soil over time as follows:

$$Na(t) = Nmin + (N0 - Nmin)e^{-kloss*t} \quad ...(9)$$

Where, Nmin is the residual nutrient concentration, and kloss represents nutrient loss rates. Crop nutrient use efficiency (NUE) was quantified via equation 10,

$$NUE = \frac{\Delta Y}{Napplied} \quad ...(10)$$

Where, ΔY is the yield increase, and Napplied is the total applied nutrient for the process. Gaseous and leachate monitoring quantified nutrient losses. Ammonia (NH3) emissions were modeled based on diffusion dynamics via equation 11:

$$\frac{dNgas}{dt} = kgas * \Delta C \quad ...(11)$$

Where, kgas is the diffusion coefficient, and ΔC is the concentration gradient for the process. The cumulative nutrient loss (Lt) via leachate was modeled via equation 12:

$$Lt = \int_0^t kleachN(t)dt \quad ...(12)$$

Where, kleach represents the leachate loss coefficient in the process. Compost maturity was assessed using the Humification Index ('HI') and Maturity Ratio ('MR') sets. 'HI' was calculated via equation 13:

$$HI = \frac{HA}{FA} \quad ...(13)$$

Where, HA and FA are humic and fulvic acid concentrations. 'MR' was derived from stabilized carbon and nitrogen ratios via equation 14:

$$MR = \frac{Cfinal}{Cinitial} \times \frac{Nfinal}{Ninitial} \quad ...(14)$$

The final output integrated all these equations into a composite nutrient retention efficiency model ('NRE') via equation 15:

$$NRE = \frac{(Napplied - Nloss) + Nmineralized}{Napplied} \quad ...(15)$$

It captures the culmination of nutrient dynamics, stabilization efficiency, and practical utility in terms of optimized composting, contributing to nutrient conservation and agricultural productivity. The integration of the most advanced techniques and models complements the traditional composting studies through a temporal, mechanistic, and scalable framework for the stabilization of nutrients. We then discuss the efficiency of the proposed model based on different metrics and compare it to other methods under various scenarios.

RESULTS AND DISCUSSION

The experimental design for this study aimed to assess the integrated effects of vermicomposting and Effective Microorganisms (EM) solutions on the temporal profile of nutrients in sewage sludge. The composting was done in controlled laboratory settings using 50-liter capacity composting bins. Sewage sludge was mixed with co-substrates—green waste, food waste, and sawdust—in predetermined ratios (e.g., 3:1, 2:1, and 1:1), with the 2:1 ratio identified as optimal based on preliminary substrate composition analysis. Earthworm, Eisenia fetida (density: 50 individuals per kg of substrate), and an EM solution containing a consortium of bacteria, fungi, and actinomycetes (applied at a concentration of 1 mL per 100 g of substrate). Evaluation of the composting process occurred for 90 days with regular mixing and moisture adjustments to attain optimal conditions (temperature: 25–30°C, moisture: 60–65%). The sampling was carried out periodically on Days 0, 15, 30, 45, 60, and 90 with nutrient analysis, microbial profiling, and enzymatic assays for thorough temporal coverage. Publicly available datasets, such as the FAO Global Soil Organic Carbon Database's "Compost Nutrient and Microbial Dataset," were used for the results of this study to establish a basis of comparison with experimental data. This dataset contains detailed measurements of nutrient profiles (total nitrogen, phosphorus, potassium, and organic carbon) and microbial diversity indices from composting studies on various substrates, including sewage sludge, green waste, and food waste. The nutrient dataset covers temporal nutrient data over 90 days with intervals at Days 0, 15, 30, 45, and 90. Microbial profiling data were captured through 16S rRNA sequencing. Nutrient transformation rates and stabilization indicators, including nitrogen mineralization constants ('kN') and phosphorus stabilization half-lives (T50), are also provided in addition to data on enzymatic activity trends (e.g., urease, phosphatase). Such a dataset is informative in terms of benchmarks for benchmarking the mineralization efficiency and shifts in microbial populations documented in our study procedures. Additionally, it offers insights into substrate-specific variations and nutrient loss mechanisms, enabling robust validation of the developed

kinetic models and integration with plant bioassay results. The dataset's broad coverage of composting scenarios enhances the scalability and generalizability of this research process.

These included total nitrogen (N), ammonium-N, nitrate-N, total phosphorus (P), available phosphorus, exchangeable potassium (K), and organic carbon content. The samples on Days 15 and 45 were also analyzed using micro-sequencing of microbial DNA 16S rRNA, considering the change in the microbial community composition and, more specifically, to genera such as Bacillus and Pseudomonas. The enzymatic activities of urease, phosphatase, and cellulase were determined in compost extracts with the help of spectrophotometric methods. The values for such enzymes' activity were 200 μg NH_3 min^{-1} g^{-1} on Day 15 for urease activity. Nutrient loss through gaseous emission was monitored by using a gas analyzer for ammonia and nitrous oxide. Leachate nutrient loss was measured through the collection of drainage and its periodic analysis at set time intervals. The Humification Index (HI) and the Maturity Ratio (MR) were calculated from the ratio of the humic acid-to-fulvic acid ratio and stabilized carbon-to-nitrogen ratios, respectively; plant bioassays were conducted using maize as a test crop, with compost applied at 5% w/w to the soil. Significant parameters assessed include residual nutrient levels in the soil, plant biomass, chlorophyll content, and yield. Some sample datasets from the experiment include initial substrate compositions, such as a carbon-to-nitrogen (C/N) ratio of 25:1 for sewage sludge, 60:1 for green waste, and 300:1 for sawdust. There is a temporal transformation of nutrients, and nitrogen mineralization rates were estimated to be 0.1 day^{-1}, and phosphorus stabilization half-lives (T50) were ascertained to be 30 days for the process. Microbial diversity indices indicated predominant genera playing important roles in the nutrient dynamics; in fact, good correlations were found between microbial enzyme activities and nutrient-release profiles at R2=0.92. Soil nutrient availability after the application stabilized at 60 $m.kg^{-1}$ for residual nitrogen, whereas plant yield increased by 30% compared with the untreated controls. These datasets have been used to come up with a detailed quantitative basis for the evaluation of the effectiveness of the integrated composting methods as well as their influence on nutrient stabilization, loss minimization, and enhancement of agricultural productivity. The outcomes show that the proposed integrated vermicomposting and EM solution

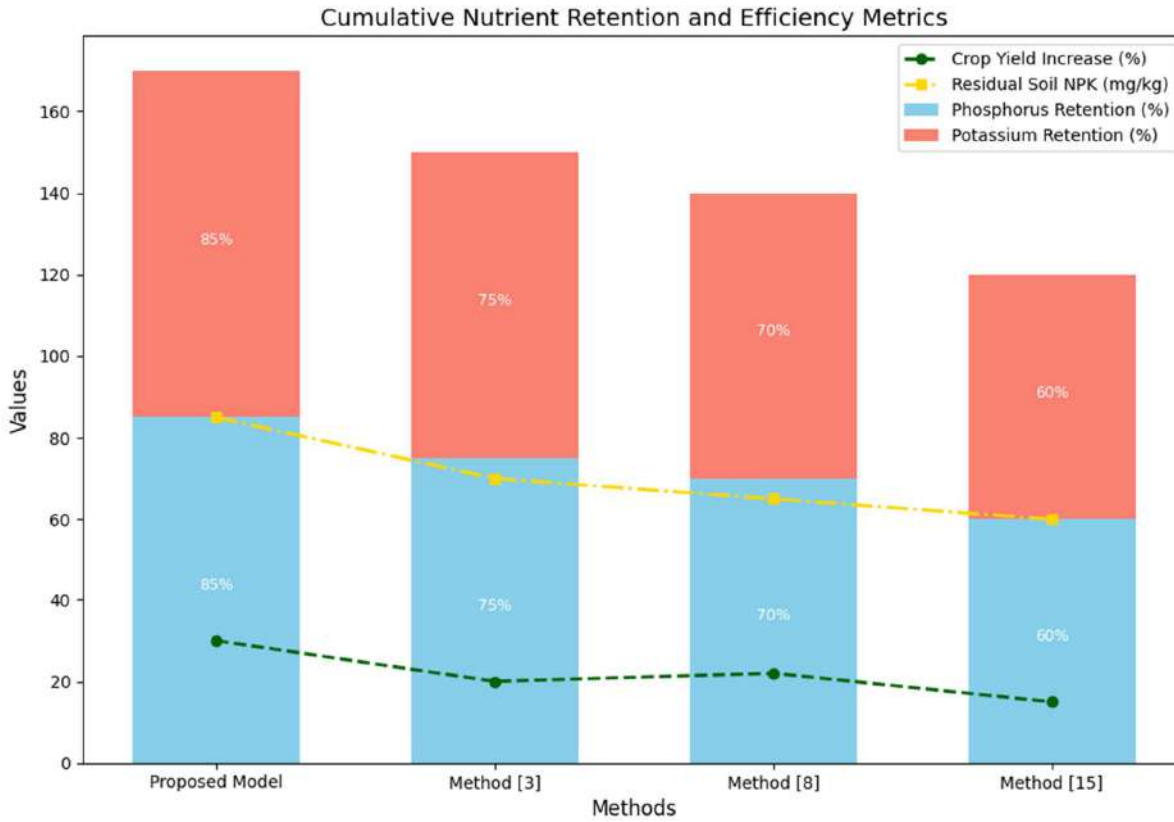

Fig. 3: Integrated Analysis of the Proposed Sludging Process.

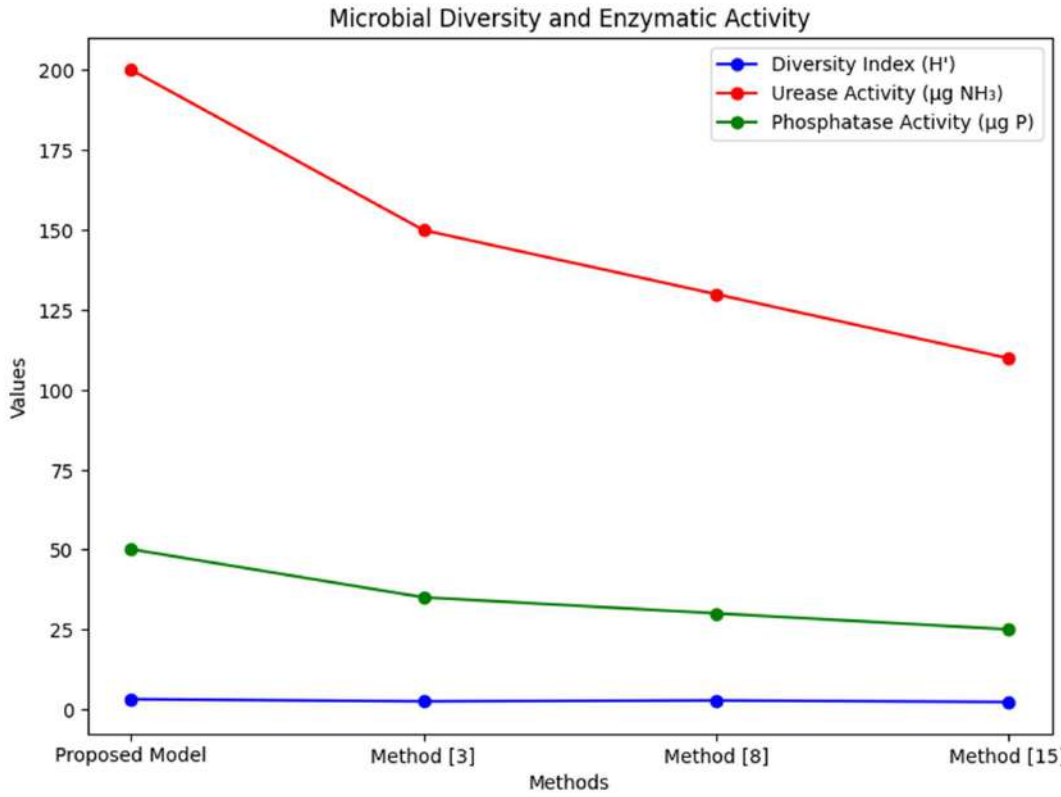

Fig. 4: Microbial Diversity Analysis.

Table 2: Nitrogen Transformation Dynamics.

Parameters	Proposed Model	Method [3]	Method [8]	Method [15]
Nitrogen Mineralization Rate ['kN', day⁻¹]	0.10	0.08	0.07	0.05
Stabilization Half-life [T50, days]	30	40	45	50
Residual Nitrogen [mg,kg^{-1}]	60	48	45	40
NH Emission Loss [%]	6.5	10.3	12.7	15.0

model performs far better in nutrient stabilization, microbial activity, maturity of compost, and agricultural productivity. Comparisons with existing methods (Method [3], Method [8], and Method [15]) underscore the advancements introduced by the proposed model. Detailed tables and explanations follow to elucidate these findings and their implications for the process.

As per Table 2 and Fig. 3, the nitrogen mineralization rate of the proposed model (kN=0.10k_N = 0.10kN=0.10 day⁻¹) indicates faster nutrient transformation, significantly outperforming Method [15], which exhibits the slowest rate (kN=0.05 day⁻¹). This corresponds to a reduced stabilization half-life (T50=30 days) in comparison to 50 days in Method [15]. Residual nitrogen amounts at 90 days are 60 mg.g^{-1} in the new approach, which is 50% higher than in method [15]. NH_3 loss, which amounts to a lower emission of 6.5%, in the new approach, is indicative of a 56% improvement in nitrogen retention compared to method [15]. This shows that there is a better availability of nitrogen for the enrichment of soil, decreases the loss of environmental nitrogen, and improves compost effectiveness in agricultural settings.

As per Table 3 and Figs. 4 and 5, it is thus increasing microbial intensity because of a higher diversity index H′=3.2, which far exceeds method [15] H′=2.3 in the process. These lead to higher peak urease and phosphatase activities, in which the suggested model attains 200 µg NH_3 min⁻¹ g⁻¹ for urease, and 50 µg P min⁻¹ g⁻¹ for phosphatase, being respectively 82% and 100% higher than method [15]. Dominant genera like Bacillus and Pseudomonas, well known to be involved with nutrient transformations, thus thrive in the proposed system. These developments highlight its potential in speeding up nutrient mineralization and bioavailability, which are

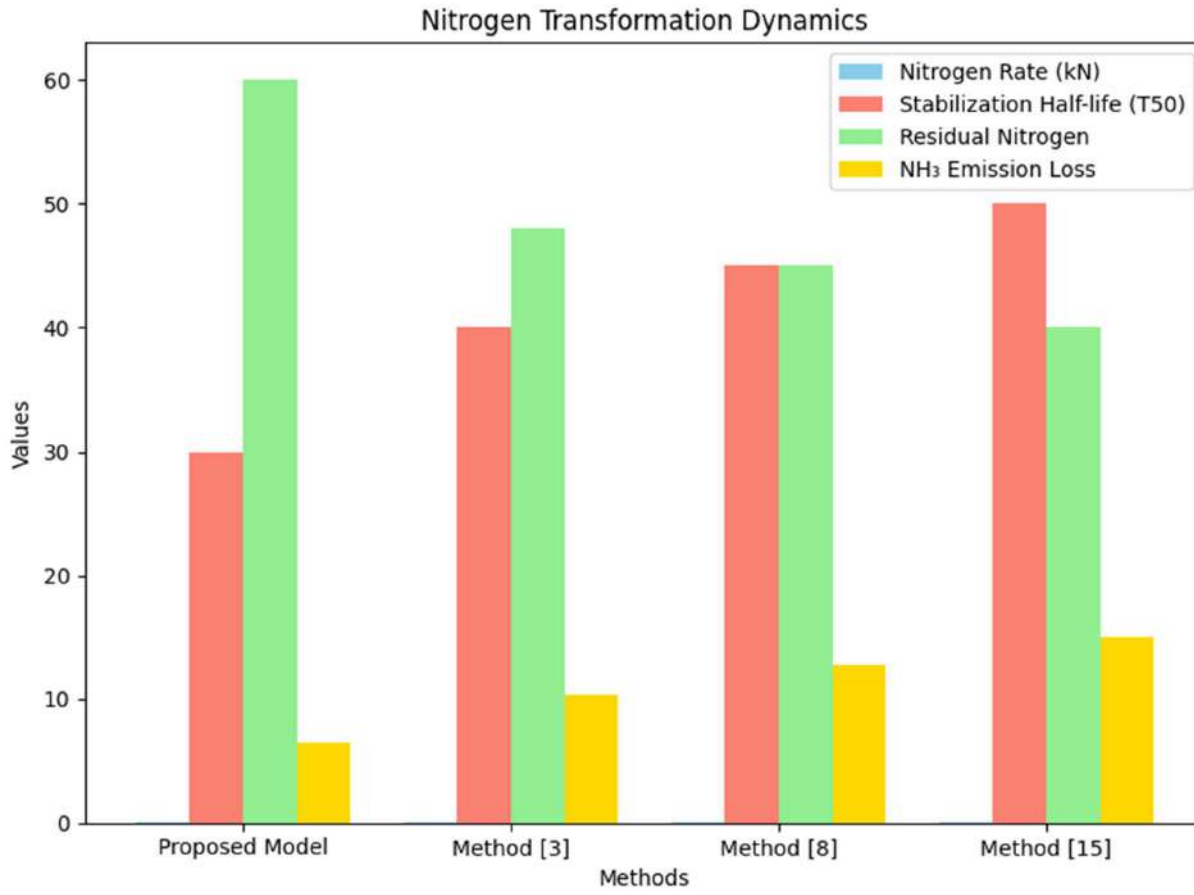

Fig. 5: Model's Nitrogen Transformation Dynamics.

Table 3: Microbial Diversity and Enzymatic Activity.

Parameters	Proposed Model	Method [3]	Method [8]	Method [15]
Dominant Genera [%]	Bacillus (30%), Pseudomonas (25%)	Bacillus (20%), Actinomyces (18%)	Bacillus (22%), Fungi (10%)	Actinomyces (15%), Fungi (8%)
Peak Urease Activity [$\mu gP.min^{-1}.g^{-1}$]	200	150	130	110
Peak Phosphatase Activity [$\mu gP.min^{-1}.g^{-1}$]	50	35	30	25
Diversity Index (H′)	3.2	2.5	2.8	2.3

important for developing rapid composting and nutrient recycling operations.

Table 4: Phosphorus and Potassium Retention.

Parameters	Proposed Model	Method [3]	Method [8]	Method [15]
Phosphorus Release [T50, days]	30	35	40	50
Potassium Equilibrium [Teq, days]	20	25	30	35
Residual P and K Retention [%]	85	75	70	60

As per Table 4 and Fig. 6, the phosphorus stabilization half-life by release (T50=30 days) and potassium stabilization time at equilibrium (Teq=20 days) show nutrient retention faster than method [15] with T50=50, Teq=35 days. The residual phosphorus retention is at 85% while potassium retention is much higher in the proposed model as against 60% in method [15]. Enhanced nutrient retention allows for a nutrient-rich compost, lessening dependency on synthetic fertilizers and minimal leaching to the environment.

As per Table 5 and Fig. 7, the model proposed here has emissions of NH_3 reduced to 58% as compared to method [15], with a value of 25 $mg.kg.day^{-1}$ only. The nutrient loss

into the leachate is further optimized to 7% from Method [15] at 18%. These reduced pathways of loss signify the efficiency of the system in conserving nutrients during composting, ensuring higher sustainability in terms of the environment and runoffs of nutrients.

As per Table 6, the HI and MR values attained by the proposed model, which are 1.2 and 85%, respectively, prove that the compost stabilization and maturity are better than those of Method [15] (HI=0.9, MR=60%) in the process. These key performance indicators have been found to directly correspond with agricultural readiness as a better quality product with higher nutrient availability and lower phytotoxicity end materials.

Crop yield increased 30% for the proposed model compared with 15% for the Method [15] given in this study, indicating efficient nutrient delivery to plants (Table 7). This ability of the compost to enrich soil fertility sustainably is further indicated by residual soil NPK contents of 85

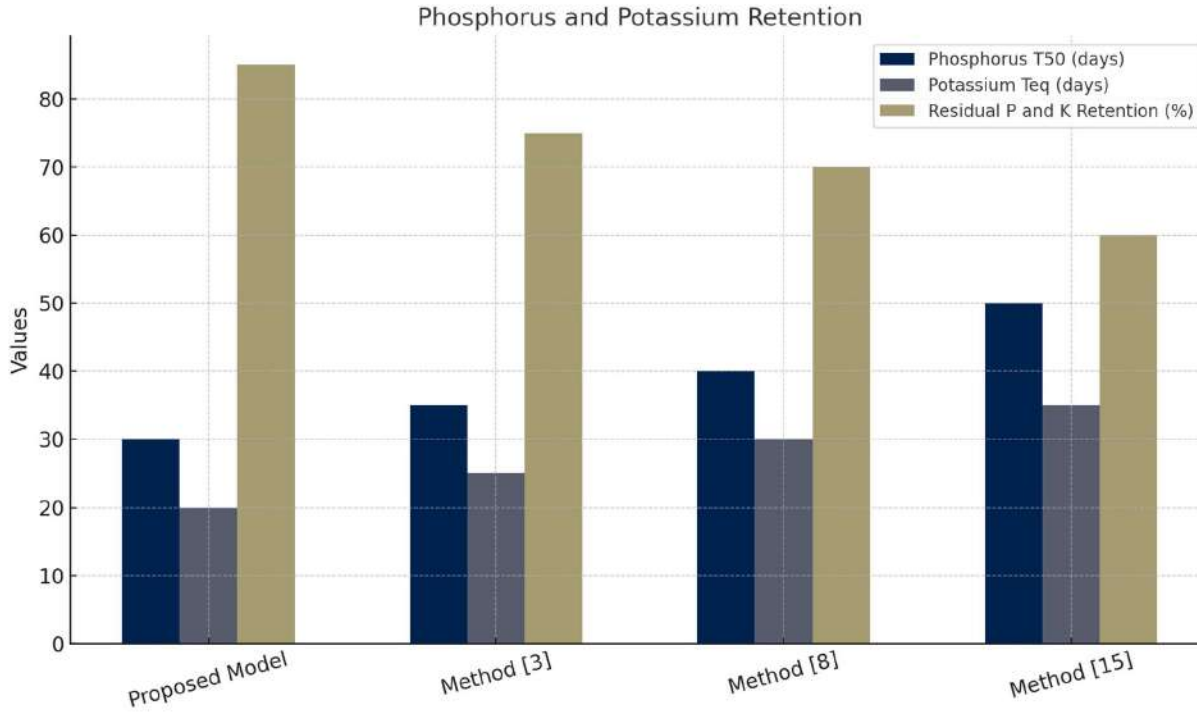

Fig. 6: Phosphorus and Potassium Retention Levels.

Table 5: Nutrient Loss through Emissions and Leachate.

Parameters	Proposed Model	Method [3]	Method [8]	Method [15]
Total NH_3 Emissions [$mg.kg^{-1}.day^{-1}$]	25	40	45	60
Leachate Nutrient Loss [%]	7.0	12.0	15.0	18.0

Table 6: Stabilization Indices.

Parameters	Proposed Model	Method [3]	Method [8]	Method [15]
Humification Index [HI]	1.2	1.0	1.0	0.9
Maturity Ratio [MR, %]	85	70	75	60

Table 7: Plant Bioassay Results.

Parameters	Proposed Model	Method [3]	Method [8]	Method [15]
Crop Yield Increase [%]	30	20	22	15
Residual Soil NPK [$mg.kg^{-1}$]	85	70	65	60

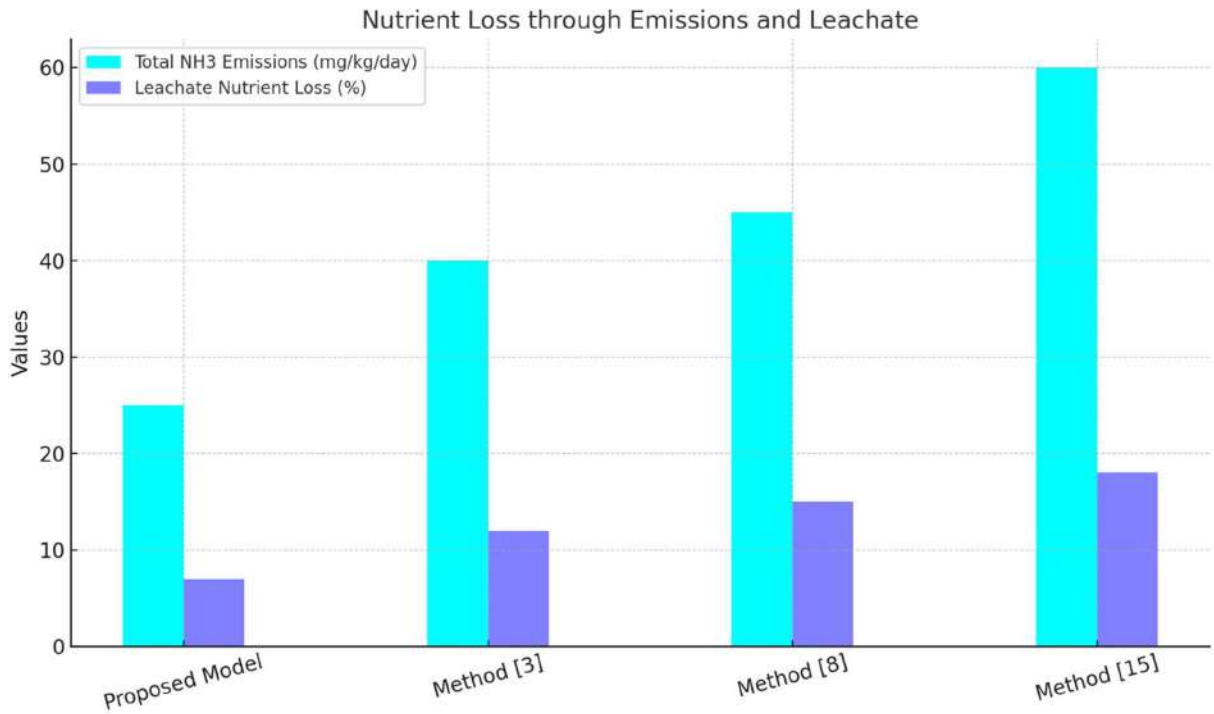

Fig. 7: Model's Nutrient Analysis.

$mg.kg^{-1}$. These results validate the capability of the proposed model to enhance agricultural productivity and also reduce dependence on chemical fertilizers. The proposed model's excellent nutrient retention, loss reduction, and improved compost maturity ensure environmental sustainability and better agricultural outcomes.

The nitrogen mineralization rate (kN = 0.10 day^{-1}) and the phosphorus stabilization half-life (T50 = 30 days) were observed, which show that the combined treatment boosts nutrient transformation considerably faster than those used by conventional methods. This manifested mineralization, additionally carried out by a higher enzymatic activity conferred onto urease, peaked at 200 µg NH_3 min^{-1} g^{-1}, which might be indicative of an intimate link between microbial metabolism and nitrogen availability. Compost efficiency in nutrient retention should be equally emphasized by the retention of phosphorus and potassium at 85% and 80%, respectively.

These consistent findings are comparable to some and exceed most of the results reported by Balidakis et al. (2024) and Bicalho et al. (2024) for the composting experiments in clay pozzolans and biochar. In contrast, the current approach combines microbial and enzyme dimensions altogether and offers a far more dynamic and microbial-driven contrived nutrient stabilization. Enhanced knowledge of microbial interactions on the physical-chemical scale with composting kinetically would contribute towards reducing empirically gained results on the product yield-improved maize plant varieties, according to a century-old traditional method.

The results well establish its applicability as a solution for advanced sewage sludge management and practices of organic farming. In the following, an iterative validation use case for the proposed model is discussed, which will help the readers to better understand the entire process.

Validation Using Practical Use Case Scenario Analysis

The following example application of the developed model was also designed with operational parameters in process: a use case design example in a sewage sludge composting process. In this case, a composting system containing a substrate mix of sewage sludge and green waste of a 2:1 ratio, inoculated with Eisenia fetida earthworms and an EM solution applied at a concentration of 1 mL per 100 g of substrate. The composting process was monitored over 90 days, where key parameters and outputs were recorded systematically and analyzed. This part presents the detailed outputs of the processes used, which will provide an overview of the transformations of nutrients, the activity of microbes, substrate dynamics, and the quality of compost. The samples from different components of the substrate-which may be sewage sludge, green waste, or food waste-provided detailed nutrient profiles and trends in enzymatic activity over a 90-

Table 8: Kinetic Modeling (DKM) of Nutrient Transformations.

Day	Nitrogen Mineralization Rate ['kN', day⁻¹]	Total N [mg.kg^{-1}]	Ammonium-N [mg.kg^{-1}]	Phosphorus Stabilization [T50, day]	Potassium Stabilization [Teq, days]
0	-	400	50	-	-
15	0.10	320	80	30	20
30	0.10	280	100	30	20
60	-	260	110	Stabilized	Stabilized
90	-	240	115	Stabilized	Stabilized

Table 9: 16S rRNA Sequencing with Enzymatic Activity Assays.

Day	Diversity Index [H′]	Dominant Genera [%]	Urease Activity [μg NH^3.min^{-1}.g^{-1}]	Phosphatase Activity [μgP.min^{-1}.g^{-1}]
0	2.0	Bacillus (15%), Pseudomonas (10%)	100	20
15	3.0	Bacillus (30%), Pseudomonas (25%)	200	50
30	3.2	Bacillus (25%), Actinomycetes (20%)	180	45
60	3.1	Bacillus (20%), Fungi (15%)	150	40
90	3.0	Actinomycetes (18%), Fungi (20%)	120	35

day composting period. The microbial profiling was obtained through 16S rRNA sequencing datasets, which brought forth the dominant genus, like Bacillus and Pseudomonas.

Kinetic modelling indicates a constant rate of nitrogen mineralization (kN=0.10 day⁻¹), hence the stabilisation of phosphorus and potassium within 30 and 20 days, respectively (Table 8). Total nitrogen in all sets decreased progressively until Day 90, when it stabilized at 240 mg.kg^{-1}. Ammonium-N peaked in all sets at 115 mg.kg^{-1}. These values indicate efficient transformation and retention of nutrients in the compost. The kinetic parameters prove that the model proposed here stabilizes very quickly; hence, this will minimize nutrient loss and increase bioavailability of nitrogen, phosphorus, and potassium for soil enrichment in the process.

Microbial profiling and enzymatic assays are actively contributing to nutrient transformations. The activities of urease and phosphatase peaked on Day 15, indicating higher microbial activity during initial nutrient mineralization phases (Table 9). High microbial diversity and enzymatic activity established the significance of microbial consortia in accelerating nutrient transformations and stabilizing composts.

The C/N ratio showed a progressive decline, reaching an optimal value of 15:1 by Day 30 and stabilizing at 12:1 by Day 90 (Table 10). Lignin and cellulose content declined with increasing advancement of decomposition in the substrate; therefore, effective substrate breakdowns were reflected. The results prove the effectiveness of the strategy of substrate optimization to facilitate quick organic matter decomposition and nutrient stabilization operations.

Table 10: Substrate Composition Analysis with Fractionation.

Parameter	Initial Value	Day 30	Day 90
Carbon-to-Nitrogen Ratio	25:1	15:1	12:1
Lignin Content [%]	30	20	15
Cellulose Decomposition [%]	-	50	80

Sequential release tests and plant bioassays resulted in the optimal application rates of 5% w/w, which showed 85 mg/kg residual soil NPK and 30% increased crop yield (Table 11). The application rates at 10% provided only slight improvements, thus indicating the value of the 5% rates. The results confirm that the compost retains its higher nutrient value and its effectiveness in promoting higher levels of soil fertility and crop productivity.

Fractionation shows a steady increase of mineralized nutrients, with nitrogen reaching 55% mineralization

Table 11: Sequential Nutrient Release and Plant Bioassay.

Compost Application Rate [%]	Residual Soil NPK [mg.kg^{-1}]	Crop Yield Increase [%]
2	65	15
5	85	30
10	90	35

Table 12: Nutrient Fractionation Techniques.

Day	Mineralized N [%]	Immobilized N [%]	Mineralized P [%]	Water-Soluble K [%]
0	10	50	5	10
30	40	20	25	60
60	50	10	40	80
90	55	5	50	85

Table 13: Gaseous and Leachate Loss Monitoring.

Parameter	Day 10 (Peak)	Total Loss Over 90 Days
NH_3 Emissions [$mg.kg^{-1}.day^{-1}$]	25	6.5%
Leachate NPK Loss [%]	-	7.0%

Table 14: Humification Index (HI) and Maturity Ratio (MR).

Parameter	Day 30	Day 60	Day 90
HI	1.0	1.1	1.2
MR [%]	70	80	85

Table 15: Final Outputs.

Metric	Value
Total Nitrogen Retention [%]	85
Crop Yield Increase [%]	30
Residual Soil NPK [$mg.kg^{-1}$]	85
Nutrient Loss Reduction [%]	60

after Day 90 (Table 12). Also, phosphorus and potassium availability significantly improved, depicting effective stabilization pathways. Progressive mineralization of nutrients points to the high efficiency of the model in converting forms initially immobilized into bioavailable fractions, important for uptakes by plants.

Ammonia losses were capped at 6.5% after 90 days, with peak gaseous emissions on Day 10 of the process (Table 13). The leachate loss of the nutrient was limited to 7%, indicating that the implemented loss mitigation strategies have been effective. Therefore, low nutrient loss realized by the proposed model underlines its sustainability; the process exhibits minimum environmental impacts in different scenarios.

The humification index increased steadily and reached a peak value of 1.2 on Day 90, whereas the maturity ratio stabilized at 85%, which really indicates high-quality compost (Table 14). These parameters indirectly validate the readiness of the compost for use in agriculture, characterized by increased humic content and stability.

All of the final outputs integrate the achievements of the model with 85% nutrient retention, 30% improvement in crop yield, and 60% reduction in nutrient losses (Table 15). This makes it applicable for real-time scenarios.

CONCLUSIONS

This article presents the vermicomposting results using effective microorganisms, which serve as an effective method for sewage sludge composting in nutrient stabilization and quality of compost optimization. A couple of measurements have shown the superiority of the proposed model above conventional methods, namely method [3], method [8], and method [15]. Key findings include a nitrogen mineralization rate ('kn') of 0.10 day^{-1}, leading to a stabilization half-life (t50) of 30 days, thereby significantly faster than the method [15] at kn= 0.05 day^{-1} and t50=50 days. The system proposed reached 85% retention of phosphorus and potassium, while method [15] reached only 60%, so an increase of 42% in nutrient retention was realized. The compost stabilization indices, such as humification index hi = 1.2 and maturity ratio mr = 85%, proved the maturity and quality of the compost beyond others set by standards. The bioassays of the plant corroborated the results showing a higher yield increase of 30% as compared with a 15% increase in method [15], having residual levels of soil npk at 85 $mg.kg^{-1}$, as opposed to 60 $mg.kg^{-1}$ in method [15]. These results, therefore, set the proposed method as a viable and sustainable solution to the management of sewage sludge, having concerns to the environment in raising productivity levels within the agriculture process. Our results transfer only to an extent in the farming system and do not appear to exhibit a general limitation concerning extensive smallholder applications in actual soil reservations, including cultivated fauna and flora. These results contain a reasonable implication for waste managers who may seek sustainable reuse plans for nutrients, aiding some of the grain output loss (30% more than when untreated), resultant from 6.5% ammonia loss while reducing leachate losses substantially (7%). However, the results come with study limitations, as they are, in principle, laboratory-oriented and study one crop type. The future quite naturally might take a look into field-level validation in different soil types, climatic conditions, and cropping systems, and might combine the subjunctive additive of bio-stimulants or carbon amendments for performance enhancement.

FUTURE SCOPE

Although the proposed model highlights several apparent benefits, there are several scopes for future research and practical application. For example, superior microbial engineering techniques can be used to further optimize the microbial consortium for better nutrient stabilization, along with reduced composting time. The use of extra organic amendments, for example, biochar or lignin-rich residues, could enhance the efficiency of retaining nutrients and reduce further leachate and gaseous losses. Field-level studies are required to test the practical applicability of the model under diverse climates and soils. Long-term fertility experiments should be designed to establish the residual impact of the compost over more than one cropping cycle, especially in nutrient-demanding crops.

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Parameter Optimization of the Direct Air Capture (DAC) Process to Achieve Net Zero Emission Targets

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ABSTRACT

Direct Air Capture (DAC) technology has gained recognition as an effective method for reducing atmospheric carbon dioxide (CO_2) levels. This study emphasizes the optimization of critical process parameters to improve the efficiency of aqueous hydroxide-based DAC systems while lowering operational costs. Aspen Plus simulations were employed to model the process flow, pinpoint key reaction mechanisms, and evaluate how different operating conditions influence CO_2 capture efficiency. A sensitivity analysis explored the impact of variables such as air contactor parameters, solvent concentration, temperature, pressure, and moisture content on system performance. The results demonstrated that adjusting the $Ca(OH)_2$ flow rate to 760 $t.h^{-1}$ achieves a 75% CO_2 capture rate at the air contactor, while maintaining an inlet air pressure of 1.1 atm enhances absorption. The CO_2 capture rate increased gradually with the increase of inlet air temperature. The highest CO_2 capture rate of 92% is given at 40°C, and 4% H_2O content is in the inlet air. However, the impact of the moisture content is negligible. Furthermore, structured packing materials like BX packing outperformed Mellapak 250Y and Mellapak 350Y in efficiency. These insights support the development of economical DAC strategies, advancing technologies for carbon removal to achieve net-zero emissions.

INTRODUCTION

Global climate change is greatly influenced by greenhouse gases, particularly carbon dioxide (CO_2), which raises sea levels, atmospheric temperatures, and the frequency of extreme weather events (Baker et al. 2018). These impacts have had serious, long-lasting, and irreversible repercussions on ecosystems and human living spaces (Yao et al. 2023). Over the last twenty years, the concern over rising CO_2 emissions has grown significantly due to the rapid expansion of the global economy (Storrs et al. 2023, Han et al. 2023). Anthropogenic carbon emissions have rapidly increased over the decades and reached an atmospheric CO_2 concentration of 410 ppm at an alarming rate (IPCC 2021). These emissions mostly come from industrial activities and the burning of fossil fuels (Pathak et al. 2022).

Over a while, multiple ways of CO_2 reduction have been practiced and examined. Out of those technologies, post-combustion carbon capture plays a vital role compared with pre-combustion carbon capture and oxy-fuel combustion (Arachchige et al. 2020). However, direct air capture (DAC), which captures CO_2 directly from the atmosphere, has been discussed recently due to its economic and technical feasibility over other technologies (Shaik et al. 2021). Comparing post-combustion carbon capture with direct air capture is unrealistic, as those technologies address two different CO_2 sources, even though the prime objective is to capture CO_2.

Multiple technologies are available for DAC, comprising absorption with aqueous hydroxide solutions such as potassium hydroxide (KOH), adsorption with solid inorganic bases such as sodium carbonates Na_2CO_3, and adsorption with solid-supported amines (SSA) such as silica mesocellular foam (Sanz-Perez et al. 2016).

Even though there are technical differences between the aqueous hydroxide solution method and the solid sorbent method, they operate under the same concept of removal of CO_2 from the atmosphere and by contact with a liquid solution or solid surface, followed by a desorption process to liberate attached CO_2. To liberate CO_2, a high temperature will be provided, and a high-purity CO_2 stream will be obtained (Abouelnaga 2022). Once CO_2 is concentrated with the desorption process, this CO_2 can be stored underground, under the ocean, or in unmineable coal and oil fields, or used for enhanced oil recovery (EOR) to recover additional crude oil once primary and secondary oil recovery is completed (Arachchige et al. 2019).

The prime chemical used in the present study was aqueous hydroxides with multiple inter-circular material recovery stages. The main idea behind replenishing liquid chemicals is to overcome the damage to the CO_2 absorption capability with heat degradation. The air contactor is the core of the process, which is used to capture the CO_2 by contacting an aqueous solution with the atmospheric air effectively.

The simplified process flow diagram is shown in Fig. 1. The four main four-unit operations are given in boxes named Air contactor, Causticizer (pellet reactor), Calciner, and Slaker. The main reaction of CO_2 absorption proceeds in the Air contactor unit, where those is converted to potassium carbonate (K_2CO_3) and water (H_2O). Followed by the air contactor, the causticizer unit operation plays another important role in converting K_2CO_3 into KOH by reacting with calcium hydroxide ($Ca(OH)_2$) by creating a by-product of KOH and Calcium carbonate ($CaCO_3$). KOH can be recycled back to the air contactor unit, while $CaCO_3$ will transfer to the Calciner unit, where it is heated up to a higher temperature to produce calcium Oxide (CaO) and CO_2. The CO_2 generated at this stage will be recovered as the main output, which will be considered for sequestration. CaO will follow the Slaker unit to react with H_2O to produce $Ca(OH)_2$, which will then be transferred to the causticizer unit again or next cycle (Keith et al. 2018, Terlouw et al. 2021).

ADVANTAGES AND DISADVANTAGES OF DAC

Plenty of advantages are associated with the aqueous DAC capturing system, which is summarized for further

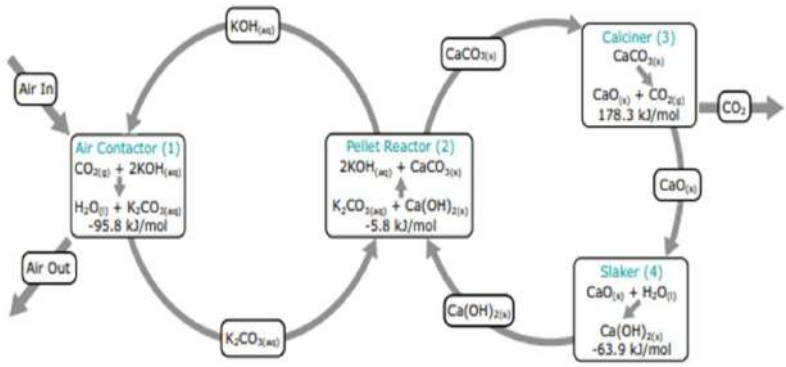

Fig. 1: Process Chemistry and Thermodynamics (Keith et al. 2018).

Table 1: Advantages and disadvantages of the DAC process for carbon removal.

Advantages	Disadvantages
The higher cumulative removal capacity compared to the other carbon dioxide removal technologies (CDR)	Higher Capital cost
The lower area of land required to install and operate the DAC system, and unproductive land can also be used.	Higher Operational cost
The amount of water required is considerably lower than the bioenergy carbon capture and storage (BECCS) mechanism.	The energy requirement to operate the process is significantly larger.
Captured CO_2 can be stored permanently for much longer without any issues.	
According to energy availability, the plant can be located as it does not require any further support or geographical considerations.	
Even with the lower concentration of CO_2, plants will operate to capture the maximum amount of available CO_2.	

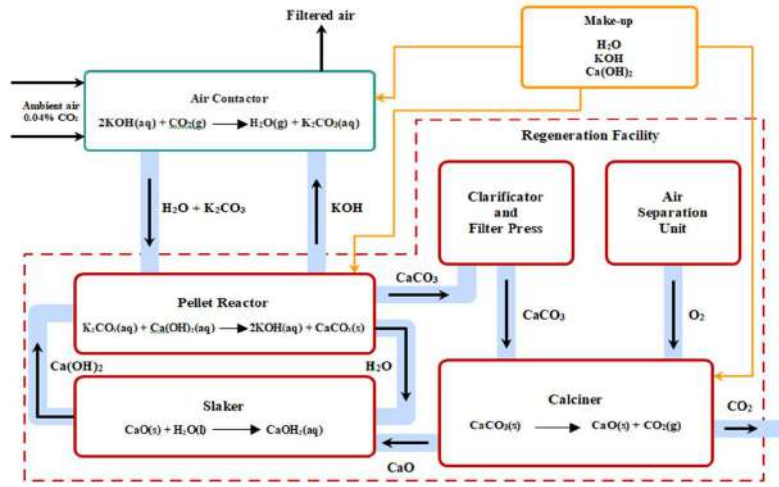

Fig. 2: The process flow diagram with important chemical reactions.

consideration for the optimization process and given in Table 1 (National Academies 2018, Keith et al. 2006, Royal Society 2018).

The main reason behind the higher operational cost is that the atmospheric concentration of CO_2 is around 0.04% compared to the CO_2 percentages in flue gases, such as 13% for coal-fired power plants, 4-5% for gas-fired power plants, and 15-30% for cement industry flue gas (Arachchige et al. 2019). The parameter optimization to minimize the required energy for the operational process of DAC is important. Therefore, the DAC process was developed in Aspen Plus and conducted simulations to identify the optimum parameters, such as inlet air pressure, moisture content of the air, inlet air temperature, liquid solvent temperature, and the packing material used in the reactor.

MATERIALS AND METHODS

The overall process model is designed with the main chemical reactions involving the four major sections: Air Contactor, Pellet unit, which is also called Causticizer, Calciner unit, and Slaker unit. The process flow diagram with important chemical reactions is given in Fig. 2.

Process Modeling

The DAC plant was developed in Aspen Plus V11 with the available literature data and support from Aspen Plus data banks. The most important chemical reactions of the four major sections are given in Equations 1 to 4.

Air Contactor:

$$2KOH_{(aq)} + CO_{2(g)} \rightarrow K_2CO_{3\,(aq)} + H_2O_{(l)} \quad \ldots (1)$$

Pellet Reactor:

$$K_2CO_{3\,(aq)} + Ca(OH)_2(s) \rightarrow 2KOH_{\,(aq)} + CaCO_{3(s)} \quad \ldots (2)$$

Calciner:

$$CaCO_{3(s)} \rightarrow CaO_{(s)} + CO_{2(g)} \quad \ldots (3)$$

Slaker:

$$CaO_{(s)} + H_2O_{(l)} \rightarrow Ca(OH)_{2(s)} \quad \ldots (4)$$

As the property method for calculations in Aspen Plus, ENRTL-RK is used with the Redlich–Kwong equation of state (RK-SOAVE) and Henry's law for vapor phase

Table 2: Operating Conditions and basic parameters of the rate-based model (An et al. 2022).

Unit: Air Contactor	
Property Method	ENRTL-RK
Model	Mixer
Operating Temperature	21°C
Pressure	1 bar
Unit: Pellet Reactor	
Property Method	ENRTL-RK
Model	Crystallizer
Operating Temperature	21°C
Pressure	1 bar
Unit: Calciner	
Property Method	PENG-ROB
Model	RStoic
Operating Temperature	900°C
Pressure	1 bar
Unit: Slaker Unit	
Property Method	PENG-ROB
Model	RStoic
Operating Temperature	300°C
Pressure	1 bar

Table 3: Multiple equilibrium reactions.

Air Contactor
$CO_2 + OH^- \rightarrow HCO_3^-$
$HCO_3^- + OH^- \rightarrow CO_3^{2-} + H_2O$
$2H_2O \rightarrow H_3O^+ + OH^-$
Pellet Reactor
$Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3$
Calciner
$CH_4 + O_2 \rightarrow CO_2 + H_2O$
$CaCO_3 \rightarrow CaO + CO_2$
$2C_2H_6 + 7O_2 \rightarrow 4CO_2 + 6H_2O$
$C_3H_8 + 5O_2 \rightarrow 3CO_2 + 4H_2O$
Slaker
$CaO + H_2O \rightarrow Ca(OH)_2$

properties. Atmospheric air composition and flow rates, as well as the other operating conditions of the DAC model, are given in Table 2. The two-film theory considers the rate-based model, which considers mass transfer analysis at the liquid–vapor interface. Other than the four major chemical reactions, multiple equilibrium reactions, salt formation, and dissociation reactions are happening in the DAC system (Table 3).

The following equilibrium reactions (Table 3), salt formation, and dissociation reactions were considered for the process modeling for the DAC system (An et al. 2022). At the same time, natural gas (NG) combustion is also included in the calcination process.

Process Layout

The key sections of the process comprise four major parts, including the Air contactor, Pellet Reactor, Calciner, and Slaker. The air contactor was designed with the Mixer unit and separator block to represent the carbon absorption process to the liquid solvent stream (Fig. 3). As the air contactor of the DAC process is an innovative component of the real process, Aspen Plus does not provide a specific unit operation model for process implementation.

AC block: Air Contactor Unit

MIX: Mixer for ambient air and flue gas

KOH: Potassium Hydroxide flow rate

To model the air contactor without complex calculations, three different Aspen blocks, a separator, and two mixer units were used to separate CO_2 from atmospheric air. The MIX block is used to mix the ambient air with flue gas CO_2, which comes from the gas turbine exhausts once they have isolated the CO_2. After that, at the AC block, CO_2 will absorb into the liquid solvent, aqueous KOH (lean solvent), composed of 2.0M K^+, 1.10 M OH^-, and 0.45 M CO_3^{2-}. Based on previous studies by Keith et al. (2018), liquid solvent flow rate and atmospheric air flow were extracted for the base-case simulation (Keith et al. 2018). Even though liquid solvent flow is introduced for the air contactor unit, in the real plant, it will come from the pellet unit as a product of the reaction with $Ca(OH)_2$. An open-loop process simulation has been developed to minimize convergence issues and reduce the complexity of the simulation process. The required solvent and air flow rates were taken from previous studies (Keith et al. 2018).

The main objective of this study was to identify optimum parameters to minimize the operational cost of the DAC process. To achieve that, optimization of the Air Contactor unit, specifically considering the amount of CO_2 absorbed by the unit, will increase overall efficiency and reduce the

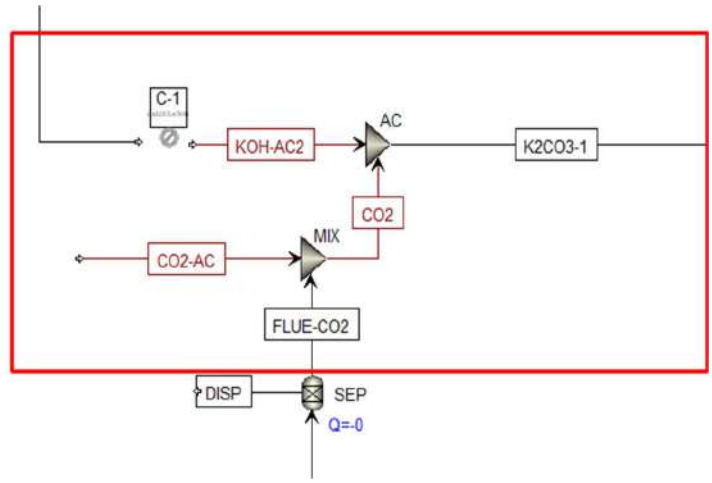

Fig. 3: Air Contactor section.

operational cost. Based on the literature (Keith et al. 2018), the total air inflow is 251,000 $t.h^{-1}$ with 0.06% CO_2 content at 21°C atmospheric pressure (total CO_2 content in the air inflow as 150.6 $t.h^{-1}$).

There are key points to consider for process optimization.

- A: $Ca(OH)_2$ flow rate and parameters to enhance the Pellet reactor operation
- B: Natural Gas (CH_4) flow rate and parameters to enhance the Calciner unit operation
- C: Natural gas (NG) flow rate and parameters to enhance the Slaker process
- D: Atmospheric air flow rate and parameters
- E: KOH solvent flow rate and parameters to enhance the Air contactor unit and the Pellet reactor
- F: H_2O flow rate and parameters to enhance the Slaker Process and CO_2 purification process

Even though water is generated during reaction 1 (air contactor unit), it will not be fully secured for reaction 4 (Slaker unit) due to evaporation and other losses during the operation. Therefore, water must be supplied to the Slaker unit as steam and cold water, and to the CO_2 purification process at the final stage of the DAC process.

Sensitivity Analysis – DAC overall Industrial Scale Plant

The abovementioned A-F, six potential key points for the process optimization, were considered to implement the DAC as a viable option for achieving net zero emission standards.

Optimization of $Ca(OH)_2$ flow rate and parameter optimization, such as the temperature and pressure, as well as the composition of the stream, were considered. With the changes in those parameters, the amount of CO_2 captured at the final stage after the Calciner was analyzed.

As the base case value 229.753 $t.h^{-1}$ rate of $Ca(OH)_2$, which 773 $t.h^{-1}$ of total $Ca(OH)_2$ stream flow rate is considered as that will be the theoretical flow rate required to react with potassium carbonate (K_2CO_3) and produce $CaCO_3$ and CO_2 with 75% capture rate at the air contactor.

It can be noticed that the amount of CO_2 captured decreased with the increase of $Ca(OH)_2$ flow rate (Fig. 4). The main reason for that is that the number of reactants required for the pellet reactor decreases, and it will not produce enough $CaCO_3$. Here, the total $Ca(OH)_2$ flow rate is evaluated in the pellet reactor. The total amount of CO_2 at the final stage of purification after the Calciner unit is given in Fig. 4. It can be concluded that the $Ca(OH)_2$ minimum flow rate should be 760 $t.h^{-1}$ to capture 75% of CO_2 (112.6 $t.h^{-1}$) at the air contactor. However, the temperature and pressure of the $Ca(OH)_2$ do not impact the CO_2 capture, varying from 1-2 bars and 21-45°C.

KOH flow rate, as well as natural gas flow rate, can be reduced to minimize the cost of operation. However, a reduction of KOH will reduce the amount of CO_2 captured at the air contactor and pellet rector, and a reduction of natural gas flow reduces the heat supply to the Calciner unit to recover captured CO_2. Therefore, both are at optimized values, such as 35000 $t.h^{-1}$ KOH flow rate to the air contactor unit and 12.6 $t.h^{-1}$ Natural gas flow rate to the Calciner unit.

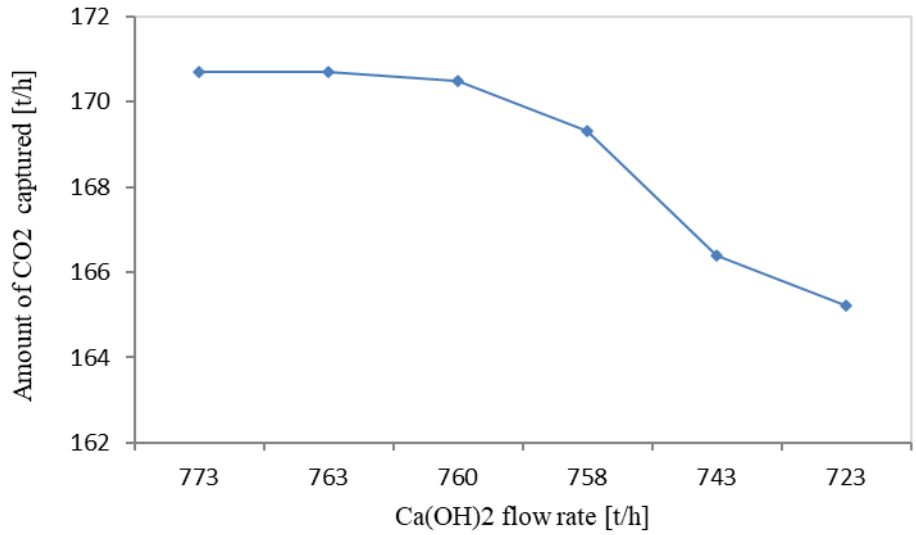

Fig. 4: Effect of $Ca(OH)_2$ flow rate on CO_2 capture efficiency.

Table 4: Operating conditions of rate-based air contactor unit.

Most important parameters for the air contactor unit	
Inlet air flow rate [$t.h^{-1}$	150.6
Inlet solvent flow rate (lean solvent flow rate) [th]	19.08
KOH concentration in the lean solvent [$moL.L^{-1}$]	1.1
K_2CO_3 concentration in the lean solvent [$moL.L^{-1}$]	0.45
Number of air contactor units	6
Reaction condition factor	0.9
Film discretization points	5
Interfacial area factor	1.2
Liquid film discretization points	5
Parameters of packing bed for base case model development	
Packing type	Sulzer 250Y
Packing bed depth [m]	1.28
Packing bed diameter [m]	5.64
Range of Inlet air operating conditions	
Inlet air temperature [°C]	0-40
Inlet air pressure [bar]	1-2
Lean solvent temperature [°C]	0-40
Lean solvent pressure [bar]	1-6
Moisture content [%]	1-4

Sensitivity Analysis – Kinetic Behavior of the Air Contactor

The impact of atmospheric air temperature on the CO_2 removal process was analyzed to identify the effect of climate conditions on the DAC process. Atmospheric air temperature varied from 0°C to 40°C, and pressure varied from 1.1 – 2 atm to identify the behavior of the air contactor unit as the amount of O_2 removal. The base case values for the rate-based model are absorbed from An et al. (2022) and Sabatino et al. (2021). The most important parameters of the inlet air stream and solvent stream are given in Table 4.

Based on the case, an Air contactor unit was developed in Aspen Plus to identify the atmospheric temperature, moisture content of the atmosphere (relative humidity), solvent temperature, and pressure of the inlet air and solvent stream. Moreover, three different packing materials were compared with a slight diameter and depth ratio. For each case, the CO_2 capture rate was calculated to compare the impact of the parameters and select the optimum values for better efficiency; the CO_2 capture rate can be calculated by the difference between the rates of CO_2 entering the column and the rate of CO_2 leaving the system, which is given in equation 5.

$$CO_2 \text{ capture rate} = (\dot{m}_{CO2-IN} - \dot{m}_{CO2-OUT})/\dot{m}_{CO2-IN} \quad \ldots (5)$$

The reaction kinetics were taken from Table 2 with the literature values for the rate of reaction parameters (Bianchi 2018, Pinsent et al. 1956).

Chemical Reactions and Kinetics

The mass transfer phenomenon between the liquid and vapor phases can be simplified in the following section. Those explanations are vital as they constitute the theoretical basis of the liquid and gaseous phase reaction process in the air contactor unit. The mass transfer mechanism can be explained using equilibrium-based stage efficiency and rate-based models.

The chemical kinetics of the main chemical reactions are listed in Tables 5 and 6, along with the equilibrium and kinetic data used for the Aspen Plus model development. For the equilibrium reactions (7-10), the equilibrium constant can be found using Equation 6:

$$\ln K_j = A_j + \frac{B_j}{T} + C_j \ln T + D_j T \quad \ldots(6)$$

Equilibrium Reactions

$$2H_2O \leftrightarrow H_3O^+ + OH^- \quad \ldots(7)$$

$$HCO_3^- + H_2O \leftrightarrow H_3O^+ + CO3^{2-} \quad \ldots(8)$$

$$H_2S + H_2O \leftrightarrow HS^- + H_3O^+ \quad \ldots(9)$$

$$HS^- + H_2O \leftrightarrow S^{2-} + H_3O^+ \quad \ldots(10)$$

Kinetic reactions are listed below, with the kinetic data in reaction numbers 11 and 12. The kinetic Equation used for mathematical calculations is defined in Aspen Plus and given in Equation (13).

Kinetic Reactions

$$CO_2 + OH^- \rightarrow HCO_3^- \quad \ldots(11)$$

$$HCO_3^- \rightarrow CO_2 + OH^- \quad \ldots(12)$$

Table 5: Constant values of equilibrium constant equations.

Parameter	Reaction 7	Reaction 8	Reaction 9	Reaction 10
A_j	132.899	216.049	214.582	-9.742
B_j	-13445.9	-12431.7	-12995.4	-8585.47
C_j	-22.4773	-35.4819	-33.5471	0
D_j	0	0	0	0

Table 6: Rate constant values.

Parameter	Reaction 11	Reaction 12
k_j	4.32e+13	2.38e+17
n_j	0	0
E_j [J.moL^{-1}]	55470913.2	123305447
T_o [K]	298	298

$$r_j = k_j \left(\frac{T}{T_0}\right)^{n_j} \exp[-\frac{E_j}{R}\,(\frac{1}{T} - \frac{1}{T_0})] \quad \ldots(13)$$

RESULTS AND DISCUSSION

Effect of the Inlet Air Temperature and Pressure

The CO_2 capture percentage was calculated with the variation of inlet air temperature and pressure while maintaining flow rate and composition at a constant value (1% H_2O). The temperature of the stream was changed from 0-40°C while the pressure changed from 1.1-2 atm. CO_2 capture at the air contactor unit is shown in Fig. 5. Moreover, the lean solvent temperature is always maintained, like the inlet air temperature during every simulation. Based on the simulation CO_2 capture rate increased gradually with the increase of the inlet air temperature. The outcome has been aligned with the previously reported analysis, which is given in Keith et al. (2018), which is 21°C temperature and 1 bar pressure, giving 75% removal efficiency.

However, the air pressure has no significant impact on CO_2 capture. The main reason is that the rate of chemical reaction is only dependent on the temperature. However, the CO_2 removal percentage decreased with increased pressure at lower temperatures. The main reason for this is that with high pressure, the percentage of air contacting with the liquid sorbent will reduce due to the higher pressure. Therefore, some air will not have enough contact time with the solvent for the reaction.

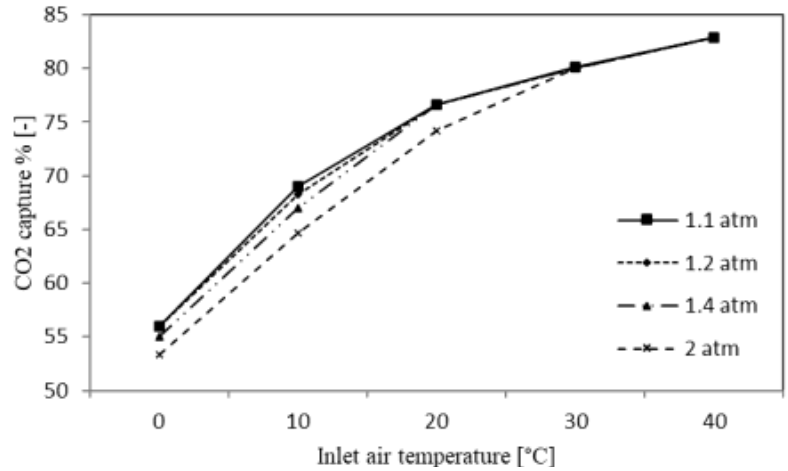

Fig. 5: Effect of inlet air temperature on CO_2 capture efficiency at different pressures.

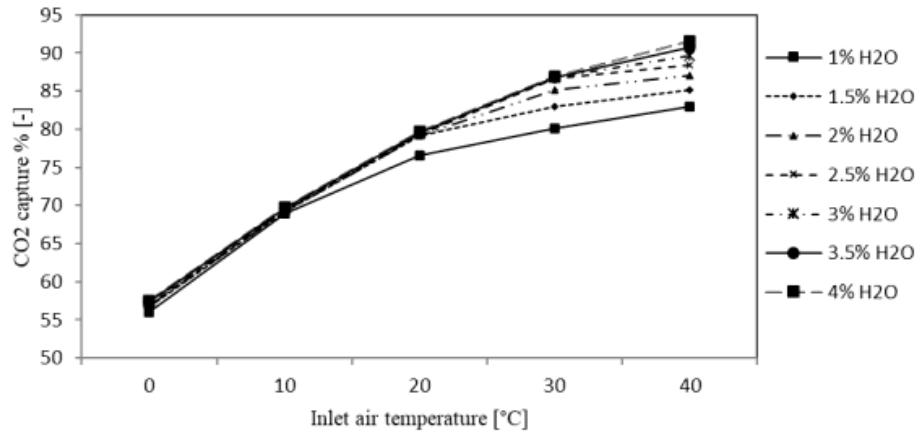

Fig. 6: Impact of Inlet Air Temperature on CO_2 Capture Efficiency Across Different Moisture Levels.

Effect of the Moisture Content of the Air

The CO_2 capture percentage was calculated with the moisture content of the inlet air. Moisture content varied from 1% to 4%, and the impact was analyzed by changing the air temperature from 0 to 40°C, as shown in Fig. 6.

According to Fig. 6, it can be seen that the moisture content of the air has a positive correlation with the CO_2 capture percentage. The highest CO_2 capture rate of 92% is given at 40°C, and 4% H_2O content is in the inlet air. At the same time, the CO_2 capture rate gradually increased with the temperature increase for every case. However, there is no significant variation in the CO_2 capture rate with the moisture percentage in the inlet air for a given temperature. Based on the analysis, it is obvious that the CO_2 capture rate will drastically drop during the winter period. However, this analysis assumes that liquid solvents will always be kept at the same atmospheric air temperature. The water in the solvent leads to water dissociation, forming more carbonate and bicarbonate at higher temperatures. The formation of more OH^- ions eventually increases the molarity, increasing the CO_2 capture rates. The results are aligned with the previous experiments, which have been published and identified that relative humidity is less significant for the DAC process (An et al. 2022).

Effect of the Liquid Solvent Temperature

However, carbon capture rate variation has been analyzed with the assumption of maintaining the solvent temperature at 21°C for the entire period. Even though atmospheric air temperature drops to 0°C, the liquid solvent supply to the air contactor unit will maintain 21°C. Fig. 7 represents the variation of the CO_2 capture rate with the air temperature when the solvent temperature is maintained at 21°C.

When the atmospheric temperature is 0-10°C, there is a slight increment in the CO_2 capture rate as the solvent temperature is higher than the air temperature. However, maintaining solvent temperature at a much higher value than atmospheric temperature will eventually increase energy consumption. Therefore, the percentage of increase in CO_2 capture rate is not significant enough compared to the energy cost. The results are aligned with the previously published results by Keith et al. 2018, which considered 21°C as the solvent temperature for multiple changes of other parameters in the DAC process.

Effect of the Packing Material

The packing section in the absorption column plays a vital role in CO_2 absorption by providing a surface area for gas and liquid phases to come into contact with the chemical reactions. Structured packing is considered for the simulation

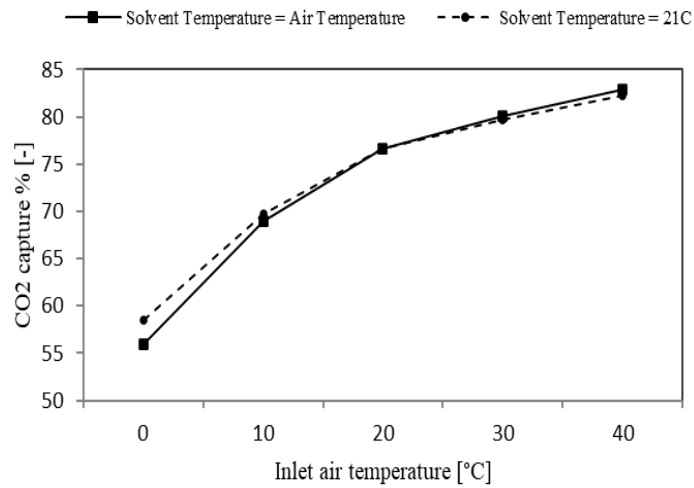

Fig. 7: Impact of Inlet Air Temperature on CO_2 Capture Efficiency at constant solvent temperature

Table 7: Information about the packing materials.

Packing Type	Size	Area [$m^2.m^{-3}$]	Voids%]	C1	C2	C3	Vendor	Reference
Mellapak	250Y	250	98	1	1	0.32	Sulzer	(Stichlmair et al. 1989)
Mellapak	350Y	350	98	1	1	0.32	Sulzer	(Stichlmair et al. 1989)
BX	-	450	86	15	2	0.35	Sulzer	(Stichlmair et al. 1989)

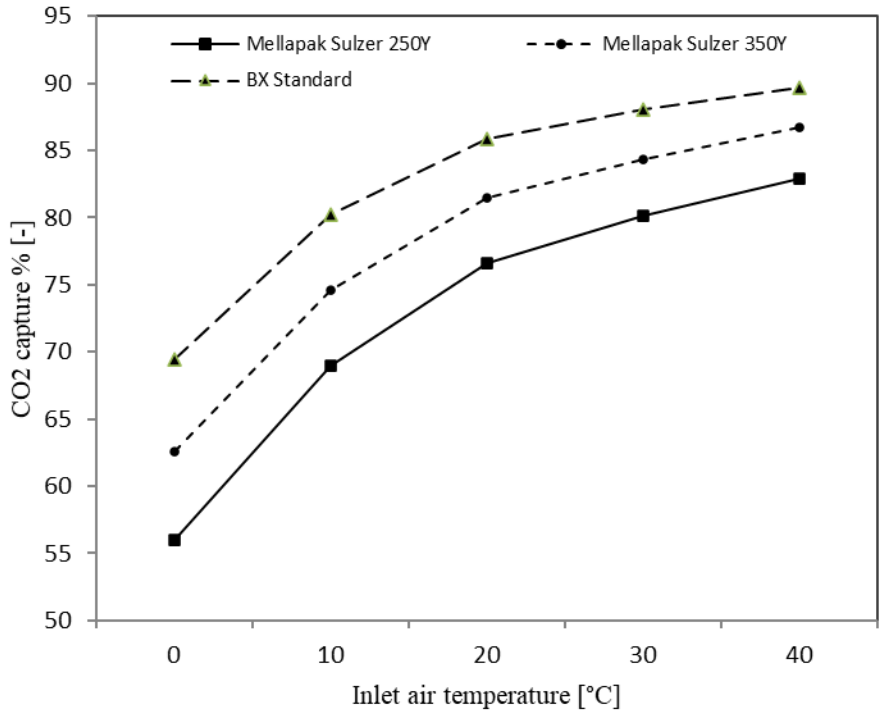

Fig. 8: CO_2 capture variation with temperature.

due to the higher mass transfer coefficient than the random packing. The most important parameters for selecting packing materials are void fraction and surface area for the reaction. Aspen Plus uses the Stichlmair Correlation for pressure drop calculations, which consists of three Stichlmair coefficients (Stichlmair et al. 1989). For the mass transfer calculations, information was taken from Bravo et al. 1985 during the simulation (Bravo et al. 1985).

Mellapak 250Y, Mellapak 350Y, and BX standard packing were considered for the analysis. During the simulations, base case values of air intake and solvent were maintained with a 1% moisture content in the atmospheric air. The CO_2 removal efficiency was calculated for each case with the five different temperature values. Information about the packing materials is given in Table 7.

CO_2 capture variation with temperature is given in Fig. 8. According to the figure, BX packing shows the highest CO_2 removal efficiency, followed by Mellapak 350Y and Mellapak 250Y. The main reason is that the higher surface area of the packing material dominates the absorption process. When the surface area is high, the available contacting area for gas–liquid reaction is higher. With the temperature increase, the CO_2 removal efficiency increased for all packing materials. The CO_2 removal efficiency is around 12% higher for the process with Mellapak 350Y and 24% higher for the process with BX packing than the Mellapak 250Y process.

However, the cost of the packing material should also be considered for the techno-economic evaluation, as that will have a significant role in capital cost calculation. Therefore, the packing material cost is also considered to identify the optimum packing material for the air contactor unit.

Moreover, packing dimensions are also considered for the process optimization, as the gas absorption process is the key component of the DAC operation. The height of the two theoretical stages, as well as the diameter of the packing bed, was considered for the simulations. However, the packing diameter impact on CO_2 capture is not significant enough to overcome the cost of the packing material.

CONCLUSIONS

This research highlights the critical role of optimizing key parameters in DAC systems to enhance CO_2 capture efficiency. The results reveal that maintaining a $Ca(OH)_2$ flow rate of 760 $t.h^{-1}$, an inlet air pressure of 1.1 atm, and utilizing packing materials with high surface area significantly boost performance. With the temperature increase, the CO_2 removal efficiency increased for all packing materials. The CO_2 removal efficiency is changed with different packing materials and is around 12% higher for the process with Mellapak 350Y and 24% higher for the process with BX packing than the Mellapak 250Y

process. Although increased moisture content improves CO_2 absorption, careful consideration is required to manage the energy demands of maintaining solvent temperatures. The operational cost, capital cost, and energy requirement for the DAC process must be analyzed with the experimental setup and validated with the simulation results. Future studies should prioritize techno-economic analyses, the integration of renewable energy sources, and strategies for scaling up to identify cost-effective approaches for large-scale DAC implementation.

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Distribution and Health Risk Assessment of Benzo[a]pyrene in Street Dust of Raniganj in Eastern India

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ABSTRACT

This study investigates the seasonal and spatial distribution of benzo[a]pyrene (BaP) in street dust across Raniganj, revealing significant variations linked to both seasonal shifts and land use types. BaP concentrations in street dust samples ranged from 82.2 ng.g^{-1} to 531.6 ng.g^{-1}, with a mean value of 262.45±75.55 ng.g^{-1}. The highest BaP levels were observed during winter, particularly in heavy traffic, coal mines, and industrial areas, suggesting contributions from industrial activities and vehicular emissions, coal chemical production, and gangue accumulation. An analysis by land use type indicated that BaP levels were highest in busy traffic areas, coal mine areas, and industrial areas, with traffic-congested sites showing the highest average concentration (328.29 ng.g^{-1}). Seasonal analysis showed that winter BaP concentrations were the highest on average (336.28±93.43 ng.g^{-1}), followed by monsoon and summer. These seasonal differences may be due to winter-specific factors, such as increased vehicular traffic, indoor heating, and atmospheric stability. In all five sampling locations, the hazard index (HI) values were moderate for both adults and children. Adults had an average overall cancer risk value of 2.89E-03, whereas children had an average of 2.61E-03, indicating that both age groups are at high risk. Samples collected from various land use types revealed a distinct difference in mean total BaP levels, as well as total cancer risk levels, with the following order observed: busy traffic area > coal mine area > industrial area > commercial area > residential area. The findings underscore the impact of anthropogenic activities and seasonal changes on BaP levels, emphasizing the need for targeted pollution management strategies in heavy-traffic and industrial regions, along with coal mining regions in Raniganj.

INTRODUCTION

Benzo(a)pyrene (BaP) is a polycyclic aromatic hydrocarbon (PAH) that has extensive biological as well as environmental significance. It is a high-molecular-weight PAH consisting of five fused benzene rings. BaP is highly stable, nonvolatile, and non-biodegradable, making it a persistent environmental pollutant (Rajput et al. 2024, Kislay et al. 2024, Ma et al. 2022). It is highly recognized for its potent carcinogenic and mutagenic properties. Owing to its high toxicity and prevalence in nature, BaP is often used as a marker of PAH pollution in the environment (Souza et al. 2016). Similar to other PAHs, BaP is emitted into the environment via several anthropogenic sources. It is mainly a byproduct of incomplete combustion of organic materials like burning of fossil fuels, vehicular emissions, domestic heating, burning of wood, various industrial activities, tobacco smoking and grilling/charring of food materials (Bukowska et al. 2022, Hellén et al. 2017, Srogi 2007). A small amount of BaP is also released into the

environment via natural processes, such as forest fires and volcanic eruptions. Once released into the environment, BaP tends to adhere to the atmospheric particulate matter as well as to the sediments of aquatic systems, where it can persist for a long time because of its hydrophobic nature and chemical stability (Maletic et al. 2019, Hussain et al. 2018). Atmospheric dry and wet deposition and the runoff process distribute BaP in soil, street dust, and water bodies, from where it enters food chains and starts bioaccumulating in the bodies of organisms (Saravanakumar et al. 2022). Due to its greater surface area and smaller particle size, street dust acts as a good harbor for BaP accumulation (Franco et al. 2017). It has a crucial direct connection to humans in day-to-day life, thus making it an important environmental matrix to investigate BaP concentration.

Ingestion, inhalation, and skin contact are the three main exposure routes through which BaP can enter the human body (Ali et al. 2021). A significant source of BaP exposure in humans can be food, particularly if it is burned or smoked. Through intricate biotransformation processes, cytochrome P450 enzymes found in the liver and other tissues gradually activate BaP into highly reactive compounds (Hand 2001). Among the several metabolites formed, BaP-7,8-diol-9,10-epoxide is a potent mutagen that can form a covalent link with DNA and produce adducts that disrupt regular cellular functions, such as replication, and induce mutations (Koh & Pan 2024). BaP is a significant material in cancer research because some of these DNA adducts have been linked to the early stages of cancer formation, specifically in the lung, liver, skin, and bladder (Zhao et al. 2024, Das & Ravi 2022). According to various epidemiological studies, BaP exposure can cause several other medical issues, such as cardiovascular diseases and developmental problems in children (Alias et al. 2022). It can impair immunity, making it more difficult for the body to eliminate several illnesses. BaP can adversely affect the female reproductive system, leading to lower birth weights, delayed child development, and decreased fertility (Jorge et al. 2021). BaP may also cause chromosomal defects, leading to genetic instability.

Several researchers throughout the globe have thoroughly researched PAHs on the street and their related health effects (Dytlow et al. 2025, Adeniran et al. 2025, Li et al. 2025). This research indicates increasing concerns over the occurrence and sources of PAHs in street dust. Similar to global trends, third-world nations such as India are becoming increasingly concerned about PAH concentrations in street dust (Nayak et al. 2023). PAHs in street dust have been found to contribute to severe human health risks through their carcinogenic, mutagenic, and toxic nature. Among these compounds, BaP is the most active and dangerous PAH and is frequently used as an indicator for evaluating the environmental toxicity of the overall PAH mixture (Kumar et al. 2013). Owing to the high impact of BaP on human health, the International Agency for Research on Cancer (IARC) has classified it as a Group 1 carcinogen. BaP is also regulated under Section 112(b) of the 1990 Clean Air Act Amendments, which is classified as a hazardous air pollutant (HAP) within the category of substances known as polycyclic organic matter, as 7-PAH (USEPA 2006). Several regulatory bodies worldwide have set limitations and guidelines for BaP exposure because of its ability to induce potential health impacts. According to the Canadian Council of Ministers of the Environment, BaP levels in residential, industrial, and agricultural soil are allowed to be between 0.015 to 0.7 $mg.kg^{-1}$, 0.7 to 2.1 $mg.kg^{-1}$ and 0.1 $mg.kg^{-1}$, respectively (CCME 2010). In India, the proposed ambient air limit for BaP is 1.0 $ng.m^{-3}$ (Gazette of India 2009). However, the situation concerning BaP remains challenging despite restrictions and regulations, particularly in regions where high levels of industrial and vehicular emissions are not effectively managed. The use of various advanced technologies and thorough monitoring of BaP levels in all environmental matrices is crucial for mitigating its long-term effects on the environment (Sushkova et al. 2016).

According to the Ministry of Coal, Government of India, 64 coalfields are currently in operation in India, with an estimated reserve of approximately 378.21 billion tonnes (National Coal Inventory 2023). Of these, 33.93 billion tonnes of the country's total coal resources belong to the state of West Bengal. Raniganj Coalfield, on the western bank of the Damodar River, is among the oldest and most historically notable coalfields in India (Mondal et al. 2018). Coal mining operations, such as extraction methods, disposal of mining waste materials, and coal processing residues, have been identified as the principal sources of PAHs in coal mine areas (Chen et al. 2019, Qian et al. 2022). Masto et al. (2015) analyzed the concentration of PAHs in the surface soils of underground mines in this region. Unfortunately, there have been no follow-up studies on PAHs in other environmental media, especially street dust. As a critical sink for PAHs and a potential health hazard due to exposure via numerous pathways, it is crucial to examine the levels of various PAH types in this matrix. Moreover, few studies have been conducted in India regarding BaP levels in ambient air (Garg et al. 2022), and no research has been conducted on the level of BaP in street dust. This is an important research gap considering BaP's toxicity and carcinogenic potential. With these research gaps taken into account, this study aims to (a) conduct a bibliometric analysis to identify research trends in India within the existing literature, (b) assess BaP concentrations by comparing seasonal variations (summer, monsoon, and winter) and spatial differences across

various land-use types, including residential, commercial, industrial, coal mining, and busy traffic areas, (c) assess the carcinogenic health risk and hazard index (HI) for two different age groups (adults and children), and (d) conduct a comparative analysis between the present findings and previously reported studies across the globe.

MATERIALS AND METHODS

Methodology: Bibliometric Analysis

The research data were collected on the 25th of February 2025 from the Web of Science. The database used was Web of Science Core Collection, and a search was done using the Advanced Search Query Builder (Laha et al. 2024, Pal et al. 2025, Gupta et al. 2024b). The search query term was as follows: TS=(PAHs) OR TS=(polycyclic aromatic hydrocarbons) OR TS=(BaP) OR TS=(Benzo[a]pyrene) AND TS=(road dust) OR TS=(street dust) AND TS=(India). Here, TS refers to the topic field. A total of 117 publications were available up to the date of the search. The authors used VOS viewer – a data analysis and visualization software first developed at the Centre for Science and Technology Studies, Leiden University, The Netherlands (Gupta et al. 2024b, Koley et al. 2023, Van Eck et al. 2010). In particular, this was a method of analyzing keyword co-occurrence networks, following the search trends in the recent literature, and pinpointing the core clusters within the literature.

Bibliometric analysis was performed using VOSviewer software to create a co-occurrence map using bibliographic data scraped from the Web of Science database advanced search. The co-occurrence relationships between the various keywords were emphasized, with all keywords selected as a unit of analysis. A full counting method was used, that is, every occurrence of a keyword was counted equally (Gupta et al. 2024a, Koley et al. 2024). To maintain accuracy and comprehensibility, the empowerment or feeding of the system was decided to be stopped at the fourth share, that is, where a keyword has at least three counts. Of the 690 keywords and co-occurrence relationships under study, 124 keywords maintained this threshold, and 66 words were eventually chosen for visualization and analysis.

Study Area

The study was conducted in the Raniganj region, situated in the Asansol-Durgapur industrial belt within the Paschim Bardhhaman district in the state of West Bengal, India. Mining operations in the Raniganj Coalfield began in 1774AD and continue extensively today. The coalfield spans areas in West Bengal and Jharkhand. Within West Bengal, it primarily covers parts of the Bardhaman district, with adjacent regions extending into the Birbhum, Bankura, and Purulia districts. In Jharkhand, portions of the coalfield are situated in the districts of Dhanbad and Santal Pargana (Manna & Maiti 2018). Coal mining, steel production, sponge iron production, and other industries contribute to diverse environmental challenges that heavily influence the region's economic landscape. Fifteen distinct sampling sites were chosen from five distinct land use patterns: residential, commercial, industrial, coal mine, and busy traffic areas (Fig. 1).

Street dust was collected during three different seasons (summer, monsoon, and winter) during the day on weekdays. Three sampling locations separated by one meter were chosen for each land use. Approximately 500 g of samples

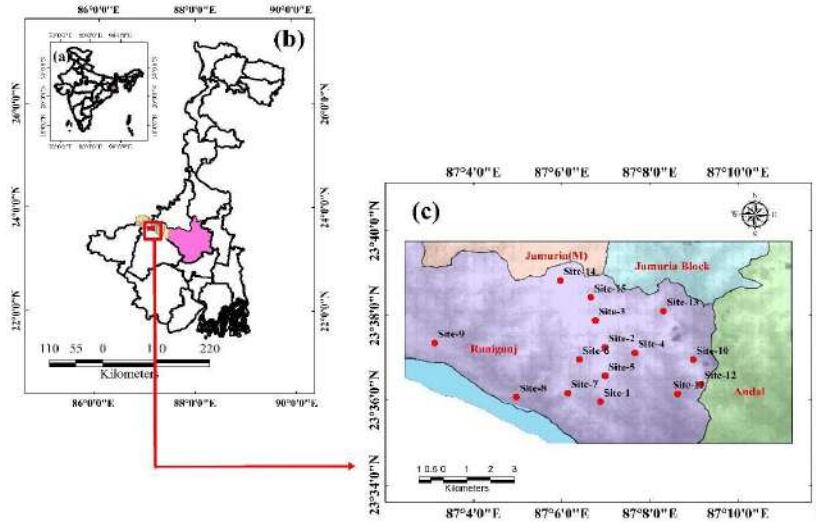

Fig. 1: Location map of the study area.

were collected from each sampling location and then combined to create a single composite sample. Three composite samples were collected from each land use type. To prevent the resuspension of extremely fine particles during the sampling process, samples were collected using a brush and swept directly into plastic bags (Gope et al. 2018). The collected samples were then cleared of extraneous items such as cigarette butts, tiny gravel, discarded plastics, metal scraps, and demolished building debris. The samples were sieved to a particle size of less than 63 µm (Gope et al. 2020). They were then kept in zip-locked plastic bags in a refrigerator at a temperature of less than 4°C until analysis.

Extraction and Analysis of PAHs

For the analysis of PAHs, samples were extracted in a Soxhlet apparatus using a 10:1 toluene: methanol mixture, as per the USEPA method 3540C (USEPA 1996a). Approximately 10 g of the sample was placed in an extraction thimble and placed in a Soxhlet apparatus, and 300 mL of the extraction solvent was placed in a 500 mL round-bottom flask containing two or three clear boiling chips. The flask was then attached to the extractor, and samples were extracted for 16-12 hours at 4-6 cycles/hour. After extraction, the samples were cooled, reduced to 2 mL using a rotary evaporator, filtered, and cleaned using silica gel column chromatography according to the USEPA method 3630C (USEPA 1996b).

After cleanup, the extracts were concentrated to 1 mL using a gentle flow of nitrogen. GC-FID (Agilent 8890-GC) analysis was performed to determine the PAHs in the extracts according to the USEPA method 610 (Hishamuddin et al. 2023). For the GC system, the initial oven temperature was 140°C (held for 3 min), then raised from 140°C to 250°C at 6°C.min^{-1} (held for 6 min), then further to 300°C at 5°C.min.$^{-1}$, and finally held for 5 min. With a total flow of 24 mL.min^{-1} and a pressure of 7.0856 psi, the injector was set to 320°C in the split mode with a split ratio of 20:1. The septum purge flow was 3 mL.min^{-1}. The FID heater was operated at 320°C and 30 mL.min^{-1} of hydrogen gas flow. The makeup flow (N_2) was 28.8 mL.min^{-1}. A typical HP-5MS capillary column (30 m length, 320 µm diameter, and 0.25 µm film) from Agilent was utilized. The injection volume was set to 1µL with a column flow of 1 mL.min^{-1}, and the USEPA-recommended 16 PAHs were analyzed.

Health Risk Assessment

A risk assessment model was developed by the United States Environmental Protection Agency (USEPA 2002, USEPA 1989) to ascertain the risk to both adults and children. In this model, humans are exposed to contaminated road dust particles through three major pathways: (1) ingestion via oral intake, (2) dermal absorption via skin contact, and (3) inhalation via the respiratory tract. The chronic exposure risk for benzo [a]pyrene (BaP) was determined from the average daily dose (ADD) calculated for these various exposure pathways.

$$ADD_{Ingestion} = \frac{CS \times IR_{Ingestion} \times EF \times ED}{BW \times AT \times 10^6} \quad \ldots(1)$$

$$ADD_{Dermal} = \frac{CS \times SA \times AF \times ABS \times EF \times ED}{BW \times AT \times 10^6} \quad \ldots(2)$$

$$ADD_{Inhalation} = \frac{CS \times IR_{Inhalation} \times EF \times ED}{BW \times AT \times PEF} \quad \ldots(3)$$

Where CS is the BaP concentration in street dust (ng.g^{-1}), and BW is the body weight (kg). AT is the average lifespan (days). EF is the exposure frequency (day.year^{-1}). ED is the exposure duration (in years). $IR_{Inhalation}$ is the inhalation rate (m^3.day^{-1}). $IR_{Ingestion}$ is the soil intake rate (mg.day^{-1}). SA is the dermal surface exposure (cm^2). AF is the dermal adherence factor (mg.cm^{-2}). ABS is the dermal adsorption fraction of the chemical. PEF is the particle emission factor (m^3.kg^{-1}). 10^6 is the conversion factor. The values of the variables are listed in Supplementary Table 1 for both adults and children.

The level of danger for BaP was determined by the ratio of ADD (for three different exposure routes) to the reference dose (RfD).

$$HQ = \frac{ADD}{RfD} \quad \ldots(4)$$

The reference dose (RfD) is defined as the estimated daily human exposure to an agent that is unlikely to cause adverse health effects in humans. It is based on the best available science and is intended to protect both present and future generations from the development of any potential pathological changes or diseases (Kosheleva et al. 2023). Reference doses have been estimated for BaP for the three exposure routes. For ingestion and dermal exposure, it is 3×10^{-4} mg.kg^{-1} (USEPA 2002), and for inhalation, it is 2×10^{-6} mg.kg^{-1} (USEPA 1989).

$$HI = \sum HQ \quad \ldots(5)$$

The HI indicates the extent to which combined health hazards are presented by several exposure pathways through which hazardous dust particles can enter the human body. Four categories of negligible (<0.1), low (0.1-1), moderate (1-10), and high (>10) health risk levels are derived from the overall HI (Kosheleva et al. 2023, USEP 2002).

To assess the age-specific potential cancer risks (children and adults) of human exposure to BaP, the incremental lifetime cancer risk (ILCR) model is used (USEPA 1989, USEPA 2002)

$$ILCRs_{(Ingestion,inhalation,dermal)} = ADD \times CSF \times \sqrt[3]{\frac{BW}{70}} \quad \ldots(6)$$

Where CSF is the cancer slope factor in $mg.kg^{-1}.\ day^{-1}$ for the three different exposure routes.

Total Crcinogenic Risk=

$$ILCR_{Ingestion} + ILCR_{Inhalation} + ILCR_{Dermal} \quad \ldots(7)$$

Generally, the acceptable risk range for total carcinogens is set to 10^{-6} to 10^{-4} by the USEPA (USEPA 2001), and risks below 10^{-6} do not require any additional action, while risks above 10^{-4} are considered to be of concern and require further action to reduce the exposure and resulting risk (USEPA 2008).

Preparation of the Spatial Pattern of BaP

Inverse Distance Weighting (IDW) is a spatial interpolation technique that is prepared in a GIS (Geographical Information System (GIS) environment to plot the missing value in a region using known data points (Lu 2008). In this study, spatial interpolation was a beneficial method not only to identify the distribution of BaP in dust or carcinogenic risk distribution, but also to correlate with other components. The major principle of IDW is that the prediction capacity of a known value decreases with increasing distance (Moussa & Abboud 2024, Achilleos 2008). The estimated value has been calculated with the help of the following formula.

$$Z_s = \frac{\sum_{i=1}^{n=1} w_i Z_i}{\sum_{i=1}^{n} w_i} \quad \ldots(8)$$

Where, Z_s = Estimated value of unknown location, Z_i = Known neighboring value, W_i = Weight assigned to each known point, n = Number of neighboring points.

The weight is computed as

$$W_i = \frac{1}{d_i^p} \quad \ldots(9)$$

Where, d_i = distance between the known and unknown point, p = Power value regulating the effect of distance (typically set between 1 and 3). IDW is a widely used interpolation method in GIS applications due to its simplicity and effectiveness. But before that, we must make a strong data set to show this spatial variability.

IDW is a popular method of interpolation. This helps to determine the spatial variability of components in a more or less accurate manner. However, there are several drawbacks to this method; for example, it assumes that the spatial relationship is not the same in all directions, so it does not address directional trends (Barbulescu & Saliba 2024). Moreover, the precision of the IDW depends on the density and distribution of the sample points. If the points are more random, there will be an error in that area (Achilleos 2011). IDW offers no indication of a forecast as other methods do (Achilleos 2008, Benmoshe 2025). IDW ignores topographic and physical barriers that may affect spatial variation, as it only depends on mathematical distance (Lu 2008). But it may be a very useful tool for air-water quality analysis, as we have done in this research (Jumaah et al. 2019, Yadav & Ganguly 2024)

RESULTS AND DISCUSSION

Current Scenario of BaP in Street Dust: A Bibliometric

A bibliometric overview of research on polycyclic aromatic hydrocarbons (PAHs) in Indian street dust identified seven distinct thematic clusters (Fig. 2). Cluster 1 (Red) consists of 27 keywords such as "PAHs", "polyaromatic hydrocarbon", "source apportionment", "PM_{10}", "$PM_{2.5}$", "black carbon", "emission", and "urban site" This group is basically concerned with territories which link the PAHs with the particulate matter (PM_{10} and $PM_{2.5}$) in urban locations. Major sources mainly are emissions from vehicle exhaust gases, such as industrial activity, while on the other side, there can be emissions due to biomass burning and burning of fossils and fuels. In the name of source apportionment, efforts have been made to determine the main sources of PAHs in the environment. Soot is indicated by "Black carbon," meaning that this research was conducted on them through which PAHs will be available in indoor air quality. The term "urban site" is used to signify that PAH contamination is ubiquitous in highly populated areas, thereby indicating that traffic and industrial emissions are high. This cluster focused on determining the presence of PAHs in street dust, soil, and sediments and their effects on human health and ecological systems. PAHs accumulate in surface sediments and road dust, and exposure through inhalation, ingestion, and dermal contact is potentially risky.

Cluster 2 (Green) includes 24 items, i.e., "street dust", "surface dust", "indoor dust", "surface sediments", "urban street dust", "soil", "road dust", "health risk assessment", "ecological risk" and "human exposure". This cluster has focused on determining how PAHs are present in street dust, soil, and sediments and their effects on human health and ecological systems. PAHs accumulate in the surface sediments and road dust, in which exposure through inhalation, ingestion, and dermal contact is potentially risky. "Indoor dust" suggests the possibility that PAHs move into homes from outside and thus induce a higher risk for longer exposure. In this cluster, studies assess ecological risk assessments and levels of human exposure to understand the long-term effects of PAH contamination. Cluster 3 (Blue):

21 items comprise "street dust", "contaminated soil", "urban soil", "sediments", and toxic metals such as "Ni, Pb, Cu, and Cd". The presence of heavy metals like Nickel (Ni), Lead (Pb), Copper (Cu), and Cadmium (Cd) is indicative of the fact that PAH pollution is generally accompanied by metal pollution. The sources for these metals comprise industrial discharge, vehicular emissions, and urban runoff. Heavy metal pollution thus poses a serious threat to environmental and human health. The correlation between PAHs and heavy metals in urban soils gives credence to the possibility of synergistic toxicity, which aggravates the pollution. The studies in this cluster aim to study the levels of contamination in the soil, source term, and remediation measures.

Cluster 4 (Yellow): 17 items include "city", "impacts", "pollution indices", "accumulation", "urban soil", "roadside" and "Kolkata". The studies of this cluster examine the urban pollution burden of some leading Indian cities with a pronounced focus on Kolkata. The use of terms like "pollution indices" and "accumulation" indicates that quantitative evaluations of PAH pollution rely on various indices to determine the severity of contamination. The term "roadside" is indicative of traffic emissions as a major source of PAHs. Referral to "Kolkata" is indicative that studies here are focused on studying the sources and the level of various kinds of pollution in the West Bengal capital, which is an industrialized and densely populated area. Cluster 5 (Purple)-13 words include "diagnostic ratio", "health risk", "agricultural soil", "river" and "surface soil". The term "diagnostic ratio" doesn't reveal much about the type of research conducted in this cluster except that fingerprinting techniques are used to differentiate between two sources of PAHs, i.e., pyrogenic (combustion) and petrogenic (oil). The mention of "agricultural soil" and "river" in this cluster indicates that PAHs are transported out of urban centres into agroecosystems and river ecosystems and thus potentially threaten food chains through bioaccumulation and pollution of these waterways. The "health risk" in this cluster refers to PAHs exposure via contaminated soil and water in places where agricultural use is widely prevalent. Cluster 6 (Sky Blue) contains 12 items, including "carcinogenic risk", "hazard index", "source profile" and "hazard quotient". Research in this cluster mainly relates to the risk assessment methods used for evaluating PAHs-related cancer and non-cancer health risks. The mention of "children" is indicative of the concerns regarding the vulnerable population, because the children are at great risk of PAHs exposure, as they tend to put their hands in their mouths and are also exposed to contaminated dust for prolonged periods. The terms "hazard index" and "hazard quotient" point to quantitative methods of assessing levels of toxicity to define safe exposure limits. This cluster relies heavily on health interventions to derive public health policy concerning PAH risk. The last cluster (Cluster 7, Orange), containing 7 items, includes "Delhi", "West Bengal", "soils", "heavy metals" and "source". The series of regional studies on PAHs clustered here focuses on places with peak industrial and urban activities: Delhi and West Bengal. As India's capital, Delhi is notorious for immense pollution from vehicle emissions, industrial sources, and biomass burning, which has given rise to severe air quality problems. On the other hand, West Bengal has

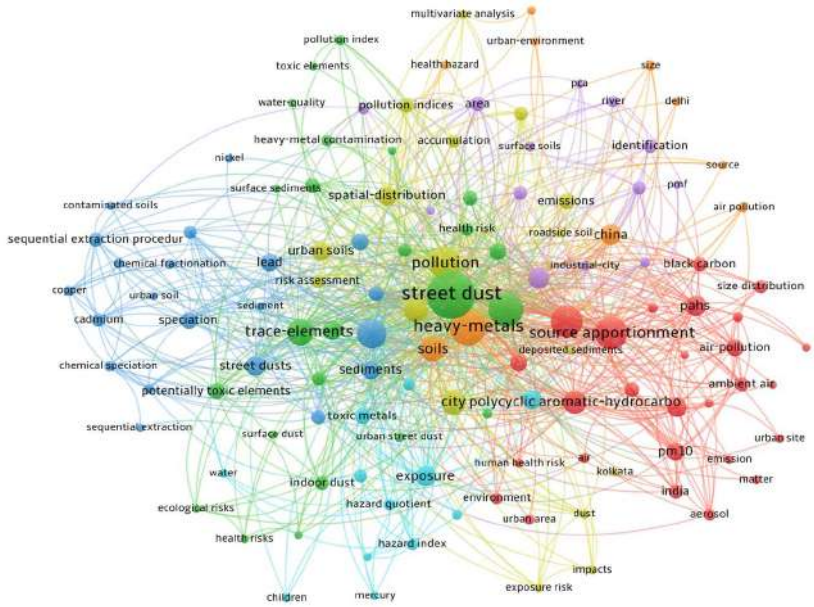

Fig. 2: Co-occurrence network.

numerous industrial bases with a huge population density that suffers heavy soil and air contamination from both PAHs and heavy metals. The keyword "source" indicates the research being done to identify the major sources responsible for PAHs pollution in the location selected.

Seasonal Variation of BaP

The total BaP content in the street dust of Raniganj ranged from 82.2 ng.g^{-1} to 531.6 ng.g^{-1} with a mean value of 262.45±75.55 ng.g^{-1}. A significant variation in BaP level was observed among different sampling sites across the study area in three different seasons (Fig. 3a, 3b, 3c). During winter highest BaP concentration was found at site 9 (531.60 ng.g^{-1}), followed by site 13 (430.00 ng.g^{-1}), site 14 (398.52 ng.g^{-1}) and site 7 (395.00 ng.g^{-1}). In summer, site 9 (290.63 ng.g^{-1}), site 11 (278.43 ng.g^{-1}), site 13 (273.80 ng.g^{-1}), and site 12 (263.25 ng.g^{-1}) showed the highest level of BaP among all the sites. While in monsoon, a maximum level of BaP was detected in site 9 (476.70 ng.g^{-1}), followed by site 13 (351.67 ng.g^{-1}), site 15 (325.90 ng.g^{-1}) and site 14 (321.70 ng.g^{-1}). In winter seasons, elevated levels of BaP in their respective sites might be due to various industrial activities (steel, sponge iron, cement, food processing) as well as vehicular exhaust emissions because of high traffic loads near some sites (Kosheleva et al. 2023). But in the monsoon, higher BaP levels in those sites can be attributed to an increase in the number of transportation activities and runoff of contaminants, along with wind deposition from surrounding polluted areas. The lowest BaP concentration was found at site 2 during winter and monsoon, whereas at site 1 in summer. The mean BaP concentration of this study area was compared with some other studies across the world. The mean BaP value was similar to the values observed from Moscow, Russia (Kosheleva et al. 2023), Kuala Lumpur, Malaysia (Hishamuddin et al. 2023), Karaj, Iran (Qishlaqi & Beiramali 2019), Trinidad, Caribbean (Mohammed et al. 2018) and Xuzhou, China (Wang 2018). The mean concentration of BaP, on the other side, was much lower than the values reported from Shenzhen, China (3089.07 ng.g^{-1}) (Ning et al. 2023), Ibadan, Nigeria (2528 ng.g^{-1}) (Yusuf et al. 2022), Jeddah, Saudi Arabia (1513.51 ng.g^{-1}) (Shabbaj et al. 2018), Tokyo, Japan (2100 ng.g^{-1}) (Khanal et al. 2018) and Ulsan, Korea (6310 ng.g^{-1}) (Lee and Dong 2010) whereas much higher than that of found in Daejeon city, South Korea (28.59 ng.g^{-1}) (Lee et al. 2023), Tyumen city, Russia (26.07 ng.g^{-1}) (Konstantinova et al. 2022), Yazd, Iran (26.10 ng.g^{-1}) (Nematollahi et al. 2021), Myanmar (45.5 ng.g^{-1}) (Mon et al. 2020) and Buenos Aires, Argentina (26.4 ng.g^{-1}) (Cappelletti et al. 2019). These findings suggest that the levels of BaP in street dust differ globally. These discrepancies may be related to changes in the volume and

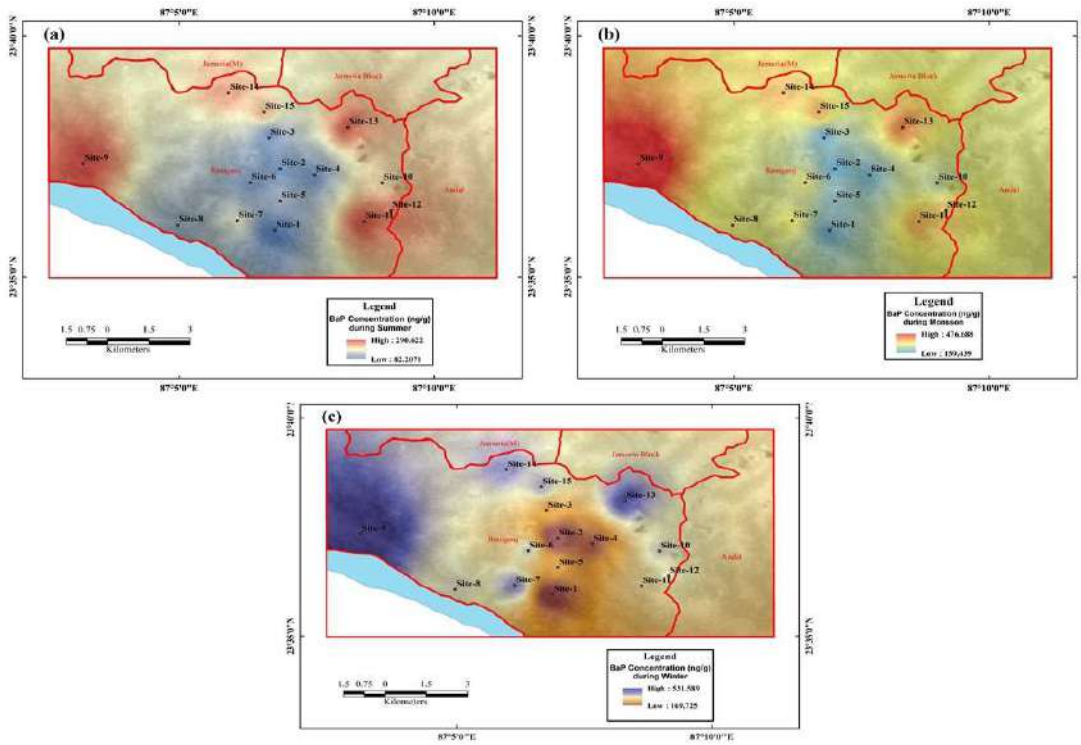

Fig. 3: Seasonal variation in BaP levels across 15 street dust samples (n= 15) from Raniganj during (a) summer, (b) monsoon, and (c) winter.

intensity of human activity, traffic loads, vehicular emissions, various technologies used, numbers of industrial activities, cooking patterns, the frequency of city street cleaning, and regional weather patterns like temperature differences, wind speeds and rainfalls that degrade, disperse and eliminate PAHs from street dust (Shabbaj et al. 2018, Hussain et al. 2015, Soltani et al. 2015).

Spatial Variation of BaP

Street dust collected from different land use types in Raniganj indicated an apparent disparity in mean total BaP levels (Fig. 4a), which was found to be in the order of busy traffic area > coal mine area > industrial area > commercial area > residential area. The highest value of BaP in busy traffic areas (328.29 ng.g^{-1}) followed by coal mine areas (323.68 ng.g^{-1}) implies the effects of high traffic congestion, vehicular emissions, coal chemical production, transportation of coal, leaching/weathering of coal beds and gangue accumulation (Gope et al. 2020, Masto et al. 2019). The lowest BaP concentrations were observed in residential areas (158.39 ng.g^{-1}), which might be due to its land use type, lower traffic loads, comparatively low vehicular emissions, minimal anthropogenic activities and absence of other non-point sources. The percentage contribution of mean total BaP levels of the residential, commercial, coal mine, industrial and busy traffic areas over the whole study area was 12.07%, 15.86%, 24.87%, 22.38% and 25.02%, respectively (Fig. 4b).

The BaP concentration in street dust of Raniganj varied significantly between the three seasons (Table 1). The mean BaP concentrations during the winter ranged from 214.23 ng.g^{-1} to 426.67 ng.g^{-1} with a mean of 336.28± 93.43 ng.g^{-1}, and during the monsoon varied from 167.44 to 344.37 ng.g^{-1} with a mean of 269.17±87.83 ng.g^{-1}, and ranged between 93.50 ng.g^{-1} and 249.90 ng.g^{-1} with a mean of 181.90±76.46 ng.g^{-1} during the summer. An increase in BaP level was observed during winter and monsoon in comparison to summer. Changes in weather patterns and existing human activity may be the cause of the varying seasonal concentrations throughout the year. Because of the thin layer of street dust and the potential for absorption and reflection of sunlight over a prolonged period by the street asphalt, along with very high temperatures, BaP is subject to photo-degradation and volatilization during summer.

Furthermore, since BaP is typically adsorbed onto very small particles, the swiftly moving vehicles on the street lift the fine dirt particles and aid in BaP dispersion by strong

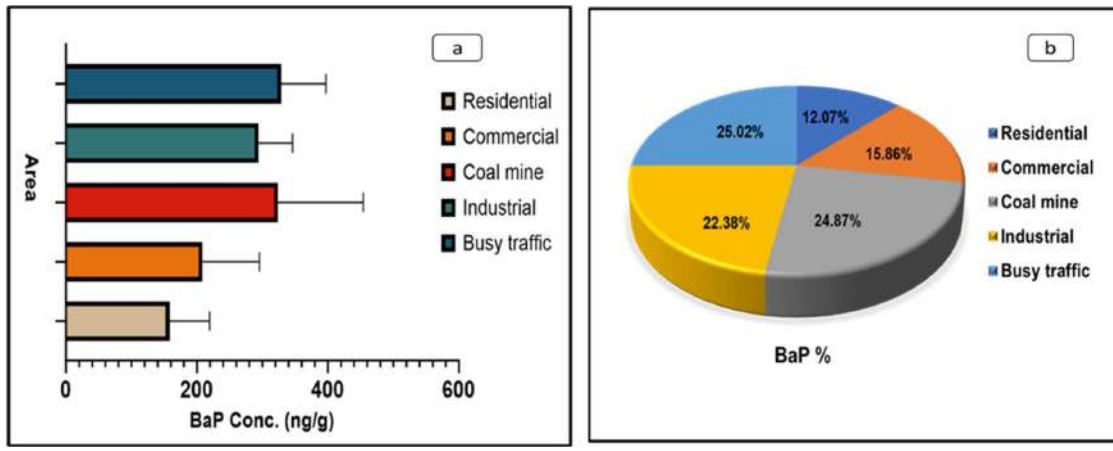

Fig. 4a: Spatial differences in BaP concentrations across various land-use types based on street dust samples (n=15) from Raniganj. Fig. 4b: BaP level percentage across various land-use types.

Table 1: Total and seasonal area-wise BaP content in street dust of Raniganj.

Sample matrix	Concentration of BaP [ng.g^{-1}]	Area	Season variation of BaP [ng.g^{-1}]		
			Winter	Monsoon	Summer
Street dust (<63 µm)	262.45±75.55	Residential	214.23±60.20	167.44±7.96	93.50±9.95
		Commercial	293.97±63.72	212.77±53.79	117.83±14.95
		Coal mine	426.693.22±7	344.37±114.77	200.01±80.91
		Industrial	344.63±13.31	288.20±46.43	248.26±39.84
		Busy traffic	401.89±26.58	333.09±16.23	249.90±23.65
		Mean total	336.293.43±8	269.17±87.83	181.90±76.46

wind, which may lead to low levels on the street side in the summer as compared to winter and monsoon (Gope et al. 2018). Primarily, an increase in vehicular traffic, indoor and outdoor heating and cooking activities during winter elevate the BaP level in winter (Manoli et al. 2016, Liu et al. 2007). Other factors such as various climatic conditions like lower temperature, high atmospheric stability, lower atmospheric mixing height, low volatilization, shorter daylight, thermal inversion, reduced vertical dispersion, low photochemical activity and increased particle adherence, could be the reason for higher BaP levels in winter in comparison to the other two seasons (Gope et al. 2020, Khillare et al. 2014).

While considering five land-use types, distinct variations in BaP levels were detected in three different seasons (Table 1). The highest BaP value was observed in the coal mine area for winter and monsoon, while in summer, it was for busy traffic areas. On the other side, the lowest BaP value was found in a residential area for all three seasons.

Risk Assessment

The assessment of adverse health effects was established for the urban population using BaP data in terms of chronic ADD from contaminated street dust. Ingestion and dermal ADDs were noted to be similar between adults and children, while values for inhalation were markedly lower. For ingestion, the estimated amounts were in the range of 2.72E-04 to 5.63E-04 and 4.53E-04 to 9.38E-04 for adults and children, respectively. Inhalation values ranged between 3.99E-08 and 8.28E-08 for adults, and for children, values were in the range of 1.66E-08 to 3.45E-08. Values of dermal absorption were recorded in the range of 1.41E-04 to 2.92E-04 and 1.65E-04 to 3.41E-04 for children. Between age groups, for ingestion and dermal exposure, children had higher ADD values than adults, whereas adults had higher values for inhalation. The HI values (Table 2) indicate moderate levels for both adults and children across the five sampling areas, with HI values varying between 1 and 10. However, it was observed that children had higher HI values than adults. Among the study areas, the highest HI values were observed for areas with high traffic density, followed by areas near coal mining activities.

According to the calculated cancer risk (Table 2), the risk for adults from ingestion and skin contact was between E-03 and E-02, which is the same as the risk for children. The risk of inhalation in this case was between E-08 and E-07, which was 10^5 times lower than the risk of ingesting and skin contact. Thus, compared to the other two exposure pathways, inhalation of dust particles from the streets was insignificant (Gope et al. 2018). This finding is consistent and similar to previously reported studies by other researchers (Ning et al. 2023, Kosheleva et al. 2023, Gope et al. 2020, Masto et al. 2019). The reason behind the higher value of ILCR for ingestion and dermal compared to Inhalation might be due to the respiratory system's innate defences that are more effective at screening out particles before they enter the bloodstream, while ingested and absorbed substances have a more direct route to internal organs (Sousa et al. 2022, Ramesh et al. 2004). The levels of cancer risk associated with ingestion and dermal contact were within the same order of magnitude, suggesting that these exposure routes significantly increased the risk of cancer in both adults and children. Nonetheless, children were far more at risk of direct consumption than adults because they were more susceptible to hand-to-mouth activity and had lower body weight, which makes it easy for them to consume contaminated dust (Gong et al. 2022).

On the other side, dermal contact induced a comparatively higher risk in adults than in children. In the present study, all 5 different areas showed significantly higher total carcinogenic risk for both adults and children, as the values exceeded ILCR> 10^{-4}. In the case of seasonal variation, winter has the highest carcinogenic risk for all five areas, followed by the monsoon and the summer. Sites 7, 8, and 9 had greater values throughout the winter (Fig. 5a, 5b) and monsoon seasons for both adults and children (Fig. 5c, 5d), while sites 13, 14, and 15 had the highest values during the summer (Fig. 5e, 5f). The carcinogenic value increased in the order of residential area < commercial area < industrial area < coal mine area < busy traffic area. Although the residential area showed a lower cancer risk than the other four areas, it fell under the high carcinogenic risk category. The mean total cancer risk values obtained for adults and children were 2.89E-03 and

Table 2: HI and results for ingestion, inhalation, dermal and total cancer risk for children and adults in the street dust of Raniganj.

Area	Child ingestion	Child inhalation	Child dermal	Total cancer risk	Adult ingestion	Adult inhalation	Adult dermal	Total cancer risk	Child HI	Adult HI
Residential	2.10E-03	4.07E-08	2.62E-03	4.72E-03	1.88E-03	1.46E-07	3.34E-03	5.23E-03	2.07	1.39
Commercial	2.76E-03	5.35E-08	3.44E-03	6.20E-03	2.47E-03	1.92E-07	4.40E-03	6.87E-03	2.72	1.83
Coal mine	4.29E-03	8.32E-08	5.35E-03	9.64E-03	3.85E-03	2.98E-07	6.84E-03	1.07E-02	4.22	2.85
Industrial	3.90E-03	7.55E-08	4.86E-03	8.75E-03	3.49E-03	2.71E-07	6.20E-03	9.69E-03	3.83	2.59
Busy traffic	4.35E-03	8.44E-08	5.43E-03	9.78E-03	3.90E-03	3.03E-07	6.93E-03	1.08E-02	4.28	2.89

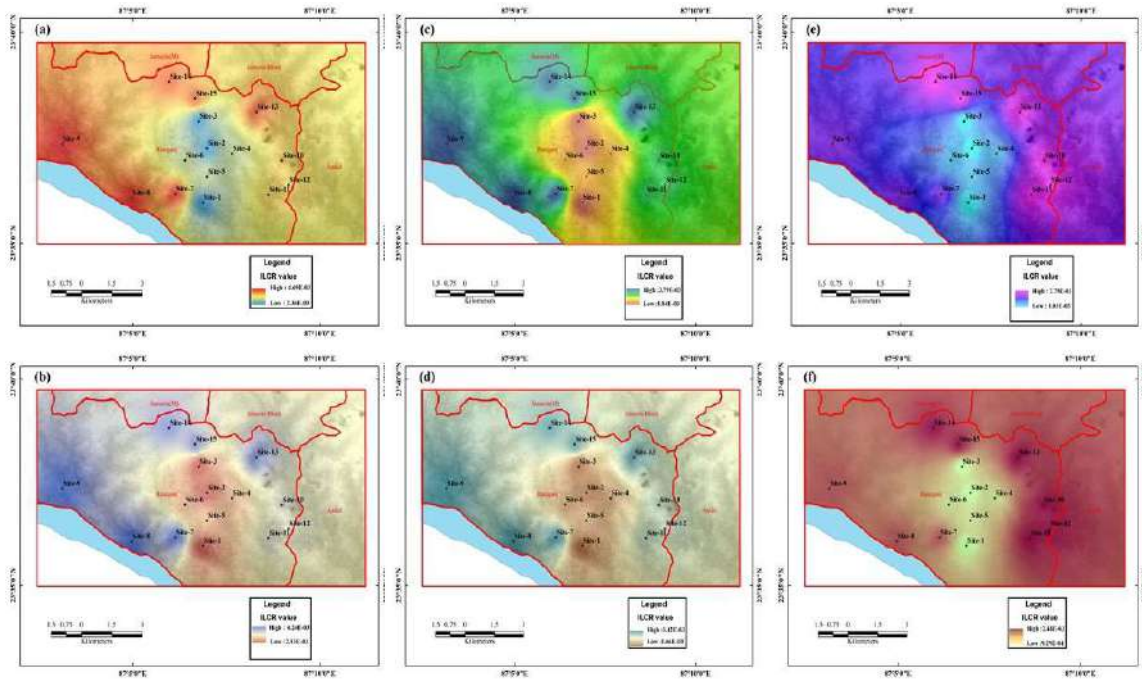

Fig. 5: Seasonal Variation (a: winter-adult, b: winter-child, c: monsoon-adult, d: monsoon-child, e: summer-adult, f: summer-child) in ILCR Levels in street dust (n=15) of Raniganj.

2.61E-03, respectively. Similar cancer risk magnitudes for both adults and children have been observed in recent studies over street dust from Kuala Lumpur, Malaysia (Hishamuddin et al. 2023), Huainan, China (Xu et al. 2022), Lagos, Nigeria (Iwebue et al. 2021), Karaj, Iran (Qishlaqi & Beiramali 2019) and Dresden, Germany (Zhang et al. 2019).

BaP in Street Dust and Associated Cancer Risk Across Different Cities: an Indian and Global Perspective

The concentration of BaP in the street dust and cancer risk of the present study has been compared with the BaP level in the street dust and ILCR value of previously reported studies across India (Table 3, Fig. 6, Fig. 7). In the present study, the BaP concentration was significantly higher than the reported concentrations of street dust in Guwahati (Hussain et al. 2015), New Delhi, and Bangalore (Tue et al. 2014). The BaP levels were 6-, 4.2-, and 5-fold higher than those in Guwahati, New Delhi, and Bangalore, respectively. The cancer risk in these three areas fell under E-04, which is one magnitude lower than the value obtained in the present study. The reported level of BaP and estimated cancer risk in street dust of Raipur (Patel et al. 2015) and Dhanbad (Tarafdar and Sinha 2019, Masto et al. 2019) were comparatively higher than the value obtained in the present study. Two separate studies from Durgapur (Gope et al. 2020) and Asansol (Gope et al. 2018), which are the adjacent towns of Raniganj, are compared for their BaP levels. Among them, Durgapur showed slightly lower BaP concentrations in street dust as compared to this study. At the same time, for Asansol, the levels were almost similar to the present study signifies the presence of similar kinds of Emission sources. The cancer risk values of Asansol and Durgapur were more or less similar to the value of the present study, which fell under E-03.

Only one study (Nayak et al. 2023) comprising three separate locations, among which street dust from Bhilai and Korba depicted extremely high BaP levels (26 to 27 times) than the level of BaP from Raniganj, while another location, Raipur, showed 7.6 times higher concentration than the present one, which is also relatively high. The ILCR value of these areas was higher than the value of Raniganj, which is E-02. Therefore, the total cancer risk estimated in all the reported studies from India ranged from E-02 to E-04, showing all the areas are at high risk of cancer for both adults and children. Thus, the high level of BaP might be due to the dominant presence of very high anthropogenic activities like vehicular emissions, diesel combustion, coke and biomass burning, etc. (Zhang et al. 2021). At the same time, the seasonal variation of BaP level in street dust is also considered. Studies from Durgapur, Guwahati and Asansol have been compared with the present study. The BaP level of current studies varies from 2.4 times to 11.6 times higher than those reported in the street dust of Guwahati in pre-monsoon, monsoon and post-monsoon. In the case of Asansol

and Durgapur, the seasonal variation in BaP concentrations is almost similar to 1.6 times lower than the seasonal BaP values of the present study. Thus, for seasonal variation, elevated levels of BaP in the street dust of Raniganj could be from several human activities besides point sources. A high amount of traffic load, several industrial activities, the presence of coal mines, and indoor and outdoor cooking activities might lead to high BaP content. Biomass burning, coke and coal combustion, vehicular emissions, wood combustion, petroleum, diesel and gasoline combustion are some major pyrogenic contributors of BaP in the street dust of the entire Raniganj area (Hayakawa 2024, Kumar et al. 2024, Ali et al. 2021). Like other studies from Dhanbad, Asansol and Durgapur, the BaP content in street dust may increase the chances of exposure and associated health risks to humans living within this region.

The analysis of the global distribution of BaP concentration and its associated risk for human cancer was conducted on a regional basis using available data (Supplementary Table 2, Figs 6 and 7). Most studies in Asia were conducted in China, followed by Iran. The highest concentration of BaP was reported in Anshan City, China, at 12,500 $ng.g^{-1}$ (Han et al. 2009), followed by Nanjing with 3,020 $ng.g^{-1}$ (Zhen et al. 2020) and Shenzhen with 3,089.07 $ng.g^{-1}$ (Ning et al. 2023). Such a concentration is many-fold greater than that recorded in the present study. The lowest BaP concentration observed in China was in Huanggang City at 67.40 $ng.g^{-1}$ (Liu et al. 2019), which was much lower than the values recorded in Raniganj. Among all the studies, Anshan had the highest cancer risk, with a magnitude of E-01 for both adults and children. Other studies from China have also indicated an elevated cancer risk among residents.

In Iran, maximum and minimum BaP concentrations were reported in Bandar Abbas and Tehran, respectively (Keshavarzi et al. 2018, Saeedi & Salmanzadeh 2012). The BaP level in Karaj City (278.10 $ng.g^{-1}$) (Qishlaqi & Beiramali 2019) was comparable to that in this study, unlike most other Iranian studies, which reported lower concentrations. The total cancer risk for both adults and children in Iran ranged between E-01 and E-05, indicating risks from very high to moderate in the population.

In Vietnam, Anh et al. (2019) found that BaP concentrations and ILCR values were similar to those found in Raniganj, India. In contrast, studies conducted by Zhen et al. (2020) in Japan, Taiwan, and Myanmar, along with Lee et al. (2023) in Daejeon Metropolitan City, South

Table 3: BaP concentration in the street dust and cancer risk from different cities in India.

Location	Concentration of BaP [$ng.g^{-1}$]	Cancer risk		Year of the Work (sampling)	Reference
		Adult	Child		
Raipur, Chhattisgarh	1988±478	2.19E-02	1.97E-02	2008-2015	Nayak et al. 2023
Bhilai, Chhattisgarh	6869±880	7.56E-02	6.82E-02		
Korba, Chhattisgarh	7094±819	7.80E-02	7.05E-02		
Durgapur (Summer)	156±7.44	1.98E-03	1.79E-03	2013-2014	Gope et al. 2020
Durgapur (Monsoon)	174±11.9				
Durgapur (Winter)	212±22.3				
Dhanbad	531±21	5.84E-03	5.27E-03	2016	Tarafdar & Sinha 2019
Dhanbad	349±71	3.84E-03	3.47E-03	2012	Masto et al. 2019
Asansol (Summer)	134±45.8	2.68E-03	2.42E-03	2013-2014	Gope et al. 2018
Asansol (Monsoon)	229±25.6				
Asansol (Winter)	369±64.8				
Guwahati city (Monsson)	29.3±35	4.88E-04	4.41E-04	2011-2012	Hussain et al. 2015
Guwahati city (Post-monsoon)	29.0±21				
Guwahati City (Pre-monsoon)	74.9±112				
Raipur city, Chhattisgarh	651.4	7.17E-03	6.47E-03	2010	Patel et al. 2015
New Delhi	62	6.82E-04	6.16E-04	2012-2013	Tue et al. 2014
Bangalore	58	6.38E-04	5.76E-04		
Raniganj (Winter)	336.28±93.43	2.89E-03	2.61E-03	2017-2018	Present study
Raniganj (Summer)	181.90±76.46				
Raniganj (Monsoon)	269.17±87.83				

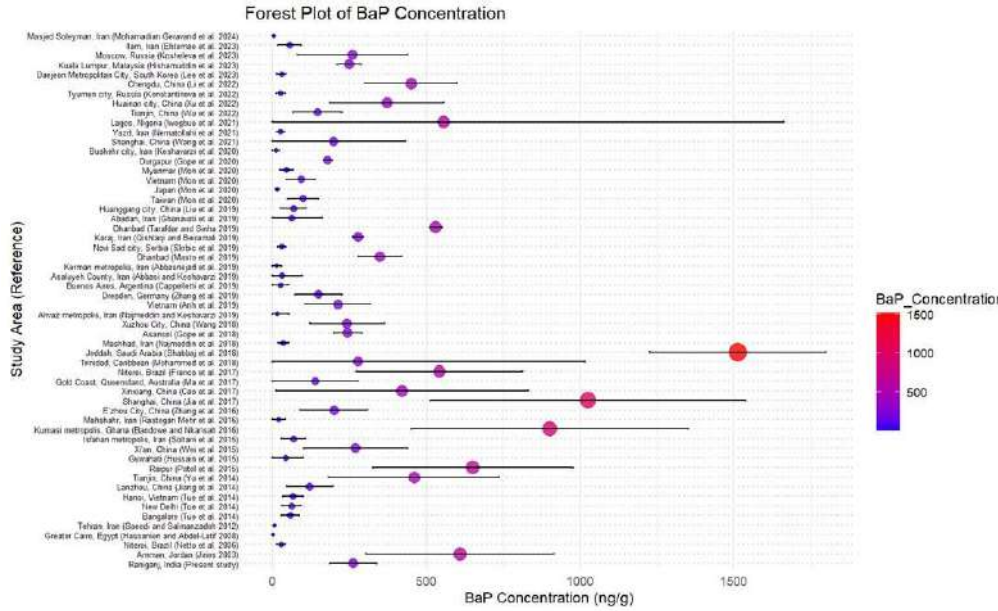

Fig. 6: BaP concentrations in street dust from different cities across India and worldwide.

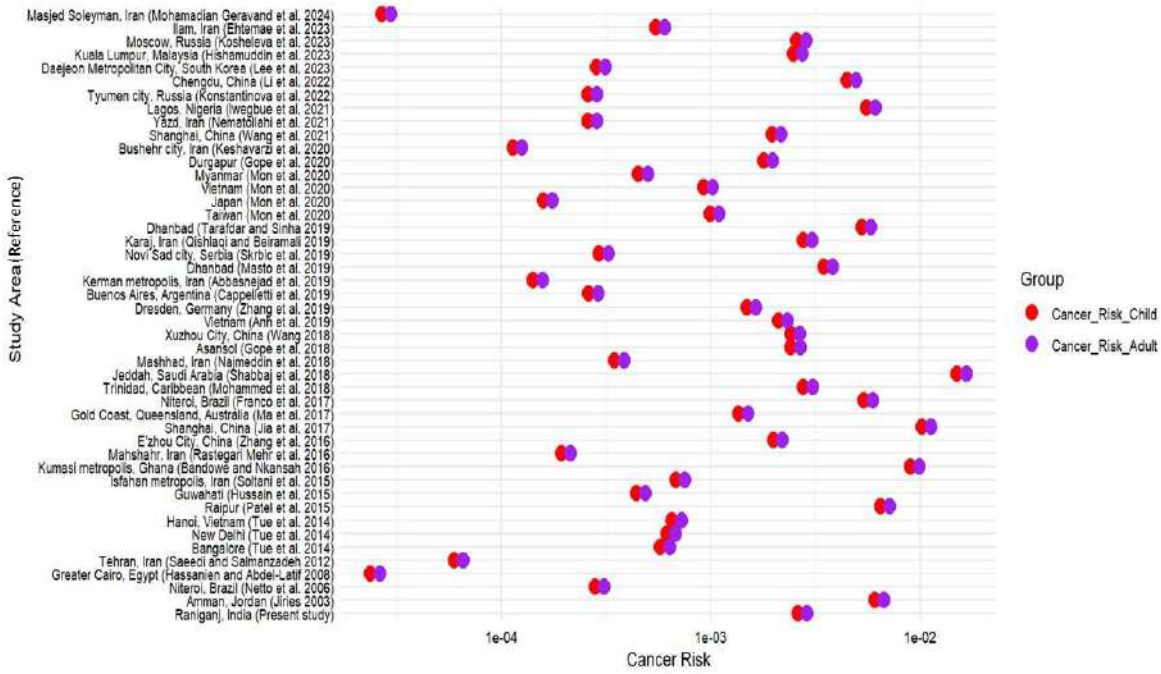

Fig. 7: Cancer risk (child and adult) in street dust from different cities across India and worldwide.

Korea, reported substantially lower BaP levels and cancer risks than those reported in the present study. Nevertheless, studies from Jeddah, Saudi Arabia, by Shabbaj et al. (2018), from Tokyo, Japan, by Khanal et al. (2018), and from Ulsan, South Korea, by Lee and Dong (2010), found the highest BaP concentrations compared to the present study (1,513 ng.g^{-1}, 2,100 ng.g^{-1}, and 6,300 ng.g^{-1}, respectively). These concentrations correspond to higher cancer risk estimates, indicating a possible health hazard for resident populations.

Four studies were conducted in Europe. One study executed by Kosheleva et al. (2023) in Moscow, Russia, recorded BaP concentrations similar to those of the present study, while the rest reported lesser amounts (Konstantinova et al. 2022, Škrbić et al. 2019, Zhang et al. 2019).

As an African Country, two different studies from Nigeria, one study from Egypt, and three different studies from Ghana were reported. A relatively high concentration of BaP was recorded in Nigeria, Ibadan, at 2,580 $ng.g^{-1}$ (Yusuf et al. 2022). Kumasi Metropolis, Ghana, recorded the highest BaP concentration at the worldwide level, which proved to be 27,900 $ng.g^{-1}$ (Essumang et al. 2006), exceeding levels in Bandar Abbas, Iran. The ILCR figures for African populations ranged from E-01 to E-05, indicating an elevated to moderate risk of developing cancer.

In the Americas, Trinidad, Caribbean (Mohammed et al. 2018), reported BaP similar to the current study, but studies conducted by Cappelletti et al. (2019) and Netto et al. (2006) in Argentina and Brazil, respectively, reported a far lower concentration of BaP in street dust and lower cancer risk estimates as compared to the values obtained for Raniganj.

The global analysis of BaP concentrations and the respective cancer risks exhibited a stark regional trend, with total cancer risk values ranging from E-01 to E-05, indicating a moderate to severe potential risk to human health. Therefore, continued environmental monitoring and the implementation of effective regulatory policies to mitigate the health hazards of BaP exposure are necessary.

LIMITATION AND FUTURE PROSPECTS OF THE STUDY

Several factors limit the present study: i) it only accounts for three seasonal periods (summer, monsoon, and winter) that might not fully represent the temporal variation of BaP concentrations. A more extensive temporal study with monthly sampling over several years would give a better insight into seasonal and inter-annual trends. ii) The number of samples (n = 15) can potentially reduce the spatial resolution and statistical strength of the findings; a larger number of sampling points could improve the representativeness and reliability of BaP monitoring of street dust. Moreover, source apportionment analysis by Principal Component Analysis (PCA) was not conducted, which limits the possibility of identifying the potential sources of BaP in the studied area.

Despite these constraints, the research offers significant guidance for future studies. Monte Carlo simulations are suggested to be integrated in order to evaluate uncertainty in human health risk assessments.

CONCLUSIONS

The present study highlights the spatial and seasonal variation of BaP levels in the street dust of Raniganj. The mean concentration of BaP in the study area was 262.45± 75.55 $ng.g^{-1}$, which might pose a risk of exposure to the residents of this area. In winter, summer and monsoon, the highest value was observed in site 9, which is a coal mine area. There was a noticeable variation in the mean total BaP levels in the street dust collected from various land use types in Raniganj. A substantial seasonal variation of BaP level was observed here, which was found to be in the order of winter (336.28 $ng.g^{-1}$) > Monsoon (269.17 $ng.g^{-1}$) > summer (181.90 $ng.g^{-1}$). The carcinogenic value and HI of the study area increased in the following order: residential area < commercial area < industrial area < coal mine area < busy traffic area. The average overall cancer risk value for children and adults was 2.61E-03 and 2.89E-03, respectively, describing the presence of a high risk of cancer. The mean HI value for children was 3.42, whereas for adults it was 2.31. Both values indicated the occurrence of a moderate level of health risk. Increased vehicular emissions, indoor and outdoor heating and cooking activities, and changes in several meteorological conditions during winter may cause a higher BaP load in street dust. To study the overall impact of BaP on the environment and human health, it is crucial to concentrate on its sources, characteristics, fate in the environment, pathways of exposure, mode of toxicity and existing regulatory controls so that government agencies and policymakers can initiate new effective policies to control automobile and industrial emissions, limit its exposure, safeguard human health and reduce the related risks. Our study will help to understand the exact scenario of BaP contamination in the Raniganj area so that effective precautionary measures can be taken by the governing body to reduce the exposure risk and to protect the health of the residents of this area from BaP contamination. For precautionary self-protection, protective masks are suggested to be worn by resident populations to minimize inhalation exposures to BaP in ambient dust. Further, enhanced indoor air circulation is recommended to minimize the concentration and accumulation of PAHs in indoor environments and, thus, lower potential health threats of long-term exposure.

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Biocells for Waste Valorization: Enhancing Methane Production and Leachate Management in the Anaerobic Degradation of Green Waste and WWTP Biosolids from Ambato, Ecuador

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ABSTRACT

The disposal of solid waste in conventional landfills poses critical environmental challenges, including uncontrolled greenhouse gas emissions and highly contaminated leachate. Bioreactor landfills with leachate recirculation offer an effective alternative, accelerating organic matter degradation and enhancing methane production for energy recovery. The present study investigates methane generation, leachate characteristics, and the stabilization of organic matter in laboratory-scale biocells that incorporate biosolids from the Ambato Wastewater Treatment Plant (WWTP), along with compost and green waste. Three replicate biocells were operated over 12 weeks with leachate recirculation to optimize nutrient removal and biogas production. Methane generation stabilized after 21 days, while phosphorus and ammonium concentrations in the leachate ranged from 10-15 $mg.L^{-1}$ and 50-80 $mg.L^{-1}$, respectively. Heavy metal concentrations significantly decreased, with final cadmium and chromium levels falling below regulatory discharge limits, reaching 0.02 $mg.L^{-1}$ and 0.05 $mg.L^{-1}$, respectively. Chemical Oxygen Demand (COD) was reduced by 85%, reaching a final concentration of approximately 300 $mg.L^{-1}$. These findings highlight the feasibility of incorporating WWTP biosolids in biocells to enhance organic solid waste degradation, sustainable landfill leachate management, and renewable energy generation in Ambato, Ecuador.

INTRODUCTION

Municipal landfills remain the predominant method for disposing of urban solid waste in developing countries due to their operational simplicity and high capacity for managing residual materials (Zhang et al. 2019). This is the case of Ambato, an Andean city in Ecuador with nearly 190,000 inhabitants and an estimated solid waste generation of approximately 300 tons per day (GAD Municipal de Ambato 2016, Instituto Nacional de Estadística y Censos (INEC) 2023). However, global solid waste generation has increased significantly-from 635 million tons in 1965 to 1,999 million tons in 2015-and is projected to reach 3,539 million tons by 2050 (Chen et al. 2020). The continued accumulation of solid waste in landfills poses serious environmental challenges, particularly concerning the generation of landfill gas (LFG) and leachate.

Waste decomposition in landfills primarily occurs through anaerobic microbial processes. These processes are facilitated by bacteria such as *Sporanaerobacter acetigenes*, *Clostridium sporogenes*, *Methanomicrobiales* and *Methanosarcinales*, which contribute to the production of gaseous compounds (Shao et al. 2021, Yang et al. 2021). The resulting LFG typically consists of CH_4 (50–60%), CO_2 (40–50%), and volatile organic compounds (VOCs). In many cases, it is either directly released

into the atmosphere or flared without adequate quality controls, exacerbating air pollution and posing risks to human health (Werkneh 2022). Methane emissions from landfills are of particular concern, given that CH_4 has a global warming potential (GWP) 28–36 times greater than CO_2 over 100 years (IPCC 2023). Landfills account for approximately 5% of global greenhouse gas emissions, contributing significantly to climate change (EPA 2024).

The disposal of household and industrial solid waste in landfills introduces various hazardous compounds that are difficult to biodegrade, including organic solvents, pesticides, herbicides, BTEX compounds (benzene, toluene, ethylbenzene, and xylene), and heavy metals such as cadmium (Cd) and hexavalent chromium (Cr^{6+}) (Essien et al. 2022, Kjeldsen et al. 2002, Vaverková 2019). Leachate can act as a transport medium for these pollutants, leading to surface and groundwater contamination and frequently migrating into surrounding soils through percolation (Hadi 2023). Leached contaminants, including Cd^{2+}, Pb^{2+}, Zn^{2+}, and Cu^{2+}, are highly toxic, carcinogenic, and resistant to degradation (Hussein et al. 2021). Furthermore, toxicological studies indicate that certain leachates exhibit mutagenic properties, reinforcing the need for effective leachate management strategies (Vaverková 2019). These strategies involve the use of enhanced landfill cover materials (Sanoop et al. 2024), leachate collection and recirculation, as well as advanced treatment processes, including biological, physicochemical, and oxidative techniques (Mojiri et al. 2021).

Despite the availability of advanced biogas management and leachate treatment technologies, their implementation is often hindered in developing countries due to financial constraints and infrastructure limitations. Consequently, methane emissions remain largely uncontrolled, and leachate frequently contaminates surrounding soil and water sources due to inadequate treatment systems (Zhang et al. 2024).

Biocells offer a promising alternative to conventional landfills, as they incorporate leachate recirculation strategies that enhance microbial activity, accelerate organic matter degradation, and promote rapid waste stabilization. This approach optimizes conditions for methane production while simultaneously improving the reduction of Chemical Oxygen Demand (COD) in leachate compared to traditional landfill treatment methods (Budihardjo et al. 2021). Leachate recirculation not only enhances bacterial metabolic activity but also facilitates more efficient biodegradation by supplying essential nutrients and microorganisms. Consequently, biocells improve waste decomposition, reclaim landfill airspace, and increase CH_4 production efficiency, which can be harnessed as a renewable energy source. Unlike conventional landfills, where biogas is often released into the environment with harmful consequences, biocells provide a controlled degradation environment that maximizes methane recovery (Japperi et al. 2021).

The primary objective of this study is to evaluate methane production and characterize the leachate generated by laboratory-scale biocells used for solid waste disposal, incorporating biosolids from the Ambato Wastewater Treatment Plant (WWTP) and green waste from local markets in Ambato. The research focuses on implementing a solid-phase biodigestion system to decompose organic matter, operating biocells over a stabilization period of 12 weeks, and assessing performance through biogas quantification, leachate characterization, and solid-phase analysis.

Integrating WWTP biosolids with green waste and intermediate compost layers in a biocell system presents challenges, particularly due to the presence of recalcitrant organic contaminants and heavy metals, which may complicate leachate management (Kjeldsen et al. 2002). Additionally, certain residues within biosolids can slow biodegradation, potentially extending the stabilization period. This study aims to address these concerns and evaluate the feasibility of biocell technology as an alternative waste management approach for Ambato. A successful implementation could reduce landfill inputs, mitigate environmental impacts, promote methane as a renewable energy source, and improve overall waste stabilization (Kumar et al. 2011).

MATERIALS AND METHODS

Biocell Design and Operation

The biocell was designed to minimize compaction within the waste mass to ensure uniform moisture distribution and adequate hydraulic conductivity, thereby preventing clogging (Aldrawsha et al. 2020). The biocells were constructed within cylindrical columns, incorporating alternating layers of compost, green waste, and WWTP biosolids (see Fig. 1). A sand-gravel layer at the base facilitated drainage, while an additional sand layer at the top ensured uniform leachate distribution. Compost was obtained from agricultural supply centers. The organic waste materials consisted of rabbit manure compost, while green waste comprised lettuce and cabbage residues. Organic waste was collected from markets in Ambato and transported to the laboratory in plastic bags. Dehydrated biosolid samples were collected from the output of the

Table 1: Weight Distribution of Layered Materials for Biocell Assembly.

Layered Material	Biocell1	Biocell 2	Biocell 3
Sand, [g]	4215	4004	3816
Compost [g]	397	363	436
Green waste [g]	311	296	270
WWTP Biosolids [g]	783	722	792
Compost [g]	422	332	497
Green waste [g]	294	290	277
WWTP biosolids [g]	758	713	746
Compost [g]	477	371	623
Sand [g]	1559	2215	1772

biosolids centrifuge at the Ambato Wastewater Treatment Plant (WWTP). To prevent decomposition prior to the incorporation of organic material into the biocell, samples were stored under refrigeration at 4°C, following the protocols established in Standard Method 1060: Collection and Preservation of Samples (APHA 2017). Table 1 presents the weights of the different material layers incorporated into the biocells.

The leachate produced by the biocell was recirculated to maintain a uniform moisture distribution throughout the waste mass, with cycles occurring approximately every two hours, driven by peristaltic pumps operating at a flow rate of 4.2 $mL.min^{-1}$. This interval was adjusted as needed based on the permeability and moisture retention capacity of the waste materials.

Leachate and biogas generation were systematically monitored weekly to evaluate the progression of the biodegradation process.

Leachate Sampling and Characterization

Leachate samples were collected weekly to monitor chemical changes within the biocells. All analyses were conducted in accordance with the Standard Methods for the Examination of Water and Wastewater (APHA 2017). All chemical analyses were performed in triplicate, and standard deviation coefficients were calculated. Conductivity and pH measurements were taken using a HANNA HI839800 multiparametric meter. Chemical analyses for sulfate, phosphate, iron, magnesium, ammonium, chemical oxygen demand (COD), sulfide, and hexavalent chromium concentrations were conducted using photometric methods with the HI83399 Hanna photometer. For heavy metal analysis, including total chromium and cadmium, Graphite Furnace Atomic Absorption Spectrometry (GFAAS) was conducted using the PG Instruments AA500 spectrometer.

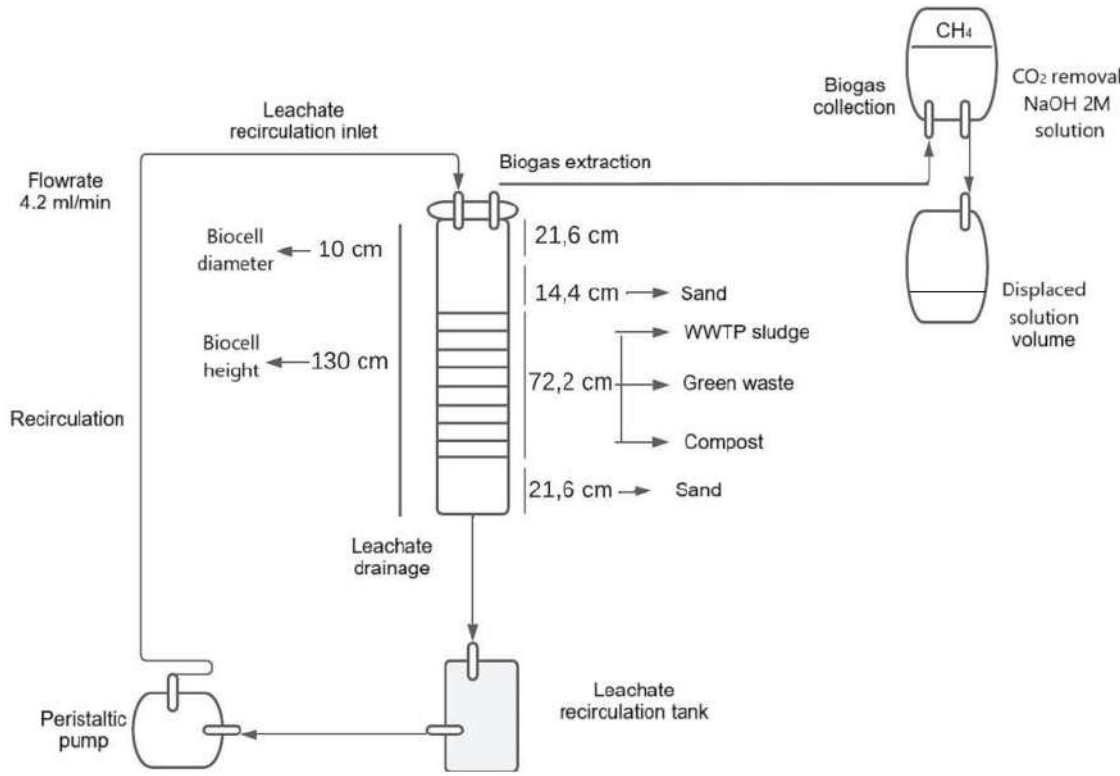


Fig. 1: Schematic of the Biocell Design for Organic Matter Decomposition.

Calibration curves with Pearson correlation coefficients ($R^2 > 0.98$) were applied.

Characterization of Waste Components (Compost, Biosolids, and Organic Matter)

The moisture content of the organic waste was determined by drying samples at 105°C for 24 h in a BINDER ED-400 oven following the methodology of 2540 D. Volatile solids were measured by igniting the dried samples at 550°C for two h in a muffle furnace, Biobase MC10-12, using the standard method SM 2540 G (APHA 2017). The biosolids samples were subjected to acid digestion to extract analytes present in the WWTP biosolids, following the EPA 3051A method (EPA 2007). Photometric methods using the HANNA HI83399 multiparameter photometer were employed to determine total phosphorus, potassium, magnesium, calcium, sulfate, manganese, iron, aluminum, copper, molybdenum, and zinc in solubilized dry biosolids. The concentrations of arsenic, cadmium, chromium, nickel, and lead in digested biosolids samples were determined by Graphite Furnace Atomic Absorption Spectrometry (GFAAS) using a PG-500 Atomic Absorption Spectrophotometer. Mono-elemental standards (AccuStandard, 1000 $\mu g.L^{-1}$) were used for calibration.

Biogas Collection and Methane Gas Quantification

Biogas was collected using a volumetric method, which consisted of a closed system with two 1-liter amber glass bottles and a 2M NaOH solution. One bottle contained the NaOH solution to capture CO_2, allowing for the collection of purified methane as biogas (Fig. 2). This setup enabled the quantification of methane output by utilizing an amber glass bottle containing a 2M NaOH solution, connected to an adjacent empty container through a tube. As biogas exerted pressure, it displaced the NaOH solution from one bottle to the other, effectively absorbing CO_2 from the biogas. The methane content was then determined by measuring the weight of the displaced NaOH solution (Nopharatana et al. 1998, Sorensen & Ahring 1993).

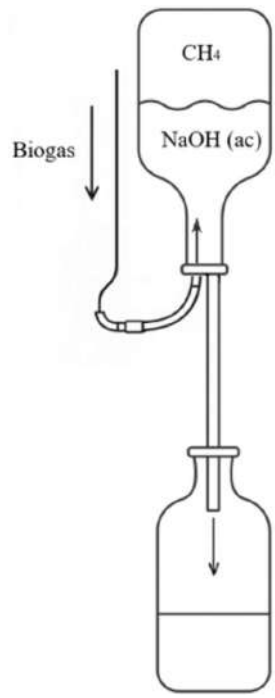


Fig. 2: Schematic of CH_4 Collection System Using NaOH Absorption.

Materials and Chemicals

All reagents were of analytical grade: NH_4Cl and $C_2H_3NaO_2$ (Sigma-Aldrich), KH_2PO_4 and H_3BO_3 (Merck). Magnesium chloride hexahydrate ($MgCl_2 \cdot 6H_2O$, 82.4 $mg.L^{-1}$), calcium chloride ($CaCl_2$, 10 $mg.L^{-1}$), sodium bicarbonate ($NaHCO_3$, 3000 $mg.L^{-1}$), and zinc chloride ($ZnCl_2$, 0.1 $mg.L^{-1}$) were supplied by Thermo Fisher Scientific. Yeast extract (10 $mg.L^{-1}$) was purchased from Becton, Dickinson, and Company (BD). Trace elements included ferrous chloride tetrahydrate ($FeCl_2 \cdot 4H_2O$, 2 $mg.L^{-1}$) and manganese chloride ($MnCl_2$, 0.1 $mg.L^{-1}$) from Sigma-Aldrich, ammonium molybdate tetrahydrate ($(NH_4)_6Mo_7O_{24} \cdot 4H_2O$, 0.1 $mg.L^{-1}$), cobalt chloride hexahydrate ($CoCl_2 \cdot 6H_2O$, 2 $mg.L^{-1}$), and aluminum sulfate hydrate ($Al_2(SO_4)_3 \cdot 18H_2O$, 0.1 $mg.L^{-1}$) from Merck, nickel nitrate hexahydrate ($Ni(NO_3)_2 \cdot 6H_2O$, 0.1 $mg.L^{-1}$), copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$, 0.1 $mg.L^{-1}$), and sodium selenite pentahydrate ($Na_2SeO_3 \cdot 5H_2O$, 0.1 $mg.L^{-1}$) from Sigma-Aldrich. EDTA (1 $mg.L^{-1}$) was also obtained from Sigma-Aldrich. The pH was adjusted using hydrochloric acid (HCl, 0.001 $mg.L^{-1}$) from Merck.

Preparation of Basal Medium

The basal medium contained 280 $mg.L^{-1}$ of ammonium chloride (NH_4Cl), 250 $mg.L^{-1}$ of monopotassium phosphate (KH_2PO_4), 82.4 $mg.L^{-1}$ of magnesium chloride hexahydrate ($MgCl_2 \cdot 6H_2O$), 10 $mg.L^{-1}$ of calcium chloride ($CaCl_2$), 3000 $mg.L^{-1}$ of sodium bicarbonate ($NaHCO_3$), and 10 $mg.L^{-1}$ of yeast extract. To promote the methanogenic activity of microorganisms, 3204.5 $mg.L^{-1}$ of anhydrous sodium acetate ($C_2H_3NaO_2$) was added. The pH of the basal medium was adjusted to a range of 7.1 to 7.3 and measured using an OAKTON N16M3F pH meter.

Trace elements and micronutrients were also incorporated, including 0.1 $mg.L^{-1}$ of boric acid (H_3BO_3), zinc chloride ($ZnCl_2$), manganese chloride ($MnCl_2$), ammonium molybdate tetrahydrate ($(NH_4)_6Mo_7O_{24} \cdot 4H_2O$), aluminum sulfate hydrate ($Al_2(SO_4)_3 \cdot 18H_2O$), nickel nitrate

hexahydrate ($Ni(NO_3)_2 \cdot 6H_2O$), copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$), and sodium selenite pentahydrate ($Na_2SeO_3 \cdot 5H_2O$). The medium also contained 2 $mg.L^{-1}$ of ferrous chloride tetrahydrate ($FeCl_2 \cdot 4H_2O$) and cobalt chloride hexahydrate ($CoCl_2 \cdot 6H_2O$). Additionally, 1 $mg.L^{-1}$ of EDTA and 0.001 $mg.L^{-1}$ of hydrochloric acid (HCl) were included to enhance nutrient stability and bioavailability.

Statistical Analysis

Repeated measures ANOVA was applied to assess whether statistically significant changes occurred over the 12 weeks within each biocell for all monitored parameters. This analysis was performed using the AnovaRM function from Python's *statsmodels* library (v0.14.0), following a within-subjects design based on weekly triplicate measurements, which allowed for the evaluation of temporal trends. In parallel, a one-way ANOVA was performed using the f_oneway function from Python's *scipy*. *Stats* module to determine whether the mean values differed significantly among the three biocells.

RESULTS AND DISCUSSION

Characteristics of Waste Components: Compost, WWTP Biosolids, and Organic Matter

The biocells were designed to accelerate the decomposition of solid waste and enhance biogas production through recirculation. Organic matter plays a key role in this process by serving as the primary substrate for anaerobic digestion. Table 2 summarises the physicochemical characteristics of the three organic substrates used in the biocell system, i.e., WWTP biosolids, compost, and green waste. The table presents their pH, moisture content (wet basis), percentage of volatile solids, and the total nitrogen and phosphorus content (dry basis). These parameters help assess the suitability and biodegradability of each substrate within the anaerobic environment.

As shown in the data, compost has a slightly basic pH of 8.6 ± 0.5, indicating a natural buffering capacity that can support pH stability within the system and potentially enhance methanogenic activity. In contrast, WWTP biosolids (7.7 ± 0.1) and green waste (7.2 ± 0.1) provide a more neutral environment, supporting microbial stability but potentially becoming susceptible to acidification under high organic loads. Moisture content is an important factor influencing hydrolysis efficiency, and in this study, green waste showed a moisture level of 80.6%. This value is slightly lower than those reported in the literature, which range from 90% to 94.7% (Sanchez-Salvador et al. 2022). In contrast, compost had the lowest moisture content at 14.1%, providing structural stability within the biocell and helping prevent excessive acid accumulation, thereby supporting stable methane generation. Centrifuged WWTP biosolids generally contain 72%–78% moisture (EPA 2000), aligning closely with the 75.4% measured in this study. The total volatile solids (VS) content further indicates the proportion of degradable organic matter present in each substrate. The VS values are 85.4%, 82.5%, and 80.6% for WWTP biosolids, compost, and green waste, respectively, indicating their high organic load. WWTP biosolids contain 1.8% total nitrogen (N) and 1.3% total phosphorus (P), providing essential nutrients for microbial growth but also posing a risk of ammonia accumulation, which may inhibit methanogenesis. These values are consistent with the typical concentrations of organic matter and nutrients found in biosolids. For example, Yang et al. (2023) reported biosolids from Jiujiang City containing 38.5% organic matter, 3.4% nitrogen, and 1.1% phosphorus. In comparison, compost (0.2% N, 0.1% P) and green waste (0.8% N, 0.02% P) are more carbon-rich but significantly lower in nutrient content. The higher nitrogen and phosphorus levels in biosolids help compensate for this nutrient deficiency, making them a more balanced substrate for anaerobic processes.

Baseline nutrient and contaminant levels in biosolids, compost, and green waste are presented in Table 3.

The macroelements in the Ambato WWTP biosolids, such as calcium (4.57 ± 0.32%), magnesium (4.86 ± 0.15%), and potassium (0.77 ± 0.06%), fall within expected ranges for biosolids used as soil amendments (Onchoke & Fateru 2021). The sulfate concentration (0.52 ± 0.11%) is relatively moderate, which suggests a potential buffering effect but also raises concerns about sulfate-associated metal mobilization. The presence of heavy metals in biosolids is a major concern. In the Ambato WWTP biosolids, aluminum (759.43 ± 7.52 $mg.kg^{-1}$), chromium (1166 ± 26 $mg.kg^{-1}$), and zinc (2683 ± 91 $mg.kg^{-1}$) are significantly high compared to the findings of literature (Martínez Durán et al. 2023, Onchoke & Fateru 2021). Arsenic (5.58 ± 1.37 $mg.kg^{-1}$)

Table 2: Physicochemical properties of three organic substrates-WWTP biosolids, compost, and green waste—utilized within the biocell system.

	pH	% Humidity wet basis	% Volatile solids	% Total N dry basis	% Total P dry basis
WWTP Biosolids	7.7 ± 0.1	75.4 ± 0.2	85.4 ± 0.1	1.8 ± 0	1.3 ± 0
Compost	8.6 ± 0.5	14.1 ± 0.1	82.5 ± 0	0.2 ± 0	0.1 ± 0
Green waste	7.2 ± 0.1	89.6 ± 0.3	80.6 ± 0.1	0.8 ± 0.1	0.02 ± 0

Table 3: Composition of Ambato WWTP Biosolids.

Component	WWTP Biosolids
Potassium (K□O) [%]	0.77 ± 0.06
Calcium (CaO) [%]	4.57 ± 0.32
Magnesium (MgO) v	4.86 ± 0.15
Sulfate [%]	0.52 ± 0.11
Copper [%]	0.002 ± 0.0
Iron [%]	0.26 ± 0.04
Manganese [%]	0.10 ± 0.01
Molybdenum [%]	0.0 ± 0.0
Zinc [%]	0.53 ± 0.02
Aluminum [mg.kg^{-1}]	759.43 ± 7.52
Arsenic [mg.kg^{-1}]	5.58 ± 1.37
Cadmium [mg.kg^{-1}]	0.88 ± 0.10
Chromium [mg.kg^{-1}]	1166 ± 25
Cobalt [mg.kg^{-1}]	65.75 ± 1.65
Copper [mg.kg^{-1}]	171.44 ± 5.04
Nickel [mg.kg^{-1}]	42.75 ± 0.18
Lead [mg.kg^{-1}]	21.69 ± 2.19
Zinc [mg.kg^{-1}]	2683 ± 91

and lead (21.69 ± 2.19 mg.kg^{-1}) remain below hazardous levels but should be continuously monitored due to their potential bioaccumulation. The relatively high zinc content suggests that these biosolids could serve as a micronutrient supplement for crops, as zinc is essential for plant enzyme functions (Saleem et al. 2022).

Biocell Operation

Leachate recirculation in biocells sustains methane production and influences microbial activity and system stability. However, moisture retention capacity may decline over time due to physical and biological transformations within the system, potentially leading to uneven waste biodegradation, decreased methane yield, and reduced overall efficiency.

The results of the statistical analyses indicate that all physicochemical and biological parameters measured in the biocells over the 12-week operational period exhibited statistically significant temporal variation, as determined by repeated measures ANOVA ($p < 0.0001$). This consistent trend across variables-including methane generation, pH, COD, sulfates, phosphates, ammonium, sulfide gas, iron, magnesium, Cr(VI), total chromium, and cadmium-underscores the dynamic metabolic and geochemical processes occurring within each biocell over time. Furthermore, one-way ANOVA revealed statistically significant differences among biocells for ammonium, leachate sulfide, iron, magnesium, total chromium, and cadmium, indicating that these variables are particularly sensitive to inter-biocell variability. In contrast, parameters such as methane generation, pH, COD, sulfates, phosphates, sulfide gas, and Cr(VI) did not exhibit statistically significant differences among biocells during the operational period. These findings may be attributed to the inherent heterogeneity of the materials used in each biocell, which can differentially influence the progression of microbial activity and physicochemical transformations across systems (Kjeldsen et al. 2002). To better understand these trends, the following section presents and discusses in detail the temporal evolution of each measured parameter throughout the 12-week operational period.

The leachate pH variation in the three biocells, shown in Fig. 3, reflects distinct microbial activity and waste degradation patterns. Initially, the pH ranged from 8.0 to 8.2, however, in the following weeks, it exhibited an increasing trend. Oscillations occurred throughout the weeks, with peaks and dips, reaching values between 8.4 and 8.6 by week 12, suggesting a dynamic system with a tendency toward alkaline conditions.

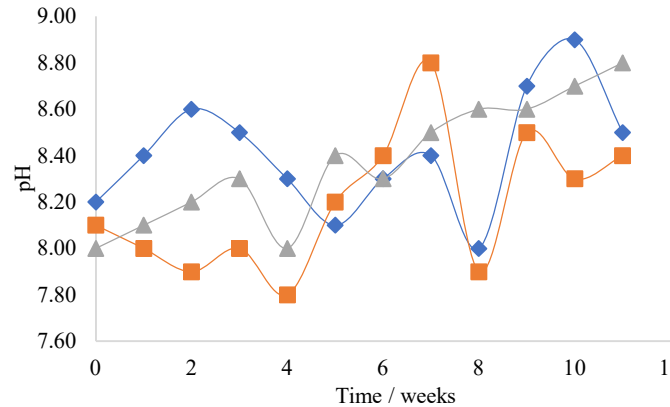


Fig. 3: pH monitoring from each biocell: (blue diamond) biocell 1; (orange square) biocell 2; and (gray triangle) biocell 3.

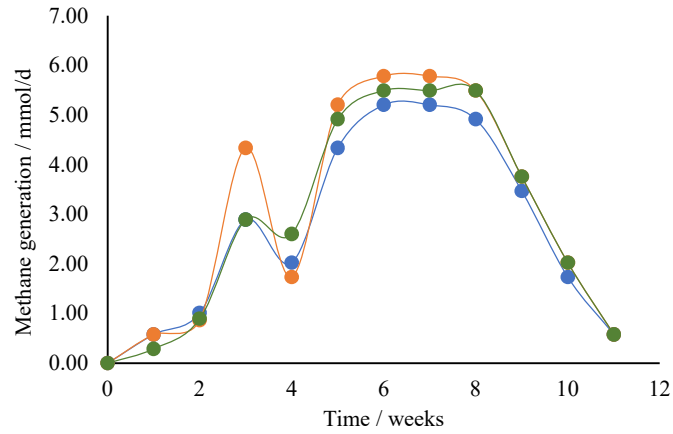


Fig. 4: Quantified methane gas from each biocell, (blue dot) biocell 1; (orange dot) biocell 2; and (green dot) biocell 3.

The pH range of 7.8 to 8.8 observed in the biocells aligns with conditions favorable for anaerobic and facultative microbial activities. While methanogenic bacteria typically thrive in a pH range of 6.6 to 7.6, other anaerobic microorganisms can function effectively in more alkaline environments. For instance, some anaerobic bacteria have been found to grow at pH levels up to 8.8, indicating that the pH conditions in the biocells are conducive to the activity of a diverse microbial community involved in organic matter degradation (Gerardi 2003).

Fig. 4 shows the quantified methane generation from each laboratory-scale biocell, measured at an average atmospheric pressure of 0.7 atm and a laboratory temperature of 22 °C. The data follow a characteristic curve with an initial phase of increasing biogas production, a steady methane generation phase between weeks 5 and 8, and a declining phase from weeks 8 to 12. The initial phase (0-1 week) of methane production in all three biocells exhibits a lag period characterized by low CH_4 volumes.

This lag period suggests that microbial communities are in an acclimatization phase, where hydrolytic and fermentative bacteria begin breaking down complex organic matter into simpler intermediates such as sugars, amino acids, and volatile fatty acids (VFAs) (Schievano et al. 2018). The delay in methane production indicates that methanogenic archaea have not yet reached full activity, as they rely on the byproducts of acidogenesis and acetogenesis, primarily acetate and hydrogen (H_2). At this stage, the similar methane production patterns among the three biocells suggest that their initial microbial inoculum or substrate composition did not differ significantly.

As described by He et al. (2019), the rapid rise in methane production observed around week 2-particularly in biocell 1-corresponds with established patterns of Methanosarcina barkeri activity and acetoclastic methanogenesis. Their findings indicate that Methanosarcina species perform efficiently under elevated acetate concentrations, which supports the early methane peaks recorded in this study. The delayed response in biocell 2 suggests a slower microbial adaptation, as the research indicates that acetate stress can hinder quorum sensing and slow methanogenesis. The lower peak in biocell 3 reflects a more balanced microbial adaptation, consistent with *Methanosarcina's* ability to shift pathways based on environmental stressors.

Following this peak, methane production experiences a noticeable decline between weeks 2.5 and 4 across all biocells. This temporary drop in biogas production can be attributed to several factors. One possible explanation is the depletion of readily biodegradable substrates, leading to a period where more complex compounds require enzymatic hydrolysis before being converted into methane precursors. Another likely cause is the accumulation of VFAs, which, if produced faster than methanogens can consume, can lead to a pH drop and partial inhibition of methanogenic activity. A microbial shift may occur, where acetoclastic methanogens are replaced or supplemented by hydrogenotrophic methanogens that utilize CO_2 and H_2 for methane production. This transition can introduce a temporary imbalance in the anaerobic digestion process, leading to fluctuations in CH_4 output (Hettiaratchi et al. 2015, Latif 2021).

The bioreactor landfill study at Sudokwon (Choi & Rhee 2024), the biocell methane production trends, and the metagenomic analysis of microbial methane cycling in landfills emphasize key factors influencing methane generation, microbial adaptation, and substrate utilization. In the Sudokwon study, leachate recirculation increased moisture content (38-39%), resulting in continuous landfill gas (LFG) production, whereas the reference zone (3D) with lower moisture (~26–29%) showed minimal LFG generation. This pattern is reflected in the biocell experiment, where

from weeks 4 to 9, methane production stabilized at 5.5 mmoL $CH_4.d^{-1}$, indicating that hydrolysis, acidogenesis, acetogenesis, and methanogenesis had reached equilibrium. The metagenomic landfill study further supports this by demonstrating that methanogenic microbial communities are more abundant and metabolically versatile in newer waste, leading to rapid methane production early in landfill life cycles. In comparison, older waste enters a phase of slower methanogenesis due to substrate depletion. The Sudokwon landfill study also found that the cellulose-to-lignin ($Ce.L^{-1}$) ratio exhibited a significantly higher reaction rate than the C/N ratio, indicating that cellulose decomposes more rapidly than nitrogen-based compounds. This corresponds to the period between weeks 9 and 11 in the biocell experiment, during which methane production declines as the readily degradable organic matter becomes depleted, leaving behind more recalcitrant components such as lignin.

Additionally, the delayed methane production in biocell 2 aligns with the reference zone (3D) in the Sudokwon landfill, where lower moisture slowed microbial adaptation and waste degradation. The metagenomic analysis of landfill methanogens indicates that oxygen-limited conditions and substrate availability shape microbial community succession, influencing methane yield over time. The decline in methane production observed in weeks 9-11 of the biocell study could be due to substrate depletion, microbial aging, and inhibitory byproducts such as NH_4^+, a trend also noted in the landfill study, where older waste exhibited reduced methanogenic activity and increased potential for methane oxidation (Grégoire et al. 2023).

Comparing the three biocells, biocell 1 demonstrated the fastest initial methane production but also exhibited a more pronounced decline post-peak, suggesting a system optimized for rapid substrate utilization but with limited long-term stability. In contrast, biocell 2 had a delayed peak but achieved methane levels comparable to the other biocells in the steady-state phase, indicating a possible higher proportion of complex organic matter in its substrate. Biocell 3, which showed a more gradual and balanced response throughout, appears to have the most stable performance, potentially due to a well-maintained microbial equilibrium or better buffering capacity within the system.

The Chemical Oxygen Demand (COD) profile in Fig. 5, biocell 1, biocell 2, and biocell 3, provides crucial insights into the organic matter degradation process and its correlation with pH fluctuations and methane (CH_4) production. COD represents the concentration of biodegradable and non-biodegradable organic matter, and its variation over time reflects the efficiency of microbial activity in anaerobic digestion.

The COD decline from weeks 0-2, particularly the sharp drop in biocell 2 (~1300 ppm to ~800 ppm), suggests rapid degradation of easily hydrolyzable organic matter, which is consistent with the high organic load degradation observed in food waste anaerobic digestion (FW-AD) (Perin et al. 2020). This degradation aligns with the early pH drop observed in biocell 2, where an accumulation of volatile fatty acids (VFAs) likely occurred. In contrast, biocell 1 and biocell 3 show a more gradual COD reduction, suggesting a slower but more controlled hydrolysis process. The previously analyzed methane production data indicated that biocell 1 exhibited the highest initial CH_4 peak, which corresponds to a moderate COD decrease, confirming that methanogenesis efficiently converted intermediates into methane (Dang et al. 2023). However, in biocell 2, the lower initial methane yield and unstable pH suggest that excessive VFAs may

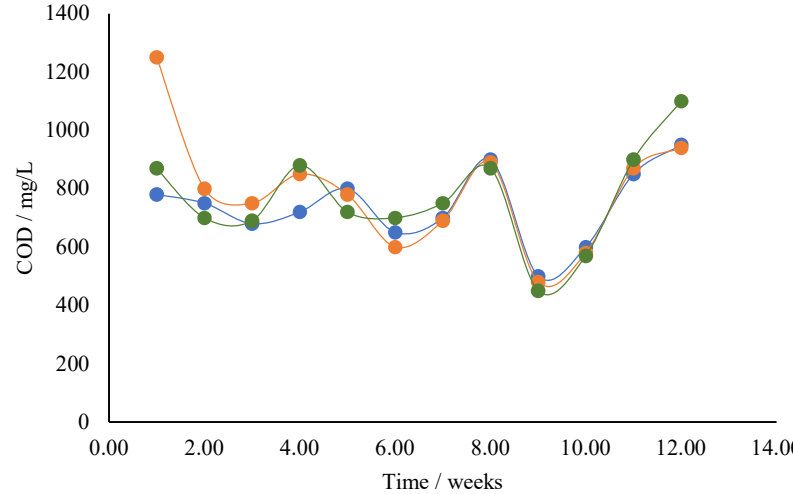
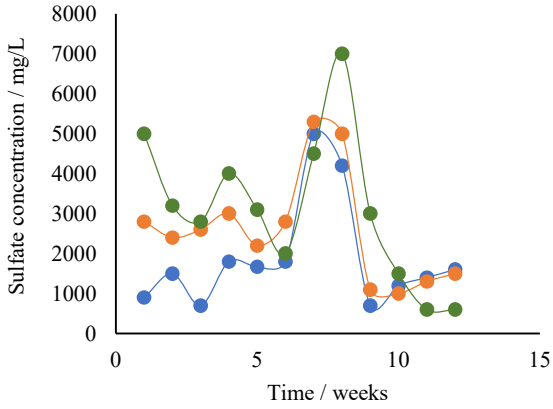


Fig. 5: Weekly COD monitoring: (blue dot) biocell 1; (orange dot) biocell 2; and (green dot) biocell 3.

have temporarily inhibited methanogenesis, leading to higher residual COD concentrations in the subsequent weeks.

Between weeks 2 and 8, all three biocells exhibit COD fluctuations, which align with pH oscillations and methane production variations. These fluctuations suggest intermittent substrate hydrolysis and VFA accumulation phases, followed by methanogenic activity consuming these intermediates. Around weeks 6-8, a COD decline is observed, corresponding to stable methane production and increasing pH in biocell 1 and biocell 3, indicating efficient organic matter conversion. In contrast, biocell 2 exhibits (Zhang et al. 2011) more erratic COD values, correlating with its observed pH drops and methane dips, suggesting ongoing acidification stress. Finally, from weeks 8-12, all



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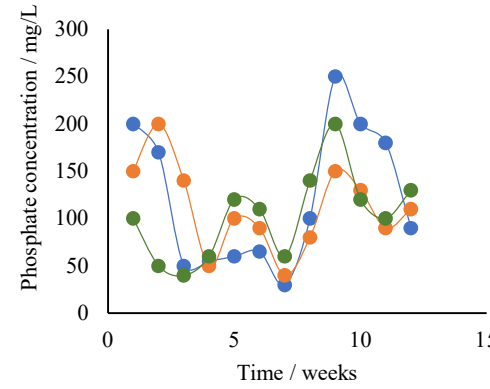

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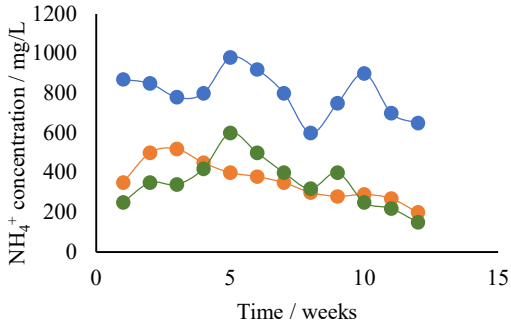

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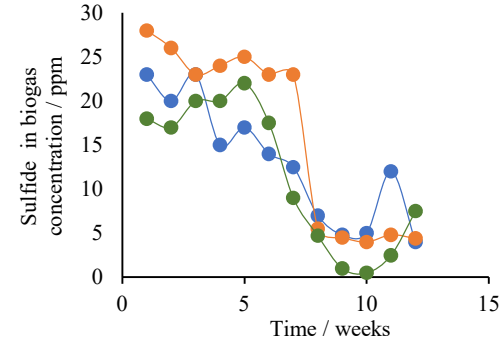

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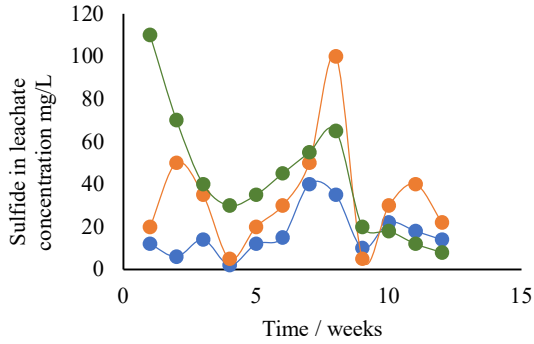

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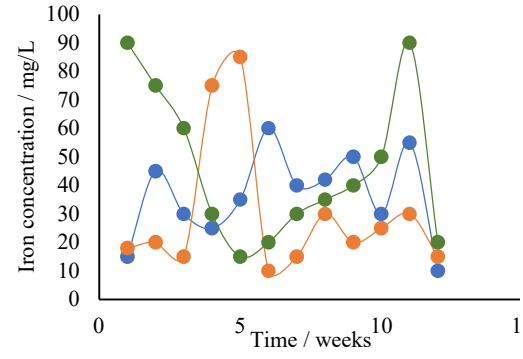

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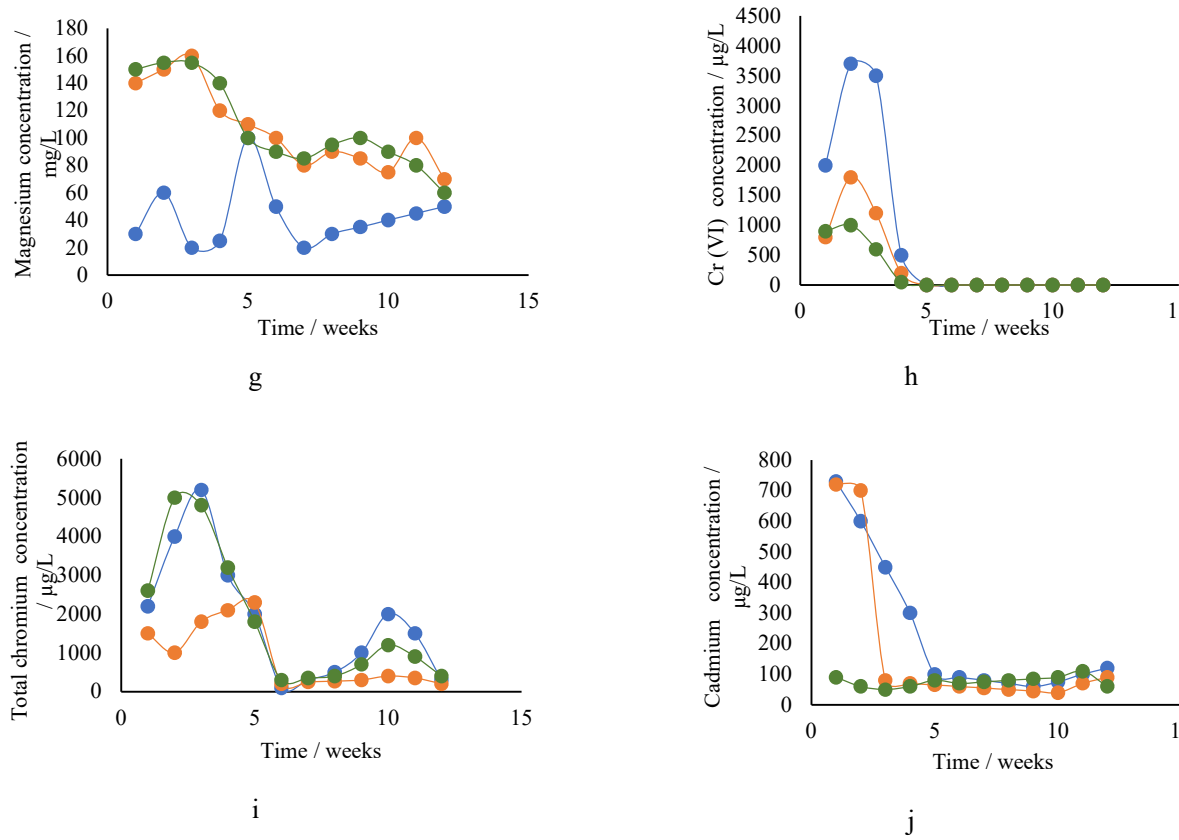

Fig. 6: Weekly Monitoring of Inorganic Compounds and Heavy Metals in Biocells: a) sulfate, b) phosphate monitoring, c) ammonium, d) sulfide in biogas, e) sulfide in leachate, f) iron, g) magnesium, h) chromium VI, i) total chromium, j) cadmium where (blue dot) biocell 1; (orange dot) biocell 2; and (green dot) biocell 3.

biocells experienced a COD increase, likely due to substrate depletion and reduced microbial activity. This final COD rise corresponds to the late-stage pH increase in biocell 1 and the decline in methane production, reinforcing the hypothesis that microbial activity is slowing at this stage, and residual recalcitrant organic matter remains unprocessed. These results show that stable pH regulation is crucial for efficient COD removal and sustained methane generation.

In Fig. 6a, sulfate concentrations show substantial fluctuations across all biocells. Biocell 2 starts with a high sulfate concentration (~5000 $mg.L^{-1}$) and rapidly declines, while biocell 1 and biocell 3 show a more gradual reduction after week 3. This trend suggests active sulfate-reducing bacteria (SRB) (Xia et al. 2014), which convert SO_4^{2-} into hydrogen sulfide (H_2S) under anaerobic conditions. The sharp drop in biocell 2 may indicate intense sulfate reduction activity, potentially linked to VFA accumulation and pH drops observed previously. The slower sulfate consumption in biocell 1 and biocell 3 suggests a more controlled microbial response, potentially reducing the risk of H_2S toxicity to methanogens (Jin et al. 2020, Ying et al. 2019).

The study on iron oxide-biochar composites by Wu et al. (2020) provides insight into the phosphate stability observed in the biocell landfill simulation. In Fig. 6b, phosphate concentrations remain stable (40–250 $mg.L^{-1}$), suggesting a balance between microbial uptake, solubilization, and potential adsorption onto mineral surfaces. The increase in phosphate levels in weeks 8–12 likely results from cell lysis and microbial turnover, releasing phosphorus into the system. The variability in biocell 2 may be linked to acidification effects on phosphate solubility, similar to Fe(III) biochar findings, which remain stable under pH fluctuations. The biochar study's 86.4% reduction in phosphate leaching suggests that iron-phosphate interactions could play a role in nutrient retention within the biocells, minimizing phosphorus loss into leachate. Iron-mediated adsorption and microbial cycling likely regulate phosphorus availability in the biocells, contributing to long-term stability and reduced leaching potential.

Fig. 6c, ammonium levels start high (~800-1000 $mg.L^{-1}$) and decline steadily in all biocells. This decreasing trend suggests effective ammonium uptake by

microbial communities or volatilization under alkaline conditions. Biocell 2 has the highest initial ammonium concentration (~1000 $mg.L^{-1}$), but its reduction rate is slower than biocell 1 and biocell 3, indicating a delayed microbial adaptation in biocell 2 or VFA accumulation inhibiting nitrification processes. By week 10, ammonium levels in biocell 1 and biocell 3 stabilize at ~250 $mg.L^{-1}$, which coincides with reduced methane production, suggesting substrate exhaustion and declining microbial activity (González-Cortés et al. 2021).

Fig. 6d and 6e, the higher sulfide concentrations in biogas compared to leachate in early weeks suggest that sulfate reduction was more active in the gas phase initially, whereas in leachate, sulfate-to-sulfide conversion varied depending on cell conditions. The delayed sulfide peak in biocell 2's leachate (week 8) corresponds with its relatively stable sulfide levels in biogas, suggesting that sulfate was retained in solution longer before microbial reduction became dominant. Biocell 3 exhibited the highest initial sulfide accumulation in leachate, which declined steadily, whereas biocell 1 demonstrated more stable sulfide trends in biogas and leachate. These differences indicate variable sulfate reduction rates, substrate availability, and microbial adaptation between biocells, affecting the balance between sulfide retention in the liquid phase and release as hydrogen sulfide in biogas (Long et al. 2016).

Fig. 6f, iron concentrations fluctuate across all biocells, with a steady decline from ~100 $mg.L^{-1}$ to ~20 $mg.L^{-1}$ by week 10, suggesting that iron is being consumed by iron-reducing bacteria (IRB) or precipitated as FeS due to sulfide interactions. Biocell 1 and biocell 3 show a more rapid iron decrease, suggesting stronger iron-dependent microbial activity, potentially enhancing redox reactions that influence electron flow in anaerobic digestion. Biocell 2 maintains residual Fe levels, possibly helping to buffer against excessive sulfide accumulation.

Fig. 6g, magnesium concentrations remain relatively stable in all biocells but drop slightly after week 6 (~90 to ~50 $mg.L^{-1}$). This decline suggests incorporation into microbial biomass (Liu et al. 2019) or precipitation as $MgNH_4PO_4$ (struvite), a common byproduct in anaerobic digestion (Muhmood et al. 2019). Stabilization in biocell 3 aligns with more controlled pH trends, reinforcing the importance of alkalinity in preventing nutrient losses.

Fig. 6h and 6i, comparing total chromium (Cr-total) and hexavalent chromium (Cr-VI) concentrations across the biocells, reveal distinct reduction trends and transformation dynamics during the experimental period. Initially, Cr-total concentrations in all biocells are high, with peaks occurring at week 3 (biocell 1: 5200 $\mu g.L^{-1}$, biocell 2: 1800 $\mu g.L^{-1}$, biocell 3: 4800 $\mu g.L^{-1}$), indicating significant chromium mobilization, likely from the degradation of organic matter or dissolution of metal complexes. Over time, Cr-total concentrations decrease substantially, reaching below 500 $\mu g.L^{-1}$ in all biocells by week 12, suggesting progressive chromium precipitation or microbial-mediated immobilization. In contrast, Cr-VI shows a sharp decline much earlier, reaching undetectable levels by week 5, with initial peaks in biocell 1 (3700 $\mu g.L^{-1}$ at week 2), biocell 2 (1800 $\mu g.L^{-1}$ at week 2), and biocell 3 (1000 $\mu g.L^{-1}$ at week 2), followed by rapid depletion by week 4 due to an effective reduction process, likely driven by anaerobic microbial activity facilitating electron transfer and Cr-VI reduction to the less toxic Cr-III form. Cr-total persistence beyond week 5 suggests that a portion remains in less bioavailable fractions, possibly as precipitates (e.g., chromium hydroxides) or bound within organic matrices. The faster reduction of Cr-VI compared to Cr-total suggests that biogeochemical conditions within the biocells favor detoxification mechanisms, potentially involving sulfate-reducing bacteria (SRB) (Yang et al. 2021) or iron-mediated reduction pathways, which are known to facilitate Cr-VI to Cr-III conversion in anaerobic environments.

In Fig. 6j, cadmium concentrations exhibit a progressive decline from ~120 $\mu g.L^{-1}$ to ~50 $\mu g.L^{-1}$ across all biocells, indicating effective metal immobilization through adsorption onto biomass, precipitation, or complexation with sulfides. Biocell 3 demonstrates slightly lower Cd levels in later weeks, suggesting enhanced removal efficiency, potentially driven by a more diverse microbial community or stronger interactions with sulfide and iron species. This trend aligns with the findings of Zhang et al. 2023), who investigated landfill leachate sludge-derived biochar (LLSDB) and reported that low Cd concentrations follow a linear adsorption model (physical adsorption). In contrast, higher concentrations engage in chemical adsorption mechanisms. The observed cadmium reduction in the biocells suggests a transition from initial physical adsorption onto organic matrices to subsequent chemical stabilization via sulfide precipitation or complexation with iron oxides, reinforcing the role of microbial and geochemical processes in heavy metal remediation.

The characterization of the solid phase at the end of biogas production, Table 4, provides crucial insights into the retention, transformation, and environmental behavior of key inorganic compounds, including sulfates, phosphates, magnesium, chromium, and cadmium (Frank et al. 2017). Sulfate concentrations were highest in biosolids (156.1 $g.kg^{-1}$ in biocell 1), indicating significant retention. At the same time, lower levels in compost and plant matter suggest partial sulfate reduction via sulfate-reducing bacteria (SRB)

Table 4: The concentration of key inorganic compounds (sulfates, phosphates, magnesium, chromium VI, total chromium, and cadmium) in the solid phase (biosolids, compost, and green waste) of the three biocells at the end of biogas production.

Compound	Biocell	WWTP Biosolids	Compost	Green waste
	1	156.1 ± 4.71	74.02 ± 0	54.1 ± 0
Sulfate, $mg.kg^{-1}$	2	112.3 ± 4.71	69.6 ± 5.18	30.7 ± 0
	3	115.9 ± 5.77	90.6 ± 5.77	26.2 ± 0
	1	8.8 ± 0.94	8.9 ± 1.41	3.7 ± 0.81
Phosphate, $mg.kg^{-1}$	2	6.7 ± 0.47	6.04 ± 0.94	0.7 ± 0.47
	3	8.2 ± 0.78	6.1 ± 0.31	0.9 ± 1.24
	1	4.5 ± 0.8	2.3 ± 0.4	0.8 ± 0
Magnesium, $mg.kg^{-1}$	2	4.4 ± 0.7	1.7 ± 0.8	1.2 ± 0.3
	3	4.5 ± 0.7	1.7 ± 0.3	0.7 ± 0.1
	1	1.4 ± 0.7	1.1 ± 0.3	0.6 ± 0.7
Total Chromium, $mg.kg^{-1}$	2	1.2 ± 0.5	0.9 ± 0.1	0.5 ± 0.1
	3	1.5 ± 0.5	1.1 ± 0.2	0.7 ± 0.1
	1	0.10 ± 0.06	0.08 ± 0.03	0.06 ± 0001
Cadmium, $mg.kg^{-1}$	2	0.10 ± 0,07	0.07 ± 0.04	0.06 ± 0.09
	3	0.10 ± 0.08	0.07 ± 0.06	0.07 ± 0.02

(Ghosh et al. 2020). Phosphates remained stable in biosolids and compost but were significantly lower in green waste, demonstrating that phosphorus is primarily retained within the solid matrix. Magnesium exhibited a progressive decline from biosolids to compost to green waste, likely due to mineral precipitation as struvite ($MgNH_4PO_4 \cdot 6H_2O$).

Total chromium and cadmium concentrations were low across all biocells, but trace cadmium was detected in plant residues (~0.06–0.07 $mg.kg^{-1}$), suggesting a potential bioaccumulation risk. Comparatively, biocell 3 exhibited the highest retention of sulfate, phosphate, magnesium, and chromium, indicating greater stabilization efficiency. These findings emphasize the need for optimized redox control, sulfate management to limit excess sulfide formation, and enhanced phosphorus recovery strategies, while cadmium retention in plant biomass warrants further investigation into its environmental risks and mitigation strategies.

CONCLUSIONS

This study demonstrates the crucial role of biocell design and operational parameters in optimizing anaerobic waste degradation and biogas production. The strategic integration of WWTP biosolids, compost, and green waste promoted a balanced microbial ecosystem, facilitating efficient methanogenesis and organic matter stabilization. The results confirm that a well-structured layering system and controlled leachate recirculation and moisture management enhance substrate bioavailability, directly impacting methane yields and leachate composition. The variability in methane production across biocells highlights the influence of waste composition, microbial adaptation, and nutrient availability in determining bioreactor performance.

Methane generation followed a characteristic pattern, with biocell 1 achieving rapid initial methane production but experiencing early substrate depletion and unstable long-term performance. In contrast, biocell 2 exhibited a delayed but sustained methane peak, indicative of the gradual breakdown of complex organic matter and a slower microbial adaptation process. Biocell 3 demonstrated the most stable methane output, suggesting that its composition and buffering capacity supported microbial equilibrium over time. The decline in methane production after week nine across all biocells suggests substrate depletion and potential microbial inhibition due to ammonia accumulation, reinforcing the need for optimized nutrient ratios to sustain methanogenesis.

Leachate recirculation was vital in sustaining methane production, influencing microbial activity, and affecting system longevity. The high initial leachate retention (~65%) facilitated methanogenic bacterial proliferation, but retention capacity declined significantly after week two (~11-13%), highlighting changes in system permeability and moisture absorption. These findings emphasize the necessity for adaptive leachate management strategies to sustain high retention rates and prevent excessive leachate loss, which could lead to uneven waste degradation and reduced biogas yields. The pH dynamics in the biocells further underscored the complexity of microbial interactions, with biocell 3 exhibiting the most stable pH profile, supporting

consistent methanogenesis, while biocell 1 and biocell 2 experienced fluctuations corresponding to microbial stress and acidification.

Nutrient cycling and heavy metal immobilization were also significantly influenced by biocell conditions. WWTP biosolids were crucial in supplying essential nutrients such as nitrogen, phosphorus, and potassium, enhancing microbial metabolic activity. Chromium (VI) was effectively reduced to the less toxic Cr(III), confirming that microbial-mediated detoxification processes were active within the biocells. Additionally, cadmium bioavailability decreased over time, suggesting successful immobilization through sulfide precipitation and iron oxide complexation. The differences in heavy metal retention between biocells indicate that optimizing redox conditions and microbial community composition could further enhance metal stabilization.

Several optimization strategies should be considered to improve biocell performance. Organic substrate composition should be adjusted to balance the C/N ratio and sustain microbial activity and methane production. Moisture management and leachate recirculation must be dynamically adjusted based on real-time microbial activity and substrate degradation monitoring to prevent excessive leachate loss and maintain optimal moisture levels. Furthermore, enhancing microbial consortia through bioaugmentation strategies could improve both methane yields and heavy metal immobilization, making biocell technology a more effective tool for landfill waste stabilization.

In summary, this study assesses the viability of biocell technology as a sustainable waste management alternative for Ambato, highlighting its potential to improve waste stabilization and reduce environmental impact. The results underscore the need for optimizing operational parameters, microbial dynamics, and nutrient balance to maintain efficient methanogenesis and effective leachate management. Successful implementation could enhance methane recovery as a renewable energy source while limiting heavy metal mobility and ensuring long-term environmental stability.

Future research should focus on developing adaptive biocell designs that integrate bioaugmentation with specialized microbial consortia and enzymatic catalysts to enhance organic matter degradation, optimize methane production, prevent contaminant migration, and strengthen the overall environmental sustainability of waste management systems. Additionally, since the biocells were evaluated under laboratory-scale conditions, pilot-scale field studies are necessary to address the operational and environmental complexities of real landfill settings. Such validation would also help assess long-term performance, scalability, and adaptability under diverse waste compositions and climatic conditions. Addressing these aspects in future research will facilitate the transition from laboratory-scale experimentation to practical, full-scale implementation.

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From Sea to Science: Unveiling the Diverse Biotechnological Applications of Marine Endophytic *Bacillus aerius*

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ABSTRACT

Marine endophytic bacteria are a promising source of bioactive compounds with diverse applications. This study investigated the multifunctional properties of *Bacillus aerius* PMRU2.8, isolated from the marine red alga *Gracilaria sp*. collected from the coastal region of Tamil Nadu, India. The bacterium demonstrated significant antimicrobial activity against multiple human pathogens, with its ethyl acetate extract containing bioactive compounds, including indoles and ketones. Molecular docking analysis revealed potential binding mechanisms of the compounds to bacterial proteins. Additionally, *B. aerius* efficiently synthesized silver nanoparticles (AgNPs) with enhanced antimicrobial efficacy compared with the crude extract. The bacterium also exhibited remarkable bioremediation capability, decolorizing up to 92.5% of the Direct Blue 6 azo dye within 48 h. Cytotoxicity assays confirmed the potential therapeutic applications of both the extract and the biosynthesized AgNPs. These findings highlight *B. aerius* as a valuable resource for pharmaceutical development, nanobiotechnology, and environmental remediation.

INTRODUCTION

Marine organisms constitute a rich source of diverse bioactive metabolites exhibiting antimicrobial, antifungal, antiviral, and anticancer activities (Zhang et al. 2019). These properties present significant potential for developing novel drugs and therapeutic agents. Investigating the diversity and ecological roles of marine bacteria associated with algal samples is of paramount importance (Kaur et al. 2023, Gu et al. 2023). These bacteria often inhabit extreme environments and possess unique metabolic adaptations, producing secondary metabolites for defense (Petersen et al. 2020, Karthikeyan et al. 2022). Recent studies have successfully isolated novel bioactive compounds from these marine bacteria, underscoring their potential as therapeutics (Machado et al. 2015, Brito et al. 2018). Algae-associated bacteria produce antibacterial, antiviral, and anticancer compounds. Research has demonstrated that extracts from these bacteria exhibit antimicrobial activity, including efficacy against methicillin-resistant *Staphylococcus aureus* (MRSA) (Kranjec et al., 2020). The development of new antibacterial drugs is imperative to address the increasing prevalence of antibiotic resistance (Butler et al. 2022). Silver nanoparticles (AgNPs) have demonstrated considerable promise as broad-spectrum antimicrobial agents because of their small size and high surface area. AgNPs synthesized using bacteria have been evaluated for their antimicrobial effects (Yin et al. 2020). However, the cytotoxicity of AgNPs remains a significant concern in biomedical applications. Textile dyes are a major source of global water pollution (Kishor et al. 2021). Azo dyes are particularly concerning because of their stability and resistance to degradation (Varjani et al. 2020). Bioremediation using microorganisms is a promising method for removing azo dyes from wastewater

(Chen et al. 2021). Bacteria capable of degrading azo dyes have been isolated from textile wastewater and textile sludge. However, degradation efficiency varies significantly depending on the bacterial strain and dye structure (Paba et al. 2021).

Despite these advances, a significant research gap exists in the comprehensive characterization of marine red algae-associated bacteria with multifunctional capabilities. While isolated studies have explored antimicrobial properties (Huang et al. 2023), nanoparticle synthesis, and dye degradation separately (Jamil et al. 2024), few investigations have systematically evaluated all these functionalities within the same bacterial isolates from marine red algae. Furthermore, the molecular mechanisms underlying these bioactivities, particularly through chemical profiling and in silico analysis of active compounds, remain insufficiently explored in this ecological niche on the southeast coast of Tamil Nadu.

Therefore, the present study aimed to isolate bacteria associated with marine red algae and systematically evaluate their multifaceted bioactive potential, including antimicrobial, dye decolorization, and cytotoxic activities. Additionally, this study investigated the biosynthesis of silver nanoparticles and conducted chemical profiling of antibacterial extracts from the isolates. These results provide insights into the biotechnological applications of algae-associated bacteria as sources of therapeutic natural products and environmentally sustainable processes.

MATERIALS AND METHODS

Isolation of Endophytic Bacteria

Marine bacteria associated with the red algae *Gracilaria* sp., collected from the rocky beach of Manapaadu, Tamil Nadu, India, were isolated and enumerated using a standardized serial dilution and plating technique (Shen & Zhang 2023). Algal tissues were surface sterilized and homogenized in sterile seawater (Mangun et al. 2023). The homogenate was serially diluted and subsequently plated onto Zobell Marine agar medium (Prabhakara & Kuehn 2023). Following a 72-hour incubation period at 27°C in the dark, bacterial colonies were enumerated, subcultured, and preserved as pure cultures.

Preliminary Screening

Isolates were cultured in Zobell marine broth for 96 h, and the fermented supernatant was collected from each culture. These supernatants were concentrated and subsequently assayed for antibacterial activity against human pathogens, including Escherichia coli, Klebsiella pneumoniae, Pseudomonas aeruginosa, Enterococcus faecalis, Serratia marcescens, Proteus mirabilis, Salmonella typhimurium, Staphylococcus aureus, Vibrio sp. and Aeromonas hydrophila, utilizing the paper disc diffusion method (Carvalho et al. 2018). Isolates demonstrating maximum antibacterial activity were selected for further experiments.

Polyphasic Recognition of Potential Isolates

Morphological Characterization: In accordance with Bergey's Manual of Determinative Bacteriology (Buchanan & Gibbons, 1974), colony morphology was evaluated on nutrient agar, encompassing shape, form, edge, elevation, and pigmentation of the colonies. Cell size and shape were observed microscopically at 10x and 100x magnification. Gram staining (Bullock & Aslanzadeh 2012) and the hanging drop method were utilized to determine Gram reaction and motility, respectively.

Molecular Identification of Bacterial Isolates: The Genomic DNA of the bacterial isolates was extracted using the Nucleo Spin Tissue kit. The 16S rRNA gene was amplified via PCR using universal primers P63f and P1378r, Phire PCR master mix, primers, water, and template DNA. The thermal cycling protocol comprised initial denaturation at 95°C for 5 min, followed by 35 cycles of denaturation (95°C, 40 s), annealing (60°C, 40 s), extension (72°C, 60 s), and a final extension at 72°C for 7 min. Amplification was confirmed by agarose gel electrophoresis with ethidium bromide staining and UV imaging. Sequencing was performed using the Big Dye Terminator v3.1 kit on an ABI 3500 genetic analyzer, with quality assessment conducted via Sequence Scanner v1 and alignment performed using Geneious Pro v5.1. Identification was accomplished through a BLAST search against the NCBI GenBank database, confirming 100% sequence similarity to the prototype strains.

Secondary Metabolite Extraction and Spectroscopy Evaluation from Potent Isolates

The bacterial isolate was cultured in starch casein media at 29°C with agitation for 48 h. Subsequently, a 2% inoculum was transferred to fresh production media (8 L) and incubated under similar conditions for 8-12 days. Following fermentation, the biomass was separated via filtration. The filtrate was combined with ethyl acetate, and after overnight agitation, the bioactive compounds partitioned into the organic layer (Xie et al. 2021). The organic layer was subsequently collected and concentrated using a vacuum rotary evaporator to remove ethyl acetate, resulting in purified bioactive compounds. Gas chromatography-mass spectrometry (GC-MS) and Fourier-transform infrared spectroscopy (FTIR) were employed to identify the antimicrobial compounds and functional groups,

facilitating their characterization.

Molecular Docking Analysis

Molecular docking simulations were conducted using Auto-Dock Vina. Ligand structures were obtained from PubChem. The sdf format underwent energy minimization using the ChemBio3D software. The three-dimensional structures of the receptor proteins (PDB ID: 3K8E, 1N67, and 1T2P) were obtained from the Protein Data Bank and prepared by eliminating co-crystallized ligands, water molecules, and cofactors. Auto-Dock 4.2 (MGL tools 1.5.6) prepared files, and Auto-Dock Vina was used to execute the docking simulations. The docking grid encompassed the entire protein target area, exploring nine ligand conformations. Ligand-receptor interactions were analyzed, with an emphasis on favorable binding energies and key residues, using Discovery Studio Visualizer in three-dimensional formats.

ADMET Analysis

Using the Swiss ADME online tool, computational predictions of citronellal and terpinen-4-ol ADME, drug-likeness, and pharmacokinetics were conducted. The following physicochemical parameters and ADME features were determined: molecular weight (MW), hydrogen bond acceptor (HBA), hydrogen bond donor (HBD), topological polar surface area (TPSA), octanol/water partition coefficient (LogP), and rotatable bond count (RB).

Silver Nanoparticle Biosynthesis

Silver nanoparticles were synthesized utilizing cell-free supernatants of a potent bacterial isolate cultured in Zobell marine broth 2216 (Ghodake et al. 2020). The broth was centrifuged, and the resulting supernatant was combined with 1mM silver nitrate solution in a 1:10 ratio. The formation of AgNPs was indicated by a color change from white to brown and subsequently confirmed through UV-vis spectroscopy using a Shimadzu UV1800 spectrophotometer, with spectra recorded from 200-1100 nm at 24-h intervals. The nanoparticles were characterized by centrifugation at 5000 rpm, followed by washing with ethanol and drying for SEM analysis (Chakraborty et al. 2023).

Antibacterial Activity

The antibacterial activity of the ethyl acetate extracts and AgNPs of the selected isolates was evaluated using the disc diffusion assay (Bauer et al. 1966) against clinically isolated bacterial pathogens obtained from the Scudder Microbiology Laboratory, Nagercoil, India. Mueller-Hinton agar plates were inoculated with pathogen cultures grown to a 0.5 McFarland standard. Filter paper discs were impregnated with extracts, silver nanoparticles, positive control (ampicillin), and negative controls (DMSO and ethyl acetate). The plates were incubated at 37°C for 18-24 h, and the inhibition zones were measured (Bauer et al. 1966). The percentage of inhibition was calculated using Equation (1).

$$PI = \frac{100 \times (X - Y)}{(Z - Y)} \quad ...(1)$$

Where X = Mean test extract,

Y = Mean negative control,

Z = Mean positive control.

Azo Dye Degradation

Several azo dyes have been evaluated for biodegradation by marine bacteria. The dyes examined included Brilliant Green ($C_{27}H_{34}N_2O_4S$, CAS: 633-03-4, λ max- 244 nm), Sudan black B ($C_{29}H_{24}N_6$, CAS: 4197-25-5, λ max- 454 nm), Direct Blue 6 ($C_{32}H_{20}N_6Na_4O_{14}S_4$, CAS: 2602-46-2, λ max- 566 nm), and Disperse Red 1 ($C_{16}H_{18}N_4O_3$, CAS: 2872-52-8, □ max- 480 nm). Zobell marine broth 2216 was prepared (30 mL.tube^{-1}), sterilized, and inoculated with the bacterial isolate. Dye degradation experiments were conducted by adding dyes (2 mg.mL^{-1}) to 24 h cultures. The decolorization was visually monitored. Cell-free supernatants were analyzed using UV-vis spectrophotometry on a Shimadzu UV 1800 instrument. The maximum absorbance wavelengths were determined by scanning from 200 to 1100 nm. Abiotic and uninoculated controls were also included. The decolorization percentage and average rates were calculated using Equations (2) and (3), respectively.

$$\text{Percentage of decolorization} = \frac{(Initial\ absorbance - Final\ absorbance)}{Initial\ absorbance} \times 100 \quad ...(2)$$

$$\text{Average decolorization rate} = \frac{C \times \%D}{100 \times t}\ \text{mg/L/h} \quad ...(3)$$

Where C is the initial dye concentration,

% D is the percentage of dye decolorized after time t.

Brine Shrimp Lethality Bioassay

The cytotoxicity of the bacterial extracts was evaluated using the brine shrimp lethality assay (Meyer et al., 1982). Brine shrimp larvae were hatched in artificial seawater under oxygenation conditions. Groups of 10 nauplii were subjected to varying concentrations (1-1000 µg.mL^{-1}) of ethyl acetate extract (EAE) and silver nanoparticles (AgNPs) from the potent isolate B. aerius PMRU2.8 dissolved in seawater with 1% DMSO. Each concentration was replicated three times, with 1% DMSO serving as the negative control. After 24 h, the surviving nauplii were enumerated, and the percentage

mortality was calculated, adjusting for control mortality. The median lethal concentration (LC_{50}) was determined through probit analysis using the SPSS software (version 22, IBM Corp.). For the analysis, mortality percentage data were plotted against the logarithm of the concentration, and linear regression analysis was performed to generate the regression equations. The LC_{50} values were calculated by interpolating the 50% mortality point on these regression lines. An LC_{50} value exceeding 1000 $\mu g.mL^{-1}$ was considered indicative of non-toxicity.

A schematic of the experimental pipeline is shown in Fig. 1.

RESULTS AND DISCUSSION

Preliminary Screening

A total of 48 bacterial strains were isolated as endophytes from the marine red alga *Gracilaria* sp. and evaluated for their antimicrobial activity against human pathogens. The isolates exhibiting inhibitory activity against the pathogens are listed in Table 1, which indicates that isolate MR-60 demonstrated superior activity among the isolates. The morphological and biochemical characteristics of these potent isolates are systematically presented in Table 2.

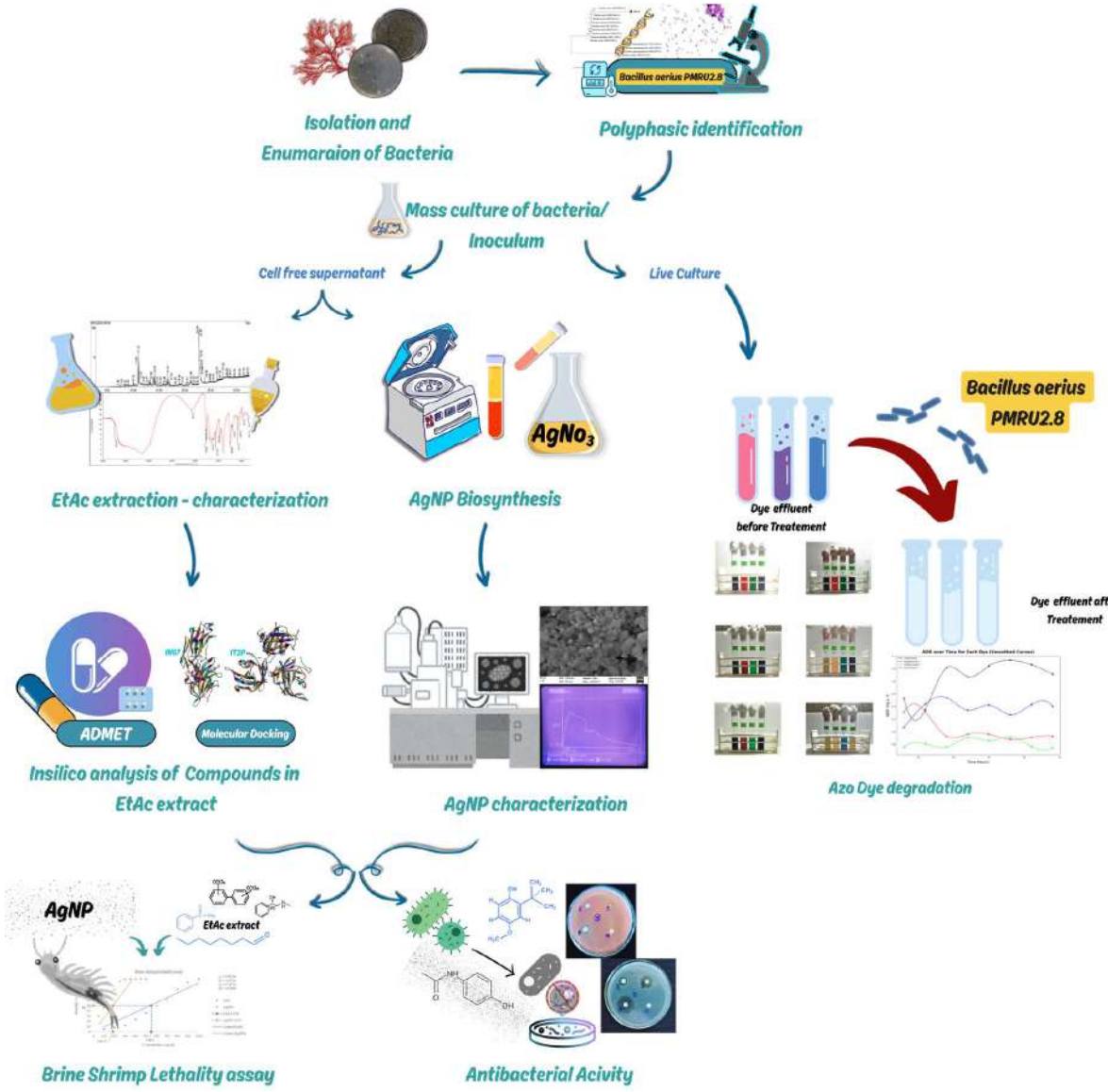


Fig. 1: Schematic Diagram of the Experimental Pipeline.

Table 1: Preliminary screening of potent isolates.

Pathogens	MR 08	MR 14	MR 26	MR 32	MR 39	MR 41	MR 53	MR 60	MR 63	MR 65
Escherichia coli	++	-	-	-	-	+	+	+++	-	+
klebsiella pneumoniae	+	-	-	-	+	-	-	+++	+	-
Pseudomonas aeruginosa	-	+++	++	-	++	+++	+	-	++	-
Enterococcus faecalis	-	+	-	++	+	-	-	++	-	+
Serratia marcescens	-	-	-	+	+	+	-	-	-	+
Proteus mirabilis	+	-	+++	+	-	+	-	+++	-	-
Salmonella typhimurium	-	+++	-	++	-	+	+++	+	+	-
Staphylococcus aureus	-	-	-	-	-	+++	-	+	-	-
Vibrio sp.	+	+	+++	-	+	-	+	++	-	+
Aeromonas hydrophila	+	-	+	-	+++	-	+	++	+	-
Total	5	4	4	4	6	6	5	8	4	4

'+++' – High activity, '++' Medium activity, '+'low activity, '-no activity.

Table 2: Morphological and Biochemical Characterizations.

Isolates	MR 08	MR 14	MR 26	MR 32	MR 39	MR 41	MR 53	MR 60	MR 63	MR 65
Morphology										
Gram's stain	-	-	+	-	+	+	-	+	-	+
Motility	+	-	+	-	-	+	+	+	+	+
Pigment	White	White	White	Yellow	Gray	White	White	White	White	White
Shape	Rod	Rod	Rod	Rod	Rod	Rod	Rod	Rod	Rod	Rod
Biochemical tests										
Indole	+	-	-	-	-	-	-	-	-	-
Methyl red	-	-	+	-	-	-	-	-	-	-
Voges Poskauer	-	-	-	-	-	-	-	+	+	-
Citrate	+	+	+	-	-	-	+	+	+	-
Oxidase	+	+	-	+	-	-	-	-	+	+
Catalase	+	+	+	+	+	+	+	+	+	+
H_2S Production	-	-	+	-	-	-	-	+	-	+
Starch	-	-	-	-	+	-	-	+	+	+
Nitrate reduction test	-	+	+	+	+	+	-	+	+	-
Gelatinase	+	+	-	-	+	-	-	+	-	-
Sugar Utilization										
Sucrose	+	+	-	+	+	+	-	+	+	+
Glucose	+	+	-	+	+	-	-	+	-	-
Rhamnose	-	-	-	-	-	-	-	-	-	-
Maltose	-	+	-	+	+	+	-	+	+	-
Lactose	+	+	-	-	+	+	-	-	+	-
Mannitol	+	+	-	+	+	+	-	+	-	-
Identified organisms	*Vibrio* sp.	*Aeromonas* sp.	*Bacillus* sp.	*Pseudomonas* sp.	*Clostiridium* sp.	*Lactobacillus* sp.	*Acinetobacter* sp.	*Bacillus* sp.	*Photobacterium* sp.	*Lysinibacillus* sp.

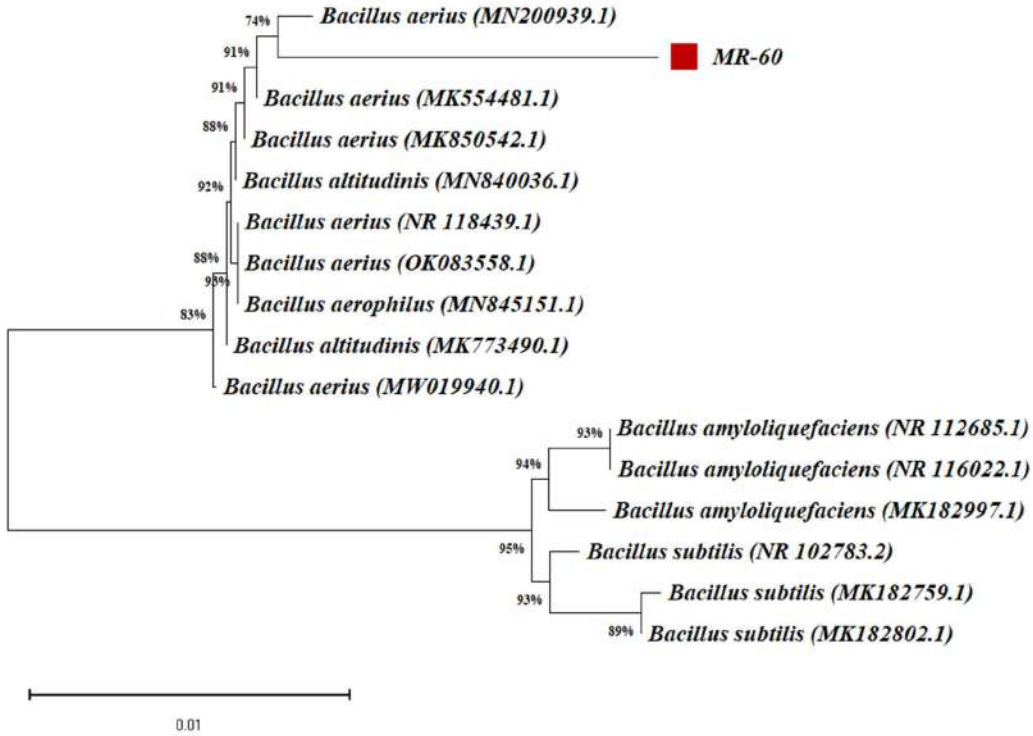


Fig. 2: Phylogenetic analysis of B. aerius PMRU2.8.

Molecular identification of the isolate exhibiting the highest activity, MR-60, was performed through 16S rRNA gene sequencing. The sequence exhibited maximum similarity to *Bacillus aerius* strain 24 K (MN200939.1), with 100% query coverage and 98.12% identity. Phylogenetic analysis indicated that isolate MR-60 formed a closely related clade with *Bacillus aerius* type sequences, whereas *B. subtilis* and B. *amyloliquefaciens* served as outgroups (Fig. 2). Based on phenotypic, biochemical, and molecular characterization, isolate MR-60 was confirmed to be *Bacillus aerius*. The sequence was subsequently submitted to the NCBI with the GenBank accession number PP415878 as *Bacillus aerius* PMRU2.8.

Analysis of Ethyl Acetate Extract of *B. aerius* PMRU2.8

The ethyl acetate extract of *B. aerius* PMRU2.8 was analyzed using FTIR spectroscopy and GC-MS, revealing a diverse chemical profile (Table 3, Figs. 3 & 4). FTIR spectroscopy identified key functional groups (carboxylic acids, ketones, esters, aromatics, and alkanes), whereas GC-MS confirmed the presence of specific compounds, including heterocyclic aromatics (indole, 2,5-dimethylpyrazine), ketones, esters, carboxylic acids, sulfur compounds (dimethyl disulfide), and alkanes (octadecane). Several compounds, particularly indole derivatives and nitrogen-containing aromatics,

Table 3: Analysis of Ethyl Acetate Extract of B. aerius PMRU2.8.

CID	GCMS		FT-IR	
	Compound Name	Mol. Formula - Mol. Weight	Functional Group	Wavenumber Range with Bond Stretch
798	Indole	C_8H_7N - 117.15	Aromatic Amine	1450-1600 (C=C), 3300-3500 (broad, N-H)
31252	2,5-Dimethylpyrazine	$C_6H_8N_2$ - 108.14	Aromatic ring - N	1450-1600 (C=C)
8034	5-Methyl-2-hexanone	$C_7H_{14}O$ - 114.19	Ketone	1700-1750 (C=O)
12232	Dimethyl disulfide	$C_2H_6S_2$ - 94.2	Sulfide	600-700 (C-S)
24020	Ethyl 2-methylbutanoate	$C_7H_{14}O_2$ - 130.18	Ester	1720-1770 (C=O), 1200-1300 (C-O)
10430	3-Methyl-butanoic acid	$C_5H_{10}O_2$ -102.13	Carboxylic acid	2500-3300 (broad, O-H), 1700-1750 (C=O)
7909	Methyl isobutyl ketone	$C_6H_{12}O$ - 100.16	Ketone	1700-1750 (C=O)
11635	Octadecane	$C_{18}H_{38}$ - 254.5	Alkane	2800-2950 (C-H)

possess antimicrobial properties. This chemical diversity demonstrates the complex metabolic capabilities of the isolate and suggests potential applications in the agricultural, pharmaceutical, and industrial fields.

Molecular Docking Analysis

Molecular docking analysis was used to assess the binding affinities of the eight volatile compounds against three bacterial proteins critical for pathogenesis (Table 4). For CMP-Kdo synthetase (PDB ID: 3K8E), indole exhibited the strongest binding, with a docking score of -6.3, whereas dimethyl disulfide showed the weakest interaction, with a score of -2.0. In the case of Clumping Factor A (PDB ID: 1N67), indole again demonstrated superior binding affinity with a score of -7.0, while 3-methyl-butanoic acid had the highest score of -4.6, indicating weaker binding. Regarding SrtA (PDB ID: 1T2P), octadecane displayed the strongest binding affinity with a score of -6.3, whereas 2,5-dimethylpyrazine exhibited the weakest interaction with a score of -4.2. These findings suggest that indole may inhibit CMP-Kdo synthetase and Clumping Factor A, whereas octadecane shows potential as a Sortase A inhibitor. Conversely, dimethyl disulfide demonstrated poor binding affinity for all targets, indicating its limited antimicrobial potential.

ADMET Analysis

Ethyl 2-methylbutanoate demonstrated the highest number of hydrogen bond acceptors (HBA: 5) and donors (HBD: 1), indicating favorable solubility and binding properties. However, its topological polar surface area (TPSA: 72.83 Å²) may restrict membrane permeability. Octadecane exhibited significant lipophilicity (LogP: 7.18) but limited aqueous solubility, along with a substantial number of rotatable bonds (Rb: 15), suggesting its structural flexibility. Dimethyl disulfide and ethyl 2-methylbutanoate both presented a relatively high TPSA (50.60 Å²), whereas

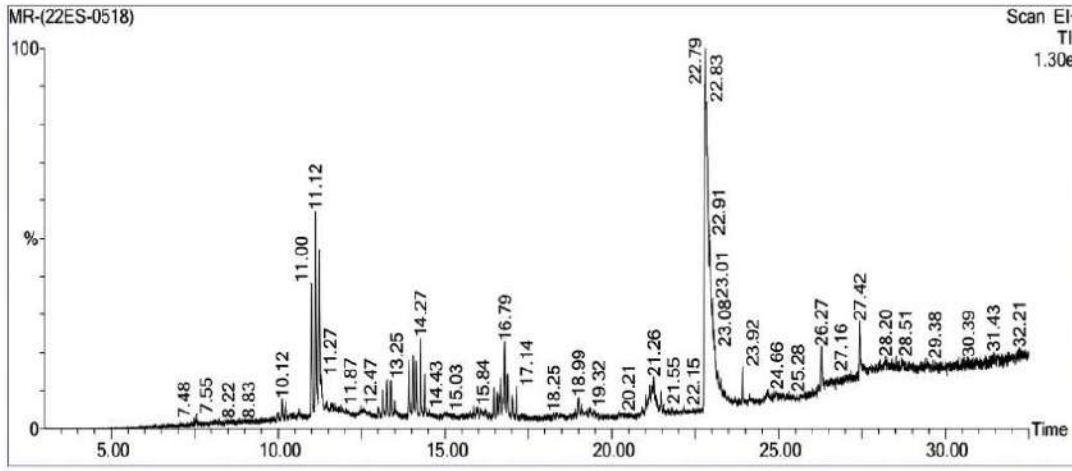


Fig. 3: GC-MS Analysis of Ethyl Acetate Extract.

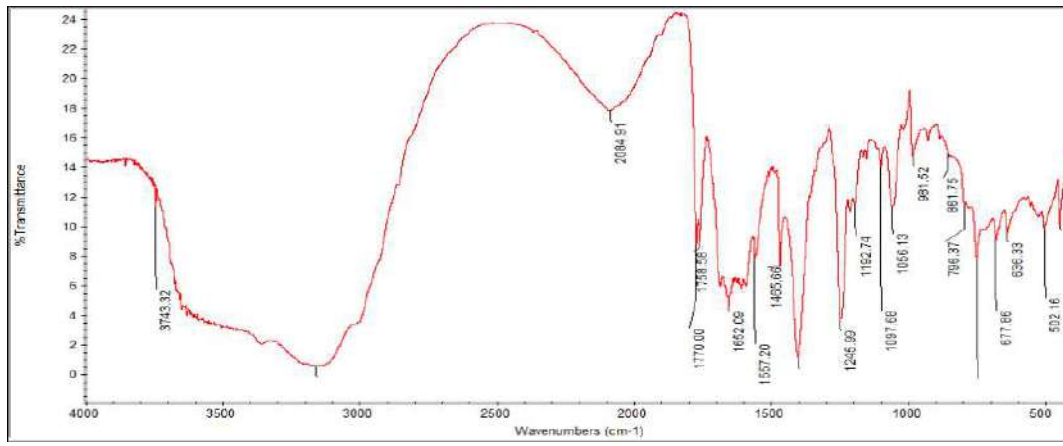


Fig. 4: FT-IR Analysis of Ethyl Acetate Extract.

Table 4: Molecular Docking Analysis of Compounds in Ethyl Acetate Extract of *B. aerius* PMRU2.8.

Compounds	3K8E	1N67	1T2P
2-5-Dimethylpyrazine	-4.6	-5.1	-4.2
3-Methyl-butanoic_acid	-4.2	-4.6	-4.2
5-Methyl-2-hexanone	-4.6	-4.5	-5.7
Dimethyl_disulfide	-2.0	-2.4	-1.9
Ethyl_2-methylbutanoate	-4.3	-4.5	-5.2
Indole	-5.3	-7.0	-6.3
Methyl_isobutyl_ketone	-4.2	-4.3	-4.0
Octadecane	-4.3	-5.3	-6.3

indole and 2,5-dimethylpyrazine showed lower TPSA values, implying enhanced membrane permeability. These findings underscore the structural diversity and physicochemical properties that affect the pharmacokinetic behavior of these compounds (Table 5).

Silver Nano Particle Biosynthesis

The synthesis of AgNPs was achieved through the reduction of silver nitrate by *B. aerius* PMRU2.8, as evidenced by the color transition from yellow to white over a 24-h period. UV-vis spectroscopy identified an absorption peak at 445 nm, corroborating the formation of spherical nanoparticles. The observed blue shift in the peak position indicated a reduction in nanoparticle size. SEM analysis further substantiated the synthesis of silver nanoparticles, revealing an average diameter of approximately 150 nm with a uniform orientation centered around 340 degrees (Fig. 5).

Antibacterial Activity

The antibacterial efficacy of *B. aerius* PMRU2.8 ethyl acetate extract and its Silver Nanoparticles (SNPs) was assessed against ten bacterial pathogens (Table 6). SNPs (25 µg) exhibited superior inhibitory effects compared to the crude extract (100 µg) in 9 out of 10 pathogens tested.

Table 5: ADMET analysis of Ethyl acetate extract.

Compound name	HBA	HBD	TPSA	Log P	Rb
Indole	0	0	15.79 Å²	1.98	0
2,5-Dimethyl pyrazine	2	0	25.78 Å²	0.99	0
5-Methyl-2- hexanone	1	0	17.07 Å²	1.87	3
Dimethyl disulfide	0	0	50.60 Å²	1.34	1
Ethyl 2-methylbutanoate	5	1	72.83 Å²	3.88	7
3-Methylbutanoic acid	2	1	37.30 Å²	0.98	2
Methyl isobutyl ketone	1	0	17.07 Å²	1.48	2
Octadecane	0	0	0.00 Å²	7.18	15

HBA - Hydrogen Bond Acceptor, HBD - Hydrogen Bond Donor, TPSA - Topological Polar Surface Area, Log P- Lipophilicity, Rb - Rotatable bond.

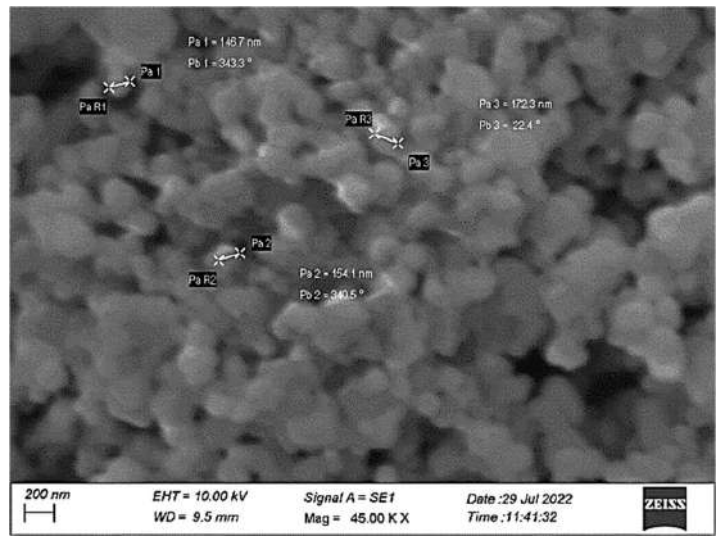


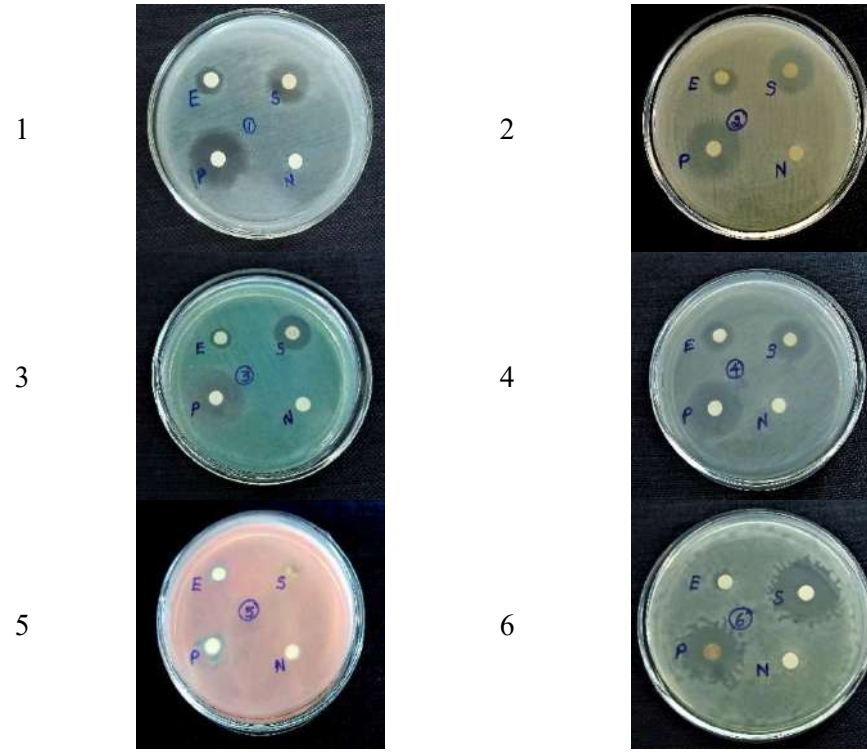
Fig. 5: SEM photograph of *B. aerius* PMRU2.8 synthesized SNP.

Inhibition zones for SNPs ranged from 7.83 to 23.8 mm, while those for the ethyl acetate extract ranged from 8.1 to 18.5 mm. Enterococcus faecalis showed the highest susceptibility to SNPs, with an inhibition zone of 23.8 mm (99.2% inhibition relative to streptomycin). Klebsiella pneumoniae also exhibited notable sensitivity to SNPs, with a mean inhibition zone of 20.5 mm (86.7% inhibition). Vibrio sp. was the only pathogen more sensitive to the crude extract (18.5 mm, 84.0% inhibition) than SNPs (17.1 mm, 77.6% inhibition). Statistical analysis confirmed significant

Table 6: Antibacterial activity of Ethyl acetate extract of *B. aerius* PMRU2.8 and its Silver Nano Particles.

S. No.	Pathogen	Samples	Zone Of Inhibition [mm]	Percentage of inhibition [%]
1	*Escherichia coli*	E	9.00 ± 0.54 a	35.9
		S	16.8 ± 0.41 bcd	69.7
		P	23.8 ± 0.86 b	-
		N	0.66 ± 0.30 a	-
2	*klebsiella pneumoniae*	E	11.8 ± 0.39 bc	48.3
		S	20.5 ± 0.25 e	86.7
		P	23.5 ± 0.68 b	-
		N	0.83 ± 0.40 a	-
3	*Pseudomonas aeruginosa*	E	8.5 ± 0.38 a	33.8
		S	15.3 ± 0.26 b	63.3
		P	23.8 ± 0.80 b	-
		N	0.66 ± 0.54 a	-
4	*Enterococcus faecalis*	E	12.1 ± 0.40 c	47.4
		S	23.8 ± 0.15 cde	99.2
		P	24 ± 0.31 b	-
		N	1.5 ± 0.15 a	-
5	*Serratia marcescens*	E	8.8 ± 0.25 a	60.2
		S	7.83 ± 0.15 a	52.6
		P	13.8 ± 0.41 a	-
		N	1.16 ± 0.41 a	-
6	*Proteus mirabilis*	E	8.8 ± 0.15 a	34.1
		S	21.5 ± 0.40 e	92.3
		P	23.1 ± 1.17 b	-
		N	1.33 ± 0.55 a	-
7	*Salmonella typhimurium*	E	8.1 ± 0.15 a	34.5
		S	19.5 ± 0.54 de	85.1
		P	22.8 ± 0.68 b	-
		N	0.33 ± 0.83 a	-
8	*Staphylococcus aureus*	E	10.3 ± 0.30 abc	40.1
		S	16.16 ± 0.40 bc	64.5
		P	24.6 ± 0.15 b	-
		N	0.66 ± 0.55 a	-
9	*Vibrio* sp.	E	18.5 ± 0.26 d	84.0
		S	17.1 ± 0.41 bcd	77.6
		P	21.8 ± 0.85 b	-
		N	1 ± 0.53 a	-
10	*Aeromonas hydrophila*	E	9.66 ± 0.79 ab	43.5
		S	14 ± 0.53 b	64.5
		P	21.3 ± 0.79 b	-
		N	0.66 ± 0.3 a	-

E- Ethyl acetate extract (100 µg), S- Silver Nanoparticle (25 µg), P- Positive control (Streptomycin 10 µg), N-Negative control (Ethyl acetate-100 µL). Statistics: dF = 9,1 (ESPN), p =>0.001(ESPN), F= (E=42.175), (S=37.404), (P=17.921) (N =0.995)



E- Ethyl acetate extract (100 µg),
S- Silver Nanoparticle (25 µg),
P- Positive control (Streptomycin 10 µg),
N-Negative control (Ethyl acetate-100 µL).

Fig. 6: Antibacterial activity of Ethyl acetate extract and SNP of B. aerius PMRU2.8.

Table 7: Azo-Dye degradation of B. aerius PMRU2.8.

Time (Hrs)	Sudan Black		Disperse red 1		Brilliant Green		Direct Blue 6	
	%D	ADR	%D	ADR	%D	ADR	%D	ADR
0	0	-	0	-	0	-	0	-
6	9.18 ± 2.03	1.53	12.1 ± 2.26	1.31	9.26 ± 1.26	0.76	12 ± 1.25	1.07
12	16.3 ± 1.26	1.35	21.6 ± 1.02	1.32	17.6 ± 3.25	0.81	25.4 ± 2.25	1.44
18	36.2 ± 1.02	2.01	33.7± 1.03	0.93	25.8 ± 2.02	0.76	39.8 ± 1.25	1.54
24	45.1 ± 3.26	1.87	43.7 ± 1.03	0.96	39.8 ± 1.65	0.91	53.6 ± 1.54	1.34
30	61.3 ± 1.02	2.04	59.3 ± 3.26	0.96	51.2 ± 1.26	0.86	72.7 ± 2.02	1.42
36	76.9 ± 3.02	2.13	67.7 ± 1.02	0.88	61.8 ± 2.65	0.92	79.9 ± 1.52	1.27
42	86.9 ± 1.00	2.06	79.4 ± 2.15	0.91	62.9 ± 3.29	0.77	88.9 ± 3.59	1.43
48	91.9 ± 2.03	1.91	86.2 ± 1.02	0.93	65.3 ± 1.26	0.75	92.5 ± 1.25	1.40

% D – Percentage of Colorization, ADR – Average colorization rate (mg L^{-1})

differences between treatments ($p<0.001$), with F-values of 42.175, 37.404, and 17.921 for the extract, SNPs, and positive control, respectively (Fig. 6).

Azo Dye Degradation

The bacterial isolate *B. aerius* PMRU2.8 showed notable decolorization capabilities against four distinct azo dyes over 48 h, as shown in Table 7 and Fig. 7 & Fig. 8 . Direct Blue 6 had the highest decolorization rate, achieving 92.5% after 48 h, with an average decolorization rate (ADR) of 1.40 $mg.L^{-1}$. Sudan Black followed with 91.9% decolorization and an ADR of 1.91 $mg.L^{-1}$. Disperse Red 1 reached 86.2%

decolorization with an ADR of 0.93 mg.L^{-1}, while Brilliant Green had the lowest at 65.3% with an ADR of 0.75 mg.L^{-1}. The decolorization process was time-dependent for all dyes. Initial rates were slow, with only a 9.18-12.1% reduction after 6 h. A significant increase occurred between 18-36 h. For Sudan Black, decolorization rose from 36.2% at 18 h to 76.9% at 36 h. Similarly, Direct Blue 6 increased from 39.8% at 18 h to 79.9% at 36 h. The rate plateaued between 42-48 h for most dyes, indicating nearing maximum decolorization capacity. ADRs varied significantly, with Sudan Black consistently showing the highest ADR, reaching 2.13 mg L^{-1} at 36 h, while Brilliant Green consistently had the lowest ADR, never exceeding 0.92 mg L^{-1}.

Brine Shrimp Lethality Bioassay

The brine shrimp lethality assay (BSLA) was employed to assess the cytotoxic potential of *B. aerius* PMRU2.8 samples, specifically the ethyl acetate extract (EAE) and

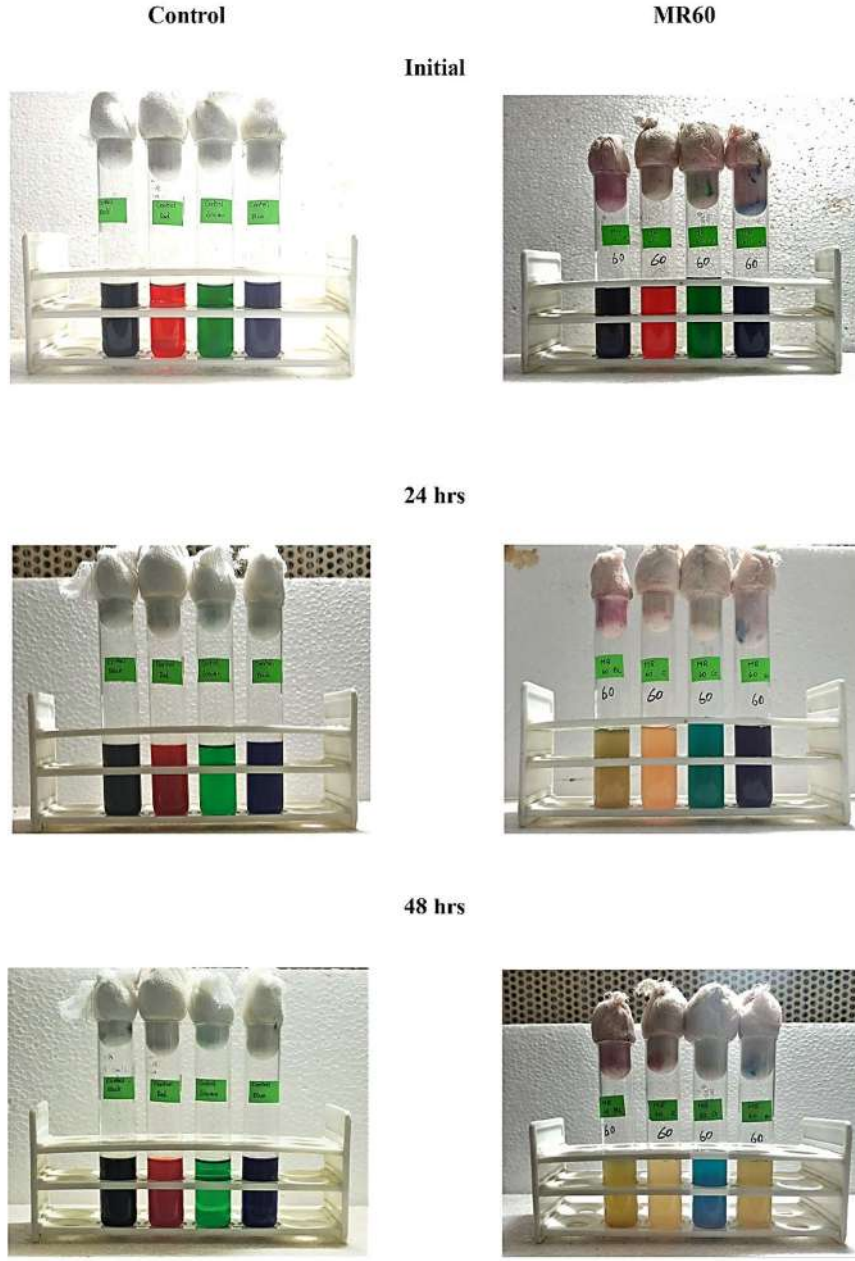


Fig. 7: Azo-Dye degradation assay of *B. aerius* PMRU2.8.

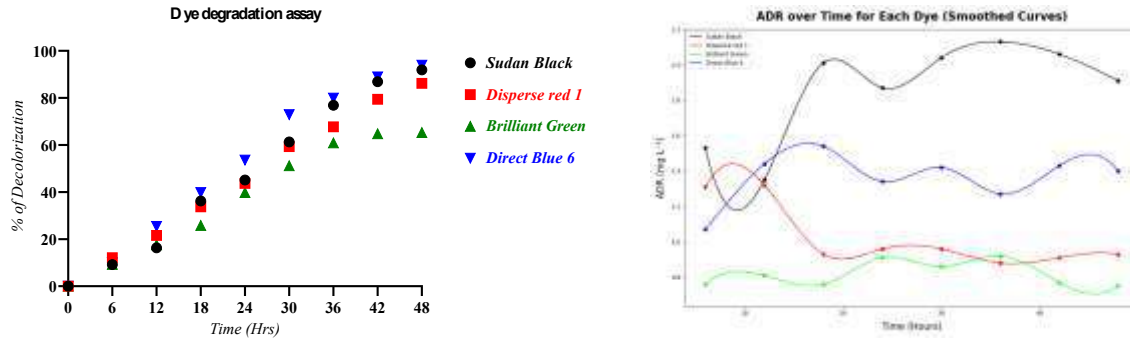


Fig. 8: Azo-Dye degradation assay of B. aerius PMRU2.8.

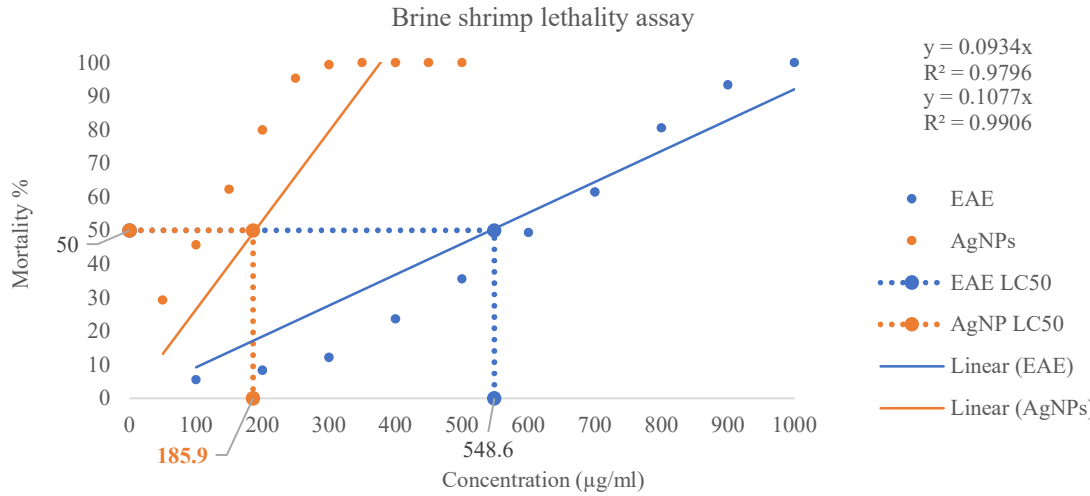


Fig. 9: Brine shrimp lethality bioassay.

silver nanoparticles (AgNPs) (Fig. 9). The assay results indicated variations in cytotoxicity among the samples. The AgNPs exhibited a higher level of cytotoxic activity, with an LC50 value of 310 µg.mL^{-1}, whereas the EAE exhibited moderate cytotoxicity, with an LC50 value of 548.6 µg.mL^{-1}. This finding suggests that AgNPs were approximately 1.77 times more potent than the crude extract in the brine shrimp lethality model. The lower LC50 value of the AgNPs indicates that a smaller concentration was necessary to achieve 50% mortality of the brine shrimp nauplii compared to the EAE, thereby demonstrating the enhanced cytotoxic potential of the nanoparticle formulation.

DISCUSSION

The ethyl acetate extract of *B. aerius* PMRU2.8 contains bioactive compounds with antibacterial properties. Indole derivatives affect E. coli and S. aureus via aromatic amine interactions (Bhagat et al. 2019). 5-methyl-2-hexanone inhibits S. aureus and Klebsiella pneumoniae by disrupting their membranes (Zhang et al. 2010). Carboxylic acids, such as 3-methylbutanoic acid, cause cytoplasmic acidification and membrane damage (Kim et al. 2012). Alkanes, such as octadecane, compromise membrane integrity (Malanovic et al. 2020).

Molecular docking analysis highlighted volatile compounds, particularly indole and octadecane, as potential inhibitors of bacterial proteins. The strong binding of indole to CMP-Kdo synthetase suggests interference with lipopolysaccharide biosynthesis in gram-negative bacteria, compromising cell wall integrity (Raetz & Whitfield 2002). This finding supports the study by Gao et al. (2018), which focused on bacterial growth inhibition through CMP-Kdo synthetase targeting. Indole also binds strongly to Clumping Factor A in Staphylococcus aureus, disrupting adherence and biofilm formation, supporting Fernandez-Calvo et al.'s (2024) findings. Ganesh et al.'s (2008) model reinforces ClfA-fibrinogen interactions in S. aureus pathogenesis. The binding of octadecane to Sortase A suggests the inhibition of this transpeptidase, which is crucial for bacterial virulence

factor display (Mazmanian et al. 1999); its inhibition could impair pathogenesis (Abujubara et al. 2023). The poor binding of dimethyl disulfide suggests that it is unlikely to be effective. The structure-activity relationship provides insights into drug design targeting these proteins.

ADMET properties of the tested compounds suggest varying potentials for activity and membrane permeability. Ethyl 2-methylbutanoate, with high hydrogen bond acceptors (HBA) and donors (HBD), indicates good solubility and potential for hydrogen bonding. However, its elevated topological polar surface area (TPSA) can hinder diffusion across membranes, affecting bioavailability. This aligns with the observation that increased polarity can impede permeability (Kenny 2022). The high lipophilicity and rotatable bonds of octadecane suggest that it may readily interact with hydrophobic environments, such as membranes; however, its poor solubility could limit absorption. Compounds with high lipophilicity often require formulation strategies to enhance their bioavailability (Argikar et al. 2022). Indole and 2,5-dimethylpyrazine, which have low TPSA values, likely have improved membrane permeability, aiding intracellular activity. These findings underscore the complex interplay of molecular characteristics in determining the pharmacokinetic and safety profiles.

The color change during AgNP synthesis indicated successful reduction, consistent with previous microbial biosynthesis studies. The UV-vis absorption peak at 445 nm matches the reported values for spherical silver nanoparticles, typically around 400 nm. The blue shift suggests a decrease in the particle size, which is consistent with Mie theory. SEM analysis confirmed the uniform distribution of nanoparticles, supporting the synthesis potential of *B. aerius PMRU2.8*. These findings align with studies highlighting microbial synthesis as an eco-friendly nanoparticle production method (Danischewski et al. 2023, Panariello et al. 2020, Rasskazov et al. 2020). The superior antibacterial efficacy of silver nanoparticles compared to the crude extract is due to their nanoscale dimensions and enhanced properties. Larger inhibition zones by SNPs at lower concentrations (25 µg versus 100 µg) align with Rai et al. (2012) and Dakal et al. (2016), who established that increased surface area to volume ratio facilitates efficient bacterial cell membrane interactions through electrostatic attraction and penetration.

The exceptional susceptibility of Enterococcus faecalis to SNPs (99.2% inhibition relative to streptomycin) is significant given its increasing antibiotic resistance. Ultrastructural studies suggest that gram-positive bacteria may experience more severe cell wall disruption from silver nanoparticles due to their peptidoglycan architecture. The pronounced inhibition of Klebsiella pneumoniae (86.7%) by SNPs indicates promising activity against a critical respiratory pathogen. Varying susceptibility patterns across bacterial species likely reflect differences in cell envelope composition, efflux pump mechanisms, and intrinsic resistance determinants, as documented by Hogan et al.(2024) and Suma et al.(2023). The sensitivity

Table 8: Mechanisms of action of key compounds in bacterial inhibition.

Compound	Molecular Target	Proposed Mechanism of Action	Potential Antimicrobial Effect	Reference
Indole	CMP-Kdo synthetase (Gram-negative bacteria)	Strong binding affinity, interference with lipopolysaccharide biosynthesis	Compromised cell wall integrity in Gram-negative bacteria	(Nitulescu et al. 2021)
Indole	Clumping Factor A (S. aureus)	Strong binding affinity, disruption of bacterial adherence	Inhibited biofilm formation and colonization	(Claes et al. 2017)
Octadecane	Sortase A	Inhibition of this transpeptidase enzyme	Impaired display of virulence factors on the bacterial cell surface	(Alharthi et al. 2021)
Octadecane	Cell membrane	Compromise of membrane integrity due to high lipophilicity	Membrane destabilization and potential cell death	(Kolaričet al. 2021)
5-Methyl-2-hexanone	Cell membrane	Disruption of membrane integrity	Particularly effective against S. aureus and K. pneumoniae	-
3-Methylbutanoic acid	Cytoplasm and cell membrane	Cytoplasmic acidification and membrane damage	Disruption of cellular homeostasis	(Ketchamet al. 2022)
Ethyl 2-methylbutanoate	Multiple targets	Enhanced solubility and hydrogen bonding potential (high HBA/HBD)	Broad interaction with cellular components, limited by higher TPSA	(Riel et al. 2019)
Dimethyl disulfide	Multiple targets	Poor binding affinity demonstrated in molecular docking	Limited antibacterial efficacy predicted	-
2,5-Dimethylpyrazine	Cell membrane	Improved membrane permeability (low TPSA)	Enhanced intracellular activity	-

of Vibrio sp. to the crude extract rather than SNPs presents an intriguing counterpoint, possibly explained by specific bioactive compounds targeting Vibrio-specific pathways. Raza et al. (2016) elucidated multiple antimicrobial mechanisms for SNPs, including ROS generation, protein denaturation, and DNA replication inhibition, which may be differentially effective depending on bacterial characteristics. The differential efficacy between gram-positive and gram-negative bacteria could be attributed to variations in cell wall permeability, as suggested by the statistical significance ($p<0.001$) in treatment responses. The F-values (E=42.175, S=37.404, P=17.921) indicate substantial variance between treatment groups, supporting the hypothesis that nanoparticle-mediated antibacterial activity involves multiple targets and mechanisms. The broad-spectrum activity of *B. aerius* PMRU2.8-derived SNPs against clinically relevant pathogens indicates promising applications in developing novel antimicrobial formulations. These findings underscore the complex interplay between nanoparticle characteristics, bacterial physiology, and antimicrobial response. The mechanism of action of each compound in the inhibition of pathogenic bacteria is summarized in Table 8.

The differential decolorization efficacy across four azo dyes is due to their structural characteristics and enzymatic systems in strain PMRU2.8. Superior decolorization of Direct Blue 6 (92.5%) aligns with Guo et al. (2021), who noted di-azo-linked naphthol groups are susceptible to bacterial azo-reductase. Sudan Black and Disperse Red 1 showed Substantial decolorization (91.9% and 86.2%), suggesting the possibility of multiple azo-reductase enzymes. Priyanka et al. (2022) suggest that oxidative enzymes like laccase contribute to decolorization, explaining the ability to degrade diverse azo compounds. Lower decolorization for Brilliant Green (65.3%) is due to its triphenylmethane structure, which resists azo-reductase cleavage. Wanyonyi et al. (2017) reported that triphenylmethane dye decolorization relies more on peroxidase activity, limited in Bacillus species. The time-dependent decolorization suggests an adaptation period for bacterial cells to induce enzymatic machinery (Eskandari et al. 2019). The acceleration between 18-36 h suggests upregulation of enzymes or optimal metabolic conditions. Higher ADR values for Sudan Black (2.13 mg L^{-1}) suggest the enzymatic systems may have evolved preferential activity toward certain azo dye structures. Near-complete decolorization (>90%) for Direct Blue 6 and Sudan Black demonstrates *B. aerius* PMRU2.8's bioremediation potential for textile effluent treatment.

Enhanced cytotoxicity of silver nanoparticles (AgNPs) compared to the ethyl acetate extract (EAE) of *B. aerius* PMRU2.8 is due to their unique properties that increase cellular interactions. Akter et al. (2017) and Liao & Tjong (2019) noted AgNPs are internalized by cells through mechanisms like scavenger receptor-mediated phagocytosis and lipid-raft-mediated endocytosis. Piao et al. (2011) showed AgNPs can localize in the cytoplasm and reach the nucleus, potentially interfering with DNA replication. This may explain the lower LC50 value (310 µg/mL) for AgNPs compared to the extract (548.6 µg.mL^{-1}). A key mechanism of AgNP toxicity is intracellular dissolution, releasing silver ions (Ag+). Akter et al. (2017) and Liao & Tjong (2019) emphasized that these ions are central to AgNP toxicity, interacting with proteins, enzymes, and nucleic acids. Continuous release of silver ions creates a sustained cytotoxic effect not seen with conventional extracts. AgNP cytotoxicity involves oxidative stress pathways. Hackenberg et al. (2011) reported that AgNPs generate reactive oxygen species (ROS) and suppress glutathione levels, causing oxidative imbalance and damage to cellular components. This contributes to their greater cytotoxic potency in the brine shrimp lethality assay. The cytotoxicity pattern suggests potential biomedical applications, particularly in anticancer agent development, but raises concerns about environmental and human safety, necessitating comprehensive toxicological assessments before practical applications.

Study Limitations and Future Directions

While this study provides insights into *B. aerius PMRU2* antimicrobial properties and dye decolorization capabilities of B. aerius PMRU2.8, certain aspects warrant further investigation. Cytotoxicity assessment can be complemented with mammalian cell line testing for clinical applications. Although B. aerius PMRU2.8 demonstrated stability, long-term studies across generations would strengthen its industrial applicability. The azo dye biodegradation assay showed high decolorization rates in this study. Future research should investigate enzymatic pathways in dye degradation, focusing on azo-reductase and oxidative enzymes. Pilot-scale studies using textile effluents would better reflect the industrial applications. Field testing would validate the real-world efficacy. Molecular docking analyses provide mechanistic hypotheses that can be validated through enzyme inhibition assays. Such studies would build upon the findings of novel antimicrobial development. Future investigations should explore the metabolic fate of degraded intermediates, scale-up parameters, and immobilized systems for continuous treatment. The multifunctionality of B. aerius PMRU2.8 suggests its potential as a versatile bioremediation agent, meriting further study.

CONCLUSIONS

This study elucidated the antibacterial efficacy of *Bacillus aerius PMRU2.8*, isolated from marine red algae, against human pathogens. Molecular and biochemical analyses

confirmed its taxonomic identity. Both the ethyl acetate extract and silver nanoparticles exhibited antibacterial activity, with the latter showing enhanced potency. Strain PMRU2.8 also displayed azo dye degradation capabilities, indicating its potential for environmental remediation. The brine shrimp lethality bioassay revealed the cytotoxicity profile of AgNPs, suggesting promising biomedical applications. These findings open avenues for future commercial nanoformulations incorporating PMRU2.8-derived compounds as antimicrobial agents in healthcare. The robust dye degradation ability of this strain positions it for scale-up in environmental pilot studies targeting textile effluent treatment. Additionally, synthetic biology approaches can enhance the production of key bioactive compounds and optimize enzymatic pathways for improved bioremediation efficiency. The multifunctional capabilities of B. aerius PMRU2.8 present significant potential for addressing both clinical antimicrobial resistance and environmental pollution challenges through sustainable biotechnological applications in the future.

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Biofuel Pellet Production and Characterization from Rice Production Waste

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ABSTRACT

Biofuel pellet materials are a key renewable alternative to fossil fuels. Evaluating biomass quality is essential for both operational efficiency and environmental impact. This study aimed to produce and characterize biofuel pellets made from rice husks and straw. The pellets were analyzed using ASTM methods and compared against the ISO 17225-6 standard. The results indicated a low moisture content (2.53 ± 0.04 %) and a relatively high ash content (11.96 ± 0.05%). Thermally, the net calorific value was 3,951 ± 7.21 Cal/g. In terms of elemental composition, nitrogen (0.29 ± 0.02%), sulfur (0.15 ± 0.02%), and chlorine (0.34 ± 0.04%) contents were in line with the ISO 17225-6 standard. Additionally, the pellets made from both biomass met durability, length, and diameter specifications. The results indicate that mixing these rice-based biomass improves pellet quality and combustion performance. Lastly, this research supports SDG 7 (ensuring access to affordable, reliable, sustainable, and modern energy for all), SDG 12.2 (promoting the sustainable management and efficient utilization of natural resources), and SDG 12.5 (minimizing waste generation through prevention, reduction, recycling, and reuse).

INTRODUCTION

Rice is one of the most widely grown and consumed cereal grains globally. During 2023-2024, the total rice production in Thailand reached 26,933,808 tons (Office of Agricultural Economics 2024), ranking the country sixth among world rice exporters (Sowcharoensuk 2024). While high rice production contributes to economic growth, it also poses significant environmental challenges due to the associated rise in energy demand (Cetinkaya et al. 2024). One of the main contributors to global greenhouse gas emissions (GHG) is rice cultivation and the unsustainable disposal of rice straw. Approximately 30 million tons of methane (CH_4) are released annually worldwide from rice cultivation, accounting for 8% of all CH_4 emissions (Roder et al. 2024). Rice husk, a by-product generated in large volumes at rice mills, is often underutilized (Phonphuak & Chindaprasirt 2015). Therefore, effective management methods are essential for substantial quantities of rice straw and husk produced (Jyothsna et al. 2024).

Rice husks and rice straws are classified as lignocellulosic materials. Rice husk is composed of approximately 50% cellulose, 25–30% lignin, and 15–20% silica (Fathurahman & Surjosatyo 2021). In comparison, rice straw contains 36.5% cellulose, 33.8% hemicellulose, and 12.3% lignin (Nie et al. 2013). The presence of cellulose, hemicellulose, and lignin in these materials enables them to function as natural binders in biofuel pellets, enhancing heat resistance and combustion efficiency (Gil et al. 2010; Rios-Badarn et al. 2020).

In terms of calorific value, rice straw has a higher heating value (HHV) ranging between 14.08 and 15.09 $MJ.kg^{-1}$, while rice husks exhibit an HHV of

Fig. 1: (A) Rice husks and (B) Rice straws.

approximately 14.2 MJ.kg^{-1} (Gummert et al. 2019). Rice straw contains a high proportion of volatile matter (VOM), approximately 60.55-69.70%, comparable to other biomass sources such as sugar cane bagasse and corn straw. In terms of volatiles, rice husk is around 73.41% (Biswas et al. 2017). High VOM content in bioenergy materials offers advantages such as easier ignition and better combustibility; however, it may also result in rapid and difficult-to-control combustion (Liu et al. 2011; Gummert et al. 2019). Moisture content is important for biofuel production. Rice straw typically contains around 11.4% moisture, whereas rice husk moisture content ranges between 6–10% (Jusoh et al. 2013, Hezam Saeed et al. 2021).

Globally, the biomass pellet market is projected to grow at a compound annual growth rate (CAGR) of 5.4% from 2024 to 2033, rising from an estimated $9.5 billion in 2023 to approximately $16.0 billion by 2033. Biofuels pellets offer a more environmentally friendly and cleaner substitute to traditional fossil fuels. They are cost-effective, reduce pollution, and provide clean-burning, sustainable energy. Moreover, biofuel pellets are easy to transport, produce low carbon emissions, and are dependable and efficient energy sources (Singh & Brar 2021, Kumari & Prasad 2024).

Therefore, the objective of this research is to produce and characterize biofuel pellets using rice husk (a by-product of rice milling) and rice straw (a by-product of rice harvesting). Biofuel pellet production offers a potential solution to reduce open-field burning of agricultural residues and the large volumes of waste generated from rice production. This can help in reducing carbon dioxide (CO_2) emissions and encouraging more sustainable waste management strategies.

MATERIALS AND METHODS

Biomass Collection and Preparation

Rice husks (Fig. 1A) were collected from Thanyakit Nakhonpathom (2521) Co., Ltd., located in Nakhon Pathom Province, Thailand. Rice straws (Fig. 1B) were collected from paddy fields within the same province. After mashing and removing the damaged rice straws and rice husks, samples were homogenized using a 3 mm mesh screen to ensure uniform particle size (Rios-Badran et al. 2020).

Used cooking oil was obtained from a collection station in Nakhon Pathom Province. Its heating value was reported as 3.99×10^4 kJ/kg (Saniso et al. 2008).

Biofuel Pellet Production

Rice husks and straws were chopped using a 7.5 HP hammer mill equipped with a 3 mm mesh screen. The processed biomass materials were then mixed with water and used cooking oil in appropriate proportions, maintaining a 1:1 ratio of rice husk to rice straw. The mixture was compressed into biofuel pellets (Fig. 2) using a pellet press, with each sample undergoing two pressing cycles before the analysis of pellet properties.

Biofuel Pellet Characterization Analysis

1. Physical Analysis

The physical properties of biofuel pellets were evaluated

Fig. 2: Rice husk and rice straw pellets.

by measuring their diameter and length using a vernier caliper. Bulk density was determined by three replicates and calculated using Equation 1.

$$Bulk\ density = \frac{M}{V} \quad \ldots(1)$$

Where: Bulk density = Bulk density of pellets (kg/m^3)

M = Total mass of pellets (kg)

V = Volume of the container (m^3)

2. Proximate Analysis

The proximate analysis was conducted to determine the average moisture content, volatile matter, and ash content of the biofuel pellets. Each parameter was measured in triplicate, following the procedures specified in the ASTM D7582-15 (2015) standard.

Moisture Content: The moisture content was determined by weighing the sample before and after oven drying. A biofuel pellet sample was weighed and then dried in an oven at 105 °C for 2 hours. After cooling in a desiccator, the sample was reweighed. The moisture content was calculated using Equation 2.

$$\text{Moisture content} = \left(\frac{A-B}{A}\right) \times 100 \quad \ldots(2)$$

Where: A = Weight of the sample before oven drying(g)

B = Weight of the sample after oven drying (g)

Volatile Matter: To determine volatile matter, a 2 g sample was ground, placed in a crucible, and oven-dried for 2 hours. The dried sample was then transferred to a furnace at 550°C for 10 minutes. After cooling in a desiccator, the sample was weighed. The volatile matter was calculated using Equation 3:

$$\text{Volatile matter} = \left(\frac{B-C}{B}\right) \times 100 \quad \ldots(3)$$

Where: B = Weight of the sample after drying in the oven (g)

C = Weight of the sample after furnace treatment for 10 minutes (g)

Ash Content: The ash content was determined by placing a 2 g sample in a furnace at 550°C for 4 hours. After cooling in a desiccator, the sample was weighed. The ash content was calculated using Equation 4:

$$\text{Ash content} = \frac{D}{B} \times 100 \quad \ldots(4)$$

Where: B = Weight of the sample after drying in the oven (g)

D = Weight of the sample after furnace treatment for four hours (g)

Fixed Carbon: The fixed carbon content was calculated by subtracting the sum of ash content and volatile matter from the total mass, as represented in Equation 5:

Fixed carbon=100% − (Ash content + Volatile matter) …(5)

3. Elemental and Thermal Analysis

The elemental composition of the biofuel pellets including carbon, hydrogen, nitrogen, sulfur, and chlorine were determined in accordance with ASTM standards: ASTM D5373-16 (2016) for carbon, hydrogen, and nitrogen; ASTM D4239-17 (2017) for sulfur; and ASTM D6721-01 (2015) for chlorine. For thermal analysis, the net and gross calorific values were determined using the ASTM D 5865-13:2013 standard.

4. Statistical Analysis

All experimental measurements were conducted in triplicate. The results were analyzed statistically by calculating the mean values and standard deviations to ensure the reliability and consistency of the data.

Results and Discussion

Physical and Proximal Analysis

The physical and proximate characteristics of biofuel pellets (Fig. 3), produced from rice husk and rice straw, are presented in Table 1. The results were compared to the ISO 17225-6 standard for non-woody biomass.

The biofuel pellet in this study exhibited a higher volatile matter (68.20 ± 0.40%) and fixed carbon content (15.70 ± 0.30%) compared to previous findings by Efomah & Gbabo (2015). Combining different biomass types can improve the biomass energy properties of solid biofuels (Liu et al. 2013). Due to their high cellulose and lignin content, rice husks and straws have a high calorific power attributed to

Fig. 3: Length of biofuel pellet.

Table 1: Physical and proximal analysis.

Parameters of Biofuel pellet analysis	Biofuel pellet	ISO 17225-6	
		Class A	Class B
Diameter	8.16 ± 0.03 mm	6-10 mm	6-10 mm
Length	24.69 ± 0.27 mm	3.15-40 mm	3.15-40 mm
Bulk Density	600.64 ± 0.43 kg/m^3	≥ 600 kg/m^3	≥ 550 kg/m^3
Moisture content	2.53 ± 0.04 %	≤ 12%	≤ 15%
Ash content	11.96 ± 0.05%	≤ 6	≤ 10

Table 2: Elemental and thermal analysis.

Parameters of Biofuel pellet analysis	Biofuel pellet	ISO 17225-6	
		Class A	Class B
Nitrogen	0.29 ± 0.02%	≤ 1.5%	≤ 2.0%
Sulfur	0.15 ± 0.02%	≤ 0.20%	≤ 0.30%
Chlorine	0.34 ± 0.04%	≤ 0.10%	≤ 0.40%

depolymerization reactions that occur when these chemical components are exposed to high temperatures (Bridgeman & Jones 2008).

Moisture content of the pellets in this study was notably low (2.53%), in comparison to earlier research by Rios-Badran et al. (2020), which reported a moisture content of 15.95%. Moisture content is a critical parameter affecting the combustion efficiency of biofuels, with lower moisture levels generally enhancing combustion performance.

All evaluated parameters of the biofuel pellets, i.e., diameter, length, bulk density, and moisture content, met the ISO 17225-6 standard for non-woody biomass, with the exception of ash content. The ash content was higher than Class A and B pellets, primarily due to the inclusion of rice husk, which typically contains high ash levels (approximately 16.0 wt%) (Puri et al. 2024, Islam et al. 2021). High ash content can lead to machinery problems such as fouling and slag deposition. Therefore, pretreatment methods to reduce ash content may be necessary prior to thermochemical processes like pyrolysis (Puri et al. 2024, Islam et al. 2021).

Although the bio-pellet from rice husk and straw had high ash content, the other parameters contribute to making it an effective biofuel pellet. These results show that bio-pellet production not only utilizes wastes from rice harvest and production but also enables more efficient use of biomass, all of which align with the biomass densification process.

Elemental and Thermal Analysis

The elemental and thermal properties of biofuel pellets produced from rice husk and rice straw are presented in Table 2. The results were compared to the ISO 17225-6 standard for non-woody biomass. Among the parameters, nitrogen, sulfur, and chlorine contents in the biofuel pellets conformed to the ISO 17225-6 standard.

Chlorine is a critical element in fuel as it contributes to machinery corrosion, accelerates oxidation, and leads to metal wastage (Nielsen et al. 2000). Although the chlorine content in the bio-pellets met the Class B ISO 17225-6 standard, it may still cause operational issues in machinery over time.

The carbon-to-nitrogen (C/N) mass ratio serves as both a qualitative and environmental indicator, reflecting the combustion potential of biomass. High content can lead to increased NOx emissions during combustion (Toscano et al. 2023). However, the nitrogen content observed in this study (0.29%) was lower than in various types of biomass reported in earlier research: herbaceous (0.9%), fruit (1.5%), and woody biomass (0.3%), indicating reduced NOx emission potential and minimal environmental impact.

The carbon (44.50 ± 0.44%) and hydrogen (6.54 ± 0.13%) contents were similar to those reported by Efomah & Gbabo (2015), where carbon and hydrogen contents were 45.20% and 5.80%, respectively. Thermal analysis revealed that the biofuel pellets had a net calorific value of 3,951 ± 7.21 Cal/g and a gross calorific value of 4,274 ± 5.29 Cal/g; the calorific value was higher than previous research (Nipa Palm 4092.7 kcal/kg) (Harun et al. 2020), confirming their energy potential as a viable biofuel source.

CONCLUSIONS

This study successfully produced and characterized biofuel pellets from rice husks and rice straws. The results indicate that a 1:1 blend of rice husks and rice straws, combined with water and used cooking oil, meets the ISO 17225-6 standard for non-woody biomass. Additionally, the resulting pellets

also fulfilled the standard requirements for durability, length, and diameter.

Our findings suggest that blending these two types of agricultural waste enhances the quality and combustion properties of the resulting bio-pellets, making them a viable source of renewable energy and a sustainable alternative to fossil fuels. This work contributes directly to Sustainable Development Goals (SDGs), particularly SDG 7 (ensuring access to affordable, reliable, sustainable, and modern energy for all), SDG 12.2 (promoting the sustainable management and efficient use of natural resources), and SDG 12.5 (reducing waste generation through prevention, reduction, recycling, and reuse).

Additionally, biofuel pellet production helps reduce open-field burning of paddy residues, supporting efforts to lower greenhouse gas (GHG) emissions and contributing to climate change mitigation and adaptation. Future work will focus on evaluating combustion efficiency, long-term storage stability, and emissions analysis to address the remaining challenges associated with biofuel pellet utilization.

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Duckweed as a Circular Economy Solution for Treating Agro-Industrial Wastewaters

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ABSTRACT

Water pollution resulting from nutrient-rich wastewater (WW) discharged by industries such as distilleries, sugar mills, and dairy farms poses significant ecological and public health challenges. Conventional treatment methods often fail to effectively reduce nutrient loads, contributing to environmental degradation. This review critically examines the use of duckweed (Lemna spp.), a fast-growing aquatic plant, as a sustainable solution for wastewater remediation. Duckweed demonstrates a high capacity for nutrient uptake, particularly nitrogen (N) and phosphorus (P), while simultaneously producing protein-rich biomass suitable for animal feed. The review synthesizes findings on the effectiveness of duckweed-based systems in reducing chemical oxygen demand (COD) and biological oxygen demand (BOD), and explores their integration into circular economy models that couple wastewater treatment with resource recovery. Additionally, it addresses current limitations in system design, scalability, and long-term implementation, highlighting areas requiring further research. Overall, duckweed-based wastewater treatment offers a cost-effective, eco-friendly strategy to enhance environmental sustainability and food-feed security.

INTRODUCTION

Water is vital for all life-sustaining activities, and the adage "no life without water" emphasizes its universal importance. Water can be sourced from natural reservoirs such as rivers, lakes, ponds, and wells. The anthropogenic activities and external factors often compromise its quality, necessitating treatment before use for drinking or other purposes (Katko 2017). The increasing demand for water in domestic, agricultural, industrial, and commercial sectors highlights the importance of maintaining its quality. Industrial activities, while consuming relatively small quantities of water, significantly impact its quality, often discharging untreated effluents into surface and groundwater systems (Wang & Yang 2016). Domestic sewage and industrial effluents, which constitute over three-quarters of the freshwater drawn, profoundly influence the degradation of water quality (Mukherjee et al. 2006). Wastewater is adversely affected by human use, making it unfit for direct consumption or discharge into the environment without treatment. It typically originates from domestic, industrial, agricultural, or commercial activities and contains a broad spectrum of pollutants, such as nutrients, organic matter, pathogens, heavy metals, chemicals, and suspended solids (Musa Yahaya et al. 2023). This poorly treated WW poses numerous health, environmental, and ecological risks. It can lead to eutrophication, ecological damage, and severe health consequences, particularly in areas with inadequate wastewater management (González-Mariño et al. 2016, Wang & Yang 2016).

This issue is exacerbated by population growth, rapid urbanization, and the increasing demand for water in food production, which relies on water for nearly

40% of the global food supply (Department of Environmental Engineering 2015). As global water quality management struggles to keep pace with these challenges, innovative and cost-effective solutions are critical to resolve the limitations of conventional WW treatment systems (Saha et al. 2017, Sharifinia et al. 2016). Duckweed (Lemna spp.) (Fig.1) has been found to be a promising, eco-friendly alternative for WW treatment. Its ability to thrive on various wastewater sources—including municipal, industrial, and agricultural effluents such as dairy, sugar, and distillery wastewater—makes it a cheap and justifiable resolution for nutrient recovery and pollution mitigation (Cheng et al. 2002, Landesman et al. 2005). It effectively removes N and P and lessens BOD and COD in wastewater (Jadhav et al. 2024).

Furthermore, its nutrient-rich biomass, with a protein content of 15–45% dependent on progress conditions, can be repurposed as animal feed or bioenergy, creating a circular economy (Chantiratikul et al. 2010, Culley Jr, & Epps 1973). This article emphasizes the application of duckweed in WW treatment and its potential to address environmental and economic challenges associated with traditional methods. It explores duckweed's nutrient removal efficiency, protein content variability based on wastewater characteristics, and its scalability for diverse wastewater sources. The review also highlights the need for further research to optimize duckweed-based systems, emphasizing their cost-effectiveness and sustainability for community-level implementation.

MATERIALS AND METHODS

This review followed a systematic screening approach. A total of 137 articles were identified from databases such as PubMed, Web of Science, and Scopus. After removing duplicates and applying inclusion criteria (industrial wastewater characterization, duckweed application), 58 studies were included.

Fig. 1: Top-view photograph of *Lemna minor* cultured in nutrient medium under controlled laboratory conditions.

The inclusion criteria: i) Articles based on the characteristics, challenges, and case studies of industrial wastewater. It also included Duckweed applications, nutritional compositions, and the mechanism of pollutant removal. ii) This paper also explored operational challenges, research gaps, and future investigation.

The exclusion criteria: Articles lacking specifics on the other natural plant strategy for pollutant removal, and domestic sewage were excluded.

Characterization of Industrial Wastewaters

Dairy, sugar, and distillery industries generate wastewater rich in organic matter, nutrients, and sometimes toxic compounds. Dairy wastewater typically contains lactose, fats, proteins, and detergents, contributing to high BOD and COD. Duckweed has shown up to 90% efficiency in nutrient removal from such effluents. Sugar industry wastewater includes organic acids and high salinity, often treated with duckweed species like Lemna gibba, achieving 75–80% BOD and COD reduction. Distillery wastewater is highly polluting due to melanoidins and phenolics. Duckweed treatment systems have achieved up to 85% BOD removal and significant reductions in color and toxicity. Duckweed's role in these sectors combines simplicity, cost-effectiveness, and biomass generation potential, making it a scalable phytoremediation option (Fenandes et al. 2023a).

Dairy Wastewater (DWW)

DWW typically comprises maximum levels of organic matter, nutrients as mentioned earlier, and suspended solids. It arises from processes such as milk processing, cleaning, and sanitizing operations (Botondi et al. 2023). The main components of DWW include lactose, proteins, fats, and minerals, which contribute to its high BOD and COD values. The high BOD and COD can significantly impact aquatic ecosystems if discharged untreated (Kolev Slavov 2017). The presence of these organic compounds poses significant challenges for treatment, as they require efficient removal to prevent environmental contamination (Ting & Praveena 2017). Biological treatment methods, including aerobic and anaerobic digestion, have been extensively researched for their efficacy in degrading organic matter in dairy wastewater (Dereli et al. 2012). However, these methods frequently face restrictions related to operational stability as well as efficiency under varying load conditions (Gao et al. 2011). To overcome these limitations, the duckweed plant plays an important part in the treatment of dairy wastewater (Zhou et al. 2023a).

Multiple recent studies have highlighted the potential of duckweed in managing DWW due to its ability to assimilate higher concentrations of nutrients and organic matter. Duckweed systems offer advantages such as low operational costs, ease of maintenance, and the production of valuable biomass (Chen et al. 2020, Xu et al. 2023). Several studies have demonstrated the effectiveness of duckweed in treating DWW. For example, Muradov et al. (2014) examined the usage of *Lemna minor* in a pilot-scale treatment for dairy wastewater. The results displayed that duckweed could achieve over 90% effluent of N & P, significantly reducing the nutrient load of the treated effluent.

Additionally, the duckweed biomass produced during the treatment process was found to have high protein content, making it appropriate for animal feed (Muradov et al. 2014). Sreehari et al. highlighted this plant's potential in eliminating organic matter and nutrients from DWW. Researchers reported that duckweed-based systems achieved significant reductions in BOD and COD values, with removal efficiencies exceeding 80%. The treated effluent met the discharge standards set by regulatory authorities, demonstrating the feasibility of duckweed for dairy wastewater treatment (Sreehari et al. 2017). *El-Sheekh et al*. further confirmed the effectiveness of duckweed in DWW treatment. The study showed that duckweed could effectively remove nutrients and organic matter from dairy effluents, with removal efficiencies comparable to conventional treatment methods. The harvested biomass was analyzed for its nutritional composition and found to be rich in proteins and other essential nutrients, making it a valuable feedstock for animal feed production (El-Sheekh et al. 2019). These studies have demonstrated its effectiveness in removing various pollutants (Hemalatha & Venkata Mohan 2022). The phytoremediation performance of duckweed is particularly effective in diluted wastewater, with removal efficiencies reaching up to 87% for phosphorus and 65% for nitrogen (Sahi & Megateli 2023) and effectively removing 74% of organic carbon from dairy wastewater.

- The duckweed biomass grew significantly over time, with an amplifying time of 0.87 days and a final dry weight of 3.73 g.
- The pretreated duckweed biomass was used as a substrate for microbial protein production using *Bacillus subtilis*, resulting in a high protein content (60%) and carbohydrate content (21%) in the final microbial protein product.

Sugar Industry Wastewater (SWW)

It poses significant environmental threats if discharged untreated (Malik et al. 2019). The wastewater generation rate is approximately 1,000 L per ton of sugarcane crushed in India. Various treatment methods have been explored, including aerobic, anaerobic, and physico-chemical processes (Kushwaha 2015). The wastewater contains significant amounts of organic and inorganic materials. These complexes contribute to the high organic load and make it challenging to treat using conventional methods (Sahu et al. 2008). Anaerobic digestion and aerobic treatment processes are commonly employed for treating sugar industry wastewater, but they often require extensive infrastructure, high operational costs, and limitations in degrading oil and grease (Dahiya et al. 2001, Pant & Adholeya 2007).

Additionally, the presence of refractory compounds and high salinity levels can inhibit microbial activity, reducing treatment efficiency (Mohana et al. 2009). Advanced treatment includes filtration, flocculation, UV disinfection, etc (Yadav et al. 2021). Batch reactor studies have shown that the wastewater is amenable to aerobic biodegradation. The quality and features of SWW depend on the region and water consumption practices (Ahmad & Mahmoud 1982). The water pinch methodology has been proposed to minimize fresh water consumption in sugar industries (Poddar & Sahu 2017).

Duckweed-based treatment systems have shown promise in effectively removing organic matter and nutrients from sugar industry wastewater (Patel et al. 2017, Verma & Suthar 2015). Duckweed's rapid growth rate and nutrient uptake ability make it an ideal candidate for bioremediation, offering an alternative option to traditional treatment methods (Leng 1999). The application of duckweed in treating sugar industry wastewater has also been explored. Patel et al. (2017) discovered research on the treatment of sugar mill effluent using duckweed species such as *Lemna gibba* and *Spirodela polyrhiza* (Cheng et al. 2002). The study reported significant reductions in COD and BOD, with removal efficacies of up to 80%. The treated effluent met the discharge standards set by regulatory authorities, highlighting the prospect of duckweed-based systems for industrial wastewater treatment (Patel et al. 2017). Ali et al. (2016) investigated the usage of duckweed for the management of SWW from a sugar refinery. The results showed that duckweed could achieve substantial reductions in organic matter and nutrient concentrations, with removal efficiencies exceeding 75%. The researchers also highlighted the economic benefits of using duckweed, as it provided a low-cost and sustainable treatment option (Ali et al. 2016). Further research by Kumar et al. (2020) demonstrated the potential of duckweed in treating wastewater from sugarcane processing. The study reported that duckweed-based systems could effectively reduce BOD, COD, and nutrient levels,

with removal efficiencies comparable to conventional treatment methods. The harvested biomass was analyzed for its nutritional composition and found to be suitable for use as animal feed (Kumar et al. 2020).

Distillery Wastewater

Distillery wastewater is the effluent generated during the production of alcoholic beverages such as ethanol, whisky, beer, and spirits from the fermentation of sugars. It is one of the most polluting wastewaters, which offers characteristics such as:

Distillery wastewater poses significant environmental hazards, leading to issues such as eutrophication. This process adversely affects human health and disrupts the flora and fauna in water bodies. Molasses-based wastewater contains toxic substances such as melanoidins, phenolics, and heavy metals, which cause hazards to human health, animals, and ecosystems. (Kumar et al. 2020). The presence of phenolic compounds and other toxic substances further complicates the treatment process. Hence, there is a need for effective treatment methods that are critical due to these environmental concerns (Raj et al. 2020). Traditional treatment methods, such as anaerobic digestion, aerobic treatment, and physicochemical processes, are used to manage distillery wastewater (Pant & Adholeya 2007, Satyawali & Balakrishnan 2008). Though these methods often face challenges related to high operational costs, sludge generation, and the inability to completely remove color and refractory compounds (Kumar et al. 2019).

Duckweed's ability to treat distillery wastewater has been highlighted in several studies. Kumar et al. (2017) evaluated the performance of *Spirodela polyrhiza* in treating distillery effluent in a laboratory-scale experiment. The results indicated a lessening of COD and BOD, along with the removal of phenolic compounds. The duckweed biomass harvested from the treatment system was analyzed for its nutritional composition and found to be rich in proteins and other essential nutrients, suggesting its potential use as animal feed (Kumar et al. 2019). Joshi et al. (2017) investigated the use of duckweed sewage treatment from a molasses-based distillery. The study reported significant reductions in organic matter and color, with removal efficiencies

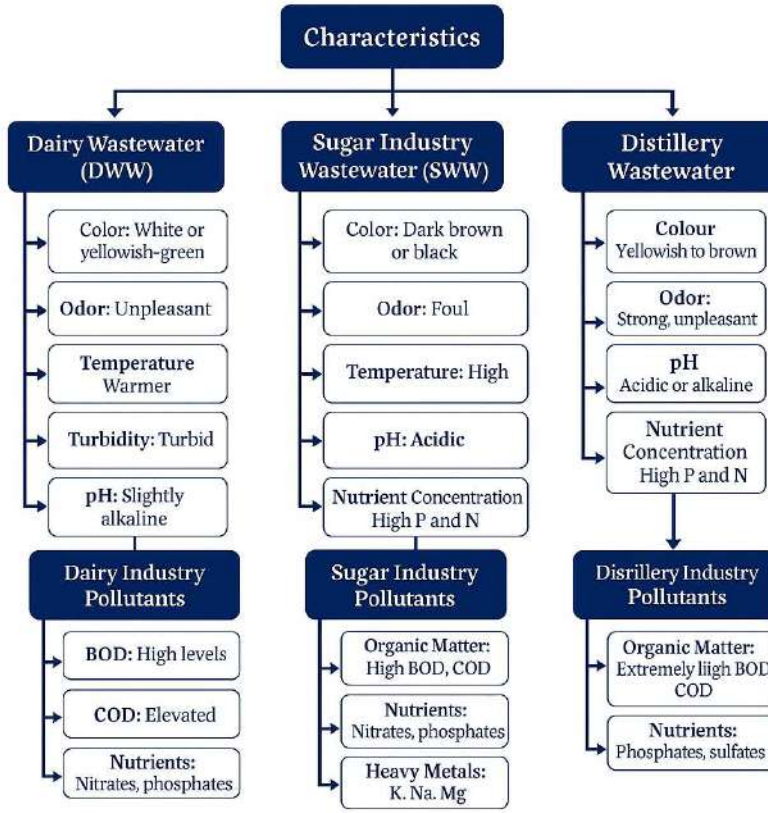


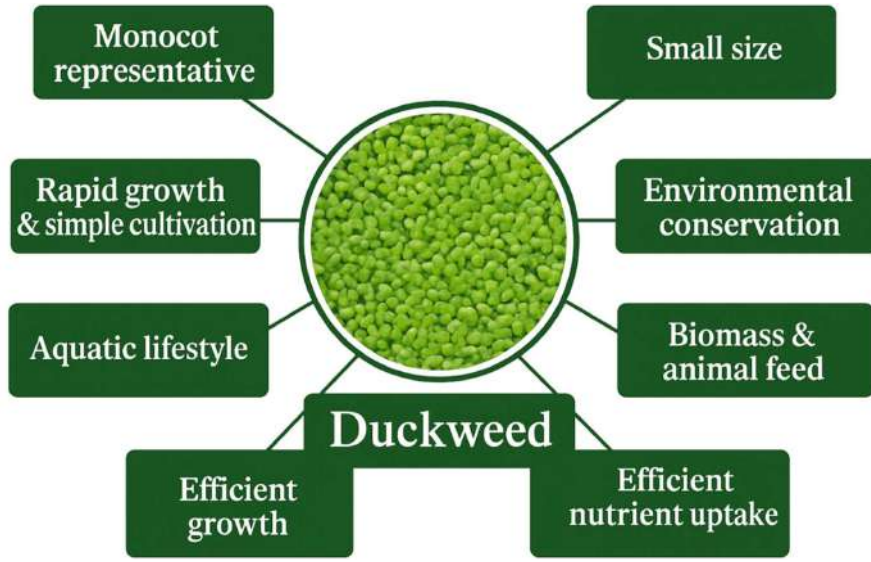
Fig. 2: Comparative physicochemical characteristics and major pollutants found in dairy (DWW), sugar industry (SWW), and distillery wastewater (DStWW).Source: Own creation based on compiled data from Kolev Slavov (2017), Malik et al. (2019), Kumar et al. (2020), Shashikanth et al. (2019), Fito et al. (2019), Ahmad and Mahmoud (1982), Sahu et al. (2008).

exceeding 80%. The treated effluent met the discharge standards set by regulatory authorities, demonstrating the feasibility of duckweed for distillery wastewater treatment (Joshi et al. 2017). Singh et al. (2015) further confirmed the effectiveness of duckweed in distillery wastewater treatment. The researchers reported that duckweed could substantially reduce BOD, COD, and phenolic compounds, with removal efficiencies comparable to conventional treatment methods. The harvested biomass was analyzed for its nutritional composition and found to be suitable for use as animal feed (Singh et al. 2015).

Duckweed Treatment Mechanism

Growth characteristics and nutrient uptake: Lemnaceae, the official name for duckweed (genus Lemna), are monocots (like grasses and palms) that are classified into five genera: Landoltia, Wolffiella, Spirodela, Lemna, and Wolffia (Asolekar et al. 2014, Thingujam et al. 2024). It floats on the surface of still-moving lakes, ponds, and sloughs and is no longer than ¼ of an inch. It exhibits rapid growth, with some species capable of doubling their biomass in just a few days under optimal conditions (Fig. 2). Duckweed is highly efficient in nutrient uptake, particularly nitrogen and phosphorus, making it an excellent candidate for wastewater treatment (Xu et al. 2023). It can thrive in varied environmental conditions, further enhancing its potential for use in wastewater treatment (Cheng et al. 2002, Culley Jr & Epps 1973, Hillman & Culley 1978). Duckweed's high nutrient uptake capacity is attributed to its physiological characteristics, including a high surface area-to-volume ratio and efficient nutrient transport mechanisms (Landolt & Kandeler 1987, Leng 1999). Duckweed can absorb nutrients directly from the water column, assimilating them into its biomass and reducing the nutrient load in the wastewater (Verma & Suthar 2015). In high-strength swine wastewater, Cheng & Stomp (2009) found that *L. minor* grew at a rate of about 29 $gm.^{-2}.day^{-1}$, but duckweed absorbed 90% of the total Kjeldahl Nitrogen (TKN) and 88.6% of the total Phosphorus (TP) (Cheng & Stomp 2009). Another study revealed the phosphorus uptake of 13 to 58 mg P m^{-2} d^{-1} and found that phosphorus uptake by duckweed was dependent on nitrogen concentration and the depth of the growth pond (Landolt & Kandeler 1987). Typical duckweed can have protein concentrations of up to 45% of its dry bulk. The gathered duckweed has a high protein content, making it a viable food source for animal and human feed (Iqbal et al. 2019).

Duckweed mechanisms of pollutant removal: Duckweed is highly effective in removing pollutants from water through various mechanisms, as supported by extensive research. These mechanisms include direct assimilation of nutrients into its biomass, microbial degradation of organic compounds in the rhizosphere (root zone), and sedimentation of particulate matter. The floating mat of duckweed also helps reduce light penetration, thereby limiting the growth of algae and other phototrophic organisms (Zhao et al. 2020, 2014). Duckweed root-associated microbial communities are essential for the breakdown of organic materials and the conversion of nutrients. These microbial processes enhance the whole pollutant removal efficacy, making duckweed-



Source: Own creation, adapted from Coughlan et al. (2022), Xu et al. (2023), and Zhou et al. (2023a).

Fig. 3: Key characteristics of duckweed (*Lemna* spp.).

based systems highly effective for wastewater treatment (Landesman et al. 2005).

Research has demonstrated the effectiveness of duckweed in eradicating a wide range of contaminants (Muradov et al. 2014). The combination of direct nutrient uptake, microbial degradation, and physical filtration makes duckweed a versatile and efficient biological treatment agent. Duckweed enhances organic matter degradation through oxygen supply and provides an exterior for bacterial growth, leading to substantial N and P removal (Körner et al. 2003). The growth rates of various *L. Minor* species vary, influencing their efficiency in wastewater treatment (Khare et al. 2021). Duckweed is an effective tool for phytoremediation of wastewater, capable of removing nutrients and pollutants like nitrate, phosphate, BOD, and ammonia with high efficiency (Frédéric et al. 2006). Duckweed is easy to harvest and has a high protein content, making it a valuable byproduct of the phytoremediation process. - Duckweed-based systems can remove 73-97% of nitrate and 63-99% of phosphate, as well as 96% of BOD and 99% of ammonia within 3 days (Gupta & Prakash 2013). These mechanisms collectively enable duckweed systems to achieve 85–95% pollutant removal efficiency**,** making them a cost-effective solution for wastewater treatment, eutrophication control, and phytoremediation.

Case Studies and Comparative Analysis

Nutritional composition of duckweed: The most successful decades for research on duckweed as a protein source were the 1970s and 1980s (Cheng & Stomp 2009), making it a fantastic option for animal feed. It contains 20–40% protein on a dry weight basis (Hughey 1995), with a high-quality amino acid (AA) profile comparable to soybean, including vital AAs like lysine, methionine, and tryptophan, which are vital for animal growth (Appenroth et al. 2018). Duckweed also provides 4–7% lipids, including omega-3 and omega-6, which improve animal health and product quality, such as enriched eggs or milk. Its carbohydrate content ranges from 25–35%, serving as a reliable energy source, while its 5–15% fiber aids digestion (Sońta et al. 2019, Xu et al. 2023). Additionally, it is rich in vitamins (A, B-complex, and C) and minerals like calcium, phosphorus, potassium, and magnesium, and trace elements such as zinc and iron (Pagliuso et al. 2022). Duckweed also contains beneficial phytochemicals like polyphenols and flavonoids, which offer antioxidant benefits. With low levels of anti-nutritional factors, it is highly digestible and suitable for various animals. The possible uses of duckweeds in aquaculture and animal feed may be affected by the presence of carotenoids such as lutein, astaxanthin, and β-carotene (Zhao et al. 2020). It is widely used in livestock, including poultry, fish, and ruminants, and in aquaculture. Duckweed also supports sustainable farming practices due to its rapid development, minimal land requirements, and ability to utilize nutrients from wastewater (Coughlan et al. 2022). Appenroth et al. have shown duckweed's nutritional worth as animal feed. The research reported that its biomass contains major amounts of protein, fiber, and essential nutrients, making it a suitable feedstock for poultry and fish. The researchers also highlighted the environmental benefits of using duckweed as feed, as it reduces the reliance on conventional feed ingredients and promotes sustainable agriculture (Appenroth et al. 2018). Cheng & Stomp (2009) explained that the grown duckweed can be used to make value-added products such as fuel ethanol and animal feed, and it can also be a good source of proteins and carbs. When yeast was used to ferment the hydrolysate, 25.8% of the initial dry duckweed biomass was converted to ethanol. According to these findings, duckweed biomass can yield sizable amounts of starch that are easily transformed into ethanol (Cheng & Stomp 2009).

Processing techniques: Processing techniques are crucial for converting duckweed biomass into safe, nutritious, and cost-effective animal feed while minimizing waste and maximizing sustainability (Ujong et al. 2025). Steps such as drying, grinding, and pelletizing enhance feed quality by preserving nutrients, reducing moisture content to prevent spoilage, and transforming raw biomass into a uniform, easy-to-consume format that reduces wastage. Additional methods like fermentation, ensiling, extrusion (Ujong et al. 2025, Zhao et al. 2014), and fortification improve digestibility and nutritional value while cleaning, disinfection, and testing ensure compliance with safety standards. Efficient processing not only optimizes the use of harvested duckweed but also positions it as a sustainable alternative to conventional feed sources like soy or fishmeal (Takács et al. 2025). Using a Box-Behnken experimental design, Mirón-Mérida et al. (2024) improved the alkaline protein extraction from *Lemna minor* using ultrasound.

Additionally, an investigation of how ultrasound affects the extracted protein's morphological, structural, and functional characteristics was conducted. They concluded that more protein may be extracted because of the cellular disturbance caused by ultrasonography. Additionally, the use of ultrasonography during the protein extraction process altered the duckweed protein's structure and color, improving its characteristics.

When taking into account the use of duckweed in various food products, these findings are encouraging (Mirón-Mérida et al. 2024). A non-thermal processing method called high hydrostatic pressure technology (HHPT) has demonstrated

potential in improving the bioavailability and bioaccessibility of bioactive substances in a range of food systems. However, little research has been done on how high hydrostatic pressure affects duckweed's bioactive qualities (Chen et al. 2018). Abdullahi AI et al. showed how various processing techniques affected Lemna paucicostata's nutritional makeup and anti-nutritional characteristics. Four different processing techniques were applied to Lemna paucicostata. While all of the processing techniques employed in this study decreased L. paucicostata's anti-nutritional components, the blanching procedure is the most effective at enhancing the plant's nutritious content (Abdullahi et al. 2022). Lam et al. (2017) investigated the use of extrusion technology to process duckweed biomass into pellets. The study reported that extrusion significantly improved the texture and nutritional composition of duckweed feed, making it suitable for use in livestock diets. The researchers also highlighted the economic benefits of using extrusion technology, as it reduces processing costs and enhances the shelf life of duckweed feed (Lam et al. 2017). Table 1 lists the comparison of duckweed with other nature-based phytoremediation solutions.

Biomass Valorization

Environmental impact: Duckweed has a profound positive impact on the environment due to its ability to address multiple ecological challenges. It improves water quality by efficiently absorbing excess nutrients such as N and P, which helps prevent eutrophication and supports healthier aquatic ecosystems (Fig. 3). Its role in wastewater treatment is particularly notable, as it thrives in nutrient-rich environments and removes contaminants, heavy metals, and organic pollutants, making water safe for reuse (Jaimes Prada et al. 2024). Duckweed's rapid growth rate enables it to act as a carbon sink, absorbing major amounts of carbon dioxide and causative to climate change mitigation. As a sustainable, high-protein biomass, it serves as an eco-friendly option to conventional feed sources like soy and fishmeal, thereby reducing overfishing, deforestation, and greenhouse gas emissions associated with traditional feed production.

Additionally, its cultivation requires minimal freshwater and no arable land, making it a resource-efficient crop that aligns with global efforts to conserve natural resources and promote sustainable agriculture (Vu et al. 2020). Utilizing duckweed to produce biomass and remediate wastewater contributes to reducing nutrient pollution in water bodies, mitigating eutrophication, and providing a sustainable alternative to chemical fertilizers. Additionally, biomass production provides a renewable source of nutrients that can be utilized as animal feed or organic fertilizer, reducing the reliance on synthetic fertilizers and promoting sustainable agriculture (Zhou et al. 2023a).

Economic Viability

Duckweed demonstrates strong economic viability with significant potential for revenue generation across multiple

Table 1: Comparison of duckweed with other nature-based phytoremediation solutions.

Parameter	Duckweed (*Lemna spp.*)	Algae	Water Hyacinth	Constructed Wetlands	References
Growth Rate	Very fast (doubling time ~1.5–3 days)	Fast, but strain-dependent	Slower (7–14 days)	Slow (~weeks)	Cheng and Stomp (2009), Zhao et al. (2020), Hillman and Culley (1978)
Nutrient Uptake	High N & P removal (up to 90–95%)	High N uptake, limited P in some species	Moderate N and P	Variable, depends on plant species and substrate	Gupta and Prakash (2013), Körner et al. (2003), Pant and Adholeya (2007)
Biomass Use	Feed, bioethanol, compost	Biofuel, compost	Compost, mulch, biogas	Rarely used, sometimes for compost	Appenroth et al. (2018), Zhao et al. (2014), Pant and Adholeya (2007)
Harvesting	Very easy, surface floating	Difficult (microscopic cells)	Bulky, manual/ mechanical	Not practical, rooted plants	Zhou et al. (2023a), Gupta and Prakash (2013)
Space Requirement	Low (compact growth in small ponds)	High (needs open, sunlit area)	Moderate	High (land-intensive)	Coughlan et al. (2022), Pant and Adholeya (2007)
Pollutant Removal Efficiency	85–95% for nutrients and organics	70–90% depending on species	60–85%	50–80%	Gupta and Prakash (2013), Zhao et al. (2014), Pant and Adholeya (2007)
Operational Cost	Low setup and maintenance	Moderate (needs light, nutrients)	Moderate, but biomass needs disposal	High (construction and maintenance)	Calicioglu et al. (2021), Pant and Adholeya (2007)
Challenges	Sensitive to pH and seasonal variation	Prone to crashes, harvesting issues	Invasive species risk	Clogging, mosquito breeding	Zhou et al. (2023a), Pant & Adholeya (2007), Gupta & Prakash (2013)

industries due to its low production costs, high biomass yield, and versatile applications (Calicioglu et al. 2021). The global duckweed protein market was valued at USD 72.7 million in 2024 and is projected to grow at a compound annual growth rate (CAGR) of over 7.9% during 2025–2034 (Global Market Insights, 2024). Similarly, overall duckweed sales are expected to grow at a CAGR of 10.8% between 2023 and 2033, reaching approximately USD 195.4 million by 2033 (Future Market Insights, 2023; Fig. 4 & 5). The global market for sustainable animal feed ingredients, biofuels, and organic fertilizers is expanding rapidly, creating a lucrative opportunity for duckweed-based products. As a high-protein biomass, it provides a cost-effective alternative to traditional feed ingredients like soy and fishmeal, meeting the rising demand in the livestock and aquaculture sectors (Pagliuso et al. 2022). The biofuel market, valued at over $100 billion, offers additional revenue streams, as duckweed can serve as an efficient feedstock for bioethanol production. Furthermore, its use in wastewater treatment and bioplastics aligns with the increasing global demand for eco-friendly solutions, supported by government incentives and corporate investments in green technologies. Duckweed farming also

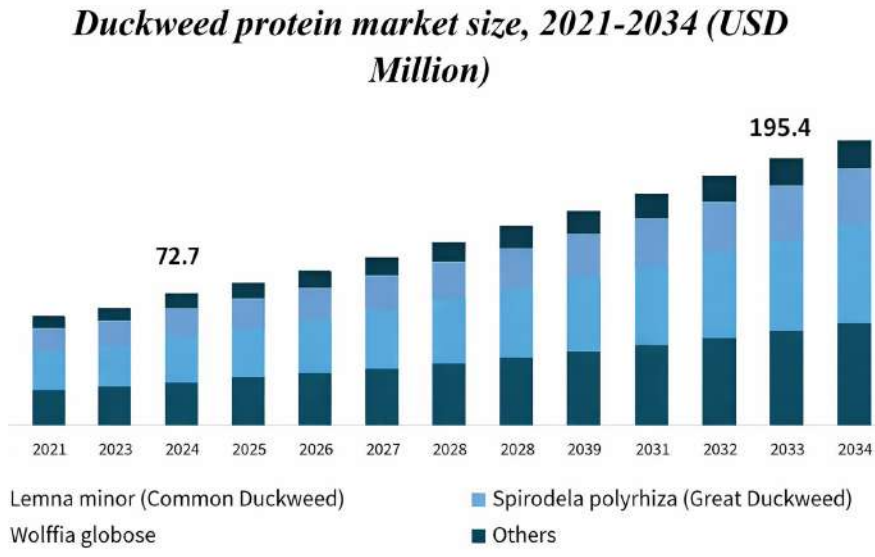


Fig. 4: Duckweed protein market size (Own creation) (https://www.futuremarketinsights.com/reports/duckweed-market).

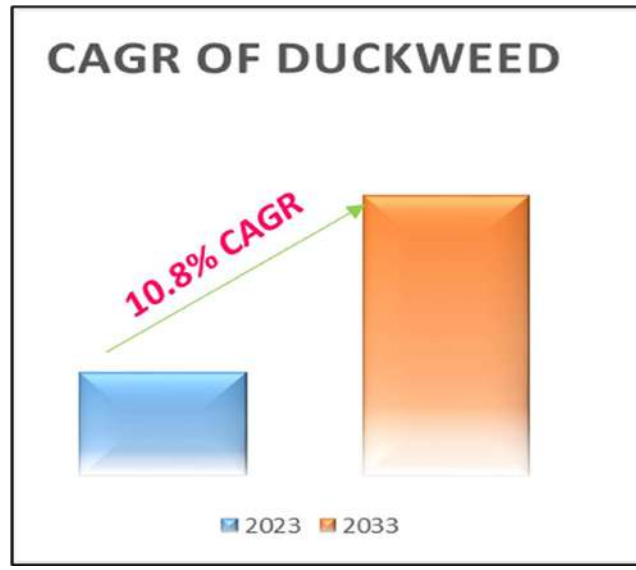


Fig. 5: Growth rate of duckweed (Own creation). (https://www.futuremarketinsights.com/reports/duckweed-market).

creates opportunities for rural development, employment, and export potential, particularly in regions with limited agricultural resources (Yang 2022). Comprehensive market analysis indicates that increasing awareness of sustainable practices and the rising cost of conventional feed and energy sources will continue to drive the adoption of duckweed, ensuring its long-term profitability and market expansion.

Challenges and Future Perspectives

Operational challenges: Duckweed wastewater treatment, though promising, faces several operational challenges that impact its scalability and efficiency. Duckweed growth is highly sensitive to environmental conditions such as temperature, light, pH, and nutrient availability, which may ensure optimal performance (Pasos-Panqueva et al. 2024). Harvesting and managing excess biomass is another hurdle, as overgrowth may lead to oxygen depletion, and disposal or utilization of the biomass requires further processing (Zhou et al. 2023a). Variability in water quality, contamination risks from absorbed pollutants like heavy metals, and difficulties in integrating with conventional treatment methods add to the complexity. Seasonal variations, especially in colder climates, can further hinder consistent performance.

Additionally, the economic viability of scaling up such systems is questionable, as it requires frequent monitoring, control, and maintenance to ensure optimal conditions for efficient treatment (Coughlan et al. 2022). The differences in the distribution and amount of bioactive chemicals within duckweed present another difficulty. Growth conditions, processing techniques, and the source or species can all have a big impact on these chemicals' concentration and bioavailability (Takács et al. 2025). Addressing these challenges is essential for the broader adoption of duckweed-based wastewater treatment.

Research gaps and future directions: Despite its potential, several research gaps limit the full-scale application of duckweed in wastewater treatment, paving the way for future exploration. Limited studies focus on optimizing duckweed's growth under diverse wastewater conditions, including high pollutant loads and variable climates, to enhance its resilience and efficiency. More research is needed on nutrient recovery mechanisms and the fate of absorbed contaminants to ensure environmental safety. Additionally, scalable and cost-effective systems for biomass harvesting, processing, and utilization, such as bioenergy production or bioproduct synthesis, remain underexplored. The integration of duckweed treatment with conventional technologies and its application in decentralized wastewater systems also requires further investigation. Integrating advanced technologies, such as hydroponic systems and automated harvesting, could enhance the efficiency of duckweed-based wastewater treatment. Forthcoming investigations should focus on genetic engineering to improve growth rates, pollutant uptake, and stress tolerance while leveraging advanced monitoring systems for automation and real-time control. Expanding these areas can unlock the potential of duckweed as a sustainable and scalable solution for WW treatment (Coughlan et al. 2022, Thingujam et al. 2024, Ujong et al. 2025). Further investigations into the potential of *L. minor* as a feedstock for biofuel production and bioremediation of emerging pollutants, such as pharmaceuticals and microplastics, could expand its applications. Additionally, future research should address the regulatory and policy frameworks needed to support the large-scale implementation of duckweed-based systems. Developing guidelines for the safe use of duckweed feed and establishing standards for wastewater treatment and biomass production are essential for promoting sustainable and economically viable practices (Zhou et al. 2023b).

Policy and Implementation Discussion

To enable large-scale implementation of duckweed-based treatment systems, policy frameworks must support integration with existing treatment infrastructure. Key considerations include establishing regulatory standards for effluent reuse, protocols for biomass utilization (e.g., as animal feed or fertilizer), and incentives for decentralized treatment models. Collaborative efforts between environmental agencies, municipalities, and industry stakeholders are essential to overcome institutional and economic barriers.

CONCLUSIONS

Duckweed-based wastewater treatment is a sustainable and innovative approach that effectively addresses water pollution while promoting resource recovery. Its rapid growth on nutrient-rich wastewater and high efficiency in removing pollutants such as nitrogen, phosphorus, COD, and BOD make it a promising biodegradable choice to conventional treatment approaches. Additionally, the nutrient-rich biomass produced during the process has immense potential for reuse, particularly as animal feed and biomass for bioenergy production, contributing to a circular economy. However, successful implementation of duckweed systems requires addressing challenges such as environmental variability, water quality fluctuations, and scalability. Standardized protocols for optimizing duckweed growth, nutrient absorption, and biomass utilization across diverse wastewater sources are crucial for maximizing its benefits.

Further research should also explore integrating duckweed systems with existing treatment technologies and their potential for decentralized wastewater management. By adopting duckweed-based treatment systems, communities can not only enhance water quality and reduce treatment costs but also generate valuable by-products like animal feed and biomass.

This dual-purpose approach offers a cost-effective and environmentally sustainable solution, paving the way for broader applications in wastewater management and resource recovery, ultimately contributing to global environmental and economic sustainability.

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The Impact of Chromium Contamination in Fish and Rice on Public Health Risks along the Opak River in Yogyakarta

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ABSTRACT

Access to clean water is increasingly threatened by industrial pollution, particularly from the tanning industry, which poses significant health risks and environmental challenges. This research aimed to determine Cr(VI) concentrations in water, sediment, fish, and rice samples from several sites along the river and to analyze the related health hazards. The study was conducted from March 2023 to November 2024, involving samples collected from 40 fishponds and rice fields located at different distances from the industrial area. Water, sediment, and fish samples were collected and analyzed to determine the concentration of Cr(VI) using Atomic Absorption Spectrophotometry (AAS) and spectrophotometry methods. A total of 360 samples from fishponds and 180 samples from rice fields were collected. In addition, a survey was conducted on rice and fish consumption patterns among 200 respondents from the affected areas. Cr(VI) concentrations were observed in all impacted locations, with levels significantly exceeding those found in the control area. Cr(VI) accumulation in fish and rice showed a significant increase, with health risk assessments revealing that both noncarcinogenic and carcinogenic risks surpassed safe limits. The findings indicate that industrial wastewater severely contaminates aquatic environments, posing significant health risks due to dietary exposure to Cr(VI). This study provides important insights into the prevalence of Cr(VI) contamination in agricultural and aquaculture systems, links environmental pollution to public health risks, and underscores the importance of regulatory measures to ensure food safety and public health.

INTRODUCTION

Access to clean water remains a critical challenge in the twenty-first century, impacting human health, limiting agricultural productivity, degrading ecosystem services, and constraining economic growth (UNESCO 2023). A major concern for water quality is the rising concentration of pollutants in water bodies, which can undermine the achievement of sustainable development goals (Ezbakhe 2018). Rapid industrialization in Indonesia poses a significant risk of environmental pollution, with river ecosystems being particularly vulnerable. Intensive industrialization and lax environmental regulations have led to significant pollution in many rivers (Liu et al. 2018). Accelerated economic expansion has resulted in serious environmental pollution challenges, with increased heavy metal concentration and accumulation harming freshwater ecosystems (Paschoalini & Bazzoli 2021). Approximately 80% of urban wastewater is discharged into untreated water bodies globally (WWAP 2017), while industries contribute millions of tons of heavy metals to these environments (Mateo-Sagasta et al. 2017).

The tanning industry is considered an ecological threat due to its release of hazardous waste into the environment, which contributes to environmental contamination (SMEP 2018, Suman et al. 2021). The tannery needs around 30-40 m^3 of water and 300 kg of chemicals to process one ton of leather or raw materials (Lofrano et al. 2013). Each tanning process can produce around 20% of leather goods products, while the remaining 60% consists of solid and liquid waste (Sivaram & Barik 2019), which is disposed of into the environment. Tannery significantly contributes to hexavalent chromium pollution, with wastewater exhibiting chromium concentrations ranging from 1 to 77 $mg.L^{-1}$ (Sharma et al. 2020). The discharge of liquid waste by tanneries into rivers has led to a decline in water and soil quality, with chromium contaminants widely distributed across various environmental compartments (Rahardjo et al. 2021a, 2021b, Rahardjo et al. 2023). Restoration efforts are necessary for heavily contaminated land (Irshad et al. 2021). Due to its extensive occurrence, environmental pollution resulting from hexavalent chromium is a worldwide issue (Brasili et al. 2020).

Chromium is classified as a class A carcinogen due to its significant toxicity (Sharma et al. 2021). Chromium exists in various valence states in the environment, with Cr(VI) and Cr(III) being the most stable forms, each displaying unique characteristics. Notably, hexavalent chromium (Cr(VI)) is the main contributor to pollution toxicity (Tumolo et al. 2020, Chen et al. 2022). Cr(VI) is detrimental to vegetation, aquatic species, and microorganisms. Cr(VI) is a potent epithelial irritant and a human carcinogen, ranking eighth on the ATSDR (2020) list. Cr(VI) and its metabolites, especially chromate, represent highly toxic forms that can infiltrate the human body via inhalation, ingestion, and dermal exposure. This exposure can lead to pathological changes in various organs and systems, including the respiratory tract, skin, and gastrointestinal tract, and may also increase cancer incidence and mortality rates (Sharma et al. 2022). Long-term exposure to chromium can lead to digestive disorders, respiratory complications, kidney and liver disorders, genetic alterations, and various other health disorders (Shanker et al. 2005). Chromium-induced river pollution significantly threatens ecological systems through its accumulation and biomagnification in aquatic environments, sediments, and food chains (Rahardjo et al. 2023). Excessive ingestion of chromium, when not metabolized by the body, can result in its accumulation within the intra- or extracellular compartments of organs (Briffa et al. 2020). Chromium has been detected in the tissues of fish sourced from metal-contaminated aquatic environments (Sobhanardakani et al. 2016). The accumulation of chromium in fish and rice may present a risk to both animals and humans.

Research has extensively examined the transmission of contaminants from the environment to food and ultimately to humans. Extensive research has been conducted on the health risks associated with chromium pollutants in aquatic biota and food products, including rice, vegetables, and fish in public waters (Gomah et al. 2019, Tayone et al. 2020, Wahiduzzaman et al. 2021, Xiang et al. 2021, Zulkafflee et al. 2022, Ogbuene et al. 2024). However, there has been inadequate research to assess the effects of using chromium-contaminated river water for aquaculture and rice agriculture, particularly regarding contamination, bioaccumulation, and potential health risks to residents. Therefore, it is critical to assess the concentration of chromium heavy metals in fisheries and agricultural products and to perform health risk evaluations concerning rice and fish consumption. Health risk assessment methods enable researchers to examine and measure the potential health effects of heavy metal exposure (Varol & Sünbül 2020). Human health risk assessment methods can evaluate both non-carcinogenic and carcinogenic health risks, specifically Risk Quotient (RQ) and Excess Cancer Risk (ECR). This study analyzed the quantity and frequency of rice and fish consumption to evaluate the potential health impacts of heavy metal exposure (USEPA 2011, 2012, 2018).

The expanding industrial activities along the downstream area of the Opak River contribute significantly to economic growth, job creation, and regional development. However, without effective governance, monitoring, enforcement, and compliance with environmental regulations, these activities give rise to environmental pollution. The discharge of liquid waste from industrial areas is the primary source of chromium contamination in the downstream section of the Opak River. Weak supervision, inadequate enforcement, and the absence of effective pollution prevention and water quality management programs have perpetuated this issue. Chromium pollution in the Opak River poses a serious threat to food security and public health, yet chromium has not been included in river water quality monitoring standards, resulting in limited assessment of its environmental impact to date. This study addresses a critical knowledge gap concerning the impact of chromium contamination on food security and public health, particularly through the consumption of rice and fish. Although prior research has explored the health risks of chromium in aquatic environments and food products, specific data on the effects of using chromium-contaminated river water for aquaculture and agriculture remain limited. This study aimed to fill this gap by assessing chromium concentrations in fisheries and agricultural products and evaluating the potential health risks for local residents. The research specifically investigated the impact of Cr(VI) contamination from tannery activities

on rice fields and aquaculture ponds adjacent to the Opak River. The findings revealed that Cr(VI) accumulation in fish and rice exceeded acceptable intake thresholds, posing significant non-carcinogenic and carcinogenic health risks for populations reliant on these food sources. The results underscore the urgent need for improved industrial waste management and regulatory measures to mitigate the adverse effects of chromium pollution, highlighting the essential role of monitoring and protecting aquatic environments.

MATERIALS AND METHODS

Characterization of Temporal Characteristics and Location

The study was carried out from March 2023 to November 2024 in rice paddies and aquaculture ponds reliant on the Opak River for water and fishing activities. Pollution from tanneries significantly increases chromium concentrations in the aquatic environments downstream of the Opak River (Rahardjo et al. 2021a, Rahardjo et al. 2021b). The increase in chromium levels presents a risk to aquatic organisms and human health, resulting in detrimental impacts on numerous species and polluting drinking water sources. Prolonged exposure to chromium pollution could reduce biodiversity, disrupt local ecosystems, and necessitate costly remediation efforts to enhance water quality and protect public health. Station (A) was located in the upper section of the Opak River, about 5 km from the industrial zone, and served as a benchmark location. The concentration of heavy metals in wastewater from tanneries was evaluated at four sites: B, C, D, and E, situated approximately 5, 10, 15, and 20 km away, respectively. Fig. 1 depicts the positioning of the industrial area within the Piyungan sub-district and the configuration of each sampling site along the Opak River.

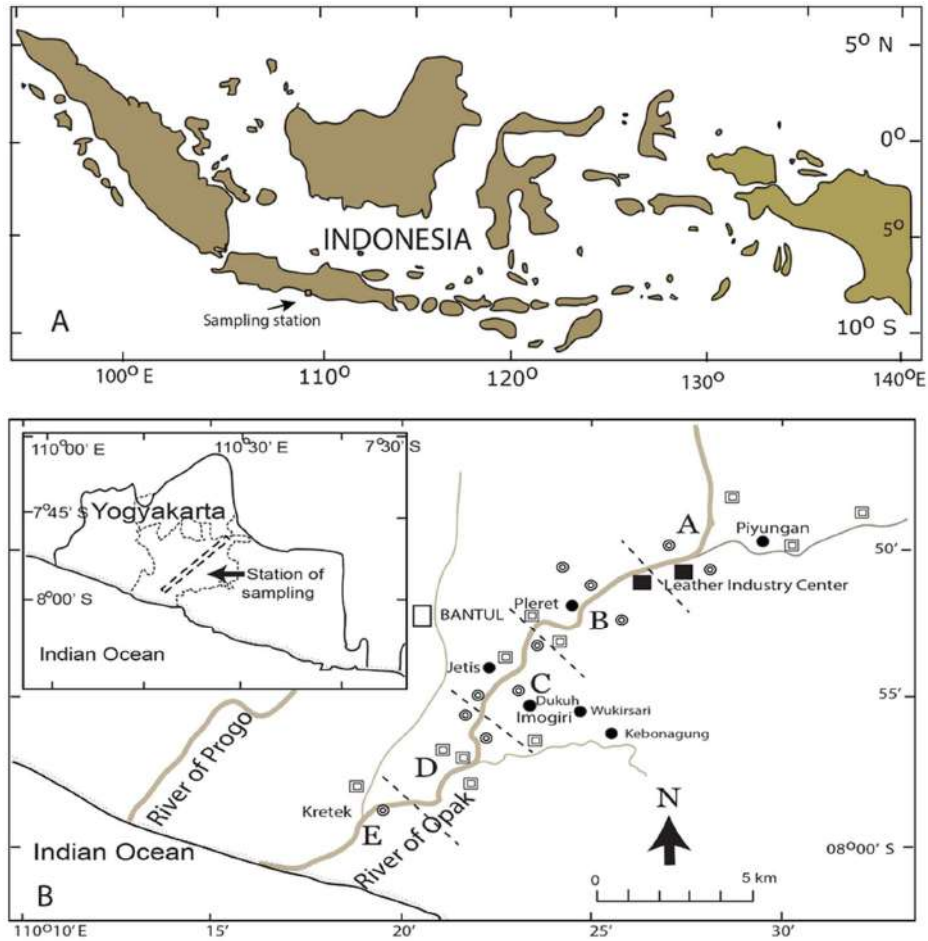

Fig. 1: Map of the territory of the Republic of Indonesia (panel A) and the location of industrial areas and distribution of sampling stations located upstream of the area 5 km away (station A), and respectively stations B, C, D, and E located downstream of the industrial area 5, 10, 15 and 20 km away (panel B).

The absence of a control station in this study was due to the use of existing upstream locations as benchmarks for comparison with downstream sites impacted by industrial pollution. Station A, positioned 5 km upstream from the industrial zone, served as the reference point for assessing the impact of chromium contamination from tanneries on aquatic environments. The study design focused on assessing the effects of pollution at various downstream distances (stations B, C, D, and E), rather than establishing a separate control station, as the upstream site was deemed sufficient for understanding baseline conditions. This approach directly compared pollution levels and related health risks without the need for additional control stations.

Sampling and Preparation

A total of 40 fish farming ponds were sampled, including four catfish ponds and four ponds of other fish species located upstream of the industrial area as control sites. The remaining 32 ponds were distributed across four sites situated 5, 10, 15, and 20 km downstream of the industrial area, which is impacted by tannery liquid waste discharge. Water, sediment, and fish samples were collected from each pond in triplicate, resulting in a total of 360 samples. Rice field samples were collected from four randomly selected areas at each location, including water, sediment, and grain, also in triplicate, yielding 180 samples. All samples were placed in sterile plastic bags and transported to the laboratory in airtight containers with ice packs. Samples were initially rinsed with tap water followed by deionized water to remove surface contaminants. Consumable portions of fish were excised using a ceramic knife, homogenized, and stored in plastic containers at –20°C.

Sample Analysis

The process for chromium removal from water samples complied with the APHA/AWWA/WEF Standard Methods, 20th Edition, 2001. The Environmental Protection Agency (2001) states that acid extraction is effective for acquiring solid materials, especially fish and detritus. The wet weight was measured with an analytical scale, and the sample was then dried in an oven at 60˚C to remove moisture. The dried weight was subsequently reevaluated, and the sample was pulverized using a mortar before being stored in a hermetically sealed container. A total of 3 g of sample was mixed with 18 mL of hydrochloric acid and 6 mL of concentrated nitric acid. The sample was then heated until it reached a volume of approximately 10 mL. The sample underwent repeated exposure to hydrogen nitrate solutions and strong hydrochloric acid before heating. The extract was subsequently filtered using filter paper that had been treated with 1% hydrogen nitrate. AAS was employed to ascertain the chromium content of the extract in accordance with the procedures outlined in SNI 06-6989.17-2004. The Perkin Elmer AAS PinAAcle 900T was employed to perform an analytical operation. All glassware and polyethylene bottles used in this study were pre-soaked with 10% HNO_3 for 24 h, rinsed with ultrapure water, and then air dried before use. Three samples, including one procedural bank, one matrix spike sample, and one bank spike sample, were analyzed along with every batch of digestion samples. The accuracy of replicate analyses of reference material showed good agreement, with a recovery rate of 85% and a detection limit of 0.003 $mg.kg^{-1}$.

Data Analysis

Fish and rice consumption data was collected from respondents in four affected areas. A total of 200 respondents were randomly selected, with 50 individuals from each location. The effects of non-carcinogenic and carcinogenic health risks were analyzed, referring to the US EPA's metal risk assessment guidelines (US EPA 2012). The calculation of non-carcinogenic health risks expressed in the RQ was carried out by comparing non-carcinogenic intake with RfD (Reference Dose):

$$\boldsymbol{Non\ Carcinogenic\ Intake} = \frac{\boldsymbol{C \times R \times Fe \times Dt}}{\boldsymbol{Wb \times Tavgk}} \quad ...(1)$$

Intake refers to the daily amount of Cr(VI) concentration entering the body ($mg.kg^{-1}.day^{-1}$). In this formula, C represents the concentration of Cr(VI) in food ($mg.kg^{-1}$), R denotes the rate of consumption or the weight of food ($kg.day^{-1}$), Fe signifies the number of days of exposure each year ($days.year^{-1}$), and Dt indicates the number of years of exposure (years). Additionally, Wb denotes human body weight (Kg), while Tavgk represents the average duration of days for non-carcinogenic effects (30 years × 365 days. $year^{-1}$).

The RQ value was determined based on the following equation:

$$\boldsymbol{RQ} = \frac{\boldsymbol{Non\ Carsinogenic\ Intake}}{\boldsymbol{RfD}} \quad ...(2)$$

Where Intake is the amount of concentration of Cr(VI) that enters the body every day ($mg.kg^{-1}.day^{-1}$), and RfD is the Reference Dose of hexavalent chromium in food according to the US EPA (2018), which is 0.003 $mg.kg^{-1}.day^{-1}$.

Carcinogenic health risks were expressed in exponential numbers without units and were assessed using the ECR metric. The risk was considered safe (acceptable) if the ECR value was $\leq 1 \times 10^{-4}$ or expressed as ECR ≤ 1/10,000.

Carcinogenic health risks were deemed unsafe if the ECR value exceeded 1×10^{-4} or ECR was greater than 1/10,000 (Ministry of Health 2012). The ECR value was calculated by multiplying the carcinogenic intake by the Cancer Slope Factor (CSF) as demonstrated below:

$$\textbf{Carcinogenic Intake } (\textbf{CDI}) = \frac{\boldsymbol{C \times R \times Fe \times Dt}}{\boldsymbol{Wb \times Tavgh}} \quad ...(3)$$

CDI refers to the daily concentration of a risk agent that is absorbed by the body, measured in $mg.kg^{-1}.day^{-1}$. In this computation, C represents the concentration of risk agents in food ($mg.kg^{-1}$), R indicates the rate of consumption or the amount of food weight ($kg.day^{-1}$), Fe signifies the length of days of exposure each year ($days.year^{-1}$), and Dt denotes the number of years of exposure (years). In the denominator, Wb represents human body weight (kg), while Tavgk denotes the average duration in days for non-carcinogenic effects (70 years × 365 $days.year^{-1}$).

Furthermore, the ECR value was calculated using the equation:

$$\boldsymbol{ECR = Carcinogenic\ Intake \times CSF} \quad ...(4)$$

Where CSF stands for Cancer Slope Factor (CSF) value, and ECR stands for Excess Cancer Risk. The US EPA states that the value for chromium hexavalent is 0.5 (US EPA 2011).

A one-way analysis of variance (ANOVA) was employed to evaluate non-categorical data that followed a normal distribution, specifically focusing on Cr(VI) concentrations in samples from both control and affected sites. This analysis assessed pollution levels across multiple districts based on the collected samples, which included water, sediment, fish, rice, and other relevant components. In all analyses, statistical significance was defined as $p < 0.05$. The statistical analyses were performed using SPSS version 21.0, while data visualizations of Cr(VI) concentrations were generated using R version 4.3.3. Additionally, the relationship between independent variables-including hexavalent chromium concentration in food, intake rate, exposure duration, and body weight-and the dependent variable of health risk (RQ) was evaluated using linear regression analysis with the enter method.

RESULTS AND DISCUSSION

Cr(VI) Contamination in Aquaculture Ponds and Rice Fields

The concentrations of Cr(VI) in rice fields, ponds, and sediments in the upstream and downstream areas of the Piyungan Industrial Area are presented in Fig. 2. Water from aquaculture ponds and rice fields sourced from the downstream section of the Opak River contained chromium, showing varying levels of contamination.. There was no indication of Cr(VI) concentrations in water samples collected from fish ponds or rice fields at an upstream industrial site. Cr(VI) concentrations were detected in water samples from downstream areas of the industrial zone, with levels ranging from 0.054 to 0.143 $mg.L^{-1}$ in fish pond water samples and 0.117 to 0.197 $mg.L^{-1}$ in rice field water samples. Meanwhile, Cr(VI) levels in sediments were found in higher concentrations, ranging from 0.016-0.770 $mg.kg^{-1}$ in fish ponds and 0.016-0.320 $mg.kg^{-1}$ in rice fields.

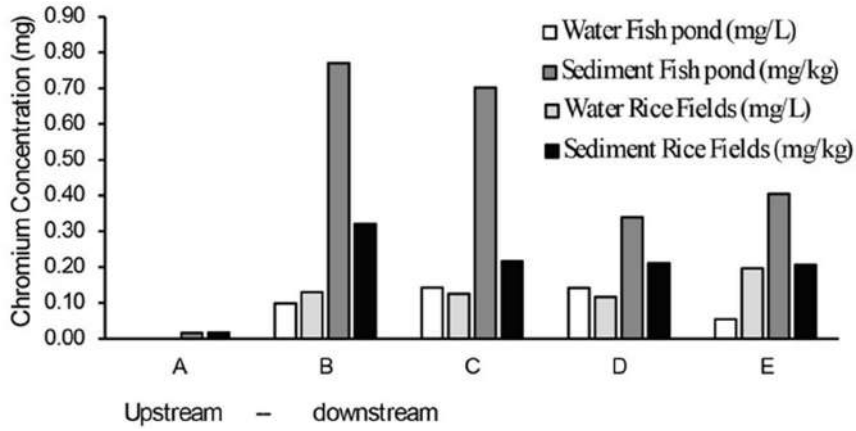

Fig. 2: The chromium concentrations at the upstream station (A) and at stations B, C, D, and E—located 5, 10, 15, and 20 km downstream of the industrial area, respectively—were measured to assess pollution patterns. Sediment samples from rice fields and aquaculture ponds contained higher levels of Cr(VI) than the corresponding water samples. Cr(VI) concentrations were highest near the wastewater discharge points at stations B and C, then decreased or fluctuated further downstream. ANOVA results showed a significant difference in Cr(VI) concentrations in both water and sediment samples between the control and affected locations, with a p-value < 0.005.

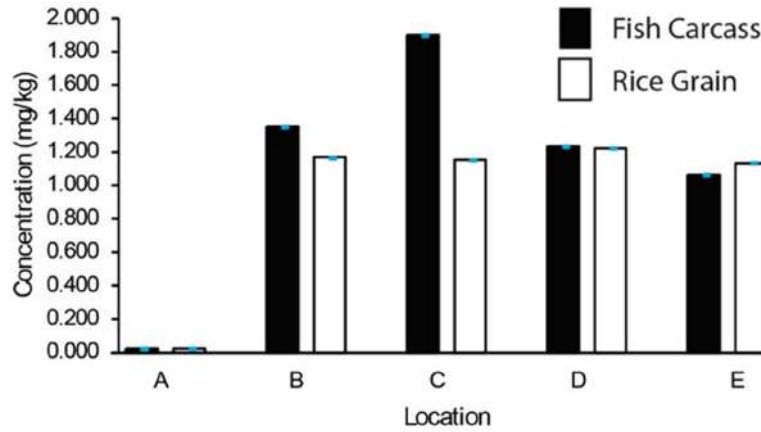

Fig. 3: Comparison of Cr(VI) accumulation in fish and rice (mg.kg^{-1}).

Chromium (VI) Accumulation in Fish and Rice

The accumulation levels of Cr(VI) in fish and rice samples from four areas downstream of the tanneries are presented in Fig. 3. Chromium pollutants contaminated all fish and rice samples in all study locations. However, the average accumulation of Cr(VI) in fish and rice in the upstream locations of the industrial area was found in very small concentrations, i.e., 0.020 mg.kg^{-1} in fish and 0.023 mg.kg^{-1} in rice. It was very different from the accumulation levels of Cr(VI) in the affected locations, which ranged from 0.860 -1.740 mg.kg^{-1} in fish and 1.132 - 1.221 mg.kg^{-1} in rice.

The mean Cr(VI) accumulation varied depending on the organism type and sampling station location. Fish samples exhibited a greater accumulation of Cr(VI) compared to rice samples. The ANOVA analysis indicated a significant difference in Cr(VI) concentration between fish and rice samples from control and affected zones (p-value < 0.005).

Consumption Rate, Estimated Daily Intake, and Health Risk

Table 1 displays the distribution of rice and fish consumption levels within the community, along with daily intake statistics. The rice and fish consumption patterns of the population vary across the four regions affected by tannery waste disposal practices. Rice consumption ranged from 253.00 to 312.43 g.day^{-1}, with an average of 267.50 g.day^{-1}. The daily fish consumption varied between 21.43 and 45.71 g.day^{-1}, with an average of 33.75 g.day^{-1}. The daily intake value was determined by the amount of food consumed and the concentration of chromium contained in the meal. The daily chromium intake from rice and paddy consumption was 10,000 µg.kg^{-1}.day^{-1} at station D, 8,800 µg.kg^{-1}.day^{-1} at station B, 8,200 µg.kg^{-1}.day^{-1} at station C, and 5,700 µg.kg^{-1}.day^{-1} at station E.

Health risk characterization was carried out by determining the RQ and ECR values based on the community's rice and fish consumption patterns. The daily non-carcinogenic intake values ranged from 0.0057 to 0.0101 mg.kg^{-1}.day^{-1}, with an average of 0.0083 mg.kg^{-1}.day^{-1}. The highest average non-carcinogenic intake value was found at station D, with an average value of 0.0101 mg.kg^{-1}.day^{-1}, followed by stations B, C, and E. Table 2 presents the non-carcinogenic intake, chronic daily intake, RQ, and ECR values associated with the consumption of rice and fish contaminated with chromium.

The RQ value was evaluated to determine non-carcinogenic risk, with an acceptable limit of one (USEPA

Table 1: Consumption levels and estimated daily intake values of chromium in rice and fish.

Location	Consumption Rate [g.day^{-1}]		Total Consumption Rate [g.day^{-1}]	Chromium Concentration [mg.kg^{-1}]		Total Chromium Concentration [mg.kg^{-1}]	Body Weight [kg]	Daily Intake Rate [mg.kg^{-1}.day^{-1}]
	Fish	Rice		Fish	Rice			
B	25.00	267.00	292.00	1.47	0.248	1.718	57.00	0.0088
C	42.85	253.00	295.85	1.74	0.206	1.946	59.00	0.0100
D	45.71	259.00	304.71	1.41	0.199	1.609	57.00	0.0082
E	21.43	291.00	312.43	0.86	0.168	1.028	56.00	0.0057
Mean	33.75	267.50	301.25	1.47	0.205	1.575	57.25	0.0081

Table 2: Non-carcinogenic intake, Chronic Daily Intake, RQ, and ECR.

Location	Non-Carcinogenic Intake [mg.kg^{-1}. day^{-1}]	RQ	Chronic Daily Intake ([DI]	ECR
B	0.0088	2.93	0.0037	7.4 x 10^{-3}
C	0.0082	2.73	0.0043	8.6 x 10^{-3}
D	0.0101	3.37	0.0035	7.0 x 10^{-3}
E	0.0057	1.90	0.0024	4.8 x 10^{-3}
Mean	0.0083	2.77	0.0035	7.0 x 10^{-3}

2011). The study results showed that the RQ values at all research locations exceeded one. This indicates that the simultaneous consumption of rice and fish contaminated with chromium can pose significant non-carcinogenic health risks, rendering them unsafe for public consumption. The ECR values further demonstrated that rice and fish consumption at all sampling sites surpassed the established safe limit of 1.0×10^{-4}, thereby presenting a substantial carcinogenic risk in these areas. The intake rate and duration of exposure also influence the health risks associated with ingesting chromium-contaminated food. This was confirmed by the regression analysis, which examined the relationship between risk factors and health risk levels (Table 3). Intake rate and duration of exposure were significantly associated with health risk ($p < 0.001$). In contrast, factors such as chromium concentration, consumption amount, body weight, and age did not demonstrate statistical significance. The intake rate and duration of exposure were significant predictors of health risk, as indicated by high regression coefficients. Multicollinearity and interaction effects were not examined in the regression analysis (VIF <10).

Discussion

Water pollution, especially from industrial activities, presents a considerable risk to human health and the environment (Ogbuene et al. 2024, Yustiati et al. 2024). In developing countries like Indonesia, rapid industrialization has led to increased contamination of water bodies, particularly rivers. The Opak River, heavily impacted by the leather tanning industry, is a stark example of how industrial waste can severely degrade water quality. The findings from this research underscore the urgent need for effective monitoring and management strategies to mitigate the harmful impacts of such pollution. Implementing stricter regulations and promoting sustainable industrial practices are crucial for protecting water resources and safeguarding the health of local communities. Moreover, community engagement and education play vital roles in raising awareness about the importance of water conservation and pollution prevention. This empowers residents to advocate for cleaner practices and strengthens accountability among industries.

Chromium-contaminated river water used for agricultural irrigation and fishing activities is the main source of pollution in aquaculture ponds and paddy fields. The absence of hexavalent chromium in water samples from aquaculture ponds and rice fields located upstream of the industrial area supports this claim. In contrast, hexavalent chromium was detected in downstream regions linked to the leather tanning sector during wastewater discharge. According to Xu et al. (2023), tannery is identified as the primary source of environmental chromium contamination. The average concentration of hexavalent chromium in water samples exceeds the established limits for aquaculture quality. As per Government Regulation 82 of 2002, the allowable concentration of chromium is 0.05 mg.L^{-1}. The United Nations Environment Programme/World Health Organization has established a maximum acceptable concentration (MAC) of 0.05 mg.L^{-1} for chromium to protect aquatic ecosystems (UNEP 2008). Soil samples showed higher concentrations of hexavalent chromium compared to water samples. This study reveals that heavy metal levels are low in water but considerably higher in sediment and biota (Paller & Littrell 2007). Chromium quickly bonds with organic molecules and accumulates rapidly in sediments (Ipinmoroti et al. 2022, Ehiemere et al. 2022). Heavy metals, especially chromium, exhibit an increased propensity to associate with sediment, leading to its sequestration (Brady et al. 2015). The movement of heavy metals into sediments

Table 3: Results of linear regression analysis of risk agents and health risks (R^2=0.823).

Variables	B	Standard Error	95% Confidence Interval	p-*value*
Constant	-0.259	0.065	-0.0426 – -0.0151	<0.001
Intake Rate [R]	473.818	12.257	215.412 – 432.181	<0.001
Duration of Exposure [Dt]	0.008	0.003	0.006 – 0.009	<0.001
Hexavalent Chromium [C]	-0.242	0.092	-0.219 – 0.045	0.110
Amount of consumption	0.0007	0.002	0.00013 – 0.00019	0.432
Weight [BB]	0.003	0.001	-0.002 – 0.004	0.122
Age [A]	0.002	0.002	-0.001 – 0.004	0.279

results in elevated pollutant concentrations in soil while simultaneously lowering levels in water (Nurkhasanah 2015).

Udosen et al. (2016) observed that sand acts as a natural adsorbent for heavy metals in aquatic environments, thereby reducing the bioavailable fraction in the water. Unlike water, sediment functions as a reservoir for metals due to its distinct physicochemical characteristics. Currently, sediment is widely regarded as a major repository for heavy metals that accumulate as a result of pollution (Xia et al. 2020). It serves as both a source and a sink for the accumulation and redistribution of heavy metals (Miao et al. 2020; Wang et al. 2020). Heavy metals stored in sediments can be reintroduced into the water column, causing "secondary pollution" that adversely affects ecosystems and human health through the food chain and biological enrichment (Bing et al. 2019). Therefore, sediment is considered a sensitive indicator for evaluating the health of aquatic ecosystems (Bastami et al. 2015). Assessing river water quality based solely on heavy metal concentrations in the water is inadequate; it is essential to also quantify heavy metals in sediments. The contamination of rice paddies and aquaculture ponds with Cr(VI) leads to exposure and subsequent accumulation of Cr(VI) in both rice and fish.

Heavy metals may significantly contaminate the ecosystem as a result of chromium deposition in fish and rice (Makedonski et al. 2017). Chromium accumulation in fish and rice occurs through the uptake of water, sediment, or dietary sources, such as algae, consumed by herbivorous and omnivorous fish (Joshi et al. 2002). The accumulation of Cr(VI) in fish and rice samples is variable and influenced by many variables, including Cr(VI) concentrations in water and sediment, along with the physical and chemical characteristics of the environment at each research location. Moreover, heavy metal absorption is influenced by biota species, organism tolerance thresholds, sensitivity, and water's physical and chemical characteristics (Yousafzai et al. 2010). The variability of chromium accumulation in rice and fish may be caused by chromium concentration in sediment, bioavailability, physical and chemical characteristics of the environment, and types of organisms (Wu et al. 2021). Heavy metal contaminants in aquatic ecosystems may accumulate in fish via bioaccumulation and bioconcentration (Korkmaz et al. 2019, Arisekar et al. 2020). Factors such as sex, age, size, reproductive cycle, swimming behavior, dietary preferences, and environmental conditions significantly affect the accumulation of heavy metals in fish. The consumption of contaminated fish introduces heavy metals into the human body (Gholamhosseini et al. 2021). The accumulation of chromium in foods such as rice and fish is concerning, as its consumption may lead to health risks. Identifying chromium in rice fields, aquaculture ponds, and food, such as rice and fish, establishes a baseline for evaluating the food safety risk to consumers of these products.

Concentrations of Cr(VI) in fish and rice samples from areas affected by tannery effluent discharge were significantly higher. The findings of this research indicate chromium accumulation levels in fish that substantially exceed those reported in previous studies. Notably, the investigation by Rahman et al. (2012) in Bangladesh reported accumulation levels of 0.09 to 0.4 $mg.kg^{-1}$, while the study by Leung et al. (2014) in China indicated levels ranging from 0.2 to 0.65 $mg.kg^{-1}$ and 0.18 to 0.85 $mg.kg^{-1}$. The concentration of Cr(VI) in rice samples was significantly higher than the results reported by Gomah et al. (2019) in Monrovia, which indicated an average hexavalent chromium level of 0.4245 $mg.kg^{-1}$, Guo et al. (2019) in China, with an average of 0.31 $mg.kg^{-1}$, and Jahirudin et al. (2017), who recorded an average chromium concentration of 1,058 $mg.kg^{-1}$. However, this concentration is considered safe for consumption according to the maximum limit established by the Director General of the Food and Drug Authority, which is 2.5 $mg.kg^{-1}$ (Dirjen POM 1989). This stands in stark contrast to the concentration limits established by the WHO and the Federal Environmental Protection Agency, which specify that the maximum allowable amount of chromium in food, including fish, is 0.05-0.15 $mg.kg^{-1}$ of fish body weight (Bakshi & Panigrahi 2018).

Rice is a staple food for a large portion of the population in many Asian countries, including Indonesia. Meanwhile, freshwater fish is a preferred source of high-quality protein, chosen by many individuals to support their health. (Parvin et al. 2023). Consequently, chromium pollution in river ecosystems and the food chain can be transmitted to humans via rice and fish consumption, potentially harming human health. The rice consumption among individuals in the four research locations was notably high, varying from 253.00 to 312.43 $g.day^{-1}$, with an average of 267.50 $g.day^{-1}$. The average fish consumption was 33.75 $g.day^{-1}$, ranging from 21.43 to 45.71 $g.day^{-1}$. In the research locations, rice consumption significantly surpassed the national average of 217 $g.day^{-1}$, whereas fish consumption was considerably lower than the national average of 51 $g.day^{-1}$ (BPS 2024). The average rice consumption in the research community was 267.50 $g.day^{-1}$, significantly higher than that of several other Asian countries: China at 238 $g.day^{-1}$, Taiwan at 132 $g.day^{-1}$, and Japan at 119 $g.day^{-1}$ (Hu et al. 2016). The significant consumption of chromium-contaminated food, particularly rice, results in a daily chromium intake in the community at the research site, estimated to be between 0.0057 and 0.0100 $mg.kg^{-1}.day^{-1}$. Approximately 90% of chromium intake in humans occurs

through food consumption, rather than drinking water, skin contact, or inhalation (Zhang et al. 2020). Diet is the major source of chromium exposure. Estimated daily oral intakes for infants (1 year), children (11 years), and adults are 33-45, 123-171, and 246-343 $\mu g.person^{-1}.day^{-1}$, respectively (Rowbotham et al. 2000). The daily intake of chromium at each research site differs due to variations in consumption patterns and the level of chromium contamination in food. Consuming foods contaminated with chromium presents a public health risk (Varol & Sünbül 2020). Even in low concentrations, chromium remains dangerous because it can accumulate in the body and reach toxic levels (Chen & Chau 2019, Ustaoglu et al. 2019).

The study demonstrated that consuming food contaminated with Cr(VI) presents significant non-carcinogenic and carcinogenic health risks, as indicated by RQ values exceeding one and ECR values greater than 7.0×10^{-3}. The US EPA (2011) states that the RQ value is used to assess non-carcinogenic risk, with an acceptable maximum limit of 1 and an ECR value below 1.0×10^{-4}. Consequently, the community's consumption of rice and fish across all research regions poses significant risks of both serious non-carcinogenic and carcinogenic health effects. The intake of heavy metals, including chromium, can lead to their accumulation in body tissues such as adipose and bone tissues. Exposure to Cr(VI) may increase susceptibility to upper gastrointestinal cancer (Mensoor & Said 2018) and may reduce human life expectancy by approximately 9 to 10 years (Guerra et al. 2012). Chemically-acquired immunodeficiency syndrome (C-AIDS) refers to a weakened immune response caused by exposure to chemicals, including heavy metals. Prolonged exposure to chromium in humans may lead to gastrointestinal disorders, respiratory complications, renal and hepatic damage, and abnormalities in genetic material, among other health problems (Shanker et al. 2005). The principal pathophysiology involves DNA damage, genomic instability, and the generation of reactive oxygen species (ROS) induced by Cr(VI). Chromium (VI) increases oxidative stress and stimulates ROS production in target DNA and cellular lipids, resulting in DNA damage and lipid peroxidation (Balali-Mood et al. 2021). The cancer risk associated with Cr(VI) exposure can be influenced by various factors, including the level of Cr(VI) intake from contaminated sources and the differing concentrations of Cr(VI) present in food and drinking water. (ATSDR 2012, IARC 2012, Ukhurebor 2021).

However, the findings of this health risk analysis cannot be presented directly to the authorities for decision-making in risk management. Further efforts are required to characterise uncertainty and variability, which are essential components of health risk assessment. Health risk analysis is inherently subject to uncertainty due to variability across spatial and temporal scales (Walker et al. 2003). This variability arises from factors such as the intrinsic properties of an agent, the nature of its side effects, the characteristics of hazards, the relationship between the agent and health effects, the actual level of exposure, and the source of observed outcomes (Jansen et al. 2019). The deterministic approach to health risk assessment has limitations, particularly its tendency to underestimate or overestimate actual risk. Variability may stem from differences in metal concentrations, chromium species, consumption levels, age, sex, body weight, and physiological or metabolic parameters (Miletic et al. 2023).

Nevertheless, Cr(VI) contamination on agricultural land and aquaculture severely undermines the safe production of food crops and presents enormous latent dangers to human health. Cr(VI) pollution negatively impacts food safety and health; therefore, effective river water quality management, rigorous monitoring, and pollution prevention measures are essential to mitigate these adverse effects. These efforts can be made through better industrial waste management to prevent the discharge of pollutants into rivers, strict supervision of polluting industries, the need for regulations that limit the amount of waste that can be discharged into the environment, and increasing public knowledge and awareness of the risks of Cr(VI) to the environment and health. The findings highlight the urgent need for improved industrial waste management, stricter pollution regulations, and public awareness initiatives to mitigate the adverse effects of Cr(VI) contamination on food safety and human health. The study underscores the critical relationship between environmental pollution and public health, emphasizing the necessity for comprehensive monitoring and preventive measures.

The findings of the Opak River study highlight the urgent requirement for improved industrial waste management practices and more stringent regulatory measures to address chromium pollution. The significant health risks associated with consuming contaminated food highlight the importance of monitoring water quality and implementing effective pollution prevention strategies. Addressing these challenges is vital for safeguarding public health and ensuring food security in communities affected by industrial pollution. The research serves as a call to action for policymakers, industry stakeholders, and local communities to collaborate to mitigate environmental contamination's impacts and protect future generations.

CONCLUSIONS

There has been a significant increase in Cr(VI) contamination in water, sediment, fish, and rice downstream of the

Opak River. The findings demonstrate that all examined water, sediment, fish, and rice samples from downstream of the industrial zone exhibited varying levels of Cr(VI) contamination, exceeding the safety limits established by health authorities. The health risk assessment revealed significant non-carcinogenic and carcinogenic risks linked to the consumption of contaminated rice and fish, as indicated by RQ values exceeding one and ECR values surpassing acceptable thresholds. The results highlight the critical need for effective management of river water quality, the implementation of stricter regulations on industrial effluents, studies to assess the carrying capacity of rivers, and the establishment of maximum acceptable limits for liquid waste discharge into water bodies. To mitigate pollution and safeguard public health, Cr(VI) parameters should be incorporated into river water quality monitoring to facilitate routine assessments, including evaluations of various food commodities along the downstream Opak River.

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Diversity and Distribution Patterns of Lichens in Different Ecological Conditions in the Garhwal Himalaya, Uttarakhand

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ABSTRACT

This study investigated the distribution and diversity of lichens across different elevational zones in the Madhyamaheshwar Valley, Garhwal Himalaya. A total of 77 lichen species from 22 families and 52 genera were recorded across three altitudes: lower (1600–2300 m ASL), middle (2600–3100 m ASL), and higher (3200–3600 m ASL). Lichen diversity increased with elevation, with 48 species recorded at higher elevation sites, Madhyamaheshwar and Budha Madhyamaheshwar, 17 at middle elevation sites, Maikhamba-Chatti and Koonchatti, and 12 at lower elevation sites, Goundar Village, Lower Bantoli, Upper Bantoli, Khadarakhal, and Nanuchatti. Temperature and humidity were identified as significant factors influencing lichen diversity, with cooler conditions at higher elevations supporting more diverse communities. Slope and cardinal directions also influenced species distribution, with gentler slopes and southern cardinal directions supporting higher diversity. Lichens have a preference for tree bark as a substrate, with certain species exhibiting greater host specificity. These findings underscore the crucial role of environmental factors in shaping the distribution of lichen communities across elevational gradients in the valley.

INTRODUCTION

Lichens, as complex symbiotic systems, are globally distributed and serve as reliable indicators of ecological conditions, particularly in relation to atmospheric and climatic variations. Recent scientific investigations have deepened our insight into their physiological and distributional responses to changing climate patterns, reinforcing their role as critical bioindicators in ecological monitoring and environmental assessment (Stanton et al. 2023). Elevation gradients are regarded as one of the most significant patterns in biogeography (Lomolino 2001). The atmospheric conditions on a mountain change with altitude, resulting in variability in both the number of different species present and the specific types of species found (Körner 2007). Lichens, comparable to various other organisms, have been demonstrated to react to elevation-dependent parameters (Vetaas et al. 2019). Consequently, altitude is a key factor affecting the abundance, composition, and variety of lichen ecosystems (Baniya et al. 2010, Vittoz et al. 2010, Bässler et al. 2016, Rodríguez et al. 2017, Cleavitt et al. 2019). Lichens are adaptable organisms capable of surviving in a variety of environments, and their population patterns are shaped by factors such as moisture, temperature, air quality, and nutrient availability (Geiser et al. 2021). Their spatial distribution is strongly influenced by climatic factors, particularly temperature and humidity.

Additionally, studies have shown that the abundance of species varies significantly across different altitudinal zones (Abas & Din 2021). The distribution of lichen communities is influenced by key environmental elements, such as sunlight, humidity, temperature, and slope (Shrestha et al. 2012). Lichen communities are sensitive to variations in abiotic conditions, such as temperature, pollution levels, rainfall, and light availability. Steeper slopes tended to support fewer lichen species, suggesting that the terrain significantly affects their distribution. Additionally, microhabitat features, such as slope and light exposure, contribute to shaping lichen diversity, influencing both their richness and abundance across different environments (Cung et al. 2021). Latitude-dependent factors, such as aspect (primarily north-south) and slope, are also crucial, as they affect the amount of solar radiation received. Along with altitude, these factors determine incident solar radiation (insolation) and the extent of evapotranspiration (Pentecost 1979, Kidron & Termina 2010, Rodríguez et al. 2017). Therefore, in the southern hemisphere, areas facing south have cooler temperatures and higher humidity levels than areas facing north (Körner 1995, 2007). Lichen communities are strongly influenced by elevation, with species richness and composition varying along altitudinal gradients (Pinokiyo et al. 2008). Numerous studies have highlighted the exceptional diversity of lichens found at high-altitude locations across Europe, Asia, and India (Baniya et al. 2010, Vittoz et al. 2010, Rashmi & Rajkumar 2019; Abas & Din 2021).

In our previous study (Prabhakar et al. 2024), we provided valuable insights into the lichen flora of the Madhyamaheshwar Valley, documenting over 60 lichen species, representing 33 genera and 21 families, across eight sites along the trekking route from Goundar village to Budha Madhyamaheshwar. However, the scope of that study was confined to a limited number of key locations along the trekking path. The present study significantly broadens this scope by investigating previously unexplored regions of the valley. In contrast to our earlier research, this study incorporated additional ecological factors, including aspect, temperature, humidity, slope, and altitudinal gradients, thereby offering a more comprehensive understanding of species distribution. Moreover, several additional species that were not recorded in our previous study were identified in this study. These novel findings provide enhanced insights into the ecological and biodiversity patterns of the region, thereby advancing our understanding of lichen distribution across this ecologically sensitive region of the Himalayan gradient.

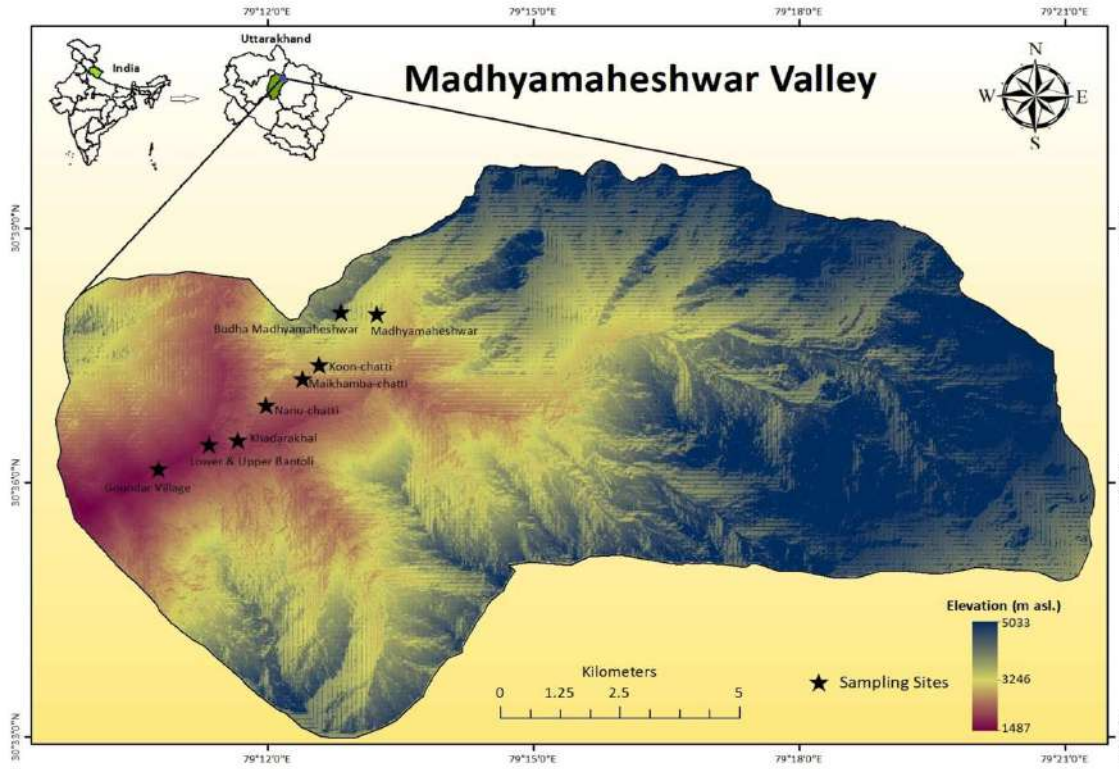

Fig. 1: Geographical map of the study area.

MATERIALS AND METHODS

Site Description

The study area, Madhyamaheshwar Valley, located in the Rudraprayag District of the Garhwal Himalayan region in Uttarakhand, is part of the Kedarnath Wildlife Sanctuary. It spans approximately 262 km^2. Lichens were collected from altitudinal ranges of 1600 to 3600 m ASL, covering an area of approximately 13.92 km^2. This area lies between latitudes 30°36'22" N and 30°37'59" N, and longitudes 79°11'12" E and 79°12'46" E (Fig. 1).

Lichen Sampling and Collection of Environmental Variables

The lichen samples were collected during May 2024 from Goundar Village, Lower Bantoli, Upper Bantoli, Khadarakhal, Nanuchatti, Maikhamba-Chatti, Koonchatti, Madhyamaheshwar, and Budha Madhyamaheshwar area of Madhyamaheshwar valley. These sites were categorized into three elevational zones: lower (1600–2300 m ASL), middle (2600–3100 m ASL), and higher (3200–3600 m ASL). Lichen samples were collected from various substrates, including tree barks, twigs, mosses, rocks, and soil. Data on elevation, slope, cardinal direction aspect, temperature, and humidity were recorded at the collection sites to ensure a thorough analysis of the environmental variables influencing lichen distribution. The study areas displayed a distinct elevation gradient, with Goundar village at 1600 m ASL Lower and Upper Bantoliranging from 1700 - 1800 m ASL**,** Khadarakhalat 2100 m ASL**,** Nanu-Chattiat2300 m ASL**,** Maikhamba-Chattiat 2600 m ASL**,** Koon-Chattiat 2900 m ASL**,** Madhyamaheshwarat 3200 m ASL**,** and the highest point, Budha Madhyamaheshwar**,** at3400 m ASL This gradient reflects a progressive increase in elevation across the study sites.

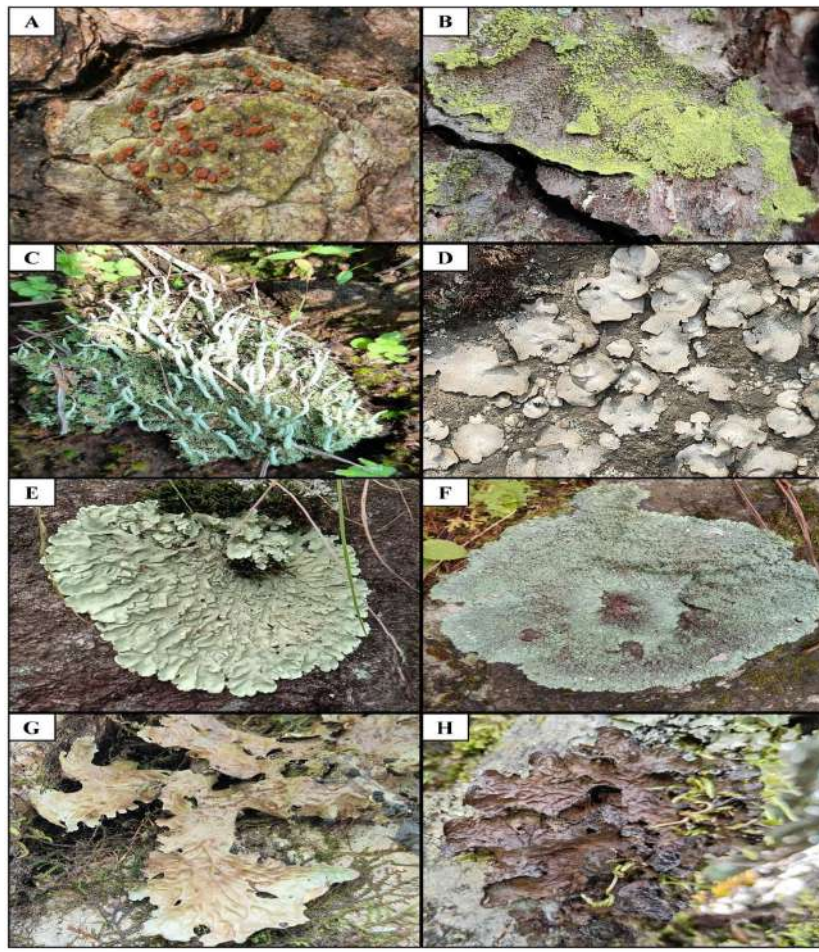

Fig. 2: Lichen species distribution across various substrates in the Madhyamaheshwar Valley **(A-H) - A.** *Caloplaca flavorubescens*, **B.** *Chrysothrix candelaris*, **C.** *Cladonia subulata*, **D.** *Dermatocarpon miniatum*, **E.** *Flavoparmelia caperata*, **F.** *Heterodermia diademata*, **G.** *Leptogium delavayi*, **H.** *Lobaria retigera*.

Table 1: Distribution of lichen taxa across different elevations, slopes, aspects, temperature ranges, and humidity levels in the Madhyamaheshwar Valley.

Lower elevation (1600 - 2300 m.ASL)			
Sites - Goundar Village, Lower Bantoli, Upper Bantoli, Khadarakhal and Nanuchatti			
Temperature (Min-max): 11.3°C to 32.1°C, Humidity (Min-max): 34% - 59%			
Family	Lichen taxa	Slope	Aspect
Teloschistaceae	*Caloplaca flavorubescens* (Huds.) J.R. Laundon	38.6°	NE
Chrysotrichaceae	*Chrysothrix candelaris* (L.) J.R. Laundon	63.7°	NE
Cladoniaceae	*Cladonia fruticulosa* Kremp.	24.7°	SE
	Cladonia subulata (L). F.H. Wigg.	11.1°	SE
Verrucariaceae	*Dermatocarpon miniatum* (L.) W. Mann	46.0°	SE
Parmeliaceae	*Flavoparmelia caperata* (L.) Hale	48.2°	E
Physciaceae	*Heterodermia diademata* (Taylor) D.D. Awasthi	20.8°	SE
Collemataceae	*Leptogium delavayi* Hue	44.2°	E
Lobariaceae	*Lobaria retigera* (Bory) Trevis	40.2°	SE
Parmeliaceae	*Parmotrema reticulatum* (Taylor) M. Choisy	57.0°	S
	Parmotrema tinctorum (Despr. ex Nyl.) Hale	40.0°	W
Pertusariaceae	*Pertusaria velata* (Turner) Nyl.	22.4°	SW
Middle elevation (2600 - 3100 m.ASL)			
Sites - Maikhamba-Chatti and Koonchatti			
Temperature (Min-max): 11.3°C to 24.8°C, Humidity (Min-max): 54% - 77%			
Cladoniaceae	*Cladonia coccifera* (L.) Willd.	22.8°	NW
	Cladonia corniculata Ahti and Kashiw	22.7°	NW
	Cladonia ramulosa (With.) J. R. Laundon	29.6°	N
Graphidaceae	*Graphis* sp. 2	14.3°	SE
Parmeliaceae	*Hypotrachyna cirrhata* (Fr.) Divakar et al.	52.5°	SW
Collemataceae	*Leptogium askotense* D.D. Awasthi	51.4°	N
	Leptogium burnetiae C.W. Dodge.	41.2°	S
	Leptogium trichophorum Müll. Arg.	50.6°	S
Lobariaceae	*Lobaria kurokawae* Yoshim	40.3°	S
Parmeliaceae	*Parmotrema nilgherrensis* (Nyl.) Hale	21.0°	E
Peltigeraceae	*Peltigera polydactylon* (Neck.) Hoffm.	28.6°	S
Physciaceae	*Polyblastidium microphyllum* (Kurok) Kalb.	45.1°	SE
Ramalinaceae	*Ramalina sinensis* Jatta	25.6°	SW
Parmeliaceae	*Sulcaria sulcata* (Lév) Bystrek ex Brodo and D. Hawksw	27.7°	W
	Usnea subfloridana Stirt.	47.5°	S
	Usnea orientalis Motyka	62.1°	S
Umbilicariaceae	*Umbilicaria vellea* (L.) Ach.	46.3°	S
Higher elevation (3200 - 3600 m.ASL)			
Sites - Madhyamaheshwar and Budha Madhyamaheshwar			
Temperature (Min-max): 7.2°C to 20.6°C, Humidity (Min-max): 29% - 66%			
Megasporaceae	*Aspicilia cinerea* (L.) Körb	32.9°	N
	Aspicilia dwaliensis R□s□nen	36.9°	NE
Parmeliaceae	*Bryoria himalayana* (Motyka) Brodo. & D. Hawksw.	52.9°	N

Table Cont....

Family	Lichen taxa	Slope	Aspect
Caliciaceae	*Buellia himalayensis* (S.R. Singh and D.D. Awasthi) A. Nordin	27.4°	NE
	Calicium adspersum subsp. *himalayense* G.Pant & D.D. Awasthi	23.4°	W
Cladoniaceae	*Cladonia fimbriata* (L.) Fr.	30.2°	W
	Cladonia laii S. Stenroos	28.5°	N
	Cladonia pocillum (Ach.) Grognot	44.2°	NW
	Cladonia pyxidata (L.) Hoffm	39.7°	W
	Cladonia squamosa Hoffm.	10.5°	E
Coccocarpiaceae	*Coccocarpia erythroxyli* (Spreng.) Swinscow & Krog	27.5°	E
Verrucariaceae	*Dermatocarpon vellereum* Zsaschke	35.9°	S
Parmeliaceae	*Dolichousnea longissima* (Ach.) Articus	36.6°	S
Graphidaceae	*Graphis cfr. duplicata* Ach.	67.8°	NE
	Graphis furcata Fée	21.2°	N
	Graphis scripta (L.) Ach.	23.1°	S
	Graphis sp. 1	25.4°	N
	Graphis sp. 3	18.6°	N
Physciaceae	*Heterodermia japonica* (Sato) Swinsc. & Krog.	42.6°	NW
Parmeliaceae	*Hypotrachyna nepalensis* (Taylor) Divakar et al.	38.6°	S
Lecanoraceae	*Lecanora caesiorubella* Ach.	42.3°	NW
	Lecanora fimbriatula Stirt.	14.2°	S
	Lecanora interjecta Müll. Arg.	13.7°	W
	Lecidella carpathica Körb.	15.1°	W
	Lecidella elaeochroma (Ach.) M. Choisy	24.8°	NW
	Lecidella euphorea (Flörke) Kremp	25.0°	W
Pertusariaceae	*Lepra leucosorodes* (Nyl.) I. Schmitt, B.G. Hodk and Lumbsch	16.8°	E
Teloschistaceae	*Loplaca pindarensis* (R□s□nen) Poelt and Hinter.	60.2°	N
Parmeliaceae	*Nephromopsis laii* (A. Thell and Randlane) Saag and A. Thell	14.2°	N
Ochrolechiaceae	*Ochrolechia subpallescens* Verseghy	58.0°	NE
Parmeliaceae	*Parmelia masonii* Essl. & Poelt.	28.4°	E
	Parmelinella wallichiana (Taylor) D.D. Awasthi	43.8°	S
	Parmotrema thomsonii (Stirt.) A. Crespo, Divakar and Hawksw	36.4°	S
Peltigeraceae	*Peltigera canina* (L.) Willd.	21.6°	SW
	Peltigera membranaceae (Ach.) Nyl	29.3°	N
Pertusariaceae	*Pertusaria composita* Zahlbr.	48.6°	S
Acarosporaceae	*Pleopsidium flavum* (Trevis) Körb.	38.1°	E
Lecideaceae	*Porpidia crustulata* (Ach.) Hertel and Knoph	62.3°	S
	Porpidia macrocarpa (DC.) Hertel and A.J Schwab	43.2°	S
Ramalinaceae	*Ramalina conduplicans* Vain	33.6°	N
	Ramalina himalayensis R□s□nen	22.9°	E
	Ramalina intermedia (Delise ex Nyl.) Nyl.	46.3°	E
Rhizocarpaceae	*Rhizocarpon geographicum* (L.) DC	44.3°	NW
Stereocaulaceae	*Stereocaulon foliolosum* var. *strictum* (C. Bab.) I.M. Lamb	57.1°	SW
	Stereocaulon myriocarpum Th. Fr	69.0°	S
Lobariaceae	*Sticta henryana* Müll. Arg.	72.0°	S
Umbilicariaceae	*Umbilicaria indica* Frey	51.9°	S
Teloschistaceae	*Xanthoria elegans* (Link)Th. Fr.	20.6°	NW
Total Families - 22	**Total number of lichen species – 77**		

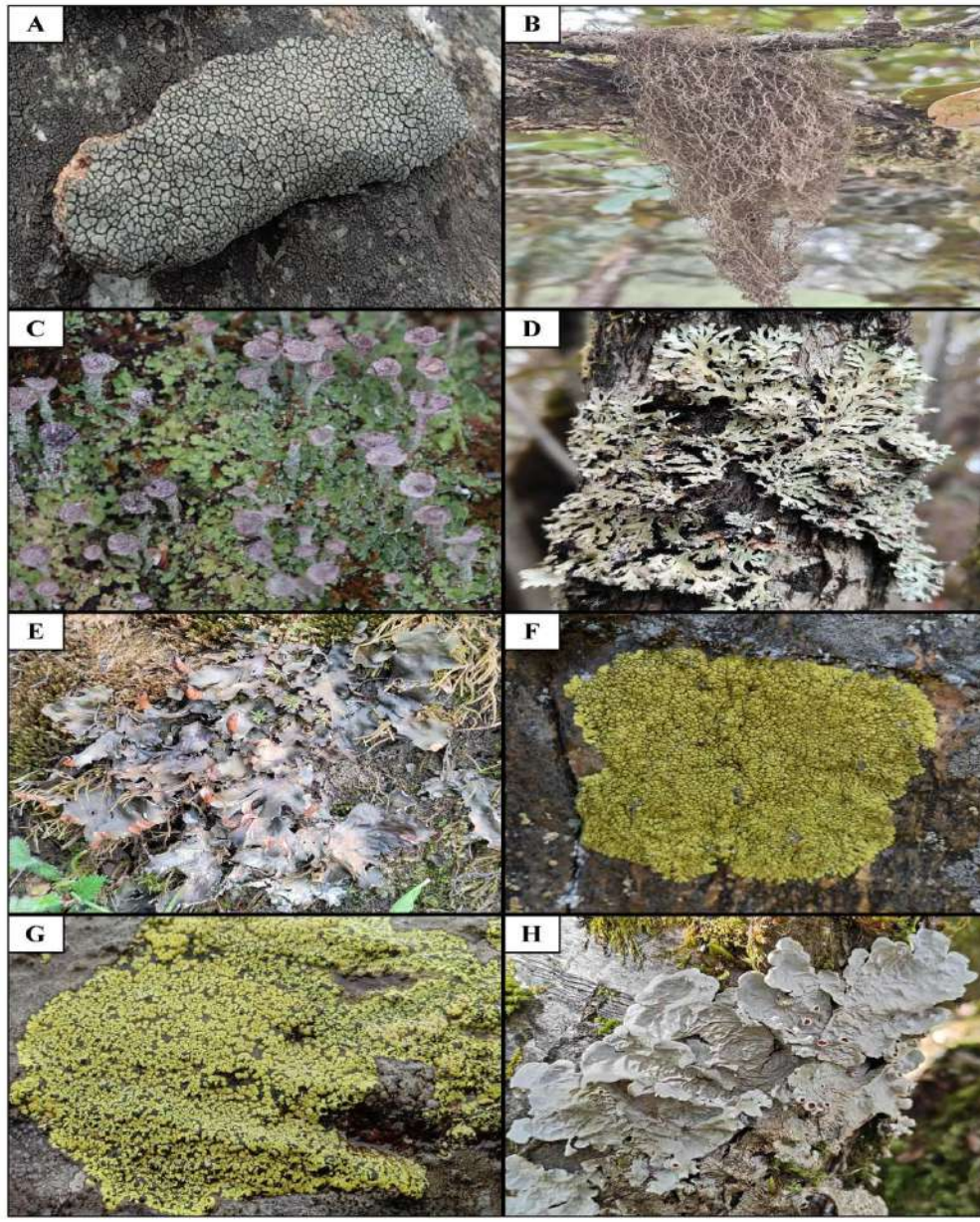

Fig. 3: Lichen species distribution across various substrates in the Madhyamaheshwar Valley **(A-H) - A.** *Aspicilia dwaliensis*, **B.** *Bryoria himalayana*, **C.** *Cladonia pocillum*, **D..***Heterodermia japonica*, **E.** *Peltigera canina*, **F.** *Pleopsidium flavum*, **G.** *Rhizocarpon geographicum*, **H.** *Sticta henryana*.

Identification of Lichens

The lichen samples were identified based on their morphological, anatomical, and chemical characteristics, following the methods described by Nayaka (2014) and the relevant literature (Awasthi 1991, 2007, Lücking et al. 2009, Singh & Sinha 2010, Singh &Arya 2019). The samples were confirmed and authenticated at the Lichenology Laboratory of the CSIR-National Botanical Research Institute in Lucknow. The identified lichen samples were deposited in the herbarium of H.N.B. Garhwal University (GUH) and the herbarium of the NBRI, Lucknow (LWG). Statistical analysis was performed using software/tools to accurately measure environmental variables: altitude was recorded with a barometric altimeter, slope was measured using a digital clinometer, aspect was determined with a compass, and humidity and temperature were quantified using a digital hygrometer. For spatial analysis and visualization, QGIS was used to process, analyze, and map the collected data, ensuring a comprehensive and scientifically rigorous assessment of the environmental parameters.

RESULTS

A total of 77 lichen species belonging to 22 families and 52 genera were identified. These included 28 crustose, 28 foliose, and 21 fruticose lichens (Table 1, Figs. 2-3).

Lichen Diversity, Temperature, and Humidity Across Different Elevation Zones

Lower elevation (1600-2300 m ASL): A total of 12lichen species were found, belonging to nine families and ten genera (three crustose, seven foliose, and two fruticose). Parmeliaceae was the dominant family. The temperatures ranged from 11.3 to 32.1°C, and the relative humidity ranged from 34% to 59% (Table 1, Figs. 4, 7, 8). These climatic conditions likely create distinct ecological niches that influence the distribution, growth, and survival of lichen species. Some species thrive in warmer and drier conditions, whereas others adapt to moderate temperatures and humidity.

Middle elevation (2600-3100 m ASL): A total of 17species were recorded from nine families and 12 genera (1 crustose, 9 foliose, and 7 fruticose). Parmeliaceae was the dominant family, followed by Cladoniaceae and Collemat. Temperatures ranged from 11.3 to 24.8°C, with humidity between 54% and 77% (Fig. 5). These moderate temperatures and higher humidity conditions likely supported greater species richness than at lower elevations. More stable climatic conditions foster a broader range of species with varying levels of tolerance to both temperature

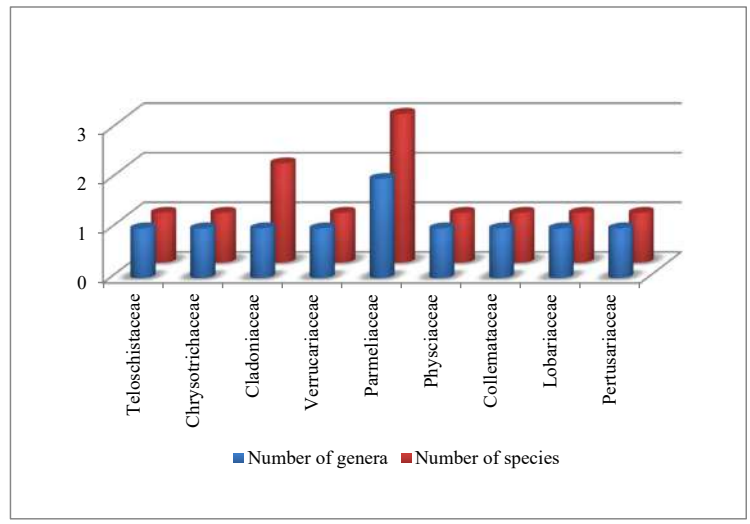

Fig. 4: Distribution of lichen families with respect to abundance of their species in lower elevation (1600 - 2300 m ASL).

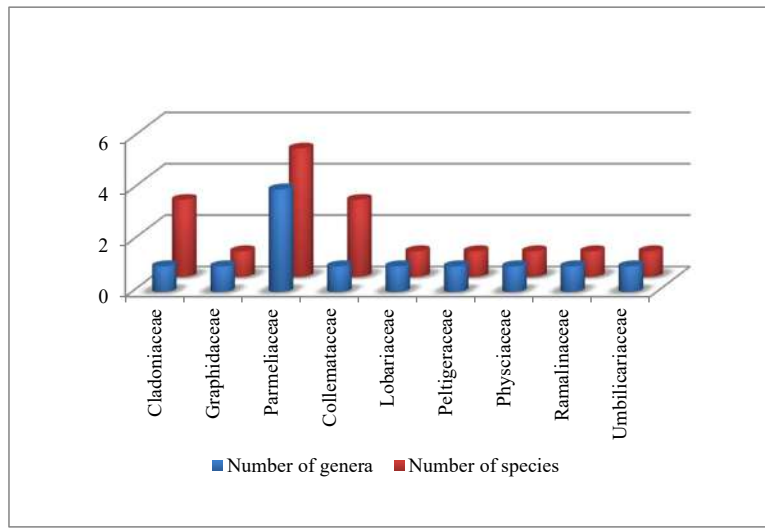

Fig. 5: Distribution of lichen families with respect to abundance of their species in the middle elevation (2600 - 3100 m ASL).

and humidity, promoting a diverse and stable ecological environment.

Higher elevation (3200-3600 m ASL): A total of 48species were recorded from 20 families and 30 genera (24 crustose, 12 foliose, and 12 fruticose). Parmeliaceae was the most represented family, followed by Lecanoraceae, Cladoniaceae, and Graphidaceae. The temperatures ranged from 7.2°C to 20.6°C, and the humidity from 29% to 66% (Fig. 6). These cooler temperatures and variable humidity levels supported a highly diverse lichen community. Some species thrived in cooler and less humid conditions, whereas others were resilient to lower humidity levels. These climatic factors, along with the specific adaptations of lichen species, play a critical role in shaping the lichen community at higher elevations.

Diversity of Lichens Across Slope Gradients

The distribution of lichens varied with slope angle

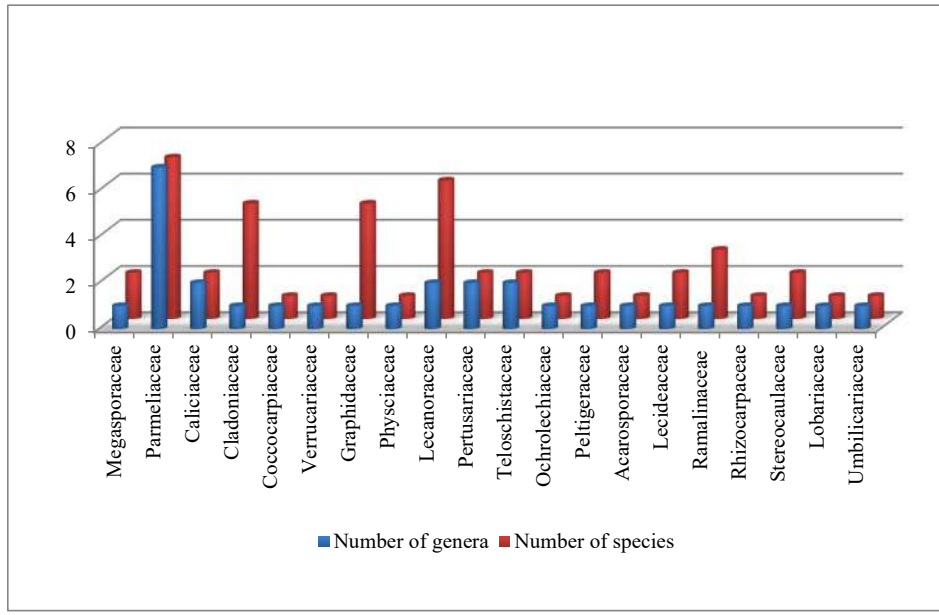

Fig. 6: Distribution of lichen families with respect to abundance of their species in higher elevation (3200 - 3600 m ASL).

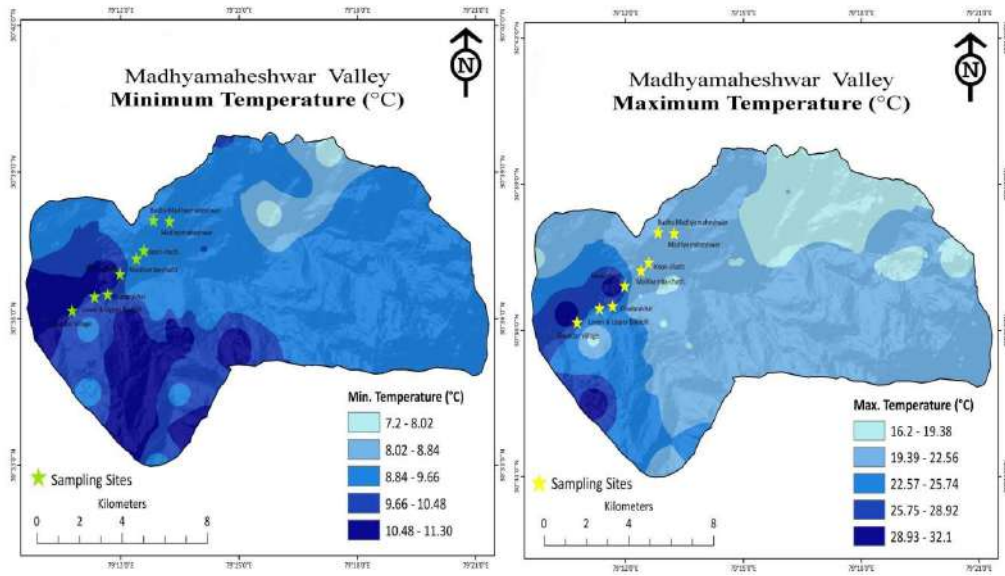

Fig. 7: Map showing the spatial variation in the maximum and minimum temperatures across the study area.

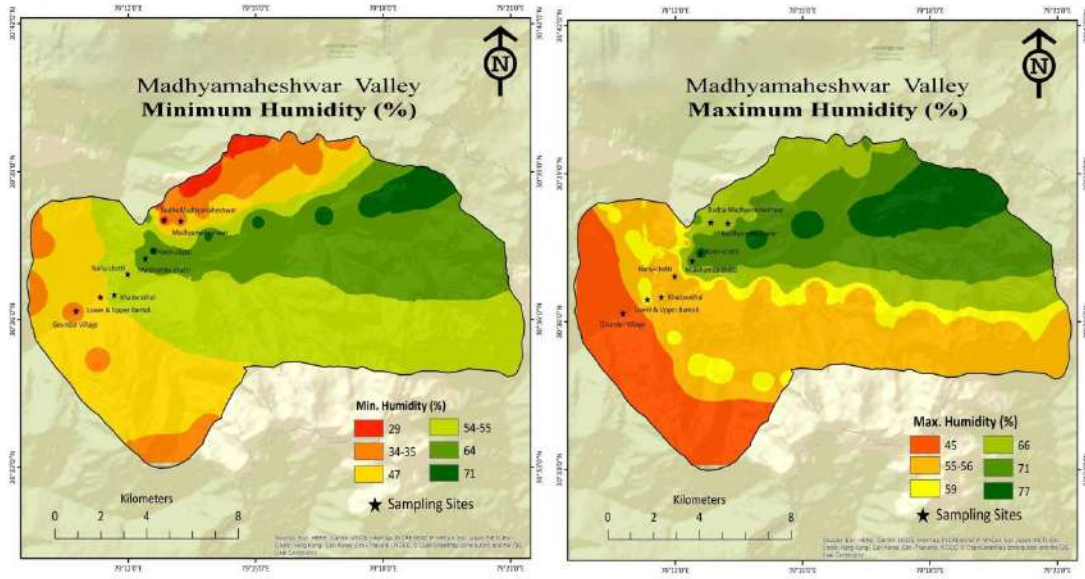

Fig. 8: Map showing the spatial variation in the maximum and minimum relative humidity across the study area.

(Table 1, Figs. 9 and 10). Gentle slopes (10.5°-30.2°) supported the highest diversity (34 species), likely because of better moisture retention. Moderate slopes (32.9°-48.6°) had 28 species, possibly affected by microclimatic variation. Steep slopes (50.6°-72.0°) supported 15 species, indicating the presence of stress-tolerant, specialized lichens. Overall, these findings highlight that lichen diversity is influenced by slope angle, with gentler slopes supporting higher diversity, whereas steeper slopes host a more specialized, lower diversity of species.

Diversity of Lichens in Different Directional Aspects

In the present study, the term 'aspect' refers to the direction a surface of a hill, mountain slope, or terrain faces relative to the sun, and it is used to describe how different orientations significantly affect lichen distribution (Table 1, Fig. 11 and 12).

The southern aspect supported the highest diversity (21 species) because of the greater sunlight and warmth. The northern aspect had 12 species, which were influenced by cooler and shaded conditions.

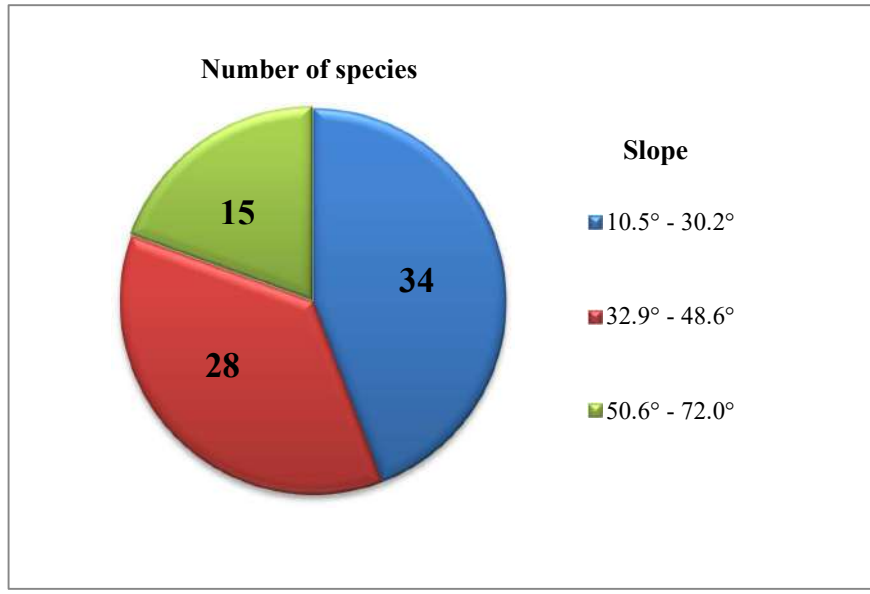

Fig. 9: Distribution of lichen species based on the slope.

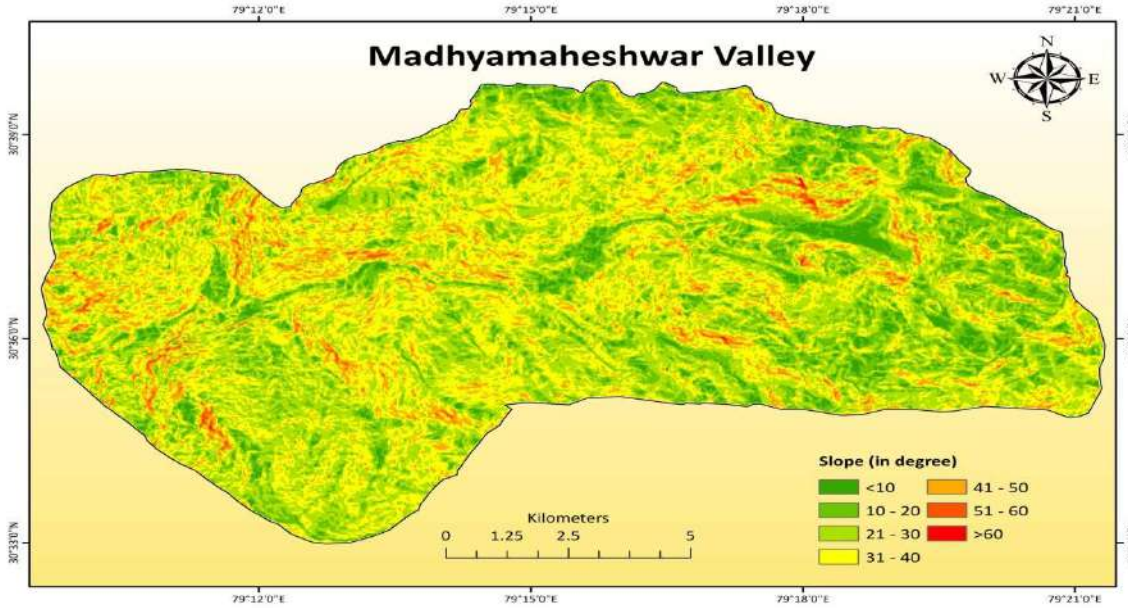

Fig. 10: Slope map (in degrees) of the study area depicting terrain steepness.

Eastern and western aspects supported 10 and 8 species, respectively, whereas intercardinal directions showed varying diversity based on differential sunlight exposure. The study found that southern aspects had the highest lichen diversity, likely because of greater sunlight and warmth. In contrast, the northern, eastern, and western aspects showed lower species richness, which was influenced by temperature, light, and moisture. These results highlight the role of directional exposure in shaping the lichen distribution in the valley.

Altitudinal Variation in Lichen Species and Substrate Preference

Substrate preference shifted with elevation. At lower elevations, foliose lichens dominated the bark, rock, and soil. At mid-elevations, foliose and fruticose forms

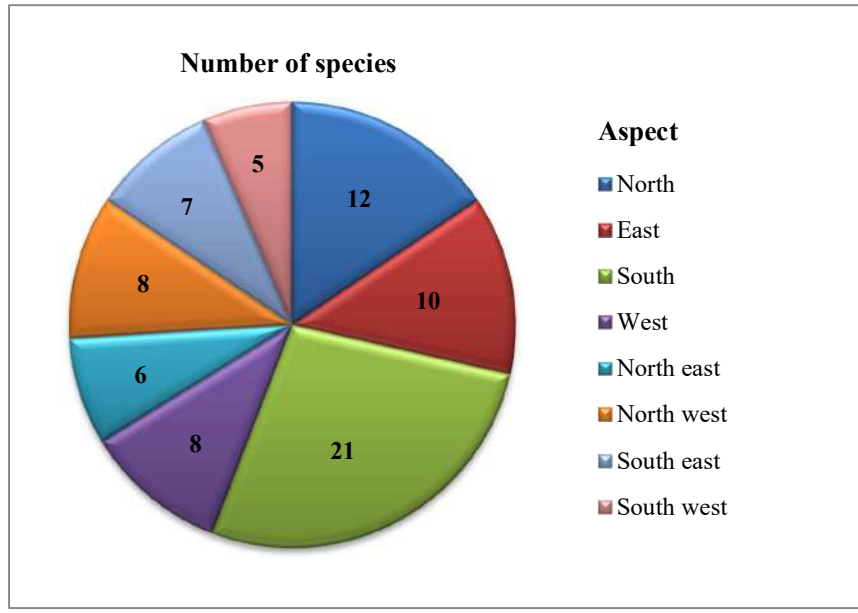

Fig. 11: Distribution of lichen species based on aspects.

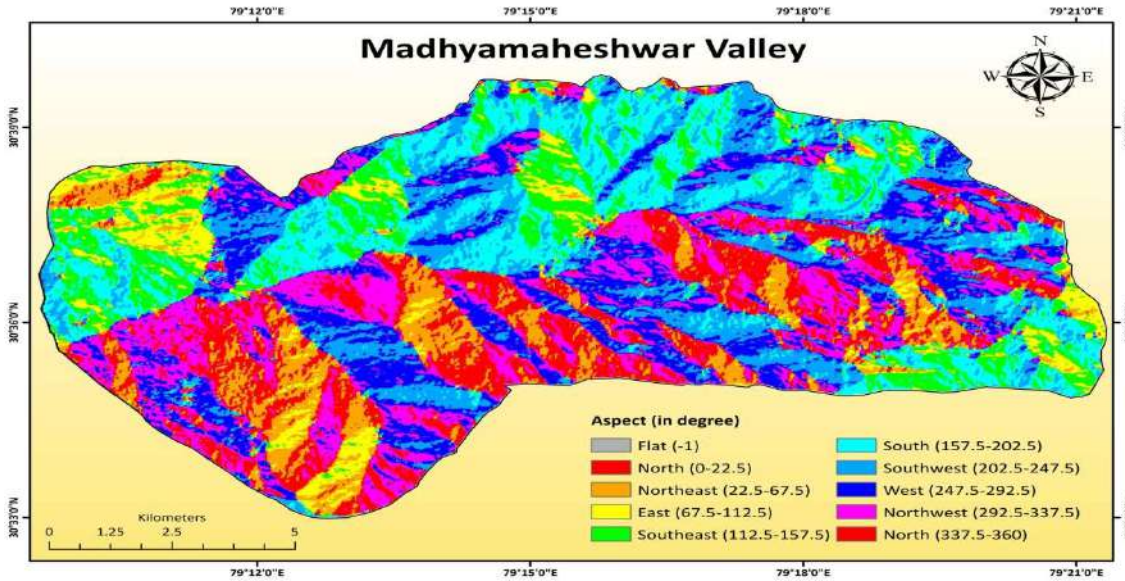

Fig. 12: Aspect map of the study area showing the terrain orientation (cardinal directions).

increased, occupying the bark, mosses, and twigs. At higher elevations, crustose lichens were more prevalent, particularly on rocks and tree trunks, reflecting their adaptation to colder, wind-exposed environments. This indicates that increasing altitude correlates with a shift in both species composition and substrate preference, favoring more specialized and resilient species at higher elevations.

Distribution Patterns of Lichens Across Elevation Zones in Madhyamaheshwar Valley

The distribution of lichen species in the Madhyamaheshwar Valley revealed distinct patterns across three elevation zones (1600-2300 m, 2600-3100 m, and 3200-3600 m ASL). Based on these zones, lichens were categorized by their presence in one, two, or all three zones (Table 2), providing insights into habitat specificity.

Lichens Present in All Three Elevational Zones

Several species occur across all elevation zones (1600-2300 m, 2600-3100 m, and 3200-3600 m ASL), exhibiting varied substrate preferences. Species such as *Dermatocarpon miniatum*, *Flavoparmelia caperata*, *Heterodermia diademata*, *Leptogium delavayi*, *Lobaria retigera*, *Parmotrema reticulatum*, and *Parmotrema tinctorum* were observed in all zones. These lichens occupy substrates, including rocks, bark, and mosses; for example, *Flavoparmelia caperata* and *Parmotrema reticulatum* were found on both bark and rock. Their widespread presence suggests a broad ecological tolerance.

Lichens Present in Two Different Elevational Zones

Lichen distribution indicated the absence of species exclusive to the lower elevation zone (1600–2300 m ASL). In contrast, species such as *Cladonia corniculata*, *Dolichousnea longissima*, and *Leptogium askotense* were found at both mid (2600-3100 m ASL) and high (3200-3600 m ASL) elevations, primarily on bark, mosses, twigs, and rocks. Their distribution reflects a preference for cooler and more stable environments typical of higher altitudes. The use of multiple substrates indicates ecological flexibility.

Lichens Present in Only One Specific Elevational Zone

Certain lichen species exhibit strong elevation-specific patterning. At lower elevations (1600-2300 m ASL), species such as *Caloplaca flavorubescens*, *Chrysothrix candelaris*, *Cladonia fruticulosa*, *Cladonia subulata*, and *Pertusaria velata* occur exclusively. Mid-elevation species included *Cladonia coccifera*, *Cladonia ramulosa*, *Graphis sp. 2*, *Hypotrachyna cirrhata*, and *Lobaria kurokawa*. At higher elevations (3200-3600 m ASL), 41 species were documented, many of which were restricted to this zone, indicating adaptation to harsher conditions. These patterns reflect the niche specialization of lichen species along an altitudinal gradient.

DISCUSSION

The study revealed that 48 lichen species were most abundant at higher elevations in the Madhyamaheshwar Valley (Table 1). This observation aligns with the findings of several

Table 2: Lichen species and their distribution across elevation zones and substrates in Madhyamaheshwar Valley.

S.No.	Name of lichens	Elevation zones (m ASL)			Substrates
		1600 – 2300 m	2600 – 3100 m	3200 – 3600 m	
1	*Aspicilia cinerea*	×	×	√	Rock
2	*Aspicilia dwaliensis*	×	×	√	Rock
3	*Bryoria himalayana*	×	×	√	Bark
4	*Buellia himalayensis*	×	×	√	Bark
5	*Calicium adspersum* subsp. *himalayense*	×	×	√	Bark
6	*Caloplaca flavorubescens*	√	×	×	Bark
7	*Chrysothrix candelaris*	√	×	×	Bark
8	*Cladonia coccifera*	×	√	×	Soil
9	*Cladonia corniculata*	×	√	√	Deadwood, mosses
10	*Cladonia fimbriata*	×	×	√	Bark
11	*Cladonia fruticulosa*	√	×	×	Soil
12	*Cladonia laii*	×	×	√	Mosses
13	*Cladonia pocillum*	×	×	√	Rock
14	*Cladonia pyxidata*	×	×	√	Mosses
15	*Cladonia ramulosa*	×	√	×	Soil
16	*Cladonia squamosa*	×	×	√	Rock
17	*Cladonia subulata*	√	×	×	Soil
18	*Coccocarpia erythroxyli*	×	×	√	Rock
19	*Dermatocarpon miniatum*	√	√	√	Rock
20	*Dermatocarpon vellereum*	×	×	√	Rock
21	*Dolichousnea longissima*	×	√	√	Twigs
22	*Flavoparmelia caperata*	√	√	√	Bark, rock
23	*Graphis cfr. duplicata*	×	×	√	Bark
24	*Graphis furcata*	×	×	√	Bark
25	*Graphis scripta*	×	×	√	Bark
26	*Graphis* sp.1	×	×	√	Bark
27	*Graphis* sp.2	×	√	×	Bark
28	*Graphis* sp.3	×	×	√	Bark
29	*Heterodermia diademata*	√	√	√	Bark, rock
30	*Heterodermia japonica*	×	×	√	Bark
31	*Hypotrachyna cirrhata*	×	√	×	Bark
32	*Hypotrachyna nepalensis*	×	×	√	Bark
33	*Lecanora caesiorubella*	×	×	√	Bark
34	*Lecanora fimbriatula*	×	√	√	Bark
35	*Lecanora interjecta*	×	×	√	Bark
36	*Lecidella carpathica*	×	×	√	Rock
37	*Lecidella elaeochroma*	×	×	√	Bark
38	*Lecidella euphorea*	×	×	√	Bark
39	*Lepra leucosorodes*	×	×	√	Bark
40	*Leptogium askotense*	×	√	√	Bark, mosses

Table Cont....

S.No.	Name of lichens	Elevation zones (m ASL)			Substrates
		1600 – 2300 m	2600 – 3100 m	3200 – 3600 m	
41	*Leptogium burnetiae*	×	√	√	Bark, mosses
42	*Leptogium delavayi*	√	√	√	Bark, mosses
43	*Leptogium trichophorum*	×	√	√	Bark, mosses
44	*Lobaria kurokawae*	×	√	×	Mosses
45	*Lobaria retigera*	√	√	√	Bark, mosses
46	*Loplaca pindarensis*	×	×	√	Rock
47	*Nephromopsis laii*	×	√	√	Bark
48	*Ochrolechia subpallescens*	×	×	√	Bark
49	*Parmelia masonii*	×	×	√	Rock
50	*Parmelinella wallichiana*	×	√	√	Bark, rock
51	*Parmotrema nilgherrensis*	×	√	√	Bark, rock
52	*Parmotrema reticulatum*	√	√	√	Bark, rock
53	*Parmotrema thomsonii*	×	√	√	Bark, Twigs rock
54	*Parmotrema tinctorum*	√	√	√	Bark, rock
55	*Peltigera canina*	×	×	√	Mosses
56	*Peltigera membranaceae*	×	×	√	Mosses
57	*Peltigera polydactylon*	×	√	√	Mosses
58	*Pertusaria composita*	×	×	√	Bark
59	*Pertusaria velata*	√	×	×	Bark
60	*Pleopsidium flavum*	×	×	√	Rock
61	*Polyblastidium microphyllum*	×	√	√	Mosses
62	*Porpidia crustulata*	×	×	√	Rock
63	*Porpidia macrocarpa*	×	×	√	Rock
64	*Ramalina conduplicans*	×	√	√	Bark, twigs
65	*Ramalina himalayensis*	×	×	√	Rock
66	*Ramalina intermedia*	×	√	√	Twigs
67	*Ramalina sinensis*	×	√	√	Twigs
68	*Rhizocarpon geographicum*	×	×	√	Rock
69	*Stereocaulon foliolosum*	×	×	√	Rock
70	*Stereocaulon myriocarpum*	×	×	√	Mosses
71	*Sticta henryana*	×	×	√	Bark
72	*Sulcaria sulcata*	×	√	√	Twigs
73	*Umbilicaria indica*	×	×	√	Rock
74	*Umbilicaria vellea*	×	√	√	Rock
75	*Usnea orientalis*	×	√	√	Twigs
76	*Usnea subfloridana*	×	√	√	Twigs
77	*Xanthoria elegans*	×	×	√	Rock

researchers who have noted greater diversity of lichens in the higher mountain ranges of Europe and Asia (Baniya et al. 2010, Vittoz et al. 2010). Variations in diversity along an elevation gradient are influenced by a combination of evolutionary adaptations and ecological factors (Rahbek 2005). Our study found that lichen abundance was greater at higher elevations, where temperatures ranged from 7.2 to 20.6°C and relative humidity fluctuated between 29%

and 66%. Similarly, an earlier study by Cobanoglu & Sevgi (2009) found that elevation significantly influences the quantity and composition of epiphytic lichen communities. Climatic factors, such as temperature, rainfall, and evaporation, are closely linked to altitude. The highest elevation zone displayed the greatest number of species.

According to the study, gentler slopes supported a higher diversity of lichen species. The findings highlight that lichen diversity is influenced by slope angle, with gentler slopes supporting higher diversity, while steeper slopes host a more specialized, lower diversity of species. As suggested by Lepp (2011), slopes with gentler angles foster higher lichen abundance and diversity because of the influence of water runoff.

Based on the cardinal direction aspect, 21 lichen species were found on the southern aspect, while 12 lichen species were found on the northern aspect, clearly indicating higher species richness on the southern aspect. Similarly, Armstrong & Welch (2007) reported that the conditions on south-facing surfaces are more favorable for the survival of competitive species, whereas only species that are resistant to unfavorable conditions are found on north-facing surfaces.

We observed that the distribution pattern differed according to elevation and substratum, with lichen species dominating tree branches and bark and being less frequent in the soil. Similarly, several authors have emphasized that most lichen species prefer tree bark as their substrate, whereas others demonstrate higher host specificity, favoring specific host trees (Sequeira & Kumar 2008, Shravanakumara et al. 2010).

In our study, we found that some lichen species were present at all three elevation levels. The main reason for this could be that these species are less responsive to the environmental conditions of their habitat. This ability may enable them to survive under various environmental conditions by maintaining their thalli structure. Similarly, Zulkifly et al. (2011) observed that certain species were prevalent in montane forests at all elevations.

CONCLUSIONS

Based on the findings of the current study in the Madhyamaheshwar Valley, lichen diversity increased with elevation, with the highest abundance at higher elevations. This pattern is linked to favorable climatic conditions such as cooler temperatures and stable humidity. Additionally, gentle slopes supported greater lichen diversity, likely due to better water retention, whereas steeper slopes hosted more specialized species. Southern aspects showed higher species richness than northern aspects, highlighting the importance of sunlight and temperature. We also observed that lichen species predominantly favored tree bark as a substrate, with fewer species found in the soil. Some species were found across all elevation zones, suggesting their adaptability to different environmental conditions. Overall, this study emphasizes how elevation, slope, aspect, and substrate type shape lichen diversity in the region. Lichens have emerged as reliable bioindicators of climate change owing to their high sensitivity to environmental perturbations. Our results indicate that spatial variations in lichen diversity across different slopes and altitudes may offer valuable insights into the impact of climatic variables, such as temperature and humidity, on ecosystem dynamics. Given that lichens are among the first organisms to respond to environmental stress, their presence or absence can serve as an early warning system for climate-induced changes. Systematic monitoring of lichen communities, particularly in ecologically sensitive regions such as the Kedarnath Wildlife Sanctuary, could be instrumental in detecting climate-driven shifts and guiding the development of targeted conservation strategies. Additionally, long-term monitoring efforts combined with citizen science initiatives could significantly strengthen our capacity to detect and mitigate the effects of climate change on biodiversity.

Future studies should emphasize the establishment of consistent, long-term monitoring protocols for lichen populations to accurately track changes over time and their responses to climate variability. Additionally, conservation priorities must include the protection and restoration of habitats within crucial elevation ranges, considering the specific microhabitat requirements of vulnerable lichen species. Incorporating lichen-based bioindicators into local climate resilience and adaptation strategies can play a vital role in safeguarding biodiversity and strengthening ecosystem stability in the Garhwal Himalayan region.

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Isolation and Genetic Identification of Lipase Producing Bacteria from Oil-Contaminated Sites

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ABSTRACT

Lipase-producing bacteria can be isolated from various environments, including industrial and agricultural waste, vegetable oils, dairy factories, and oil-contaminated soil. Lipase is the third most important enzyme in biotechnology due to its broad catalytic properties and ability to function in heterogeneous media. The current study aims to isolate, screen, and determine the prevalence of lipase-producing bacteria from various oil-contaminated soils in Basrah province, Iraq. Eleven soil samples and five water samples were collected from various oil-contaminated sites. Identification of lipase-producing bacteria was performed using the *16S rDNA* sequencing technique. A total of fifty-one lipase-producing bacterial isolates were identified. Seven isolates exhibited high efficiency in lipase enzyme production, including A5, A1, A3, J3, A4, A2, and G3, with lipase activity values of 49 $U.mL^{-1}$, 28 $U.mL^{-1}$, 24 $U.mL^{-1}$, 23 $U.mL^{-1}$, 23 $U.mL^{-1}$, 20 $U.mL^{-1}$, and 20 $U.mL^{-1}$, respectively. The isolate A5 was the most promising, exhibiting the highest activity at 49 $U.mL^{-1}$. Based on the sequencing of the *16S rDNA* gene, these seven isolates were identified as *Bacillus subtilis* strain QD517, *Bacillus velezensis* strain Bac104, *Bacillus subtilis* strain PK9, *Enterobacter cloacae* strain YY-2, *Bacillus cereus* strain RB1, *Lysinibacillus xylanilyticus* strain D, and *Brevibacillus borstelensis* strain LDH-b. Seven lipase-producing bacterial isolates were characterized as new bacterial strains, and their sequences were registered in the NCBI GenBank database. The production of large quantities of the lipase enzyme requires optimization of culture conditions using various factors to enable applications in multiple fields.

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INTRODUCTION

Pollution of the environment due to pipeline leakage, spillage, and oil disruption is common and represents a major environmental issue for communities, environmentalists, and governments (Ikuesan et al. 2017, Almansoory et al. 2019).

The ecosystem suffers considerable damage and alterations as a result of oil spills. Hydrocarbon (HC) toxicity to organisms and soil is a significant global concern today. The effects of this pollution can lead to changes in soil physicochemical and microbiological properties, resulting in soil infertility or stunted and delayed plant growth, or both. Additionally, both surface and underground water can become contaminated, posing risks to human health (Abdul-Ameer Ali et al. 2019).

The natural environment can manage minor oil spills, but only over an extended period. Consequently, there are still negative impacts on various components of the environment before it can fully regain its integrity. This has prompted scientists and environmentalists to search for effective methods to restore oil-polluted sites. The most common technologies and strategies for oil containment and removal from water include floating devices, barriers, oil collection devices, collection vessels, absorbent materials, chemical dispersants, surfactants, physical degradation, biological breakdown, and onsite burning. Cleanup efforts typically employ mechanical or physical methods (sorbents, booms, and skimmers),

chemical treatments, and biological techniques (Pete et al. 2021).

Biological techniques for oil spill remediation have generated interest in the microbiological study of oil pollution in the environment. This trend encompasses several areas, including the effects of oil spills on microbial populations, the microbial degradation and biodeterioration of crude oil and petroleum products, bioremediation of oil spills, and the production of useful products by crude oil-degrading microorganisms. Notable products include biosurfactants and enzymes (Auta et al. 2022). The demand for these enzymes is rising across various industries due to their numerous advantages, such as minimal environmental toxicity (Thabet et al. 2023).

Most microorganisms enzymatically attack pollutants, converting them into safe molecules. Biodegradation involves treating petroleum pollutants with aliphatic-degrading microorganisms that possess various enzymes, notably lipases (Aransiola et al. 2022). Lipases are among the most valuable biotechnological enzymes, produced by microorganisms (bacteria and fungi), plants, and animals. However, microbial lipases, especially those from bacteria, are more effective than their plant and animal counterparts (Thabet et al. 2023). These microorganisms are primary sources of lipolytic enzymes, making them relatively low-cost, viable, and efficient at breaking down pollutants in a short time (Abubakar et al. 2024). Additionally, microbial enzymes tend to be more stable than those from plants and animals, and their production is more convenient and safer, enhancing their importance for commercial applications. The oily environments of vegetable oil-processing factories, industrial waste sites, soil contaminated with oil, and diesel-fuel-polluted soil provide suitable habitats for lipase-producing bacteria (Alhebshi et al. 2023). Various bacterial strains isolated from these environments have shown the ability to produce lipase. The most notable genera known for their lipase production include *Acinetobacter, Bacillus, Burkholderia, Pseudomonas, Staphylococcus, Microbacterium, Lactobacillus, Serratia, Aeromonas, Arthrobacter, Stenotrophomonas*, and *Thermosyntropha* (Gururaj et al. 2016, Sethi et al. 2023).

Bacteria generally produce lipase enzymes in small quantities under natural environmental conditions. However, this production can be increased by altering nutritional sources, such as carbon and nitrogen, as well as cultivation conditions like temperature, pH, and inoculum size. These adjustments help identify the most suitable variables for enhancing bacterial growth and enzyme production. While bioremediation typically occurs in an aerobic environment, it can also take place under anaerobic conditions (Abbas et al. 2020, Alyousif et al. 2022). The study aims to investigate the diversity of lipase-producing bacteria in oil-contaminated environments in Basrah province, Iraq, and identify species using molecular tools for potential biotechnological applications.

MATERIALS AND METHODS

Collection of Samples

Eleven soil and five water samples contaminated with hydrocarbons and oil waste were collected from several areas in Basrah Province, Iraq (Table 1 and Fig. 1). The soil samples were taken at a depth of 5–10 cm below the surface. Approximately 100 g of soil was collected using a sterile laboratory spatula and placed in sterile, clean plastic containers. Simultaneously, 500 mL of water

Table 1: The sites and types of collected samples.

Type of samples	No. of Samples	Site of Samples	Samples code	Latitude [N]	Longitude [E]
Soil	2	Basrah Center	A	30˚ 30’ 0 “N	47˚ 47’0”E
			L	30˚ 30’ 0 “N	47˚ 47’0”E
Soil	5	Khor Al-Zubair	B	30˚ 13’ 0 “N	47˚ 50’0”E
	.		C	30˚ 16’ 0 “N	47˚ 46’0”E
	.		F	30˚ 13’ 0 “N	47˚ 41’0”E
	.		G	30˚ 15’ 0 “N	47˚ 42’0”E
	.		M	30˚ 18’ 0 “N	47˚ 42’0”E
Water	4.	Khor Al-Zubair	N	30˚ 13’ 0 “N	47˚ 46’0”E
			O	30˚ 15’ 0 “N	47˚ 46’0”E
			P	30˚ 13’ 0 “N	47˚ 46’0”E
			Q	30˚ 11’ 0 “N	47˚ 50’0”E
Soil	1	Al-Najebia	D	30˚ 35’ 0 “N	47˚ 47’0”E
Water	1		E	30˚ 13’ 0 “N	47˚ 42’0”E
Soil	2	Khums mile	H	30˚ 29’ 0 “N	47˚ 50’0”E
			J	30˚ 31’ 0 “N	47˚ 47’0”E
Soil	1	AL-Rumila	K	30˚ 31’ 0 “N	47˚ 18’0”E

samples were collected in sterile, clean plastic containers. The samples were transported to the laboratory in the Department of Ecology, College of Science, University of Basrah, and stored in a refrigerator at 4°C until analysis.

Isolation of Lipase-Producing Bacteria

One gram of oil-contaminated soil and 1 mL of oil-contaminated water were separately added to tubes containing 9 mL of distilled water and mixed thoroughly using a vortex. A series of serial dilutions was performed, ranging from 10^{-1} to 10^{-6}. Next, 0.1 mL of each dilution was pipetted and spread onto Petri dishes containing Tween 80 agar medium using an L-shaped spreader. The plates were then incubated at 37°C for 24 hours. Bacterial colonies that produced a clear zone around them were recorded as positive (Muhsin et al. 2016).

Screening of Lipase-Producing Bacteria

The positive isolates were purified on nutrient agar medium and activated in 5 mL of nutrient broth for 24 h. at 37°C. Then, 2.5 mL of nutrient broth was added to 100 mL of mineral salt medium (MSM) containing NaCl (0.25 $g.L^{-1}$), peptone (0.2 $g.L^{-1}$), $NH_4H_2PO_4$ (0.1 $g.L^{-1}$), $MgSO_4 \cdot 7H_2O$ (0.04 $g.L^{-1}$), $CaCl_2 \cdot 2H_2O$ (0.04 $g.L^{-1}$), Tween (1-2 drops), and olive oil (2 mL). The mixture was then incubated for 3-5 days at 37°C (Mobarak-Qamsari et al. 2011).

Lipase Enzyme Aassay

Lipase activity was estimated using a mixture of 2 mL of phosphate buffer (pH 10), prepared by dissolving 1 g of phosphate buffer in 100 mL of distilled water, 4 mL of olive oil, 2.5 mL of distilled water, 0.5 mL of $CaCl_2$ solution (2.19 g in 100 mL of distilled water), and 1 mL of diluted lipase enzyme. The mixture was incubated in a shaker at 27 °C for 15 minutes. After incubation, 1 mL of 96% ethanol and 2–3 drops of phenolphthalein (0.5 g in 50 mL of ethanol) were added. The mixture was then titrated with NaOH (0.2 g in 100 mL of distilled water), gradually adding the base until the color changed to light pink. A control sample was prepared without the diluted lipase enzyme, and the reaction was stopped by adding NaOH (Rasmey et al. 2017). The amount of fatty acids liberated in each sample was calculated based on the volume of NaOH used to reach the titration endpoint using the following equation:

$\mu mol\ fatty\ acid.mL^{-1}$ subsample (U) = [(mL NaOH for sample – mL NaOH for blank) × N × 1000] / 5 mL

U = µmol of fatty acid released/mL

N = The normality of the NaOH titrant used

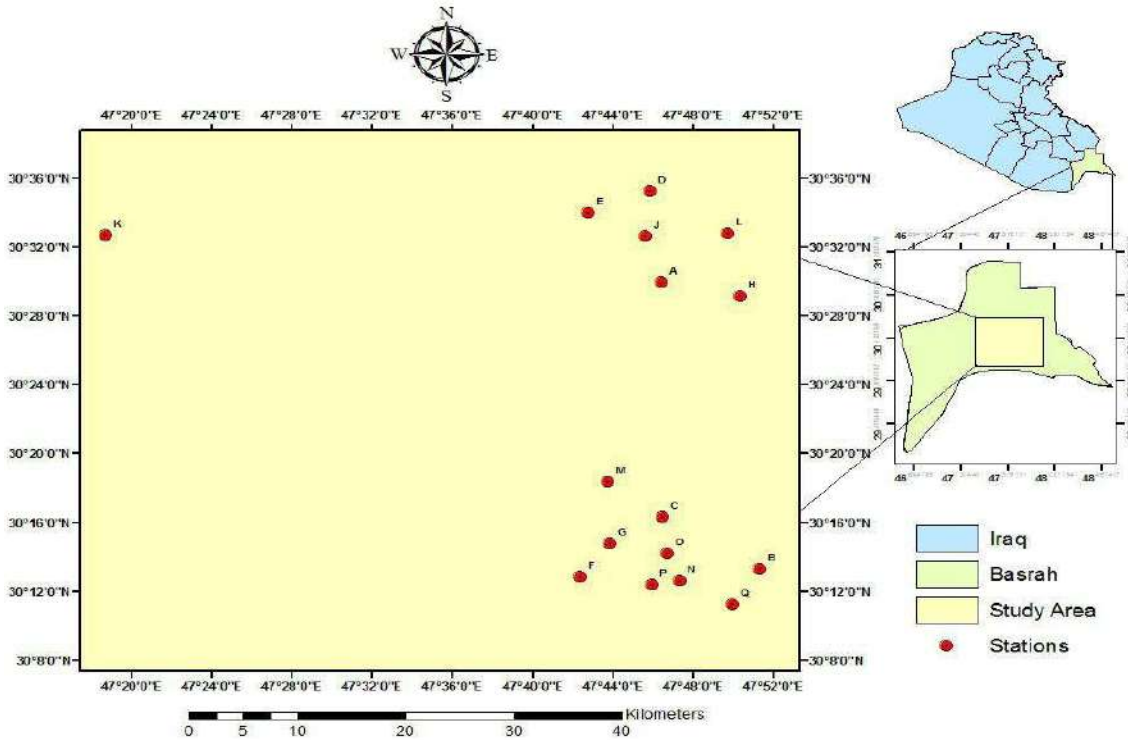

Fig. 1: The map of study areas in Al-Basrah Province, Iraq.

Identification of Lipase-Producing Bacteria by 16S rDNA Gene Sequencing

The isolated lipase-producing bacteria were identified via sequencing and analysis of the *16S rDNA* gene. Bacterial DNA was extracted using the Presto TM Mini gDNA bacteria kit (Geneaid, Taiwan). The *16S rDNA* gene was amplified with universal primers 27F and 1492R (Miyoshi et al. 2005). The PCR mixture was prepared in a total volume of 25 µL, which included 12.5 µL of master mix (Promega, USA), 2 µL of DNA, 1 µL each of forward and reverse primers, and 9.5 µL of nuclease-free water. The PCR program for amplifying the *16S rDNA* gene consisted of an initial denaturation at 96°C for 3 min, followed by 27 cycles of 96°C for 30 seconds, primer annealing at 56°C for 25 seconds, extension at 72°C for 15 seconds, and a final extension at 72°C for 10 min.

Sequencing the PCR Products of the 16S rDNA Gene

The PCR products of the 16S rDNA gene were sent to Macrogen Company in South Korea for purification and sequencing. The obtained 16S rDNA gene sequences were proofread using Chromas and aligned with nucleotide sequence databases from NCBI using BLAST tools (http://www.ncbi.nlm.nih.gov) to identify and assess the sequence homology of the lipase-producing bacterial isolates. A phylogenetic tree was generated using MEGA X, which aligned the sequences using the Clustal W program included in the software (Kumar et al. 2018).

Data Analysis

The lipase assays were done in duplicate the average of the duplicate determinations was used to represent the result of the lipase assay for all bacterial isolates.

RESULTS AND DISCUSSION

Isolation of Lipase-Producing Bacteria

Sixteen samples collected from different sites were used in the present study to isolate lipase-producing bacteria. The results showed that 51 bacterial isolates were able to produce the lipase enzyme. Gram staining indicated that most of the isolated bacterial species were Gram-positive, 42 (82.35%), while 9 (17.65%) were Gram-negative, as shown in Table 2. The occurrence and distribution of lipase-producing bacteria in each sample were reported as 7 bacterial isolates from sample A, 4 isolates from sample B, 8 isolates from sample C, 5 isolates from sample D, 4 isolates from sample E, 1 isolate from sample F, 5 isolates from sample G, 2 isolates from sample H, 3 isolates from sample J, 2 isolates from sample K, 1 isolate from sample L, 3 isolates from sample M, 3 isolates from sample N, 1 isolate from sample O, 2 isolates from sample P, and 1 isolate from sample Q, as listed in (Table 2).

Table 2: Gram's staining characteristics of lipase-producing bacterial isolates.

Samples	Isolates code	Gram's staining	Percentage of Gr+ve	Percentage of Gr-ve
A	A1	Gr+ve	100%	0%
	A2	Gr+ve		
	A3	Gr+ve		
	A4	Gr+ve		
	A5	Gr+ve		
	A6	Gr+ve		
	A7	Gr+ve		
B	B1	Gr+ve	100%	0%
	B2	Gr+ve		
	B3	Gr+ve		
	B4	Gr+ve		
C	C1	Gr-ve	50%	50%
	C2	Gr-ve		
	C3	Gr+ve		
	C4	Gr-ve		
	C5	Gr-ve		
	C6	Gr+ve		
	C7	Gr+ve		
	C8	Gr+ve		
D	D1	Gr+ve	100%	0%
	D2	Gr+ve		
	D3	Gr+ve		
	D4	Gr+ve		
E	E1	Gr+ve	100%	0%
	E2	Gr+ve		
	E3	Gr+ve		
	E4	Gr+ve		
F	F1	Gr+ve	100%	0%
G	G1	Gr+ve	100%	0%
	G2	Gr+ve		
	G3	Gr+ve		
	G4	Gr+ve		
	G5	Gr+ve		
H	H1	Gr+ve	100%	0%
	H2	Gr+ve		
J	J1	Gr-ve	0%	100%
	J2	Gr-ve		
	J3	Gr-ve		
K	K1	Gr+ve	100%	0%
	K2	Gr+ve		
L	L1	Gr+ve	100%	0%
M	M1	Gr+ve	66.66%	33.33%
	M2	Gr+ve		
	M3	Gr-ve		
N	N1	Gr+ve	100%	0%
	N2	Gr+ve		
	N3	Gr+ve		
O	O1	Gr+ve	100%	0%
P	P1	Gr-ve	50%	50%
	P2	Gr+ve		
Q	Q1	Gr+ve	100%	0%

Table 3: Screening of bacterial isolates for the production of lipase enzyme.

Samples	Isolates code	Lipase assay [U.mL^{-1}] *
A	A1	28
	A2	20
	A3	24
	A4	23
	A5	49
	A6	24
	A7	5
B	B1	3.5
	B2	4.5
	B3	6
	B4	4.5
C	C1	13
	C2	17
	C3	7
	C4	9
	C5	7
	C6	11
	C7	12
	C8	18
D	D1	8
	D2	10
	D3	3
	D4	16
	D5	18
E	E1	11
	E2	15
	E3	10
	E4	15
F	F1	17
G	G1	18
	G2	16
	G3	20
	G4	20
	G5	11
H	H1	12
	H2	15
J	J1	12
	J2	12
	J3	23
K	K1	16
	K2	14
L	L1	9
M	M1	11
	M2	8
	M3	9
N	N1	14
	N2	12
	N3	10
O	O1	12
P	P1	10
	P2	7
Q	Q1	10

* Mean ± SD, n=2

Screening of Lipase-Producing Bacteria

Bacterial isolates displayed varying abilities to produce lipase enzymes. The isolates that yielded positive results in the preliminary screening underwent further evaluation to assess their lipase production using mineral salt medium (MSM), with olive oil as the sole carbon source and peptone as the sole nitrogen source (Fig. 2). These isolates demonstrated different lipase activity when tested with phosphate buffer, ranging from 49 U.mL^{-1} to 3 U.mL^{-1}. Seven isolates, A5, A1, A3, J3, A4, A2, and G3, emerged as the most promising lipase enzyme producers, exhibiting the highest lipase activities of 49 U.mL^{-1}, 28 U.mL^{-1}, 24 U.mL^{-1}, 23 U.mL^{-1}, 23 U.mL^{-1}, 20 U.mL^{-1}, and 20 U.mL^{-1}, respectively, out of the 51 bacterial isolates that tested positive for lipase activity. Among these, isolate A5 was identified as the most promising lipase producer with an activity of 49 U.mL^{-1}, while isolate D3 displayed the lowest activity at 3 U.mL^{-1}, as shown in Table 3.

Characterization of Lipase-Producing Bacteria by 16S rDNA Gene Sequencing

The *16S rDNA* gene of all bacterial isolates was amplified using the PCR technique, and the results were visualized by electrophoresis under a UV transilluminator, showing a band size of nearly 1500 bp when compared to the DNA ladder, as illustrated in Fig. 3. The bacterial isolates were identified through *16S rDNA* gene sequencing and analysis. Molecular identification and sequencing of the *16S rDNA* genes were conducted using the NCBI BLAST tool to characterize the lipase-producing bacterial isolates by comparing the obtained sequences with those in the NCBI database. The identification of the lipase-producing bacterial isolates was confirmed through sequencing and analysis of the *16S rDNA* gene.

Most lipase-producing bacterial isolates in the present study belonged to spore-forming, Gram-positive bacilli. Analysis of the 16S rDNA gene sequences indicated that, at the genus level, the isolates belonged to Bacillus (34 isolates), Brevibacillus (3 isolates), and two isolates each of Lysinibacillus, Achromobacter, Stutzerimonas, Enterobacter, and Exiguobacterium. Additionally, one isolate each belonged to Staphylococcus, Cronobacter, Pigmentiphaga, and Acinetobacter. The most prevalent bacterial species were Bacillus subtilis, Bacillus amyloliquefaciens, and Bacillus cereus (Table 4, Fig. 4). A phylogenetic tree was constructed using partial 16S rDNA sequences from the lipase-producing bacterial isolates to determine their evolutionary relationships. The analysis revealed two main groups: one comprising Gram-positive bacteria, predominantly spore-forming species, which were further divided into several

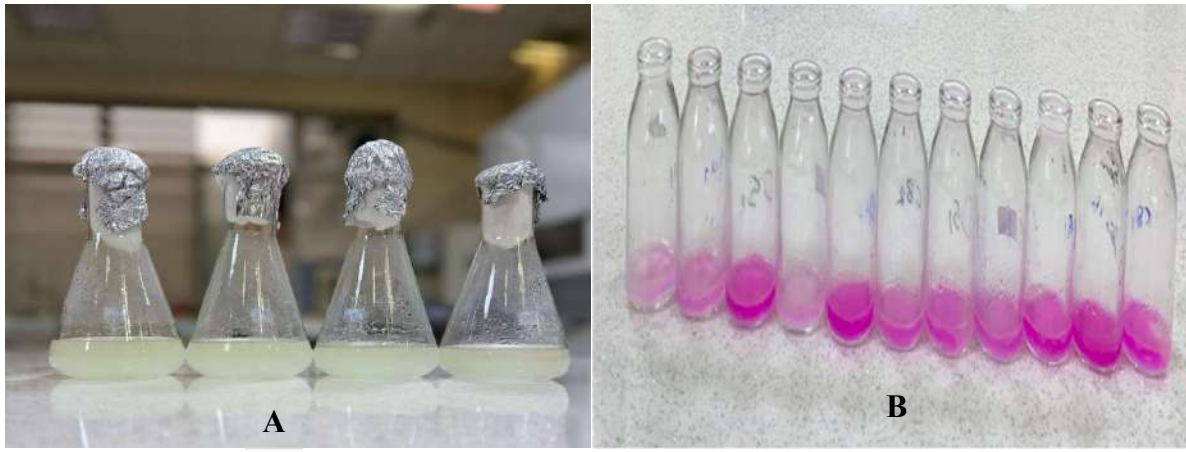

Fig. 2: Screening of bacterial isolates for the production of lipase enzyme, A: flask containing MSM with bacterial isolate**, B:** Lipase enzyme assay by the titration method**.**

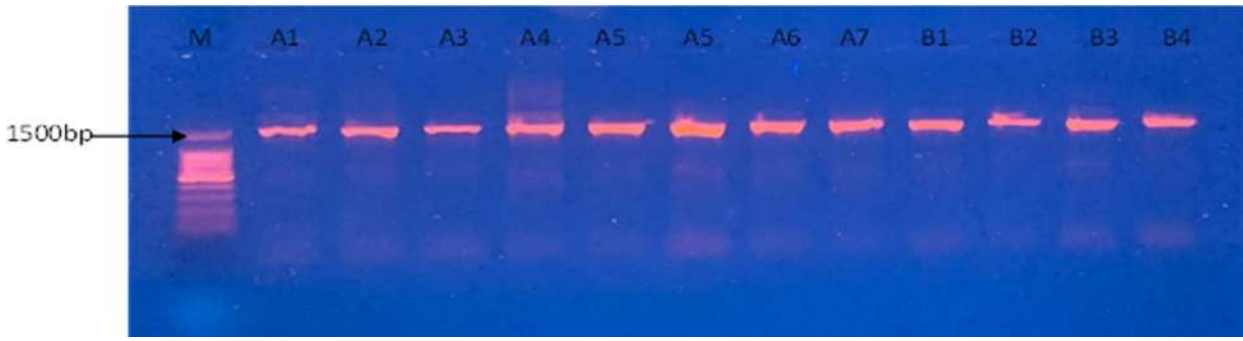

Fig. 3: Agarose gel electrophoresis for PCR products of the 16S rDNA gene (1500 bp) for lipase-producing bacteria. Lane M: DNA ladder, lane A1-B4: Bacterial isolates.

subgroups based on the degree of closeness between the isolates, irrespective of their source, and the other consisting of Gram-negative bacteria, also divided into subgroups reflecting their similarity. These subgroups indicate the phylogenetic relatedness among bacterial isolates, regardless of type or source of isolation (Fig. 5).

Recording of New Lipase-Producing Bacterial Strains

The lipase-producing bacterial isolates in the present study were 100% similar based on *16S rDNA* sequencing, except for seven bacterial isolates, namely B4, C5, D3, D4, J1, J3 and Q were characterized as a new bacterial strain. The sequences of these new bacterial strains have been recorded at the National Center for Biotechnology Information (NCBI) under the accession numbers listed in Table 5. The new lipase-producing bacterial strains that have been recorded include Gram-negative and Gram-positive bacteria.

Discussion

Lipase is the third most widely used enzyme in biotechnology, after protease and amylase, due to its broad catalytic properties and ability to function in heterogeneous media (Yao et al. 2021). This enzyme finds applications across diverse industries, including biodiesel, food and beverages, leather, textiles, detergents, pharmaceuticals, tea processing, and cosmetics. Lipase is characterised by high selectivity, catalytic efficiency, and stability (Remonatto et al. 2022). It exhibits notable resistance to temperature and pH variations and remains highly active in the presence of industrial organic solvents, metal ions, enzyme inhibitors, and commercial detergents (Zhao et al. 2021). While lipases can be isolated from bacterial, fungal, plant, and animal sources, bacterial lipases are generally more efficient and stable (Snellman et al. 2002). Lipase-producing bacteria can be obtained from diverse environments such as industrial and agricultural waste, vegetable oils, dairy factories, and oil-contaminated soils (Bashir et al. 2024). Their ability to

Table 4: Characterization of lipase-producing bacteria by *16S rDNA* gene sequencing, isolate code and similarity percentage.

Isolates code	Closest species	Accession no.	identity %
A1	*Bacillus velezensis* strain Bac104	PQ813838.1	100
A2	*Lysinibacillus xylanilyticus* strain D	KF923754.1	100
A3	*Bacillus subtilis* strain PK9	PQ289147.1	100
A4	*Bacillus cereus* strain RB1	MK418365.1	100
A5	*Bacillus subtilis strain* QD517	EF472261.1	100
A6	*Bacillus amyloliquefaciens* strain IRHB18	OP364585.1	100
A7	*Bacillus subtilis* strain QD517	EF472261.1	100
B1	*Bacillus licheniformis* strain HNXS-W29	PP092139.1	100
B2	*Staphylococcus warneri* strain HBUM06952	MF662224.1	100
B3	*Bacillus amyloliquefaciens*	KU143924.1	100
B4	*Bacillus nakamurai* strain PGPR_corkoak_AJ34	MZ700084.1	99.92
C1	*Cronobacter sakazakii* strain KYU46	GU227669.1	100
C2	*Achromobacter spanius* strain 22STR569	OR831916.1	100
C3	*Bacillus cereus* strain LT419	PQ782563.1	100
C4	*Achromobacter marplatensis* strain KLK37	MT634501.1	100
C5	*Pigmentiphaga daeguensis* strain PT-16	HQ848120.1	98.30
C6	*Lysinibacillus macroides* strain CRDT-EB-7.2	MN192407.1	100
C7	*Bacillus subtilis* strain Bs31	OQ423166.1	100
C8	*Bacillus subtilis* strain SPA N1	JQ308575.1	100
D1	*Bacillus subtilis* strain MA139	DQ415893.3	100
D2	*Bacillus badius* strain M9-20	KT382256.1	100
D3	*Bacillus amyloliquefaciens* subsp. *plantarum* strain Hk8-20	JF899260.1	99.18
D4	*Bacillus velezensis* strain FJ23	OR551414.1	99.93
E1	*Bacillus amyloliquefaciens* strain MX66	OP753626.1	100
E2	*Bacillus amyloliquefaciens* strain Ba09	MT250917.1	100
E3	*Bacillus amyloliquefaciens* strain MX66	OP753626.1	100
E4	*Bacillus encimensis* strain SGD-V-25	KF413433.2	100
F1	*Bacillus amyloliquefaciens* strain MX66	OP753626.1	100
G1	*Bacillus subtilis* strain HY-34	MZ895403.1	100
G2	*Brevibacillus borstelensis* strain UTM105	KF952566.1	100
G3	*Brevibacillus borstelensis* strain LDH-b	MW686864.1	100
G4	*Brevibacillus borstelensis* strain UTM105	KF952566.1	100
G5	*Bacillus subtilis* strain LE24	MG980567.1	100
H1	*Bacillus cereus* strain H23B00114	PQ093572.1	100
H2	*Bacillus subtilis* strain B4	MW776610.1	100
J1	*Stutzerimonas balearica* strain ROD040	OK135687.1	99.93
J2	*Enterobacter cloacae* strain HG-1	MN582993.1	100
J3	*Enterobacter cloacae* strain YY-2	OR378485.1	99.89
K1	*Bacillus oceanisediminis* strain 4S3	KM374766.1	100
K2	*Bacillus subtilis* strain L4	KU179323.1	100
L1	*Bacillus subtilis* strain EPP2 2	JQ308548.1	100
M1	*Exiguobacterium aestuarii*	MW192903.1	100
M2	*Exiguobacterium profundum* strain B09	JX112643.1	100
M3	*Acinetobacter junii* strain BASN28	OQ780942.1	100
N1	*Bacillus tropicus* strain SS9	MK467544.1	100
N2	*Bacillus subtilis* strain ME2	JQ900623.1	100
N3	*Bacillus cereus* strain ABC12	ON631071.1	100
O1	*Bacillus oceanisediminis* strain 4S3	KM374766.1	100
P1	*Stutzerimonas stutzeri* strain XX1	MW820278.1	100
P2	*Bacillus cereus* strain WX2	KF624695.1	100
Q1	*Bacillus albus* strain FA135	MK993477.1	99.81

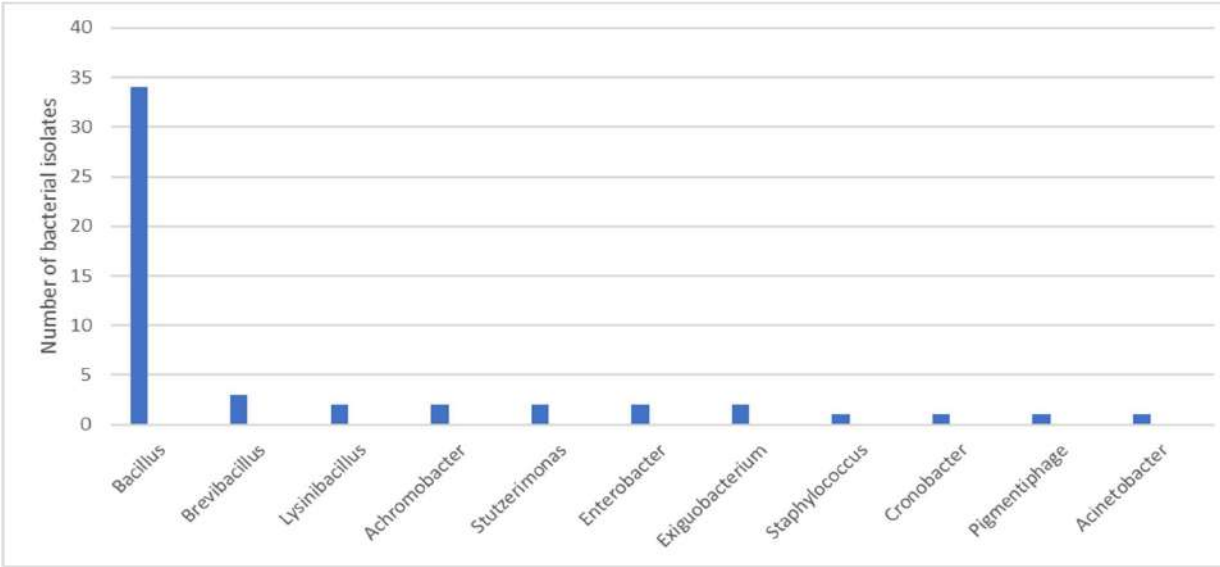

Fig. 4: The number of total lipase-producing bacteria of the respective genera (n=51).

Table 5: The lipase-producing bacterial isolates were recorded as new bacterial strains.

Samples	Isolates code	New bacterial strains	Sequence identity (%)	Accession no. of the new strain
B	B4	*Bacillus nakamurai* strain ZAINABBSRB4	99.92	PV050379.1
C	C5	*Pigmentiphaga daeguensis* strain ZAINABBSRC5	98.30	PV050380.1
D	D3	*Bacillus amyloliquefaciens* strain ZAINABBSRD3	99.18	PV050381.1
	D4	*Bacillus velezensis* strain ZAINABBSRD4	99.93	PV050382.1
J	J1	*Stutzerimonas balearica* strain ZAINABBSRJ1	99.93	PV050383.1
	J3	*Enterobacter cloacae* strain ZAINABBSRJ3	99.89	PV050384.1
Q	Q	*Bacillus albus* strain ZAINABBSRQ	99.81	PV050385.1

produce lipase allows them to thrive in areas contaminated with oil waste (Lee et al. 2015). Most bacterial species isolated in the present study, belonging to the genera *Bacillus*, *Brevibacillus*, *Lysinibacillus*, *Achromobacter*, *Stutzerimonas*, *Enterobacter*, and *Exiguobacterium*, have been previously identified in Basrah province from various environmental samples, including soil, water, sewage, and food (Alyousif 2022, Mohammad & Alyousif 2022, Al Khafaji et al. 2023, Al-Amery & Alyousif 2024). Species of the genus *Bacillus* were prevalent at 14 out of 16 sampling sites due to their ability to withstand harsh conditions, such as high temperatures, aridity, and intense solar radiation. These bacteria form thick spores, which contribute to their resistance (Alyousif 2022).

Bacillus species play essential roles in soil ecosystems, including nutrient recycling, nitrogen stabilization, and enrichment of minerals. They are also significant for industrial applications due to their production of enzymes and other valuable metabolites (Yahya et al. 2021). Lipase activity assays in the current study confirmed that *Bacillus* species have the potential to produce substantial amounts of lipase, consistent with previous reports (Al Mohaini et al. 2022, Oni et al. 2022, Saravanakumar et al. 2024).

Bacillus encimensis, Bacillus badius, Bacillus nakamurai, Bacillus oceanisediminis, and *Pigmentiphaga daeguensis* have not been previously reported for their production of lipase enzymes, nor has their ability to secrete lipase and treat oil pollution been studied. These isolates are being reported for the first time in Iraq. *B. encimensis* was isolated from a marine sediment sample in India (Dastager et al. 2015), *B. nakamurai* from soil (Dunlap et al. 2016), *B. oceanisediminis* from the South Sea in China (Zhang et al. 2010), and *P. daeguensis* from wastewater collected from a dye works in Korea (Yoon et al. 2007).

The current results indicate that several Gram-positive and Gram-negative bacteria, which do not form spores, are effective at secreting lipase enzymes in varying amounts depending on the bacterial species. Examples include *Enterobacter cloacae*, *Staphylococcus warneri*, *Achromobacter* spp., and *Stutzerimonas balearica*. These

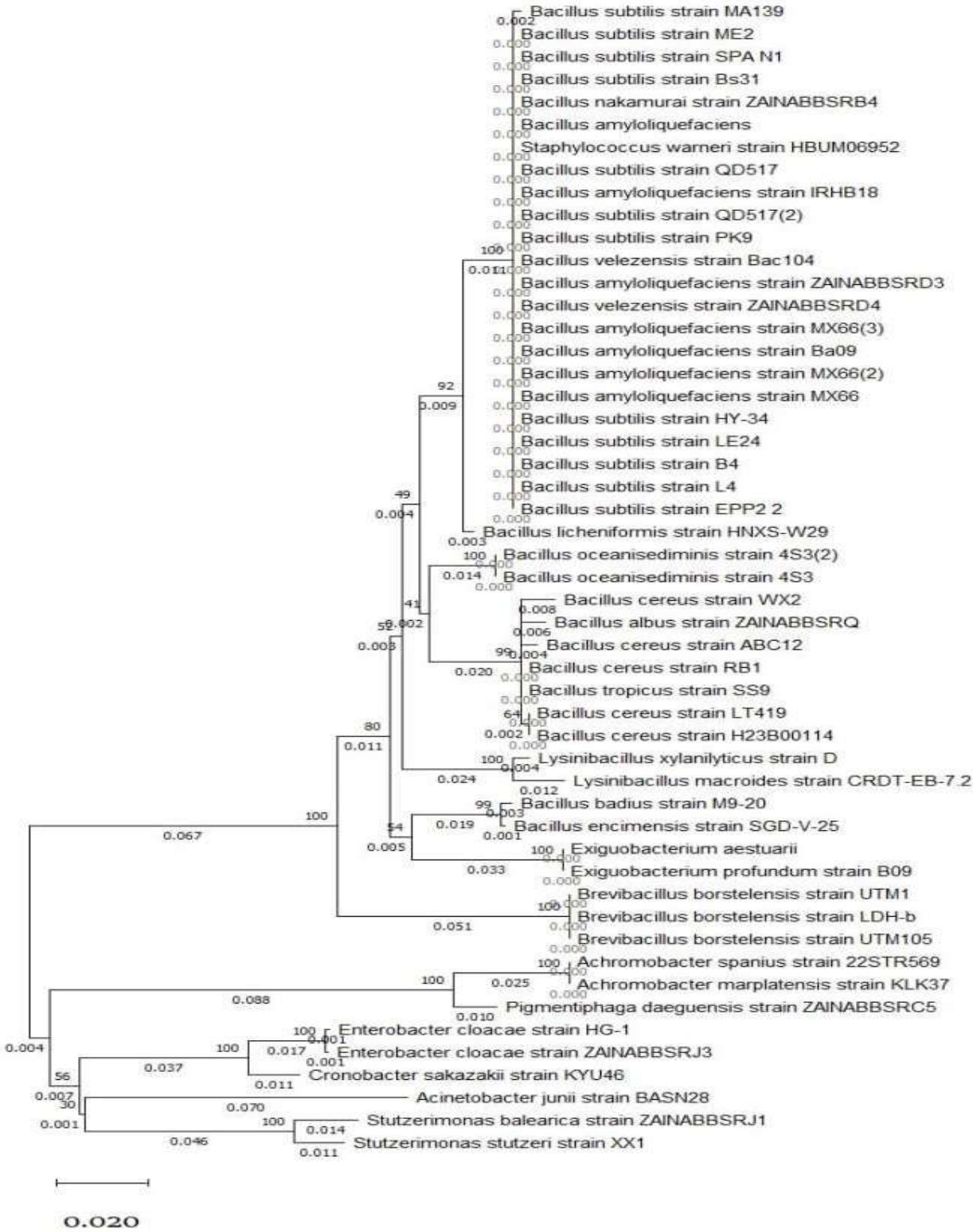

Fig. 5: Neighbor-joining phylogenetic tree illustrating the evolutionary relationships among lipase-producing bacteria.

species have been shown to secrete lipase enzymes that were isolated from various sources, including environmental and food samples (Ali et al. 2015, Yele & Desai 2015, AL-Lami & AL-Kazaz 2016, Ugras & Uzmez 2016, Iboyo et al. 2017, Issa et al. 2023).

Mubarak-Qamsari et al. (2011) studied several lipase-producing bacteria from the wastewater of an oil processing plant and found that maximum lipolytic activity occurred at 45°C and a pH of 7-10. Sagar et al. (2013) isolated lipase-producing bacteria from a waste-contaminated site. Among 18 bacterial isolates, two demonstrated significant lipolytic activity, their optimal conditions were 45°C, pH 8, and incubation times of 18 h at 37°C and 24 h, respectively. Habibolahi & Salehzadeh (2018) isolated and identified lipase-producing bacteria from the soil of oil extraction plants, identifying them as *Pseudomonas* spp. They determined that the optimal conditions for lipase production by this strain included 48 h of incubation at 37°C, a pH of 7, an agitation speed of 150 rpm, peptone as the nitrogen source, and olive oil as the carbon source.

In the current study, we recorded seven new bacterial strains through the sequencing of the *16S rDNA* gene, which have been registered in the NCBI GenBank database under specific accession numbers. The DNA sequences of these bacterial isolates may change due to exposure to mutagens such as chemicals, radiation, and other factors that can cause DNA damage and affect inheritance (Mohammad & Alyousif 2022).

Soil and water contaminated with oil and its derivatives are important sources of bacterial isolates capable of producing lipase enzymes. Lipase-producing bacteria have both biotechnological and ecological significance, making them a focus of research aimed at discovering new and more efficient isolates (Yao et al. 2021). The potential lipase-producing bacteria identified in this study are promising candidates for large-scale industrial applications in the future. Lipase enzymes are used across multiple sectors, including oil processing, detergents, food production, and pharmaceuticals. Environmentally, these enzymes can be applied to treat petroleum-contaminated soil and water, offering an economical and eco-friendly solution. To achieve high lipase yields from these candidate isolates, further optimization of culture conditions is necessary. Consequently, additional experiments on both laboratory and large scales should be conducted to evaluate their practical applications, following the initial screening performed in this study.

CONCLUSIONS

In conclusion, 51 lipase-producing bacterial isolates were obtained from oil contamination sites, all capable of producing lipase enzymes with varying activity levels. Seven isolates demonstrated high efficiency in lipase production, among these, isolate A5 was the most promising, exhibiting the highest activity at 49 $U.mL^{-1}$. Based on the sequencing of the *16S rDNA* gene, these seven isolates were identified as *Bacillus subtilis* strain QD517, *Bacillus velezensis* strain Bac104, *Bacillus subtilis* strain PK9, *Enterobacter cloacae* strain YY-2, *Bacillus cereus* strain RB1, *Lysinibacillus xylanilyticus* strain D, and *Brevibacillus borstelensis* strain LDH-b. The production of large amounts of lipase enzymes requires the optimization of culture conditions, considering various factors for application in diverse fields.

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A Machine Learning-Based Multi-Criteria Decision-Making Approach Utilizing D-Numbers for Water-Energy-Food Nexus Assessment

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ABSTRACT

The interdependency between the water and energy infrastructure represents the core challenge of resource management. Effective decision-making for water-energy-food (WEN) scenarios requires robust tools. Traditional Multi-Criteria Decision-Making (MCDM) approaches are undermined by uncertainty because they assume perfect and complete information, which rarely occurs in Water-Energy Nexus (WEN) issues. Classical models oversimplify the complex interconnections between water and energy systems and therefore result in suboptimal decision-making approaches. Although fuzzy and intuitionistic models are efforts towards uncertainty modelling, they also fall short of fully capturing the dynamics of real-world scenarios. They are inefficient in addressing conflicting and uncertain information, which hinders the practical implementation of these techniques. In addition, the lack of a platform that unites MCDM with integrated uncertainty management increases decision-making complications. To bridge these gaps, the current study proposes a new framework that integrates D-number-based multi-criteria analysis with Dempster-Shafer theory (DST) for WEN decision-making. The integration of DST rigorously enhances the ability of DST to process complete, uncertain, and conflicting information for WEN decision-making. The study also compared the performance of the Random Forest and Optimized Artificial Neural Network models.

INTRODUCTION

The Water-Energy Nexus (WEN) highlights the intimate interdependence and mutual interrelation of water and energy infrastructures. Water is vital for energy generation, from power to fuel extraction, whereas energy is required for water sourcing, treatment, distribution, and wastewater handling. This interrelationship places water and energy as essential pillars of economic development, environmental resilience, and human health. Mitigating the uncertainties in this nexus, much like in supply chain management or sustainable production models, calls for sophisticated decision-making paradigms that transcend conventional practices, fusing fuzzy logic, neutrosophic techniques, and multi-attribute decision-making to improve resource optimization. As the global population increases, industrialization and urbanization fuel the demand for water and energy, and the challenge of resource scarcity grows more intense. Climate change exacerbates the situation by causing shifts in precipitation patterns, lowering the supply of freshwater resources, and raising the demand for cooling and desalination of energy. Historically, separate infrastructures for water and energy have been integrated for efficient and sustainable resource consumption. Policymakers, researchers, and

engineers need to understand the Water-Energy Nexus to develop plans that achieve maximum utilization of resources, minimum environmental footprint, and resilience in water and energy systems. Some sustainable solutions include desalination powered by renewable energy, smart water grids, and energy-efficient wastewater treatment plants. Societies can become water- and energy-secure and minimize climate and environmental risks through an integrated approach (Ding et al. 2020). Water and energy are critical inputs for modern economies. Over the past several years, both sectors have experienced profound reforms, prompted by the imperatives of supply security, sustainability, and economic efficiency. However, their interdependence creates challenges for policymakers (Hussey & Pittock 2012). Over the past decade, the Water-Energy Nexus analysis has drawn increasing attention from the scientific community and policymakers. In a review of the recent scientific literature, 70 relevant studies were identified, and 35 were selected for a detailed case study analysis (Dai et al. 2018). A review of more than 120 studies analyzed their objectives, scope, methodology, and limitations. Additionally, 23 case studies were summarized based on their titles, objectives, and key findings, and 21 case studies were critically analyzed within the context of water-energy nexus research in urban systems (Fayiah et al. 2020). The nexus framework has become increasingly important for analyzing the challenges associated with these vital resources and comprehending the relationships between them (Al-Sumaiti et al. 2020). The most significant contribution of this research is that it offers a valuable knowledge base for successfully managing this nexus. The use of a nexus approach in tourism resource conservation is a necessary step to achieve relative and absolute reductions in resource use (Becken & McLennan 2017). The findings underscore that Ghana's exposure to electricity significantly boosts the share of renewable energy and lowers carbon emissions (Bieber et al., 2018). The results further indicate gaps in the realization of the Sustainable Development Goals. The food-energy-water nexus must be prioritized for sustainable development (Apeh & Nwulu 2024). This study provides a conceptual model for emerging technologies in the WEF sector of new cities. Its primary focal points are internal relations, external drivers, and linkage system evaluation in the WEF nexus (Abdelzaher et al. 2023). This study emphasizes the dual role of water in treatment and processing and its interdependence. The use of integrated and strategic solutions can direct research and development toward addressing local water and energy issues. Enhancing models and data engagement will better serve researchers, policymakers, and the community (Maftouh et al. 2022). This review contrasts the various policy initiatives African nations have embarked on regarding the management of the WEN, their efficiency, and areas. It further locates innovative practices and effective interventions in the African context, with insightful recommendations and local initiatives devoted to simplifying the interface between water and energy resources (Nwokediegwu et al. 2024). Approximate technical outcomes show that hybrid renewable energy plants in North African countries are the most appropriate choice for a regional transmission grid to meet future electricity demand, given the high intermittency of renewable energy sources. Lastly, achieving the aspirations of renewable energy in the region demands meticulous consideration and implementation of diverse policy strategies, particularly in light of North Africa's strong dependence on fossil fuels for national revenue (Adun et al. 2022). Carbon emissions from different energy sources and main scenarios (van Huyssteen et al. 2023). Simultaneously applying the WEF nexus perspective to WEF resource security at the same time has tremendous potential to enhance overall sustainability (Simpson et al. 2023). This article compiles an "ensemble" of water-use indicators across electricity-generation technologies from existing published research to demonstrate the level of detail or lack thereof in the extant large-scale energy-sector water-use data. Using these indicators, this study evaluated the adequacy of such estimates for modeling electricity-production water use at broader scales (Larsen & Drews 2019). Coupled with the coal-led electricity sector in the region, high water demand will remain a long-term constraint (Sun et al. 2018). This study synthesizes the interrelation between energy and water based on two primary considerations: the level of dependence of energy on water and the drivers of this dependence. This study first synthesizes the literature on the water dependence of the energy-by-energy type. It then elaborates on the primary drivers of this relationship. It further summarizes the primary methodologies in the existing literature. Finally, this paper outlines the primary advancements and gaps in the literature (Tan & Zhi 2016). It is essential to synthesize this dependence through analysis and review to design more efficient and sustainable infrastructure systems that optimize resource conservation (Simpson 2023). The study demonstrates that past policy designs have largely been cast in sector-specific mandates (Venghaus & Hake 2018). In the new model, the team members' ratings are presented in the form of D numbers (Bian et al. 2018). In this study, we present a hybrid decision-making support model in the form of D numbers and D, which are employed to acquire input parameters for computing the weight coefficients of the criteria (Božanić et al. 2021). This study expands the ANP methodology within the D-numbers framework to address three types of imprecise information (Chatterjee et al. 2018).

This article introduces a conceptual framework, the "Informational Paradigm," for addressing this issue. This study systematically explored several approaches to these uncertainties, beginning with easier cases that addressed one source and proceeding to more complex cases in which multiple sources interacted (Coppi 2008). This research can be coupled with the D-AHP (D numbers extended AHP) model developed earlier, and it is an integrated solution for MCDM (Deng et al. 2015). Consequently, fuzzy risk assessment has been an active research trend, with increasing interest in its potential to circumvent these drawbacks (Deng & Jiang 2017, 2019). An extension principle is then defined to allow the use of aggregation operators (Li & Chen 2018). To further advance knowledge integration, we developed D-number fuzzy cognitive maps (DFCMs) by integrating D-number theory with fuzzy cognitive maps (Li & Shao 2022). It integrates the strengths of D numbers in handling the weights of criteria (Mousavi-Nasab & Sotoudeh-Anvari 2020). They develop the rough D-TOPSIS approach by integrating D numbers, rough weights, and rough entropy weights, enabling the analysis of uncertain and vague information without additional assumptions (Sarwar 2020). The growing threat of chemical weapons poses a significant global risk. This study proposes a hybrid model of fuzzy VIKOR and an optimal search model to address the urgent need for the identification of illegal chemical warehouses. As such activities are highly clandestine, the issue is beset by multiple layers of uncertainty and ambiguity. Fuzzy VIKOR is applied to rank suspect warehouses according to search time and cost in a fuzzy decision environment (Sotoudeh-Anvari and Sadi-Nezhad 2022). To further test their effectiveness, a case study was conducted by applying the developed D-number method to bridge condition assessment (Xia et al. 2019). This study illustrates that ecosystems are core elements of the WEF nexus. Existing tools are resource interaction-based and largely do not account for dynamic ecosystem functions and services. Aquatic ecosystem models (AEMs) provide a useful framework for quantifying such services, including water purification, carbon sequestration, and biodiversity support. By integrating AEMs into nexus assessments, decision-making can be enhanced with a greater appreciation of how ecosystems enhance resource sustainability. Such integration would provide a more comprehensive management framework for water, energy, and food systems while maintaining ecological integrity (Hülsmann et al. 2019). This study reviews a range of methods. Interdisciplinary methods are used to integrate interrelated variables, graphically represent complex problems, evaluate significant issues and simulate system behavior. Qualitative methods, in particular, can be utilized to delineate the nexus in a specific region, employing direct research methods such as questionnaire surveys and indirect methods such as ontology engineering and integrated maps. With such methods, researchers can better comprehend WEF interdependencies and facilitate more effective decision-making and policy-making (Endo et al. 2015). These categories provide valuable hydrological insights, showing various ways in which water is linked to energy and food systems under varying environmental and socio-economic conditions. Understanding these connections is important for integrated and sustainable resource management (Endo et al. 2017). The application of systematic assessments of water, energy, and food interlinkages or the shaping of socially and politically relevant resource policies is still in its infancy (Albrecht et al. 2018). This study then examines the prospects for integrating nexus thinking more effectively within IWRM frameworks (Benson et al. 2015). This study looks through the lens of water quality to shed light on the potential for agriculture to maximize the system (Bell et al. 2016). Our most important resources are land, energy, and water; however, their utilization is a major driver of climate change. Resource systems are extremely vulnerable to climate change. Therefore, efficient management is crucial for both mitigation and adaptation. Howells et al. (2013). The models should be geographically flexible to enable resource management at the project, watershed, regional, and national levels. The integration of spatial–temporal drivers would create more integrated models, resulting in more effective policies for sustainable development, greater institutional synergies, and greater social welfare (Mabrey & Vittorio 2018). Current nexus thinking is focused primarily on the social-ecological systems paradigm, which complements adaptive responses to global change (De Grenade et al. 2016). This is counterintuitive, considering that livelihood is a foundational pillar for attaining sustainable development (Biggs et al.2015). This paper provides a conceptual overview of significant interlinkages, primarily from a developing country perspective, and analyzes case studies to define promising strategies for Nexus management. It also provides the features of a modeling framework for the nexus and information to guide more effective regulations and policies at the national level (Bazilian et al. 2011). With the world increasingly populated and urbanizing, and with material consumption-based lifestyles, greater focus is being placed on the interlinked system supplying energy, water, and food—the Nexus. Simultaneously, there is growing concern regarding climate and environmental degradation of this system, which is crucial for maintaining critical ecosystem services (Keairns et al. 2016). The research revealed twenty-four ideas on governance that underpin this literature and fall within these categories (Urbinatti et al. 2020).

LITERATURE REVIEW

A review was conducted to assess the situation in the water–energy–food nexus in terms of methodology and how the nexus approach has expanded geographically and academically. This research examines how this approach brings together several discourses, disciplines, and fields using integrated and interdisciplinary methodologies (Endo et al. 2020). This paper provides an overview of 20 case studies featured in this special issue, with an emphasis on the practice and scientific foundations of the Nexus (Taniguchi et al. 2017). The reviewed studies were categorized and evaluated based on their geographic scale and nexus scope (Dai et al. 2018). This study sets out a balanced Nexus framework and applies it to the Mekong Basin. The analysis highlights the strengths of a sectorally balanced, dynamic Nexus approach, particularly its ability to uncover new cross-sectoral relationships or shifts in such relationships as a result of single-sector interventions (Smajgl et al. 2016). Recent literature increasingly demands a shift from "nexus thinking" to "nexus action," with an emphasis on actual implementation. Nexus policies should be implemented across various geographic levels, supported by effective measures and a range of tools developed through research programs and on-the-ground projects. Far from being a one-size-fits-all solution, however, the nexus approach should be seen as a flexible framework that must be adapted to each situation in order to be effective in managing complex resource interdependencies (Simpson & Jewitt 2019). The nexus strategy is likewise concerned with addressing the needs of the most vulnerable people in the world (Leese & Meisch 2015). is increasing awareness of the need to break away from a sector-based framework in science, policy, and practice to an integrated framework that considers the interlinkages between water, food, and energy resources. This realization identifies synergies and trade-offs in managing all three resources (Reinhard et al. 2017). This research examines how nexus discourse is unfolding and being mobilized by various stakeholders in the UK natural resource discourse (Cairns & Krzywoszynska 2016). This study seeks to establish a method for comparing the perceived complexity of nexus tools identified by international organizations and primary literature sources (Dargin et al. 2019). This study investigated the problems and prospects for enhancing water efficiency in thermoelectric power plant cooling systems (Pan et al. 2018). The systems for all three resources are interdependent, and there is a need to create assessment tools that reflect their interdependencies, particularly in assessing the environmental performance of food production systems (Al-Ansari et al. 2015). As a pilot project for the multiple interlinkages among the three elements, this study explored biogas production from energy crops through anaerobic digestion. Similar to all bioenergies, greater production from energy crops raises concerns about potential negative environmental impacts, competition in food markets, and progressive land-use changes (Pacetti et al. 2015). System dynamics models provide a framework for simulating and assessing the system-wide impacts of interventions in a given area (Purwanto et al. 2021). These studies include water resources required to generate various foods and energy, energy per unit of water or agricultural product delivered, and CO_2-equivalent emissions for water and energy delivery (Sadegh et al. 2020). The integrated framework thus developed was then utilized in four case studies in irrigation: food production and energy consumption using water allocation in a Spanish irrigation project; food and bioenergy production and delivery using treated water in Germany; water allocation for food production and urban use in Kenya; and energy production and delivery for food production in Hyderabad, India (Villamayor-Tomas et al. 2015). The interdependence between developments on a global and Brazilian scale is investigated, focusing on how changes in the world and Brazil influence each other and how Brazil influences other countries and the world. Reform in the scientific approach to such issues is needed as a facilitator for strengthening science-policy bridging in sustainability policies (Mercure et al. 2019). This article sets out a generic scenario-based methodology employing the Q-Nexus Model to assess nexus effects to be addressed in WEF planning and policymaking (Karnib 2018). integrating both the production (supply) and demand aspects of WEF systems into one system-of-systems framework (Wu et al. 2021). This study examines the San Antonio Region in Texas, a high-growth resource-intensive region with expanding energy production in the Eagle Ford Shale Play and rising agricultural activity. It summarizes the findings of a survey conducted with 370 researchers and regional stakeholders from the government, non-governmental/non-profit, and business sectors in the region's water, energy, and food industries (Daher et al., 2020). This study applies a participatory modeling process to outline significant interlinkages in the Water-Energy-Food (WEF) nexus of Andalusia as a basis for constructing a system dynamics model. Using fuzzy cognitive mapping, 14 decision-makers were asked to contribute insights to enhance knowledge regarding the WEF nexus and enhance stakeholder awareness and consensus (Martinez et al. 2018). This study explored nascent vulnerabilities in Bwaise and Kanyogoga's informal settlements (Mguni et al. 2020). This technique enables quantifiable measures of stakeholder satisfaction and resource integration in economic and environmental terms. A case study was conducted in a region in Mexico due to industrial activity and challenges in meeting

resource demands with low water levels (Cansino-Loeza & Ponce-Ortega 2021). The principal scientific challenges arise from gaps in data, information, and knowledge in understanding the interlinkages in the WEF nexus. Additionally, it is constrained by a deficiency in systematic tools that can manage all interlinked trade-offs. Increasing the pool of information is a priority for future research (Liu et al. 2017). Classical MCDM approaches are confronted with the issue of uncertainty because they presume complete and perfect information, which is scarcely present in WEN contexts. Furthermore, the absence of a general platform for coupling MCDM with holistic uncertainty management contributes to decision-making complexity. To fill these research gaps, this study develops a novel framework that combines D-number-based Multi-Criteria Analysis and Dempster-Shafer Theory to enhance WEN decision-making.

The major drawback of conventional Multi-Criteria Decision-Making (MCDM) models in Water-Energy Nexus (WEN) decision-making is their presumption of complete and perfect information, which is not a reality in most real-world WEN situations. The Models cannot deal effectively with uncertainty, ambiguity, and conflicting information, and tend to provide oversimplified descriptions of the intricate interdependence among water and energy systems. Consequently, conventional MCDM techniques tend to yield suboptimal or unreliable decisions in the context of WEN. The Dempster-Shafer theory (DST) plays an important role in promoting uncertainty management using a flexible mathematical representation for reasoning in the presence of incomplete, imprecise, and contradictory information. In contrast to the conventional theory of probability, which only permits the assignment of belief to individual events and not to sets of possibilities, DST models uncertainty more effectively. In the Water-Energy Nexus (WEN) decision-making, the use of DST improves the resilience of Multi-Criteria Decision-Making (MCDM) through evidence-based information synthesis using information that may come from various uncertain sources and results in more informed and credible decisions. The integration of a strong uncertainty-handling mechanism in Multi-Criteria Decision-Making (MCDM) for Water-Energy Nexus (WEN) problems is important because WEN systems are dynamic, complex, and data-sparse in nature, usually entailing conflicting goals, incomplete data, and ambivalence among stakeholders. In these settings, traditional MCDM techniques that presume inputs to be deterministic are insufficient. A strong mechanism, such as that founded on Dempster-Shafer Theory or D-numbers, allows decision-makers to capture, represent, or analyze uncertainties in a better way, resulting in more credible, transparent, and adaptive decisions that are a true representation of the real-world complexities involved in the management of interrelated water and energy resources. The performances of the random forest and optimized ANN models were compared. It measures the degree to which the model accounts for the variation in the target variable. Higher R^2 values represent good model performance and predictability. These measures determine the accuracy, precision, and interpretability of the forecasts made by the models in the context of the Water-Energy Nexus (WEN).

Current systems simplify the complex relationships among water and energy systems to a level that leads to suboptimal decision-making approaches. In spite of the fact that fuzzy and intuitionistic models are an improvement of uncertainty representation, they also fall short in fully capturing the dynamic nature of real-life situations. They are deficient in the management of conflicting and uncertain information, which forms a barrier to the application of these approaches in practice. Furthermore, the absence of an existing platform that integrates MCDM and the management of all uncertainties escalates the complexity of decision-making.

Existing MCDM approaches within the WEF environment tend to ignore or inadequately address the uncertainty and complexity of such systems. Our research brings Dempster-Shafer Theory (DST) and MCDM to provide a strengthened approach to dealing with uncertainty and conflicting data, which has been relatively under-addressed in previous studies. While MCDM methods have been traditionally employed in WEF problems, there has been limited research on the integration of machine learning approaches for predictive decision-making. Our research fills this gap by integrating Optimized ANN and Random Forest techniques with MCDM, using a data-driven methodology to improve the accuracy of decisions. Existing studies usually address single elements of WEF systems (e.g., energy policy, water management, and food security) independently. We combine all these dimensions in one integrated decision-making context to enable a more in-depth consideration of the interdependencies in WEF systems.

MATERIALS AND METHODS

Consider a set of m WEN $S = \{S_1, S_2, \ldots, S_m\}$ scenarios and n evaluation criteria $C = \{C_1, C_2, \ldots, C_n\}$. Each scenario was assessed based on these criteria, and uncertain expert judgement was represented using D-number theory.

In D-number theory, a D-number is a generalization of the Dempster-Shafer Theory (DST), which captures incomplete, imprecise, or uncertain data.

Each expert k provided a judgment on the scenario. S_i under criterion C_j as

$$D_k(S_i, C_j) = \{(H, d_k(H), (\Omega, 1 - d_k(H))\} \quad \ldots(1)$$

Where H is the hypothesis

d_k (H) is the belief degree assigned by expert k $0 \leq d_k(H) \leq 1$

□ represents uncertainty.

$1 - d_k$ (H) represents the remaining uncertainty.

For multiple experts, the aggregated D-number for the scenario S_i under criterion C_j is

$$D(S_i, C_j) = \{(H, d(H), (\Omega, 1 - d(H))\} \quad \ldots(2)$$

Where $d(H)$ is computed as: $d(H) = \frac{1}{K}\sum_{k=1}^{K} d_k(H)$

To ensure consistency, the D-number values were normalized across all scenarios.

$$D_{norm}(S_i, C_j) = \frac{D(S_i, C_j)}{\sum_{i=1}^{m} D(S_i, C_j)} \quad \ldots(3)$$

This step removes the scale inconsistencies across the criteria.

Weighted D-Number Aggregation

Each criterion C_j has an assigned importance weight w_j, obtained via an entropy-based or expert-based weighting method:

$$W_j = \frac{-\sum_{i=1}^{m} D_{norm}(S_i, C_j) log D_{norm}(S_i, C_j)}{\sum_{j=1}^{m}(-\sum_{i=1}^{m} D_{norm}(S_i, C_j) log D_{norm}(S_i, C_j))} \quad \ldots(4)$$

Then, the D-number-based performance score is computed for each scenario. S_i

$$D_{score}(S_i) = \sum_{j=1}^{n} W_j . D_{norm}(S_i, C_j) \quad \ldots(5)$$

The scenario with the highest $D_{score}(S_i)$ is the optimal choice.

Scenario Ranking Using D-TOPSIS

To prioritize the scenarios, we integrated D-number theory with TOPSIS as follows:

Compute Positive & Negative Ideal Solution

$$D_j^+ = max_i D_{norm}(S_i, C_j) \quad D_j^- = min_i D_{norm}(S_i, C_j)$$

$$D_i^+ = \sqrt{\sum_{j=1}^{n}(D_{norm}(S_i, C_j) - D_j^+)^2} \quad \ldots(6)$$

$$D_i^- = \sqrt{\sum_{j=1}^{n}(D_{norm}(S_i, C_j) - D_j^-)^2} \quad \ldots(7)$$

Compute Relative Closeness Score

$$C_i = \frac{D_i^-}{D_i^+ + D_i^-} \quad \ldots(8)$$

Rank Scenarios

The scenario with the highest C_i is ranked as the most optimal.

Algorithm

Finds the ideal best and worst solutions.

The Euclidean distance to these solutions is computed.

Calculates the closeness score to rank the scenarios.

Ranking Output: A Higher closeness score indicates a better scenario.

Mathematical Model for D-Number-Based VIKOR

$A = \{A_1, A_2, \ldots, A_m\}$ be the set of alternatives

$C = \{C_1, C_2, \ldots, C_n\}$ be the set of criteria

xi_j be the performance value of the alternative A_i on criterion C_j

w_j be the weight of the criterion C_j, determined using D-number

D-Number representation for uncertain weight

Each criterion weight is expressed as a D Number

$$D(C_j) = (m(C_j), u(C_j)) \quad \ldots(9)$$

Where $m(C_j)$ is the belief mass assigned to the criterion C_j

$u(C_j)$ represent the uncertainty in the weight assignment

The total belief mass satisfies $\sum_{j=1}^{n} m(C_j) + u(C_j) = 1$ …(10)

The final weight of the criterion (C_j) is computed as:

Where uncertainty is evenly distributed across all criteria.

For benefit criteria:

For cost criteria $r_{ij} = \frac{x_{ij} - min_i x_{ij}}{max_i x_{ij} - min_i x_{ij}}, \quad r_{ij} = \frac{max_i x_{ij} - x_{ij}}{max_i x_{ij} - min_i x_{ij}}$ …(11)

Where r_{ij} is the normalised value of the alternative A_i for Criterion C_j

Compute Utility and Regret Measures

Utility Measure (S_i)

$$S_i = \sum_{j=1}^{n} \omega_j . (f_j^* - r_{ij})/(f_j^* - f_j^-) \quad \ldots(12)$$

Regret Measure (R_i):

$$R_i = max_j S_i = \sum_{j=1}^{n} \omega_j . (f_j^* - r_{ij})/(f_j^* - f_j^-) \quad \ldots(13)$$

Where

$f_j^* = max_i r_{ij}$ (best value for criterion C_j, $f_j^- = max_i r_{ij}$ (Worst value for the criterion C_j)

The D VIKOR index is computed.

$$Q_i = v\frac{S_i - S^-}{S^* - S^-} + (1 - v)\frac{R_I - R^-}{R^* - R^-} \quad \ldots(14)$$

Where $S^* = maxS_i, \; S^- = minS_i,$

$R^* = maxR_i, \; R^- = minR_i,$

v is the weight of the strategy (usually set to 0.5 for equal compromise between utility and regret).

Rank the alternatives

Rank alternatives based on Q_i

The best alternative A^* satisfies

Acceptable advantages $Q(A_2) - Q(A_1) \geq 1/(m-1)$

Acceptable stability: The best-ranked alternative should also be top-ranked in either S or R.

Given a D-number set:

$D = \{(A_i, d_i) | i = 1, 2, \ldots . n\}$

Where A_i is a focal d_i element and is the corresponding D-number satisfying $0 \leq d_i \leq 1, \sum_{i=1}^{n} d_i \leq 1$

(The sum can be less than 1, allowing for incomplete information.) The D-number entropy quantifies uncertainty by considering both the distribution and incompleteness. A commonly used entropy measure for D-numbers is

$$H(D) = -\sum_{i=1}^{n} d_i log d_i - (1 - \sum_{i=1}^{n} d_i) \log (1 - \sum_{i=1}^{n} d_i \quad \ldots(15)$$

The first term accounts for the uncertainty in the assigned D-values.

The second term measures the uncertainty due to incomplete information

The transition to data analysis takes place as we transform quantitative data into D-numbers and utilize the same to determine optimal decisions in light of uncertainty. Mathematical expressions form the basis of data analysis as a process, where the Euclidean distance of alternatives from the ideal solution is calculated using real data.

The theoretical model governs the calculation of compromise solutions, whereas data analysis deploys data from the decision matrix to calculate S and R values to determine ranking. The entropy calculation theory provides a formula for calculating consistency. Data analysis implements this formula to calculate the process of making decisions that coincide with the theoretical expectations derived from the input data.

Database

The dataset, sourced from open-access materials (Simpson 2023), is provided in an Excel file comprising four sheets labelled '2019,' '2020,' '2021,' and '2022.' It offers a comprehensive assessment of countries across multiple factors related to water, energy, and food security (WEF). It covers essential indices, such as the WEF Nexus Index Rank and Score, which reflect the overall rating of resource sustainability in a country. The dataset further analyzes water-related factors such as Water Pillar, Water Access, and Water Availability that reflect a nation's ability to supply sufficient and equal water resources. Similarly, energy-related factors measure factors such as the Energy Pillar, Energy Access, Electric Power Consumption (kWh/capita), and Energy Imports to reflect energy sustainability and dependency. Food-related factors integrate essential factors such as the Prevalence of Undernourishment, Malnutrition in children (wasting, stunting, obesity), Yield in cereals, and Value in Food Production to reflect a country's standing in food security. Through integrating these diverse measurements, the dataset provides an overall rating of interlinks between water, energy, and food security. The dataset utilizes various methods of decision-making to rank each alternative in terms of performance. TOPSIS (Technique for Order of Preference by Similarity to Ideal Solution) ranks alternatives in accordance with closeness to the ideal optimum solution, where a greater DTOPSIS value indicates better performance. VIKOR (VIšekriterijumska Optimizacija i Kompromisno Rešenje) takes both optimum and worst-case options into consideration by ranking alternatives in favor of the closest compromise towards an ideal solution, where better performance is indicated by a lower value in DVIKOR. Additionally, the Entropy method ranks each alternative in importance by taking uncertainty into consideration, where greater importance is indicated by a greater value in D Entropy. Utilizing these methods in an integrated manner, the dataset provides an efficient system to perform multi-criteria decision making by ranking alternatives in comparative performance.

RESULTS AND DISCUSSION

The Total WEF Nexus Index holds a mean of 57.45 with a moderate balance between the management of resources within the examined areas. The most negative recorded index is 35.00, pointing toward serious difficulties within particular areas, while the best recorded index of 80.77 identifies areas with more efficient and more sustainable linking of resources together. The index displays moderate diversity with a standard deviation of 9.20, pointing toward serious inequalities between the performance of the WEF nexus within different areas.

Table 1 shows disaggregated WEF Nexus Index figures by its three pillars, water, energy, and food, that reflect big

Table 1: Descriptive Statistics of the Pillar Scores.

	Water Pillar	Energy Pillar	Food Pillar
Mean	58.87	57.27	56.96
Min	28.23	35.07	56.96
Max	81.87	94.34	78.24

differences in resource management and access at the various regions. A mean value of 58.87 and a wide spread from 28.23 to 81.87 under the Water pillar reflect big differences in the governance of water and the availability of resources in different regions. The widespread signifies unevenness in the distribution and efficiency of water use, as it is likely to reflect some of the regions experiencing serious constraints in water security, while some have relatively efficient systems. The Energy pillar is characterized by its mean score at 57.27, with the greatest disparity, varying from 35.07 to 94.34, indicating extreme differences in access to energy, sustainability, and technology integration. This is indicative of the fact that some regions have access to superior, dependable energy infrastructures while others are far behind, potentially because of limitations in infrastructure or inefficient policymaking. In the same manner, the Food pillar, with a mean of 56.96 and ranging from 27.55 to 78.24, evidences big differences in food security, agricultural output, and resource efficiency. This is indicative of mixed adherence to sustainable food systems and supply chain resilience within regions.

Table 2 provides a nuanced assessment of critical indicators under the Water-Energy-Food (WEF) Nexus, which captures significant disparity in the availability and access to critical resources by regions. The indicator for Water Availability (Ind.07), at an average of 1,149 cubic meters, varies widely from 18.1 to 3,240 cubic meters, signaling wide geographic disparity in freshwater availability. The indicator for Energy Access (Ind.08) is at 84.09% average but has a wide range of 7.24% to 100% coverage, evidencing both advances and ongoing disparities in electrification. With the high median, many countries have made great advances toward universal access to energy. For Food Security (Ind.18), the average of 81.72, ranging from 25.70 to 144.00, captures the dispersed nature of food availability and nutritional security.

Fig. 1 shows that the WEF Nexus Index of the Top 10 countries identifies countries that perform best in the integrated management of Food, Water, and Energy resources. The best performers are Iceland, closely followed by Canada and Norway, owing to the robustness of their resources and sustainability practices. The United States, New Zealand, Sweden, and Denmark are the other leading countries that perform well in terms of efficient utilization of resources and safety. Kuwait, Australia, and Brazil are the interesting additions that find place within the top 10, with the diversity of geographical and economic resources for WEF management. The index emphasizes the need for good policies, infrastructure, and governance for the efficient use of resources in different geographical areas.

Table 2: Descriptive Statistics Of The Selected Indicators.

	Water Availability	Energy Access	Food Security
Average	1149 cubic meters per capita	84.09% population with access	81.72
Max	3240 (High water abundance)	100% (Full access)	144.00
Min	18.1 (Severe water stress)	7.24% (Severe energy deficiency)	25.70

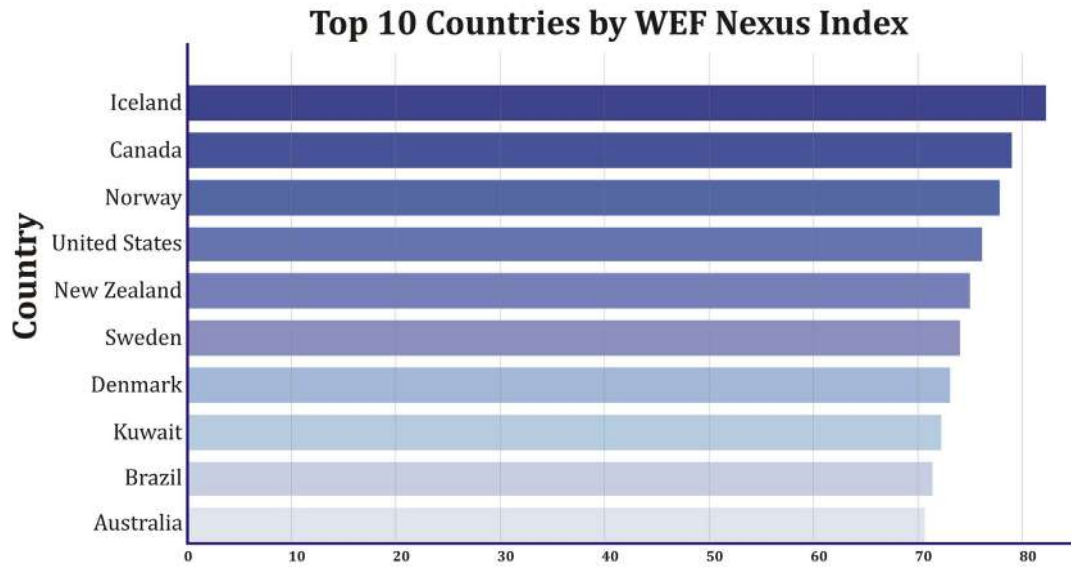

Fig. 1: WEF Nexus index.

Fig. 2 reveals that the WEF Nexus Index identifies the 10 countries with the most serious challenges with Water, Energy, and Food security. The lowest-ranked countries are the Central African Republic, South Sudan, and Chad, with the most serious scarcity of resources and infrastructural shortfalls. Haiti, Yemen, and Niger are the other countries with the poorest access to vital resources, with the added burden of political conflicts and climate vulnerability. Madagascar, Guinea-Bissau, Lebanon, and Mauritania are the countries with the most such bottlenecks that restrict the ability of these countries to manage their resources sustainably. The above ratings emphasize the necessity for the implementation of policy interventions, international support, and the implementation of sustainable development for the mitigation of critical scarcity of resources and enhancing resilience for these countries.

The WEF Nexus Index ranking illustrates the top 10 and the bottom 10 nations by the level of their resource security. The top nations (indicated by blue) have the best water, energy, and food security with excellent infrastructure, effective management of resources, and good policymaking. The bottom nations (indicated by red) are faced with severe resource challenges with shortages of resources, ineffective management of resources, and exposures due to climate change, economic sanctions, or political instability. The clear contrast highlights the need for interventions with specific solutions and the necessity of solutions that are environmentally sustainable for closing the resources gap at the global scale.

Correlation analysis of the Food p.01, Water p.02, and Energy pillars p.03 establishes the crucial interdependency of the pillars. A positive correlation of circa 0.85 between

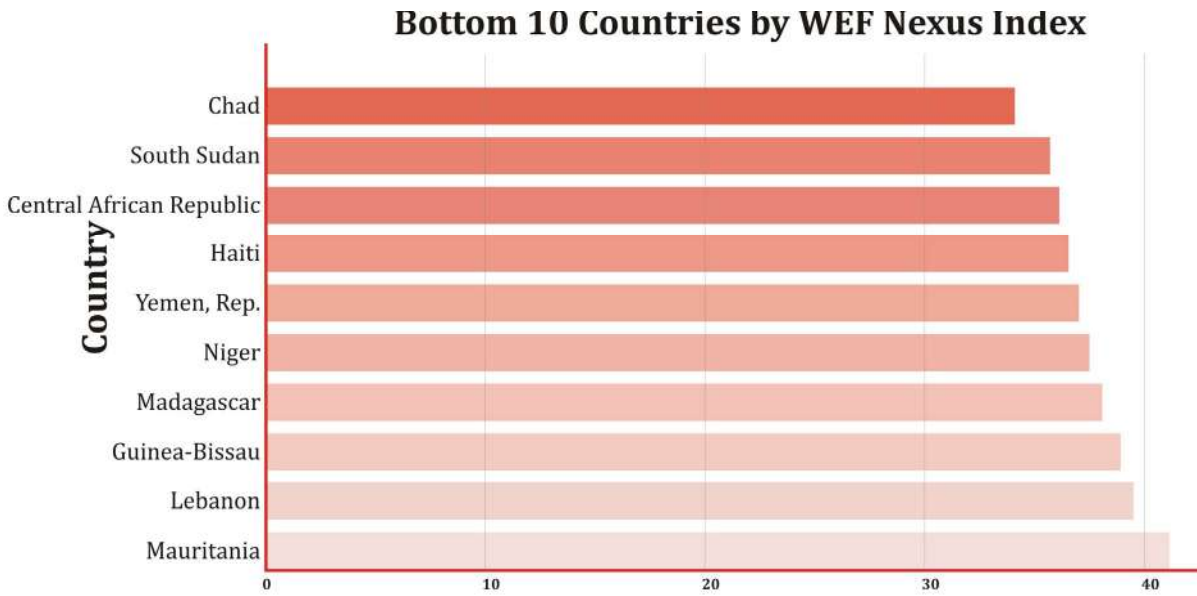

Fig. 2: Bottom countries WEF Nexus index.

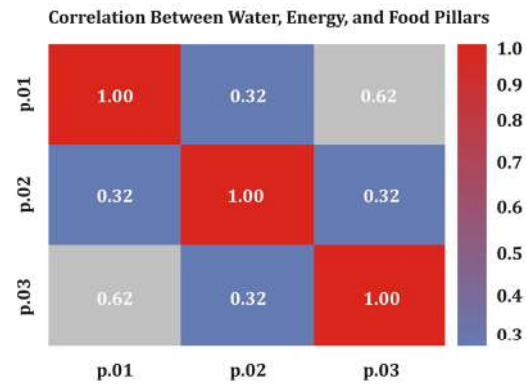

Fig. 3: correlations between water, energy and food pillars.

Food and Energy establishes that countries with good access to energy are likely to be food-secure, validating the crucial contribution of energy to the supply, storage, and production of food. A moderate positive correlation of circa 0.62 between Water and Energy suggests that while the presence of water influences the access to energy, it is just one of the determinants of the latter, since infrastructure and technology are crucial determinants of the latter. The relationship between Food and Water is moderate at circa 0.58 and suggests that the latter influences the former, but with weaker intensity compared with the Food-energy relationship. The above conclusions underscore the intrinsic dependence of the nexus between the three pillars of the nexus and the need for integrated policies for the enhancement of the nexus for enhanced efficiency and sustainability shown in Fig. 3.

The negative declining water-food relationship might be due to improvements in more efficient agriculture practices that conserve water resources or new food production processes that decrease dependence upon water resources.

WEF Nexus Index is a comprehensive indicator of sustainability that encompasses the linkages between the systems of water, energy, and food. To explore the determinants of the WEF Nexus Index, multiple regression analysis shall be utilized with the determinants of the Water Pillar, the Energy Pillar, and the Food Pillar. The analysis shall explore the separate and combined influences of these fundamental dimensions of sustainability on the WEF Nexus Index. The analysis identifies the important predictors and the extent of the effect they have, to provide insight into the processes of resource management and guide the decision for improvement of the state of sustainability. The multiple regression analysis provides an exceptionally good fit, R^2; the value of R^2 is close to one. The analysis of the multicollinearity of the variables is shown in Table 3. The perfect fit is rare and suggests that the relationship between the predictors and the outcome measure is deterministic. All three pillars of Water ($\beta = 0.3333$, $p < 0.001$), Energy ($\beta = 0.3334$, $p < 0.001$), and Food ($\beta = 0.3333$, $p < 0.001$) are highly significant and explain almost proportionally the index. The non-significant intercept of -0.0002, $p = 0.925$, indicates that the model does not require the presence of an additional constant term for the explanation of the relationship. All these findings verify that the WEF Nexus Index measure of sustainability reflects the good balance of the three pillars of water, energy, and food systems, with each pillar's contribution being equally important.

Machine Learning Model Comparison for Predicting WEF Nexus Index

Variance Inflation Factors for the Water, Energy, and Food pillars are all below the widely accepted threshold level of 5, as seen in Table 3. This demonstrates there is no multicollinearity of serious nature among the data, confirming each pillar is bringing in unique and non-redundant information to the model. The absence of multicollinearity increases the statistical accuracy of the regression analysis and upholds the independent contribution of each WEF component to the overall nexus evaluation. This finding attests to the strength of the analytical model, ensuring that the pillars' relationships with each other do not contort or distort the impact of each of them individually.

Table 3: VIF values of the water, energy and food pillars.

Feature	VIF
Water Pillar	1.568
Energy Pillar	1.149
Food Pillar	1.491

Table 4: Model performance of the metrics.

Model	RMSE (Lower is better)	MAE (Lower is better)	R^2 Score (Higher is better)
Random Forest	2.31	1.82	0.90 (Good fit)
ANN	8.57	7.29	-0.33 (Poor fit)

Table 4 illustrates how the Random Forest model far surpasses other predictive models in its performance, capturing 90% of the WEF Nexus Index's variance at an R^2 value of 0.90. This excellent level of explainability implies that Random Forest is well-suited for modeling the intricate, nonlinear relationships among the Food, Energy, and Water pillars. Its ensembled learning architecture—integrating many individual trees—improves resistance to overfitting and enables its ability to take into consideration complex variable relationships that linear methods might miss. In contrast, the Artificial Neural Network (ANN) model failed to converge, resulting in a negative R^2 value of -0.33, which implies that its predictive performance is worse than a simple mean-based model. This may reflect challenges in parameter tuning, data size, or overfitting due to the high sensitivity of ANN to training dynamics and data distribution.

Table 5 and Fig. 4 compare the year-to-year performance of the Optimized Artificial Neural Network (ANN) and Random Forest models from 2019 to 2022 in terms of RMSE, MAE, and R^2 criteria. For all four years, the Optimized ANN outperforms the Random Forest model with better predictive accuracy and consistency. In 2019, the ANN model produced significantly lower RMSE (1.79 vs. 2.31) and MAE (0.99 vs. 1.82), and with higher R^2 (0.942 vs. 0.904), demonstrating its better capability to model nonlinear relationships in the WEF Nexus Index.

Fig. 5 presents the Random Forest and Optimized ANN 2023 WEF Nexus forecast, both of which returned

Table 5: Future trend and prediction analysis.

	Year	Model	RMSE	MAE	R2
0	2019	Random Forest	2.305147	1.818624	0.903668
1	2019	Optimized ANN	1.786738	0.986328	0.942124
2	2020	Random Forest	1.944113	1.527488	0.957258
3	2020	Optimized ANN	1.916585	1.389615	0.958459
4	2021	Random Forest	2.784226	1.969276	0.917200
5	2021	Optimized ANN	2.454844	1.569940	0.935632
6	2022	Random Forest	1.614885	1.259340	0.944308
7	2022	Optimized ANN	0.759705	0.559183	0.987675

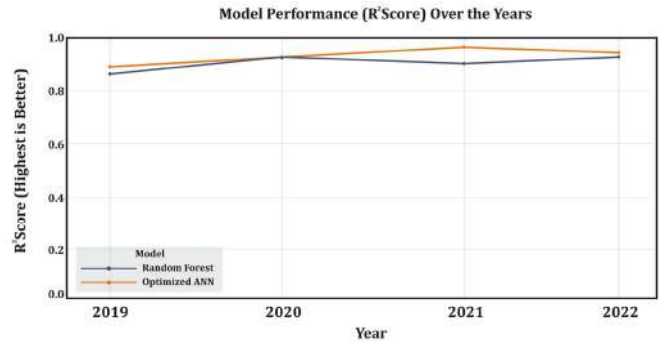

Fig. 4: Model performance.

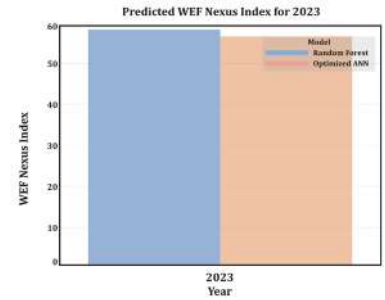

Fig. 5: Predicted WEF Nexus index for 2023.

very similar estimates—59.291 and 59.363, respectively. This near-consistency reflects the strength and overall concordance of both machine learning methods in picking up the underlying data patterns. Yet, the slightly larger forecast from the Optimized ANN aligns with its past trend of increased accuracy as reflected in its lower RMSE and MAE values from past years.

TOPSIS computes the relative closeness of each alternative to the best solution. The greater the DTOPSIS value, the more efficient the performance.

VIKOR: Compares the best and the worst cases. The more the DVIKOR value decreases, the more the alternative approaches the ideal solution.

Entropy: Measures the contribution of each alternative based on its uncertainty. A higher DEntropy score suggests higher significance.

D-TOPSIS Analysis

The most proximate countries with the largest number of D-TOPSIS values are the best performers concerning the criteria under study. The poorest performers are the countries with the most distant D-TOPSIS values. A country that ranks highly under D-TOPSIS and similarly under other approaches is a good decision-making option. High D-TOPSIS are generally Qatar, Switzerland, and Singapore. These are Afghanistan, Chad, and Yemen that are at the tail end of the D-TOPSIS ratings.

D-VIKOR Analysis

The countries with the best D-VIKOR rank are placed at the top since VIKOR favors the "closeness of the ideal solution. The farthest countries from the compromise solution are the countries with the largest D-VIKOR values. Gap between Best and Worst: A narrower gap implies homogeneity, while the wider the gap, the more it implies stark contrasts between countries. Low-scoring countries with low D-VIKOR ratings are Norway, Switzerland, and Sweden. These more highly rated countries by D-VIKOR, such as Sudan and Somalia, rank lower. A few of the countries that perform well under D-TOPSIS may perform differently with slight deviations, identifying the compromise susceptibility of VIKOR.

D-Entropy Analysis

The countries with higher D-Entropy levels are more consistent over different criteria.

Bottom Performers: Countries with lower D-Entropy values indicate higher uncertainty or worse performance on several criteria.Comparison with Alternative Approaches: D-Entropy ratings are likely to be different from TOPSIS and VIKOR since they are more sensitive to the spreading of the information over the criteria rather than performance directly. These include the USA, Canada, and Germany with high D-Entropy scores. The low-scoring nations include conflict zones like Yemen and South Sudan. It could be different from the other methods since weight assignments affect the rank differently.

Table 6 shows that the Countries consistently ranked high (Switzerland, Norway) excel across multiple decision criteria. Countries with fluctuating rankings (Germany, Canada) exhibit sensitivity to criteria weight variations. Countries consistently ranked low (Afghanistan, Sudan, Yemen) show weak performance across all metrics.

Inconsistencies in Multi-Criteria Decision-Making (MCDM) approaches have far-reaching policy-level implications for global development planning. They have the potential to result in conflicting priorities in that various approaches recommend different top-performing countries or strategies, which creates uncertainty or bias in strategic prioritization and the reallocation of resources. Inconsistencies may also erode the credibility of the planning framework and that of the decision-making process among stakeholders. In addition to this, inconsistent rankings underscore the sensitivity of results to the method and weighting schemes used and highlight the importance of transparent, sound methodological and robust approaches to facilitating even-handed and effective global development policy.

Table 6: Comparison of the models.

Method	Best-Ranked Countries	Worst-Ranked Countries
D-TOPSIS	Qatar, Switzerland, Singapore	Afghanistan, Chad, Yemen
D-VIKOR	Norway, Switzerland, Sweden	Sudan, Somalia, Yemen
D-Entropy	USA, Canada, Germany	Yemen, South Sudan, Afghanistan

CONCLUSIONS

In the current study, the performance of the 2019-2022 Optimized Artificial Neural Network (ANN) and Random Forest models was compared for predictability using the performance metrics of Root Mean Square Error (RMSE), Mean Absolute Error (MAE), and R^2. The results indicate that the Optimized ANN model outperformed the Random Forest model with steadily lower RMSE and MAE and higher R^2 levels, with the best improvement performance recorded for 2022. The 2023 forecast indicates a tight correspondence between the models, with the Optimized ANN model maintaining the slightest margin of precision over the Random Forest model.

In turn, multi-criteria decision-making techniques are implemented with the assistance of D-TOPSIS, D-VIKOR, and D-Entropy techniques for country ratings with varying performance criteria. According to D-TOPSIS, the best-performing countries are Qatar, Switzerland, and Singapore, while the poorest-performing countries are Afghanistan, Chad, and Yemen. D-VIKOR highlights the compromise sensitivity variations, while D-Entropy displays the extent of the degree of consistency over multiple criteria, with the USA, Canada, and Germany at the higher ranks, while Yemen and South Sudan are at the lower ranks. The inconsistencies of these techniques highlight the effect of weightage and the difference in the techniques upon the resultant ranks. It determines that the Optimized ANN model is a more accurate and consistent decision-making prediction model compared to the traditional model, while the proper selection of the MCDM techniques strongly affects the rank consistency. The conclusions are of great significance for the enhancement of forecast precision and comparative performance analysis between different study areas.

In summary, the findings validate the performance of the Optimized ANN model for prediction analysis and the impact of different MCDM techniques on rank consistency. The findings are the basis for decision-making for country forecasts and comparative country performance analysis. The varying water-energy relationship implies the effect of new policy developments or new technologies that affect

the production of energy and the consumption of water. The moderate increase of the energy-food relationship might be due to increased reliance upon more energy-demanding processes of food production, with the growing importance of the use of energy within new agriculture and food processing systems. The fusion of Machine Learning (ML) and Multi-Criteria Decision-Making (MCDM) approaches has importance far beyond the given setting of the Water-Energy-Food (WEF) nexus. ML models can learn from large data volumes and dynamically update weights or preferences in MCDM approaches to enable better, context-specific, and adaptive decisions. The conventional MCDM approaches have difficulty processing imprecise, fuzzy, or incomplete data. ML methods—particularly probabilistic models and fuzzy methods—can manage such data very well, facilitating MCDM.By using ML, MCDM processes have been made automated and scalable to process thousands of alternatives and criteria. In the future, to create dynamic, time-varying ML-MCDM processes that adapt inputs, preferences, and weights every time they are provided with fresh data. Incorporate climate projections and models (e.g., IPCC scenarios) into ML-MCDM pipelines to evaluate long-term WEF sustainability and resilience. Create real-time WEF dashboards using streaming data (IoT, satellites) and ML to provide ongoing optimization and alarms. Apply geospatial ML algorithms (e.g., GIS + ML) to adapt WEF decisions to fit typical regional characteristics and expositions. Use ML to identify shifting stakeholder preferences from participatory sites, social media, or feedback. Model WEF systems as multi-agent ecosystems where various sectors (e.g., agriculture, energy, water authority) interact and compete.

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Performance Assessment of E-Waste Plastic as a Sustainable Natural Aggregate Substitute in Traditional Concrete: A Comprehensive Review

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ABSTRACT

Globally, e-waste plastic recycling has emerged as a more popular and creative way to manage electronic waste, which is also being accepted since this resource is available in enormous amounts, comprises many kinds of hazardous components, and possesses a very low recycling rate. Growing urbanization, industrialization, and economic expansion are driving global concrete production, causing pollution and depleting natural resources. Using e-waste plastic as a natural aggregate presents a novel method for conserving resources and addressing the challenges of electronic waste, plastic, and concrete production. This article discusses different e-waste plastic types, techniques for producing e-plastic aggregates, and their application in traditional concrete. Additionally, this study examined the behavior of e-waste plastic aggregates, which affect various concrete characteristics. These include fresh properties such as workability, as well as hardened characteristics such as density (both fresh and dried), splitting tension strength, flexural strength, compressive strength, and durability aspects such as chloride attack and thermal resistance. Reusing electronic waste plastic as aggregates is also a new hope for protecting the environment and guaranteeing the secure disposal of the enormous amount of e-plastic waste generated. However, additional research is needed to address e-waste disposal challenges and its use in conventional concrete.

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INTRODUCTION

Advances in science and technology have driven the rapid expansion of the electronics and electrical systems sector, now the fastest-developing sector across the globe (Ilankoon et al. 2018, Wang & Xu 2014, Yadav & Upadhyay 2015). These sectors have changed how people live and influenced communication, healthcare, and defense in a way that has resulted in notable advancements over time (Danish et al. 2023, Hamsavathi et al. 2020, Ullah et al. 2022, Wang & Xu 2014, Wath et al. 2011). Technology companies constantly introduce new, eye-catching products to dominate the stable market. For instance, in 2019, the central processing unit's (CPU) life expectancy in personal computers (PCs) dropped from 6 to 4 years and from 3 to 2 years in 2005, respectively, while the lifespan of cell phones is less than 2 years (Akram et al. 2019, Babu et al. 2007, Ilankoon et al. 2018, Islam et al. 2020, Kang & Schoenung 2005, Needhidasan et al. 2014, Shamim 2015, Tipre et al. 2021, Xu et al. 2012). Modern society relies on creatively and newly designed electrical and electronic systems. Additionally, its effects reduce the price of electrical and electronic products and assist many people in developing and impoverished nations in improving their standard of living (Babu et al. 2007, Shamim, 2015). Increased demand from people utilizing new products more frequently causes massive production of electrical and electronic equipment, which further shortens device lifespans and generates an extensive amount of e-waste (Babu et al. 2007, Yong et al. 2019). E-waste, often recognized

as "E-waste," is a shorthand form of unwanted electrical and electronic devices, including copper, glass, steel, plastic, and other components, as well as electronic devices that present difficulties in recycling (Luhar & Luhar 2019). Specifically, electronic waste (e-waste) comprises items such as PCBs, televisions, DVD players, refrigerators, freezers, cell phones, MP3 players, and other electronic devices that are discarded after a relatively short period of use (Fadaei 2022, Wath et al. 2011). The main categories of e-waste are illustrated in Fig. 1. The European Union (EU) states that electronic waste is escalating annually at a rate of between 3% and 5% (Akram et al. 2019, Babu et al. 2007, Gaidajis et al. 2010, Gupta 2023, Ilankoon et al. 2018, Liu et al. 2023, Tipre et al. 2021, Tuncuk et al. 2012, Van Yken et al. 2021). E-waste is composed of 1000 diverse kinds of materials, both toxic and non-toxic, all of which pollute the environment. If toxic materials such as mercury, arsenic, cadmium, and lead are not properly managed, they will lead to health issues. Nontoxic resources such as platinum, gold, copper, and silver are recycled (Brindhadevi et al. 2023, Kurup & Kumar 2017a, Needhidasan et al. 2014, Wath et al. 2011). In 2019, only 17.4% of the total 53.6 million metric tons of e-waste produced worldwide was recycled appropriately. The balance of 82.6 percent was not officially reported or recycled. In 2019, 50 million metric tons of electronic waste were created in Asia, America, and Europe, as opposed to 0.7 and 2.9 million metric tons in Oceania and Asia, respectively. Globally, e-waste is predicted to attain 74.7 million metric tons by the year 2030 and 110 million metric tons by the year 2050 (Baldé et al. 2022, Elgarahy et al. 2024, Liu et al. 2023, Rajesh et al. 2022, Shahabuddin et al. 2023, Van Yken et al. 2021). Forecasts of global e-waste generation around the world are typically shown in Fig. 2. The total volume of electrical and electronic equipment (WEEE) constitutes approximately 8% of all municipal solid waste (Fadaei 2022). Plastic is one of the most essential components of e-waste. Tackling e-plastic waste management is the most significant challenge worldwide. The presence of flame retardants significantly hinders the recycling of these materials, despite several technological advancements (Danish et al. 2023, Hamsavathi et al. 2020, Sahajwalla & Gaikwad 2018). Many researchers have employed diverse approaches to manage e-waste plastics, one of which is their use of electronic waste plastics in the construction industry. Electronic waste, such as printed circuit boards, as illustrated in Fig. 3, recovered acrylonitrile butadiene styrene, high-impact polystyrene wastes, and other varieties of electronic waste can be utilized to create sustainable concrete (Kurup & Kumar 2017b). Concrete is the second most widely utilized construction material globally, after water (Nilimaa 2023). Conventional concrete is widely utilized because of its excellent mechanical properties, such as high compressive strength, long-lasting durability, and capability to be molded into the desired shape during casting (Kumar et al. 2025). The increasing need for infrastructure development is evident in the increasing amount of concrete produced daily. The rising requirement for concrete and its associated effects have led to the possibility that the exploitation of natural aggregates is depleting natural resources globally, hence endangering the needs of future generations. Aggregate, which constitutes more than 70% of the material's volume, is one of the most crucial ingredients used in concrete. Focusing on the conservation of natural materials is vital for minimizing the impacts of resource depletion and climate change (Padmanaban et al. 2020, Ullah et al. 2021). To reduce the use of natural aggregates as much as possible, many researchers are attempting to replace them, either entirely or partially, with waste materials such as e-waste, recycled natural aggregates, granite, marble, regular plastic, refractory bricks, and ceramic tiles. Nevertheless, recycled natural aggregates, granite, marble, regular plastic, refractory bricks, ceramic tiles, and other waste materials cannot be produced in sufficient quantities to meet the requirements of the growing building sector. In this case, the abundant generation of e-waste will be directed toward the substitution of natural aggregates to encourage the use of green concrete and preserve natural resources. After analyzing several research articles, we found that nearly all researchers have worked on e-waste recycling, management, usage in concrete, and the consequences for humans and the environment. Little research has been conducted to enhance the strength of concrete composites composed of e-plastic waste. The major aim of this detailed analysis is to scrutinize the behavior of e-waste plastic as a sustainable substitute for natural aggregates in traditional concrete, in addition to the challenges of using electronic waste plastic in lieu of natural aggregates. Additionally, it provides an in-depth exploration of issues related to e-waste plastics, such as recycling, toxic materials, environmental harm, and health concerns.

MATERIALS AND METHODS

The growing requirement for concrete and its byproducts, combined with the mounting problem of electrical waste, has prompted investigations into the viability of replacing natural aggregates in concrete with e-waste materials. This study primarily examines the behavior of e-plastic waste as a traditional aggregate in regular concrete and its effect on the mechanical and durability characteristics of the material. The most innovative research articles on e-waste plastics were considered in this review. A variety of sources, including Google Scholar, Science Direct, Springer, Taylor

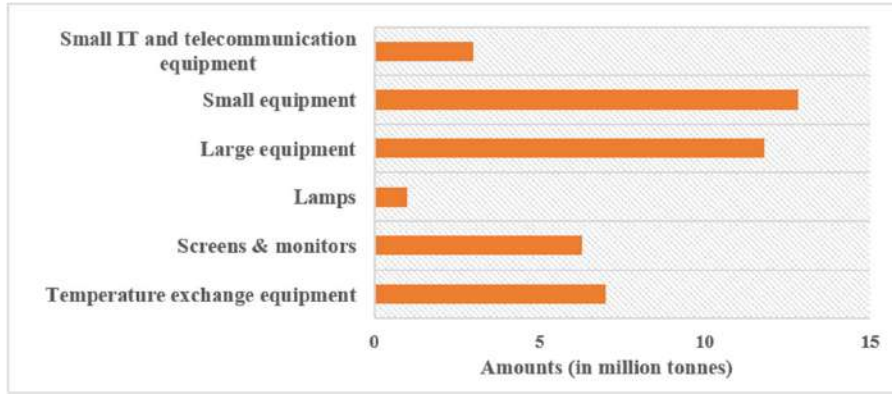

Fig. 1: Classification of e-waste (Kumar et al. 2017).

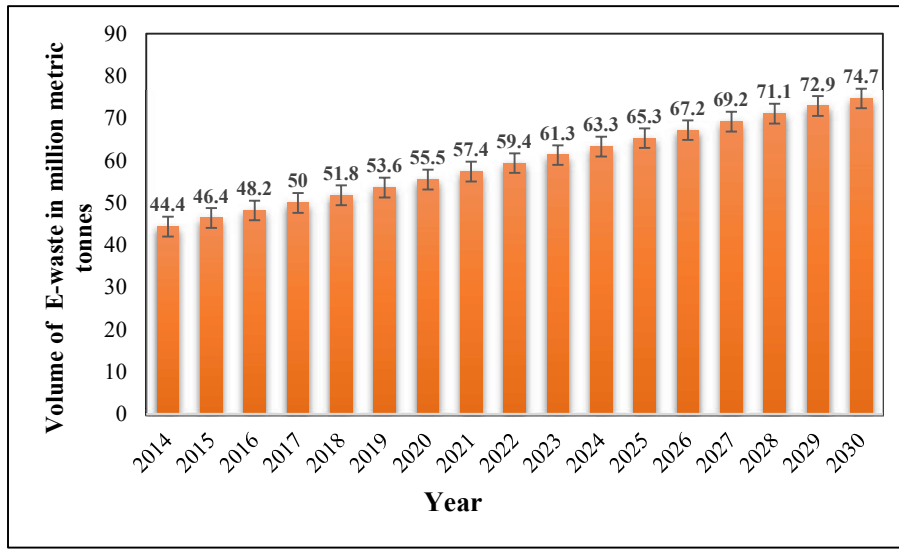

Fig. 2: E-waste production from 2014 to 2030 (Forti et al. 2020).

Fig. 3: Raw printed circuit boards (Gupta & Singh 2021).

& Francis, and other notable works that were accessible as open resources, were used to conduct the literature survey. Specialized keywords such as "electronic waste," "e-plastic aggregate," "e-waste plastic," and "e-waste aggregate" are typically used for reviews. The information was gathered such that the work reports included the major discoveries of the previous few years, including the formation technique, recycling procedure, and usage of rejected plastic as both fine and coarse aggregates in conventional concrete. A comprehensive review was conducted, drawing on a range

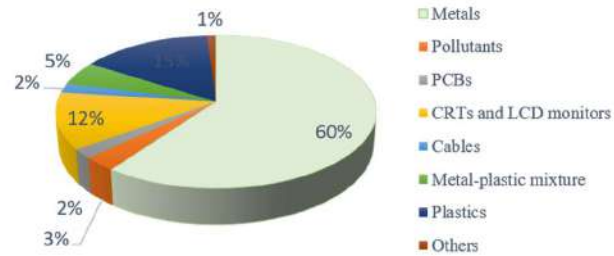

Fig. 4: Composition of e-waste (Chakraborty et al. 2022, Mtibe et al. 2023, Tipre et al. 2021).

Table 1: Environmental impacts of e-waste (Luhar & Luhar 2019, Manjunath 2016).

Component category of E-waste	Procedure used for	Possible Effects on Groundwater, Soils, Health, and Environmental Hazards
Computers, TVs with CRT screens, monitors, ATMs, cameras, and so forth.	Removal and disposal of items following a breakup	The leaching of heavy metals like lead, barium, and others introduces toxic phosphorus into underground water.
PCBs are thin, plate-like structures and a few e-constituents for mechanical support and electrical connection.	Recovering the best metal items requires eliminating soldering, outdoor fire, and an acid bath.	Gas leaks, air, surface, and subsurface water pollution, and glass dust
Chips and a couple of au-plated components.	A chemical stripping procedure using HNO_3 and HCl, along with setting the chips on fire	Fish and plants are acidified when hydrocarbons, dense metals and materials exposed to bromination, such as dioxin, seep directly into surface and subterranean waters.
Computer cords	Cu is extracted by chemical and fire stripping outside.	Discharge of hydrocarbon ash into the atmosphere, soils, and water vapor.

Table 2: Contaminates in e-waste (Akram et al. 2019, Gaidajis et al. 2010, Rao et al. 2017).

Elements	Occurrence in electronic waste	Associated health and environmental factors
Polychlorinated biphenyls	Old-fashioned light fluorescent ballasts with transformers, condensers, and capacitors.	It causes cancer, which affects the endocrine, neurological, immunological, and reproductive systems of humans.
Chlorofluorocarbon	Insulation foam and refrigerants are used.	Combustion of halogenated compounds may produce dangerous fumes.
Arsenic	They are present in trace amounts as gallium arsenide in light-emitting diodes.	Long-term exposure to it is extremely harmful to health.
Polyvinyl chloride	Cable insulation	Processes using high temperatures and wires that convert chlorine into dioxins and furans may release
Barium	Computer screens and plasma displays.	Moisture can cause combustible gases, such as hydrogen.
Beryllium	Exist in wires, power supply boxes, including silicon-controlled rectifiers, and heat sinks for computer chips.	harmful if consumed
Cadmium	Present in plastics, printer ink, and cell phones	It is extremely harmful and can ruin your health over time.
Chromium VI	Floppy disks and data tapes	Highly poisonous and detrimental to health over an extended period. Additionally, it causes allergic reactions.
Lithium	Present in Li-batteries	Capable of releasing explosive hydrogen gases when wet.
Mercury	Useful for mercury-wetted switches and fluorescent lights.	Highly toxic and detrimental to health over time.
Nickel	Like electron guns and rechargeable nickel-cadmium batteries.	It is possible to experience allergic reactions.
Zinc sulfide	Used in CRT screens in conjunction with rare earth elements.	Harmful if breathed in
Residue from toner	Printer and copier cartridges for laser devices.	Inhaling dust raises the chance of explosion.

of papers, to study the impact of waste e-plastic aggregates on the unique characteristics of ordinary concrete. Moreover, a quantitative evaluation of fragmented information was employed to examine the suitability of e-plastic aggregates as a sustainable and safe replacement for fine or coarse aggregates in concrete**.**

E-Waste Composition

E-waste is a complex mixture of potentially dangerous and helpful materials. The complex components that make up e-waste comprise a wide spectrum of both "hazardous" and "non-hazardous" substances. Inadequate handling of these materials may result in significant damage to human health and the environment. The electronic waste plastic can be categorized into eight distinct classes based on its composition, as depicted in Fig. 4. E-waste usually consists of the following materials: 60% metals, 3% contaminants, 2% printed circuit boards (PCBs), 12% cathode ray tubes (CRTs) and liquid crystal displays (LCDs), 2% cables, 5% metal-plastic blend, 15% plastics, and 1% miscellaneous goods. Factors such as the types of electronic devices, their models, producers, ages, and production dates significantly affect the formation of e-waste (Tipre et al. 2021, Yong et al. 2019). The various e-waste components and their environmental impacts are listed in Table 1. Compared to regular municipal waste, e-waste contains thousands of harmful components and metals, such as lead, phthalates, beryllium, antimony, cadmium (Cd), chromium, mercury, polyvinyl chlorides, and brominated flame retardants, making it significantly more hazardous (Elgarahy et al. 2024, Luhar & Luhar 2019). Extended exposure to the reproductive and endocrine systems, joints, organs, and brain circuits is detrimental. Some of these substances are partially neurotoxic and carcinogenic (Yadav & Upadhyay 2015). The contaminated ingredients present in e-waste are listed in Table 2.

Techniques for Recycling Plastic from Used Electronics

The first stage of plastic recycling from electronic waste is the collection, physical selection, disassembly, and shredding of electronic devices. (Ceballos & Dong 2016). Materials, both metallic and non-metallic (plastics, glass, and ceramics), are mechanically detached from shredded electronic waste (Patil & Ramakrishna 2020, Shahabuddin et al. 2023). Polyethylene, acrylonitrile-butadiene-styrene,

Fig. 5: Shredded plastic strip of e-waste.

Fig. 6: Formation of e-waste plastic aggregates (Senthil Kumar & Baskar 2015a, 2015b).

Table 3: Physical techniques for extracting metals from WEEE (Tuncuk et al. 2012).

Techniques	Criteria for separation	Sorting metals
Separation by gravity	Specific gravity	Metals derived from polymers
Separation using a magnetic	Vulnerability to magnetic fields	Ferromagnetic from a non-magnetic, ferrous substance
Coronal electrostatic separation	Conductivity of electricity	Costly metals derived from materials that are not metallic
Spread of eddy currents	Density and conductivity of electricity	Switching from non-metals to non-ferrous metals.

polycarbonate, polyesters, polyamides, polypropylene, and high-impact polystyrene are among the polymers that can be recovered from e-waste (Ilankoon et al. 2018, Wang & Xu 2014). Prior to being recycled into goods or transformed into energy, these must be graded. Although e-waste offers valuable engineering plastics such as ABS, recycling remains a challenge because of the presence of numerous polymers, BFRs, and plasticizers, and the limited understanding of the compatibility between plastics during the melt extrusion process (Mtibe et al. 2023). Large amounts of sorted e-waste plastic strips, as shown in Fig. 5, require appropriate recycling infrastructure for a well-established recycling process. The manufactured or processed e-waste plastic aggregate is shown in Fig. 6.

Primary and Secondary Recycling

Melting plastics from e-waste and forming them into new products is a frequently used technique called mechanical recycling (Sugumar & Nayak 2014). In the first recycling, recycled plastic is utilized to produce items that closely resemble virgin plastic products in appearance and performance, whereas in secondary recycling, the retrieved plastic is repurposed to produce new goods with lower functionality requirements than the original materials. The size reduction process begins with the separation of plastic fractions from electronic waste. This involves breaking the plastic waste into tiny pellets or pieces, sorting, and cleaning (based on the desired product, either optical, magnetic, or manual sorting utilizing eddy current separators, as illustrated in Table 3). After sorting, the plastics are put through various melt processing procedures, including injection molding, hot pressing, and melt extrusion (Charitopoulou et al. 2021, Das et al. 2021, Jaidev et al. 2021, Mtibe et al. 2021). A flow diagram of primary and secondary recycling is shown in Fig. 7.

Tertiary Recycling

The third step of chemical and thermal recycling involves the use of depolymerization techniques to separate chemicals and fuels from polymers made from electronic waste through thermal and chemical treatments (Sugumar & Nayak 2014). For e-plastic waste, recycling chemicals enable the processing of polluted polymers without requiring laborious pretreatment steps. Subsequently, plastic portions are repurposed to create valuable items. Catalytic cracking, pyrolysis, hydrogenation, dissolution, and gasification are common processes. The sample is heated to high temperatures (400–800°C under inert conditions) during pyrolysis to produce products such as char, oil, and combustible gases. Pyrolysis produces fewer contaminants than conventional thermal treatments (Charitopoulou et al. 2021). The optimal processing parameters, including temperature, feedstock

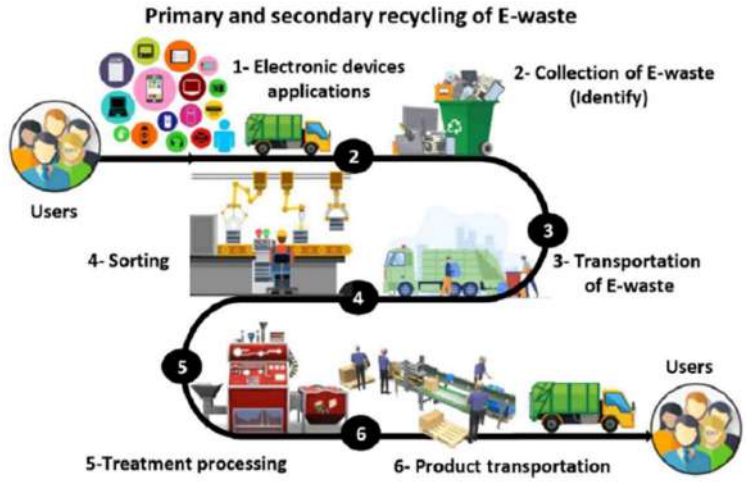

Fig. 7: Primary and secondary recycling of e-waste (Elgarahy et al. 2024).

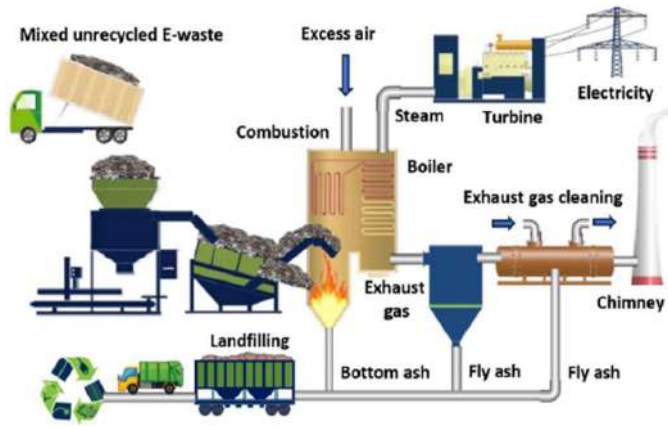

Fig. 8: Tertiary recycling diagram of e-waste (Elgarahy et al. 2024).

composition, heating rate, and time, determine the products produced during pyrolysis. Utilizing catalytic pyrolysis, which lowers the temperature and shortens the residence time, can yield high-value compounds. A flow diagram of the tertiary recycling approach for e-waste is shown in Fig. 8.

Incorporating E-Plastic Waste into Concrete Production

Various studies have been conducted on the use of plastic electronic waste in concrete manufacturing. These discarded components undergo a recycling procedure before being used to make concrete, as shown in Fig. 9, where the raw plastic components from e-waste are crushed and processed into various aggregate sizes, similar to regular aggregates, for the creation of concrete. Processed e-waste, particularly electronic waste plastic particles, is frequently utilized as an alternative to fine or coarse aggregates in concrete mixes. Novel research has highlighted that the materials were organized according to their weight, with e-plastic aggregates replacing natural coarse and fine aggregates based on weight (Arun Kumar & Senthamizh Selvan 2017, Manjunath 2016). According to past studies, e-plastic aggregates were used to substitute 5%–50% of conventional aggregates. The mechanical, physical, and long-lasting characteristics of concrete with these substitutions have been compared with those of regular concrete by previous researchers.

Characteristics of Concrete with E-Waste

Concrete's properties, both fresh and hardened, are evaluated by the physical and mechanical characteristics of its components. Durability, workability, strength, hardness, and water absorption are the primary characteristics of concrete. Multiple studies have analyzed the efficiency of e-waste plastics in enhancing the physical characteristics of

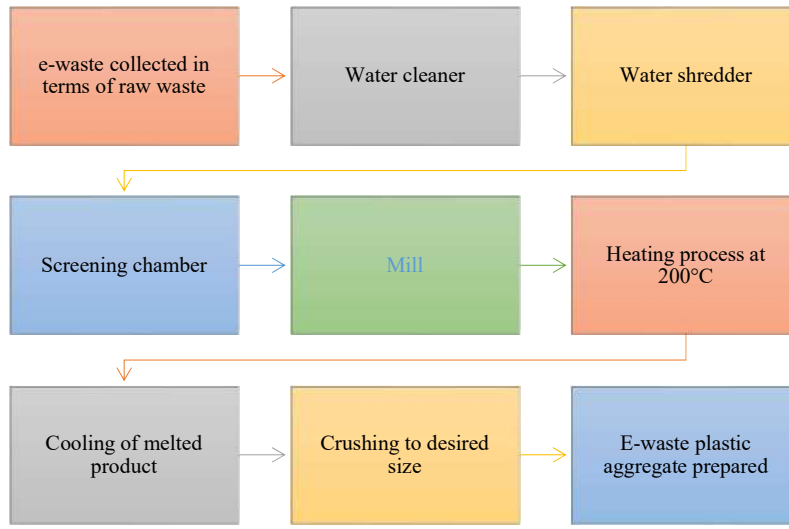

Fig. 9: Manufacturing procedure for e-waste aggregate (Bamigboye et al. 2024, Z. Ullah et al. 2021).

Table 4: Properties of e-waste plastic aggregates (Danish et al. 2023, Lakshmi & Nagan 2011).

Description of Properties	Test data
Color	White and dark
Shape	Irregular
size	4.5 mm-20mm
Specific gravity	1-1.25
Aggregate crushing value	< 2%

concrete. The physical characteristics of the e-waste plastic aggregates are shown in Table 4. The following sections critically explore the impact of e-plastic aggregates on the properties of concrete.

Workability

Published literature shows that the viability of concrete adapted with e-waste varies based on the size and form of the used e-waste. Few studies suggest that substituting coarse aggregates (CA) in concrete with e-plastics can lead to reduced workability. Kumar & Baskar (2015b) demonstrated that the addition of 10%–50% e-plastic in concrete led to a reduction in slump of 25%–65% compared to standard mixes, as the plastic obstructs other ingredients and reduces workability. Kumar & Baskar (2015a) quantified a related remark and observed that concrete mixtures with 10-50% e-plastic waste and water-to-cement ratios of 0.45, 0.49, and 0.53 showed slump reduction of 10-61%, 23-67%, and 31-73%, respectively, with respect to the control mix, with minimal change noted at 50% coarse aggregate replacement. Manjunath (2016) also noted that the use of 10-30% e-plastic waste in concrete resulted in a slump decrease of 10.93% to 41.40% compared to the standard specimen. Rohini & Padmapriya (2021) revealed that the slump value of the bacteria- and e-waste-based concrete was 50% higher at 20% e-waste and 0% to 2% bacteria compared to the control concrete.

Dry Density and Fresh Density

The quality of fresh concrete affects the properties of the prepared concrete. For instance, the slump, fluidity, and consistency of concrete are influenced by its fresh density, whereas its dry density influences its hardened properties. Research on the unique properties of e-waste plastics is limited. Kumar & Baskar (2015b) showed that adding electronic waste plastic to concrete reduced its initial density. According to their research, replacing 10-50% of the coarse particles with e-plastic waste reduced the new concrete density by 1.10-13.58% as compared to the standard mix. The fresh density decreased because the e-plastic waste aggregate had a lower density than the coarse aggregate. Kumar & Baskar (2015a) conducted a similar study using high-impact polystyrene (HIPS) electronic waste aggregates at varying weight-to-cement ratios to produce ecologically friendly concrete. In comparison to the reference specimen, investigators found that at a w/c of 0.53, adding 10-50% e-waste decreased density by 0.61-14.64%. At water-to-cement ratios of 0.45 and 0.59, incorporating similar amounts of e-waste plastic resulted in a fresh density reduction of 0.93–14.41% and 0.77–13.58%, respectively, with respect to the control sample.

Structural Properties of E-Waste Concrete

Mechanical Properties

Compressive strength: The capacity of a material to resist loads that attempt to compress or shorten its dimensions, rather than those that stretch it, is known as compression strength. Kumar & Baskar (2015b) observed that the compressive strength (CS) value drops as the proportion of e-plastic in the mixture rises (10% to 50%), and it exhibited a maximum loss of 47.41% at 50% e-plastic replacement because of one of the causes, namely, insufficient adherence of e-plastic to cement mortar. Another similar study by Manjunath (2016) utilized coarse e-waste plastic (0% to 30%) as aggregate and observed that the compressive strength dropped by 53.05% at 30% e-waste content. In contrast, Ahirwar et al. (2016) replaced 0–30% coarse aggregate with e-waste and 10–30% cement with fly ash, observing a minor drop in compressive strength with respect to the reference concrete. Rohini & Padmapriya (2021) proposed an innovative idea that focused on the addition of microbiologically induced calcite precipitation to electronic waste-treated concrete for strength enhancement. Their results showed that the compressive strength of 15% e-waste plastic concrete improved by 6.26%, 8.41%, and 5.95% with the addition of 0%, 1%, and 2% bacteria, respectively, compared with the control mix. The improvement in the compressive strength of e-waste plastic concrete with the addition of bacteria is attributed to the production of calcium carbonate and its inherent self-healing properties.

Splitting tensile strength: Analyzing the splitting tensile strength of concrete containing varying levels of e-waste plastic aggregates is vital for understanding its performance under tensile loads, particularly because concrete is naturally prone to tensile weakness owing to its brittle characteristics. Research consistently shows that increasing the proportion of e-waste aggregates tends to diminish the splitting tensile strength of concrete. For example, Kumar & Baskar (2015b) indicated that incorporating 10-50% e-waste led to a decrease in the splitting tensile strength of concrete, reducing it by 8.06% to 47.89% compared to

the control sample. Additionally, the samples containing e-waste plastic aggregates exhibited a different failure mode in their splitting behavior, unlike the typical brittle failure observed in the reference specimen. A similar remark was conveyed by Kumar & Baskar (2015a) assessed the effect of high-impact polystyrene electronic waste on the splitting tensile strength of concrete. Their results demonstrated that concrete containing e-waste plastic aggregates exhibited ductile behavior, preventing complete separation into two parts, whereas the control specimen experienced brittle failure, splitting into two distinct halves under the ultimate load. This implies that e-waste plastic aggregates can tolerate significant elastic deformation before fully breaking down. A similar study by Manjunath (2016) incorporated e-waste plastic aggregates at replacement levels ranging from 10 to 30 percent for coarse aggregates in the concrete mix. The inclusion of 20% e-waste plastic aggregates improved the 28-day splitting tensile strength of concrete by 10.20% compared to that of the reference sample. Ganesh et al. (2021) conducted a 28-day split tensile test of concrete (M20 grade) containing crushed printed circuit board as fine aggregate replacement at levels of 3% to 25% wt. The split tensile strength reached 1.51 MPa, reflecting an 11.85% increase with 15% fine aggregate replacement using PCB, compared to the control mix strength of 1.35 MPa.

Flexural strength: It measures the capability of a material

Table 5: Summary of effects of e-waste on concrete properties.

Author and Date	Percentage replacement (%)	Replacement method	Grade of Concrete	Strength after 28 days in MPa		
				CS	TS	FS
Manjunath (2016)	0%	E-plastic with FA or CA	M20	44.81	4.90	5.76
	10%			41.25	4.80	4.92
	20%			17.95	5.40	5.28
	30%			19.03	3.80	6.84
Alagusankareswari et al. (2016)	0%	Printed circuit boards with FA	M30	33.11	3.31	5.60
	10%			30.59	3.26	4.67
	20%			25.99	2.62	3.33
	30%			24.46	2.02	3.20
Needhidasan et al. (2020)	0%	E-plastic with CA	M20	45.05	3.90	4.10
	12%			41.95	3.50	4.30
	17%			44.93	4.90	4.80
	22%			41.95	6.70	5.20
Mary Treasa Shinu & Needhidasan (2020)	0%	E-plastic with CA	M40	46.25	4.63	4.54
	12%			44.85	4.09	4.20
	17%			38.24	3.82	4.01
	22%			35.15	3.01	3.84
Rajkumar et al. (2021)	Control	E-plastic with CA	M20	27.83	1.98	4.40
	5%			31.60	2.55	5.07
	10%			33.20	3.10	6.00
	15%			35.50	2.85	6.38
	20%			25.50	2.65	5.09
Ullah et al. (2021)	0%	ABS with CA	M20	34.40	2.68	4.35
	10%			32.20	2.05	4.40
	15%			31.20	1.85	4.30
	20%			28.00	1.81	2.50
Arivalagan (2020)	0%	E-plastic with CA	M30	31.00	4.90	4.40
	10%			32.73	4.40	4.40
	20%			37.50	5.50	4.50
	30%			35.00	3.75	2.90

[5] CS-Compressive strength, TS-Tensile strength, FS- Flexural strength, ABS-Acrylonitrile butadiene styrene plastic, CA-Coarse aggregate, FA-Fine aggregate

to withstand distortion under an increasing load, and numerous studies have explored this property. For instance, Kumar & Baskar (2015b) prepared concrete by replacing coarse aggregates (CA) with variable proportions (10% to 50%) of e-plastic by volume and assessed the 7 and 28-day flexural strength. As the proportion of e-plastic increased, a decline in the flexural strength of concrete was noted. The 10% replacement of coarse aggregate yielded the highest flexural strength values at 7 and 28 days compared to all other replacement percentages. Similarly, Manjunath (2016) noted a 1.14% enhancement in the 28-day flexural strength of concrete with 10% e-waste plastic aggregate compared to the control specimen.

Additionally, their results showed that concrete containing 20% e-waste plastic aggregates exhibited a flexural load capacity comparable to that of the control mix. Ahmad et al. (2022) prepared concrete with nano graphite platelets (doses of 1%, 3%, and 5% by weight of cement) and e-waste plastic coarse aggregates substituted partially at a percentage level of 25% to explore the flexural strength. The specimens with 25% plastic aggregates and 5% nano-graphite platelets exhibited a 31.42% increase in flexural strength. Sharma et al. (2022) developed M30 concrete by using HIPS electronic waste as a replacement for natural fine aggregate at levels of 5% to 25% and conducted a flexural test. The strength decreased by as much as 15.18%, 15.10%, and 16.01% at a 25% replacement level for 7, 14, and 28 days, respectively. The observations indicated that a replacement level of up to 10% e-waste plastic was viable.

Shear strength: Kurup & Kumar (2017) added e-waste fibers to the concrete mix in proportions of 0.6%, 0.8%, and 1% by OPC weight, and silica powder replaced 10% of the cement content to produce silica fiber-reinforced concrete (SFRC). The incorporation of silica powder into fiber-reinforced concrete improves its shear strength compared to that of conventional fiber-reinforced concrete. Silica fiber-reinforced concrete exhibited a 21.5% decrease in shear strength, whereas fiber-reinforced concrete experienced a 25.6% reduction compared to conventional concrete with the inclusion of 1% fiber. Although there was a decline in strength, the addition of e-waste fibers significantly minimized the brittleness of the conventional concrete. A Summary of the effects of e-waste on various concrete properties is presented in Table 5.

Durability Characteristics of Concrete Incorporating Electronic Waste

This attribute is vital for practical applications in the industry. Consequently, assessing the durability of e-waste concrete to determine its long-term performance and suitability is essential for its application. There is still a shortage of studies on the long-term behavior of e-waste concrete, as mentioned below.

Water Absorption Properties

To evaluate the suitability of e-waste concrete for construction applications, it is essential to conduct additional studies on the water absorption capabilities of this plastic-based concrete. Concrete durability is associated with lower water absorption values; however, this property has not been widely investigated. Ullah et al. (2022) conducted tests to assess the water absorption characteristics of concrete incorporating e-waste as a coarse aggregate. As the replacement of natural coarse aggregates with e-waste increased from 0% to 20%, the reduction in water absorption became more pronounced, which was linked to a decrease in the sorptivity coefficient. When coarse aggregates were replaced with e-waste at levels of 10%, 15%, and 20%, the sorptivity coefficient of the concrete decreased by 12.2%, 14.5%, and 29.0%, respectively.

Alternate Wetting and Drying

The ability of concrete to endure weathering in various wet and dry environments is assessed using sea tidal waves as stress factors. Structural durability diminishes when cracks develop due to stress and the reinforcement becomes weathered. Ullah et al. (2022) created concrete with electronic waste, demonstrating improved resistance to compressive strength deterioration after cycles of wetting and drying, with resistance increasing as electronic waste content rose, in contra st to concrete with natural coarse aggregates.

Abrasion Resistance

This has been investigated in several studies, which have improved the viability of the material. However, research on this topic is limited. Ullah et al. (2021) noted that a higher percentage of e-waste improves the abrasion resistance. The experimental findings indicated that substituting 10%, 15%, and 20% of natural coarse aggregates with e-waste enhanced the abrasion resistance by 39.8%, 44.3%, and 46.4%, respectively. This was because of the increased toughness and abrasion resistance of the e-plastic aggregates compared to those of the natural aggregates.

Ultrasonic Pulse Velocity (UPV)

This durability examination is essential for evaluating the homogeneity and consistency of concrete. Using this test, the compactness of the concrete and flaws such as pores and cracks were identified. In this context, Kurup & Kumar (2017) created fiber-reinforced concrete using PVC waste at 0.6%, 0.8%, and 1% by weight of cement and silica-reinforced concrete with 10% of the cement weight replaced by silica. It was dis-

covered that the various concrete mix types had values above 4.2 km.s.$^{-1}$ The specimen with e-waste fibers demonstrated a declining UPV value, which was attributed to the ability of the fibers to absorb pulse waves. A similar study by Ullah et al. (2021) found that as the content of the e-waste aggregate increased, the UPV of the concrete decreased, which was due to an increased air void content and irregular distribution of the plastic aggregate. When the natural coarse aggregate was replaced by 10%, 15%, and 20%, the UPV of the e-waste concrete decreased by 1.2%, 1.9%, and 3.3%, respectively. The incorporation of e-waste plastic as a coarse aggregate had a minimal impact on concrete quality, with UPV values ranging from 3660 to 4575 m.s^{-1}.

Chloride Penetration

Chloride attack must be considered when evaluating the long-term resilience of concrete, as it is a primary cause of reinforcement corrosion, which is of great importance. For instance, Kumar & Selvan (2017) conducted a rapid chloride ion penetration test using e-waste, where coarse aggregates (5%, 10%, and 15%) and fine aggregates (10%, 20%, and 30%) in fiber-reinforced green concrete were replaced with 30% GGBS instead of cement. The control concrete exhibited modest levels of chloride ion penetration, whereas the fine and coarse aggregate replacements made from e-waste showed moderate levels of chloride ion penetration, with charges passing between 3271 and 3966 coulombs.

Temperature Resistance of Electronic Waste Concrete

It is important to understand the effect of temperature on the material strength for the construction of fire-resistant structures. This test evaluates the response of a material to fire and its tendency to ignite. For instance, Lakshmi & Nagan (2010) revealed that as the amount of electronic waste plastic aggregates increases, the compressive strength of concrete decreases at elevated temperatures. Another observation by Ullah et al. (2021) developed e-plastic waste-based concrete to assess its performance at elevated temperatures ranging from 150 to 350 °C, applying e-waste at various replacement percentages for coarse aggregates. They reported a compressive strength decrease of 21-26% at 150 °C and 39% at 300 °C. The appearance of e-waste plastic concrete before and after thermal exposure is shown in Fig. 10. However, this reduction is minor compared to the strength losses observed in the standard mix. Further investigation is required to confirm that electronic waste plastics perform adequately as construction materials at high temperatures.

SEM and XRD Analysis of Electronic Waste Concrete

Balasubramanian et al. (2021) utilized e-waste plastic to replace 5% to 20% of the coarse aggregate volume in the concrete matrix for SEM and XRD analysis. SEM analysis identified darker regions associated with denser packing and lower porosity of calcium hydroxide (CH) in conventional concrete. It also revealed large hexagonal CH plates, a small fibrous crystalline C-S-H gel, and needle-like crystalline ettringite. Conversely, the bond between the waste plastic aggregates and the concrete matrix was found to be weaker, as shown in Fig. 11. In the X-ray diffraction analysis, the control specimen demonstrated prominent crystal phases at 21°, 26.7°, and 50.08°, linked to silicon dioxide and crystal phases at 28.16° and 81.72°, associated with calcite. Low-intensity peaks at 18.48° and 80.38° were also detected,

Fig. 10: Photographic view of e-waste concrete before and after exposure to high temperature (Ullah et al. 2021).

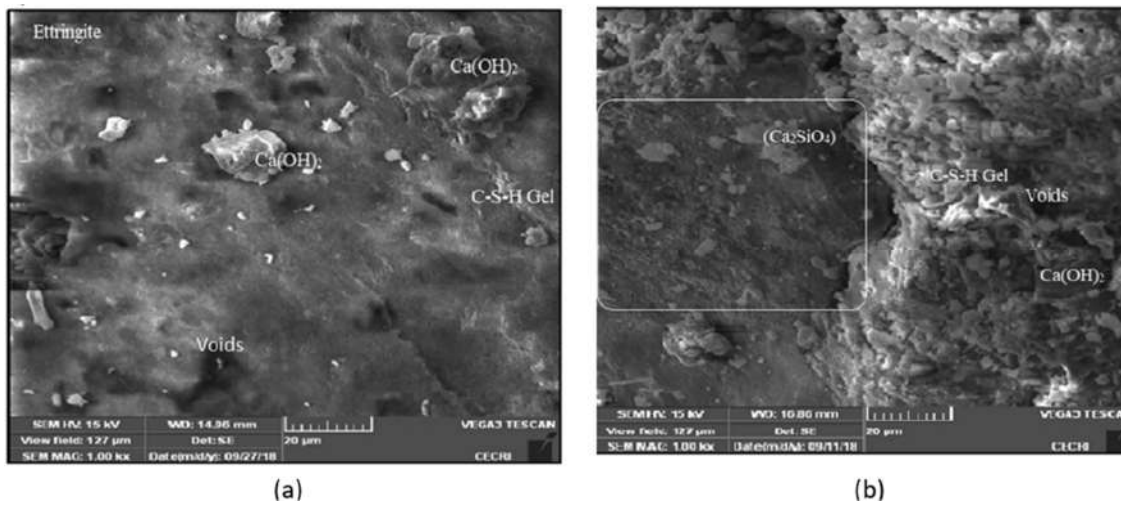

Fig. 11: SEM images of (a) normal concrete and (b) E-waste concrete (Balasubramanian et al. 2021).

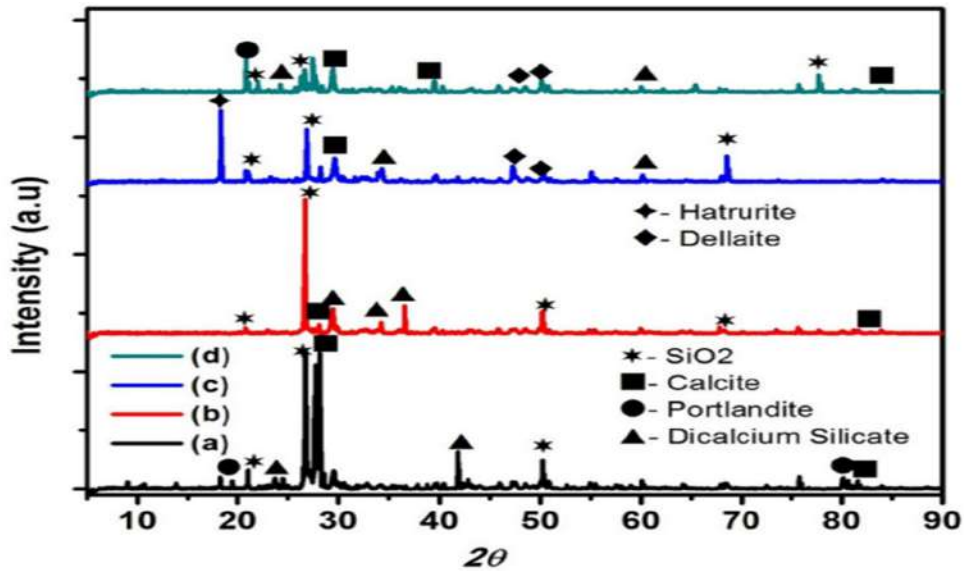

Fig. 12: XRD patterns of (a) control concrete and (c) E-waste concrete (Balasubramanian et al. 2021).

which were associated with calcium hydroxide. Conversely, the introduction of 20 % e-waste plastic resulted in a new peak for hatrurite, and the most intense crystal phases of dellaite occurred at 18.15° and 47.17°, respectively. However, the strength characteristics of the matrix were reduced owing to the expansion and fissures caused by the presence of water. Strength reduction occurred owing to the lower formation of dicalcium silicate (Ca2SiO4). The XRD pattern of the composite concrete matrix is shown in Fig. 12.

Comparison of E-Waste Concrete with Other Alternative Aggregate Concretes

Islam et al. (2025) investigated the use of e-waste as a partial replacement for natural coarse aggregate in concrete, with substitution levels varying from 10% to 20% by mass. After 28 days of curing, the study observed a decline in compressive strength by 13.41% to 25.50% and in tensile strength by 11% to 19.26% relative to conventional concrete. Afshinnia & Rangaraju (2016) observed that replacing natural coarse aggregate with coarse waste glass notably decreased both compressive and splitting tensile strengths, with a 38% drop in compressive strength. Nováková & Mikulica (2016) produced sustainable concrete by substituting natural aggregates with recycled concrete aggregates (RCA) and found that replacing raw aggregates with up to 20% RCA did not adversely affect the concrete's physical or mechanical

properties. Alaud et al. (2023) created sustainable concrete by substituting gravel with recycled rubber particles at 10%, 15%, and 20% by volume. The inclusion of rubber reduced the density of concrete, resulting in a compressive strength drop of over 26% at 20% replacement. Li et al. (2025) investigated the effects of varying rubber replacement ratios (0%, 5%, 10%, and 15%) on the fundamental mechanical, dynamic, and frost resistance properties of rubber recycled aggregate concrete. At a 15% replacement level, the results showed a 36.86% reduction in compressive strength, 44.07% decrease in axial compressive strength, and 25.76% drop in elastic modulus. Conversely, the splitting tensile strength improved by 46.29% and the impact resistance increased by 181.3 J. Based on these findings, it can be concluded that a 10–15% replacement level of alternative aggregates, such as rubber, glass, or recycled aggregates, may offer an optimal balance between mechanical performance and sustainability, making it a promising range for producing eco-friendly concrete without significantly compromising structural integrity.

Potential Strategies for Enhancing the Characteristics of E-Waste Concrete

When e-waste is mixed with concrete, the peculiar properties of the e-waste components may result in diminished strength. Researchers have proposed several solutions to overcome this issue and make concrete altered with e-waste useful as a building material. The following are crucial strategies for enhancing the characteristics of concrete containing e-waste:

1. Optimizing the Design of Concrete Mix: Researchers have suggested adjusting the water-to-cement ratio, choosing suitable e-waste aggregates, and incorporating chemical admixtures to enhance the workability and strength of e-waste concrete. Studies have shown that these changes can significantly enhance the overall strength.
2. Incorporating Admixtures: To improve the workability and mechanical strength of fresh e-waste concrete, incorporating superplasticizers and mineral admixtures such as fly ash, silica fume, and slag is recommended. Previous research has shown that superplasticizers improve the strength and fluidity of e-waste concrete.
3. Fiber reinforcement: It has been suggested that strengthening concrete treated with e-waste with fibers that are synthetic or natural could enhance the material's mechanical properties.
4. Microbial Additives in Concrete: Strength improvements in e-waste concrete can be achieved through an innovative approach that facilitates calcium carbonate precipitation, and microorganisms help reduce the negative impacts associated with e-waste aggregates.
5. Incorporating graphene oxide into concrete: Graphene oxide (GO) is a novel nanofiller that significantly improves the density and hardness of cementitious composites by reducing porosity and reinforcing the microstructure. Consequently, incorporating GO can improve the hardened characteristics of e-plastic waste concrete.

Combining these techniques will allow for improvements in e-plastic waste-modified concrete, transforming it into a more valuable and eco-friendly building material for the construction industry. Adopting these strategies will help address issues related to e-waste concrete, supporting resource conservation and broader applications in the construction industry.

Prospects and Recommendations for Further Research

Despite the difficulties in using e-waste concrete, several methods should be investigated to overcome the limitations of the material.

1. There are several ways in which e-waste integration into concrete will benefit the environment: it will produce more sustainable concrete, manage e-waste more effectively, and conserve natural aggregate resources.
2. Compared with conventional concrete, e-waste aggregate concrete demonstrated adequate sound absorption. The UPV values of the concrete specimens ranged from 3660 to 4575 $m.s^{-1}$, suggesting elevated quality and making e-waste concrete a suitable alternative, as these values are within the acceptable range.
3. Incorporating electronic waste aggregates as a 20% replacement for natural coarse aggregates in concrete increased its resistance to abrasion.
4. It is essential to recognize that the inclusion of e-waste aggregates can enhance the workability of concrete. However, using shredded e-waste components of non-uniform size should be avoided, as this can negatively affect workability, largely because of the low water absorption of e-waste.

Further investigation is essential to fully comprehend the role of E-waste aggregate concrete in construction practices. The literature review uncovered various gaps in the current knowledge. The following are suggestions for future research.

1. Mechanical properties, such as hardness (measured by aggregate abrasion), strength (assessed using the aggregate crushing value), and toughness (evaluated using the aggregate impact value), should be considered in the classification of e-waste aggregates.
2. There is insufficient data on the elastic modulus, bond

strength, Poisson's ratio, stress-strain behavior, and flexural strength of concrete using e-waste aggregates.

3. Studies on incorporating e-waste plastics into reinforced concrete remain scarce. When constructing columns, beams, and slabs, e-waste should be considered as an alternative aggregate with different substitution rates and mix compositions.
4. Additional research is necessary to explore the impacts of alkali aggregate reactions, color changes, thermal resistance beyond 300°C, post-fire characteristics, slip resistance, carbonation, chloride penetration, freeze-thaw stability, seawater and chemical resistance, shrinkage, and swelling. It is necessary to investigate the durability-related behavior of e-waste aggregate concrete because e-waste has poor bonding with cement mortar.

CONCLUSIONS

This review highlights the incorporation of e-waste in construction and infrastructure while adhering to a sustainable framework. To accomplish this, we evaluated the essential features of green concrete containing e-waste, focusing on its physical properties (both fresh and hardened), strength, durability, and thermal performance. Based on this evaluation, the following conclusions were drawn:

1. The size and shape of the aggregates largely influence the properties of the concrete containing e-waste aggregates. Finer electronic waste aggregates usually exhibit higher density, lower absorbability, and lower fineness modulus than coarser aggregates. Additionally, e-waste aggregates often have lower bulk densities and specific gravities than conventional aggregates, and they tend to absorb less water because many e-waste materials, particularly plastics, are nonabsorbent.
2. Lead, antimony, mercury, brominated flame retardants, and cadmium (Cd) not only contaminate soil, water, and air and destabilize ecosystems but also present potential health hazards to humans. Consequently, careful consideration is required when choosing e-waste for concrete manufacturing.
3. A higher proportion of e-waste in the aggregate mix enhanced the workability of the concrete. Research indicates that incorporating shredded e-waste particles of different sizes influences the workability of concrete.
4. Based on a review of multiple literature sources, it is suggested that replacing up to 15% of the original amount of coarse or fine aggregates with e-waste is the ideal replacement ratio in terms of strength.
5. An increased e-waste content in the concrete matrix reduced both the UPV value and sorptivity coefficient while improving the abrasion resistance.
6. According to thermal exposure testing, high temperatures cause e-waste concrete to compress more readily.
7. According to the literature, its strength is reduced by 39% at high temperatures (300°C). However, research on boosting strength at elevated temperatures is still lacking.

Recycled plastic aggregates sourced from e-waste provide an eco-friendly alternative, helping manage excessive e-waste, preserve the environment, and reduce traditional concrete expenses.

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Spectral Characterization and Indexing Methods for the Quality Assessment of Municipal Solid Waste Compost Prepared Using Novel Bacterial Consortia

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ABSTRACT

Composting is one of the integral components of the global circular bio-economy platform. However, traditional composting contains major limitations, including its longer time requirement and the formation of odour. Therefore, the inoculation of efficient novel bacterial consortia for compost process modification is a global concern. Furthermore, the assessment of compost quality is crucial because immature compost can cause phytotoxicity, disrupt soil structure, and damage the natural ecological balance when used in agriculture. Conversely, there is no universally applicable procedure to determine compost quality, maturity, and stability. This study focuses on assessing the quality of compost produced by five novel microbial consortia using indexing and spectroscopic methods. Clean Index (CI), Fertilizing Index (FI), Germination Index (GI) and Vigor Index (VI) were used as indexing methods to assess the phytotoxicity and compost quality. Scanning Electron Microscopy (SEM), X Ray Diffraction spectroscopy (XRD) and Energy Dispersive X-ray Spectroscopy (EDS) techniques were used for spectroscopic analysis of compost microstructure. The results revealed that out of all compost samples (including the control), the compost made by Consortium 5 (C5) recorded a significantly greater ($p<0.05$) GI, VI, FI and CI compared to the control and other treatments. Further, the GI value of C5 was recorded as 110.2 ± 2.2 %, demonstrating the possible usage of C5 compost as a phytonutrient soil amendment. Importantly, SEM, XRD and EDS spectrograms also confirmed the rapid waste degradation pattern and elemental composition alteration by the C5 consortium. Consequently, the compost by the C5 consortium was categorized into the compost quality "A" category, whereas the control compost belonged to the compost quality "D" category. In contrast, the findings of the present study confirm that the potential applicability of a prepared novel bacetrail consortium as a rapid, greener waste management approach in the circular bio-economy.

INTRODUCTION

The generation of solid waste has become a burning issue in the world, owing to rapid urbanization and industrialization (World Bank Report 2022). Currently, around 33% of the 2 billion tonnes of solid waste generated each year worldwide is not collected and treated by municipalities (Valavanidis 2023, Wijerathna et al. 2024a). Consequently, about 90% of this waste is used for uncontrolled dumping, which leads to severe ecological and public health implications. Nevertheless, the approximate global operational cost for waste management was USD 252 billion in the year 2020, and it could double to a staggering USD 640.3 billion by the year 2050 (World Bank 2022, Roy et al. 2023).

Composting is an effective, environment-friendly, integral component of waste management in the circular bio-economy platform. Further, it is a biological process

influenced by several physicochemical conditions and enzymatic degradation of different microbial communities in the environment (Sarsaiya et al. 2019, Wijerathna et al. 2023). However, traditional composting processes still have several limitations, including the prolonged time required and the difficulty in assessing compost maturity and quality. Inoculation of microbial consortia with greater extracellular hydrolytic enzyme activity (cellulase, amylase, protease, pectinase, and lipase) is a rapid biological method to facilitate the efficient transformation of Municipal Solid Waste (MSW) to valorized compost (Valavanidis 2023).

However, the final quality of MSW compost depends on its stability and maturity. Compost maturity describes how much organic matter has stabilized in composts throughout the biodegradation processes (Azim et al. 2018). Poor maturity compost can threaten soil quality and inhibit crop growth (Sarsaiya et al. 2019). Furthermore, by releasing harmful compounds and competing for oxygen in the rhizosphere, spreading premature compost may limit the growth of plants. Therefore, evaluation of compost maturity is a crucial part of composting. Several European and North American countries have adopted specific standards mainly to regulate the marketing of desirable quality composts (Esteves 2021). However, such quality control guidelines have been unable to identify different quality grades of marketable compost (Esteves 2021).

Compost maturity can be assessed using a variety of techniques. However, there is no universal method to define it. Among these methods, the Vigour Index (VI) and Seed Germination Index (GI) have been extensively employed as biological parameters to assess the compost's maturation and phytotoxicity (Yang et al. 2019). Additionally, the overall quality of compost in agriculture is assessed based on the Fertilizing Index (FI) and Clean Index (CI). The Fertilizing Index is a quantitative measure used to assess compost's fertilizing potential or nutrient content. It commonly involves analyzing the nutrient composition of the material. This analysis includes measuring key elements such as nitrogen (N), phosphorus (P), potassium (K), and other essential micronutrients (Ji et al. 2023). The fertilizing index can incorporate various parameters and factors, such as nutrient ratios, bioavailability of nutrients, and their release rates in soil, to assess the material's ability to enhance soil fertility and support plant growth. Additionally, the CI indicates a quantitative number to represent the amount of heavy metal available in the compost samples (Yang et al. 2019).

Additionally, several spectroscopic techniques, including Scanning Electron Microscopy (SEM), X Ray Diffraction Spectroscopy (XRD), and Energy-Dispersive X-ray Spectroscopy (EDS) analysis, have recently been utilized to assess compost age. These techniques can successfully acquire complementary, comparative data and a description of compost maturity. While EDS and SEM are evident methods used for the material's elemental identification and to obtain information about its quantitative composition, XRD analysis of compost evaluates the molecular and atomic composition of compost (Kataki 2017).

In Sri Lanka, there is a lack of published literature on the overall quality of MSW compost prepared using different bacterial inocula. Therefore, the application of immature compost and compost with phyto toxicity can negatively affect the agricultural plants (Yang et al. 2019). The objective of the present study evaluate systematic comparison and quality assessment of MSW compost prepared from five indigenous novel bacterial consortia using the indexing methods, including GI, VI, FI and CI, by evaluating the compost phyto toxicity of prepared compost. Further, the manuscript aims to compare the spectral characterization of prepared compost using SEM, XRD and EDAX spectra to evaluate the compost quality of prepared compost to determine the potential applicability of the prepared novel bacterial consortium as a successful composting approach of MSW.

MATERIALS AND METHODS

Isolation and Screening of Bacteria

For the study, the bacteria were isolated from the three different dump sites in Sri Lanka, including Karadiyana, Meethotamulla and Sundarapola dump sites. Leachate, solid waste and soil samples were collected from these dump sites, and standard pour plate and streak plate methods on nutrient agar were used for the isolation of bacteria. The culture plates were incubated at 30°C for 24 h under steady conditions (Wijerathna et al. 2024a, Gunaratne et al. 2024). The screening of bacteria was done based on the extracellular enzyme secretion potential of these bacteria to degrade cellulose, starch, lipids and protein substrates contained in the solid waste. The primary screening of these bacteria was done by testing their cellulase, amylase, lipase and protinase activity on clear zone formation on CMC agar media, starch agar media, casein agar media and olive oil agar medianrespectively (Sarkar & Chourasia 2017). The secondary screening was done for the positive isolates to select the best potential bacteria to degrade the solid waste by quantifying their enzyme activity following the standard enzyme assays (Wijerathna et al. 2024a, 2024b).

Preparation of Bacterial Consortia and Identification of Bacteria

The best potential bacteria were used for the consortia

preparation by evaluating their antagonistic effects. Out of all prepared consortia, the best five consortia (Table 1) were used for the field application based on their total enzyme production potential (Wijerathna et al. 2024a).

The molecular identification of bacteria was done by using the 16S rRNA molecular identification. The DNA extraction of bacteria was done by using the freeze-thaw manual method, and the sequencing was done from Macrogen Korea (Wijerathna et al. 2024b).

Experimental Setup and Bacterial Consortia Used for the Study

Table 1 represents the five developed bacterial consortia for the composting field experiment.

For the composting experiment, 18 standard compost containers (each 150 cm high and 45 cm in diameter) were used in triplicate for the control and experimental groups, comprising five different bacterial consortia configurations. All the MSW samples were collected from the Maharagama Municipal Council, Sri Lanka, from their daily organic waste collection and all the waste samples were collected at the same time from the same location. Considering the bin's capacity, 95 kg of mixed organic municipal solid trash and 2% (volume/ weight) inoculum of bacteria (depending on the waste's weight) were introduced to each bin. No additional microbial consortia were added to the control group. The spectral and indexing approaches assessed the final composts' quality.

Analysis of Compost Physicochemical Properties

The moisture content of the compost was analyzed by measuring the weight loss at 105°C. Before being examined for chemical properties, the samples were ground up, sieved through a 4 mm sieve, and dried at 70ºC. A 100 mL measuring metal core was used to calculate the bulk density (Sharma et al. 2019). The formula for total organic carbon is % C = (% VS)/1.8. Analysis for pH, total N and Total P were carried out using standard methods (Santos et al. 2020). The pH was measured using a portable pH meter, and the total nitrogen was measured following the Kjeldahl method (Santos et al. 2020). The organic carbon was measured using the dichromate wet oxidation method. Total phosphorus was determined by total digestion of the compost in the di-acid mixture ($HClO_4$: HNO_3 at 9:4 ratio) with subsequent analysis by spectrophotometer after colour development using the vanadomolybdate yellow colour method (Sharma et al. 2019, Santos et al. 2020).

Determination of Trace Metals

Metal analysis for Cd, Zn, Cr, Pb and Cu in prepared compost samples was performed using the Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES Agilent 720) (Fathabad et al. 2018).

Indexing Methods for Characterization of Prepared Compost

Seed germination index (GI): *Vigna radiata* seeds were used as the test seeds to assess the compost phytotoxicity, considering it's readily availability and susceptibility to compost toxicity. The compost water extract was used for the seed germination test (Luo et al. 2018). 10 g of new compost was added to 100 mL of deionized water, and it was stirred at 170 rpm for 30 min. Then the extract was filtered via a 0.45 µm filter paper, and the filtrate was gathered onto several petri dishes. The seeds were subjected to surface sterilization by dipping in 70% alcohol for 30 seconds, followed by an immediate washing with distilled water five times (Luo et al. 2018). Each petri dish contained 10 *Vigna radiata* seeds, water instead of compost extract, which were then incubated for 48 h (Luo et al. 2018). The GI was measured based on the following equation.

$$GI = (G\% \times RRG\ \%) \times 100$$

Where,

G % - (number of seeds germinated in a sample/number of seeds germinated in the control) × 100,

RRG % - (mean root length in a sample / mean root length in the control) × 100,

Seedling vigor index (SVI): The Vigor index of the seedlings was calculated using the following equation.

SVI = Germination (%) × Seedling Length (Mean Root Length + Shoot Length)

Table 1: Different bacterial consortia used for the experimental setup.

Bacterial consortium	Bacteria in the consortium
C1	*Bacillus haynesii* BHC1 (PP391133), *Bacillus haynesii* (PP438760), *Bacillus licheniformis* (CP027791)
C2	*Bacillus haynesii* BHC1 (PP391133), *Bacillus licheniformis* (CP027791), *Bacillus subtilis* (CP007800)
C3	*Bacillus haynesii* BHC1 (PP391133), *Bacillus safensis* (PP391033) , *Bacillus velezensis* (CP 026533)
C4	*Bacillus haynesii* BHC1 (PP391133), *Bacillus amyloliquefaciens* BAC1 (PP391056), *Bacillus subtilis* (CP007800)
C5	*Bacillus haynesii* BHC1 (PP391133), *Bacillus amyloliquefaciens* AMWC (PP391615), *Bacillus safensis* (PP391033)

Table 2: Score value chart for fertilizing index calculation.

Fertility parameter	Score value [S_i]					Weighting Factor [W_i]
	5	4	3	2	1	
TOC [% dm]	>20.0	15.1-20.0	12.1-15	9.1-12	<9.1	5
TN [% dm]	>1.25	1.01-1.25	0.81-1.00	0.51-0.080	<0.51	3
TP [% dm]	>0.60	0.41-0.060	0.21-0.40	0.11-0.20	<0.11	3
TK [% dm]	>1.00	0.76-1.00	0.51-0.75	0.26-0.50	<0.26	1
C:N ratio	<10.10	10.1-15	15.1-20	20.1-25	>25	3

Note * TOC - Total Organic Carbon, TN - Total Nitrogen, TP - Total Phosphorus, TK - Total Potassium, C:N - Carbon to Nitrogen ratio
*Source –Saha et al. (2010)

Table 3: Score value chart for clean index calculation.

Heavy metal	Score Value (S_j)						Weighting Factor (W_j)
	5	4	3	2	1	0	
Zn [$mg.kg^{-1}$]	<151	151-300	301-500	501-700	701-900	>900	1
Cu [$mg.kg^{-1}$]	<51	51-100	101-200	201-400	401-600	>600	2
Cd [$mg.kg^{-1}$]	<0.3	0.3-0.6	0.7-1.0	1.10-2.0	2.0-4.0	>0.4	5
Pb [$mg.kg^{-1}$]	<51	51-100	101-150	151-250	251-400	>400	3
Ni [$mg.kg^{-1}$]	<21	21-40	41-80	81-120	121-160	>160	1
Cr [$mg.kg^{-1}$]	<51	51-100	101-150	151-250	251-350	>350	3

*Source –Saha et al. (2010)

Fertilizing index (FI): The Fertilizing Index (FI) was used as an indicator to determine the fertilizing potential and the economic worth of the prepared compost. A rating system from 1 to 5, where 5 represents the best fertilization possibility, was used to calculate the FI.

The FI is measured based on the following formula:

Fertilizing index (FI) = $\sum_{n}^{I=1} 1(\text{SiWi}) / \sum \text{n i=1 (Wi)}$

where "Si" is the statistically calculated rating associated with total organic carbon content, Total Nitrogen content, Total Phosphorus, Total Potassium, and C: N and "Wi" is the ranking variable 5-1, which represents the quality of compost in descending order (Table 2).

Clean index: Clear index. According to the procedure shown in Table 3, ranking values were allocated to each metal based on its high toxicity to mammals. The clean index was calculated based on the following formula.

$$\text{Clean Index (CI)} = \frac{\sum_{n}^{j=1} S_j W_j}{\sum_{n}^{j=1} W_j}$$

Where 'S_j' is the score value of analytical data for heavy metal (in a five-point scale), and 'W_j' is the weighing factor of the j^{th} fertility parameter (in a five-point scale).

Compost Quality Grade

Compost quality grading criteria is given in Table 4.

Table 4: Compost quality grading criteria.

Compost category	FI Value	CI value	Compost Quality	Remarks
A	>3.5	>4.0	Complying with all heavy metal parameters	Excellent quality, with very low heavy metal contaminants content, strong potential for manurial value, and suitable for organic agriculture
B	3.1-3.5	>4.0	Complying with all heavy metal parameters	Good quality, relatively lower heavy metals, medium fertilizing ability
C	>3.5	3.1-4.0	Complying with all heavy metal parameters	Good quality High fertilizing potential
D	3.1-3.5	3.1-4.0	Complying with all heavy metal parameters	Medium quality,
RU-1	<3.1	-	Complying with all heavy metal parameters	Should not be allowed to market. However, these can be used.
RU-2	>3.5	>4.0	Not complying with all heavy metal parameters	Should not be allowed to market, can be used for growing non-crops
RU-3	>3.5	-	Not complying with all heavy metal parameters	Restricted use, marketing should be prohibited.

*Source –Saha et al. (2010)

Spectroscopic Methods for Compost Quality Assessment

SEM/EDS spectroscopy: SEM-EDS analysis of compost samples produces high-quality pictures of solid materials. The current study used SEM and element detection by EDS (SEM-EDS) to investigate the structural modifications of the composting samples. The analysis was performed based on the method described by Joseph et al. (2018).

XRD spectroscopy: An XRD device (Philips X-ray diffractometer) was used to get the spectra of the various compost specimens after they had been compressed into a fine powder. 200 mg of the specimen was used, and the specimens were exposed to Cu Kα radiation with a wavelength of 1.5406 Å (0.15406 nm) for 20 min per scan. The diffractometer was operated in continuous scanning mode over a 2θ range of 5° to 80°, with a step size of 0.02° and a counting time of 1 second per step (Sharma et al. 2019).

Statistical Analysis

Minitab 19 software was used for the statistical analysis, and a one-way ANOVA test was conducted to evaluate the effects of different compost treatments (Control, C1, C2, C3, C4, and C5) on various parameters, which were analyzed independently. Before conducting ANOVA, the assumptions, including independence of observations, were ensured through the experimental design and the normality of residuals was assessed. The homogeneity of variances was also tested, and the parameters showed significant differences ($p < 0.05$). Tukey's Honestly Significant Difference (HSD) post-hoc test was applied to identify specific group differences.

RESULTS AND DISCUSSION

MSW contains diverse toxigenic substances such as heavy metals, a broad range of organic and inorganic compounds, which negatively impact the plant growth (Sharma et al. 2022, Liyanage et al. 2024, Pal & Tiwari 2023). Table 5 represents the physicochemical results of the compost samples. Referring to the data, the pH, conductivity, bulk density and moisture of the C5 compost sample ranged between 7.2-9.5,0.76-1.12 $mscm^{-1}$, 325-680 kgm^{-3}, 40.5 %-44.6 % respectively, showing a significant difference ($p<0.05$) compared to the control and other samples.

Table 5: Compost quality characterization.

Parameters		Compost sample						Sri Lankan Standard
		C1	C2	C3	C4	C5	Control	
pH		7.6±0.2	7.2±0.3	7.3±0.2	7.4±0.1	7.2±0.1	9.5±0.2	6.5-8.5
Conductivity $mS.cm^{-1}$		0.86±0.10	0.79±0.02	0.76±0.02	0.78±0.02	0.76±0.03	1.12±0.06	$4mscm^{-1}$
Bulk density $kg.m^{-3}$		580±10	490±10	660±12	680±10	660±10	325±12	NA
Moisture %		44.6 ±0.5	42.5±0.45	44.5±1.5	40.5±0.2	42.5±0.5	45.2 ± 0.5	NA
Total P %		0.60±0.05	0.40±0.10	0.40±0.10	0.98±0.10	1.1 ±0.10	0.95±0.05	0.5%
Total N%		0.9±0.1	0.9±0.2	0.9±0.2	1.2±0.2	0.9 ±0.2	0.8±0.1	1%
Total organic C %		22±0.5	24±0.5	26±0.5	30±0.5	25±0.2	34±0.2	20%
C:N		24	26	29	25	27	42.5	10-25
Total Potassium %		0.6±0.0	0.8±0.1	0.7±0.1	0.5±0.1	0.6±0.1	0.5±0.1	1%
Faecal coliform		5	6	5	0	0	45	0
Salmonella spp.		0	0	0	0	0	5	0
Shigella spp.		0	0	0	0	0	0	NA
Sand content %		12±1	12±1	11±1	11±2	10±1	12±2	20%
Particle size (Residue particles when passing through a 2 mm sieve)		3.8±0.1%	4.2±1.2%	3.4±0.5%	1.8±0.2%	1.4±0.2%	15.2±0.1%	2%
Heavy metals (mg/kg)	Zn	64±2	72±1	45±1	40±2	42±1	46±0	N/A
	Cu	42±1	32±1	38±1	44±0	24±0	40±0	N/A
	Cd	0.52± 0.02	0.48± 0.15	0.32± 0.15	0.24± 0.05	0.26± 0.15	0.93± 0.12	3
	Pb	20±1	16±1	14±1	18±2	14±2	20±2	50
	Ni	10±1	10±2	13±2	10±2	15±1	47±4	50
	Cr	24±1	30±1	22±1	24±1	20±1	52±4	50

Further, total phosphate, total nitrogen, total organic carbon and C: N ratio were significantly different($p<0.05$) compared to the control. Importantly, the heavy metal content of Zn, Cu, Cd, Pb, Ni and Cr ranged 40-72 $mg.kg^{-1}$, 24-44 $mg.kg^{-1}$, 0.24-0.93 $mg.kg^{-1}$, 14-20 $mg.kg^{-1}$,10-50 $mg.kg^{-1}$, 20-52.4 $mg.kg^{-1}$ resulting a significant different ($p<0.05$) compared with the control sample. Similar studies done by Sharma et al. (2022) and Kutu (2019) also recorded the compost physicochemical values in the same range.

Seed Germination Results

Compost phytotoxicity is one of the critical parameters that determine the overall applicability of the compost to agricultural purposes. Referring to the results in Table 06, all the consortia inoculated compost samples (C1-C5) recorded a greater VI than was in the control compost, showing a significant difference ($p<0.05$) between the treatments. The GI value of C5 compost was recorded as 110.25 2.21% which was significantly ($p< 0.05$) higher than the control compost. Moreover, the GI value of the control (76.5 3.2%) and the C1 (72.4 4.2 %) compost was recorded below 80% which represents the presence of phytotoxic compounds in the compost. The GI values, which are less than 80% represent the presence of phytotoxicity in the compost as evidenced by the published studies conducted by Humaira et al. (2016) and Selim et al. (2012). Further, the GI values, which are greater than 100% represent that compost consists of no phyto toxicity, showing the potential applicability of compost as a phytonutrient (Humaira et al. 2016). Based on the results of the GI values (Table 6), the GI values of the control and C1 compost samples were recorded the GI values below 80% indicating the plant phytotoxicity. Further, C2 to C4 compost samples were within the acceptable range, indicating no phytotoxicity. The VI of green gram for the prepared five compost types ranged from 364.8 – 551, while the control compost sample (consortium not added) recorded the lowest VI value. This may be due to the detoxification mechanisms carried out by particularly C5 consortium, which may rapidly degrade the toxic secondary metabolites and complex compounds available in the MSW (Meena et al. 2019, Benavente et al. 2018).

Similarly, Liu et al. (2019) also reported a high level of toxicity among samples of fresh MSW compost due to the presence of heavy metals, including Cu, Cd, Pb, Ni and Cr. Even in the present study, the inoculated microbial consortium (C4 and C5) may efficiently convert the MSW into matured compost while reducing the phytotoxic effect. Moreover, the generation of short-term daughter compounds (short-chain organic fatty acids, phenols) during the initial phases of composting may also link to the record of a lower GI value in compost, particularly, which may be attributed to slower composting of the control treatment (Marouani et al. 2019, Mahongnao et al. 2024). Besides, the seed VI represents the compost maturity, the availability of nutrients and the presence of toxic substances in the compost. Referring to the VI values of the present study (Table 4), the overall greatest VI value was recorded in the C5 consortium inoculated compost, indicating the formation of quality compost. Furthermore, high seed vigor in compost often correlates with high microbial activity and a healthy microbial community (Bu et al. 2022).

Clean Index and Fertilizing Index

Table 7 represents the FI, CI and the compost class of the compost samples, which provides a comprehensive idea of compost stability and maturity. Based on the data, the FI and CI of the C5 compost samples were significantly different ($p<0.05$) compared to the control and to the other treatments. Moreover, the greatest FI was recorded in the C5 control sample, which indicates high fertilizing potential, whereas the lowest FI was recorded in the control compost sample, indicating the lower availability of nutrients. Only the C4 and C5 composts were categorized to compost class "A", which has good quality, whereas the control compost remained within the compost class "D", which has a lower quality index. Furthermore, the significant ($p<0.05$) increment of CI in the C5 inoculated compost may be attributed to the particular heavy metal detoxification mechanisms that may be undergone by the C5 consortium. The FI increment of C5 compost may be due to the complete and faster degradation of MSW to compost by the C5 consortium, which may increase the available nutrient composition in the compost.

Table 6: Seed germination and Vigour index of the prepared compost.

Parameter	Compost type					
	C1	C2	C3	C4	C5	Control
Seed Germination percentage %	85	88	94	96	100	74
Germination Index (GI)	72.4 4.2%	80.1 3.1%	84.5 2.2%	84.5 2.3%	110.2 2.2%	76.5 3.2%
Total root and shoot length (cm)	5.1 0.2	5.2 0.1	5.30.1	5.4 0.1	5.5 0.4	4.9 0.2
Vigour index (VI)	437.7	462.0	502.9	521.3	551.0	364.8

Table 7: Compost grade of prepared compost based on the clean index and fertilizing index.

Compost type	Fertilizing index	Clean index	Compost class
C1	3.2 ± 0.5	4.0 ± 0.3	C
C2	3.4 ± 0.3	4.6 ± 0.2	B
C3	3.3 ± 0.3	4.6 ± 0.5	B
C4	3.6 ± 0.2	4.6 ± 0.2	A
C5	4.5 ± 0.2	5 .0 ± 0.5	A
Control	3.0 ± 0.2	4.2 ± 0.2	D

The C2 and C3 compost types belonged to the compost class "B" whereas the control compost sample which prepared without addition of consortium belonged to the compost class "D". Importantly the overall quality of C5 compost were notably high which may be due to the synergistic interaction of C5 consortium which contains *Bacillus haynesii* (strain BHC1 -PP391133), *Bacillus amyloliquefaciens* (strain BAC1 -PP391056) and *Bacillus safensis* (Strain-PP391033). Several studies have been conducted on the FI and the CI of the MSW compost in the world (Bello et al. 2023, Aweez &

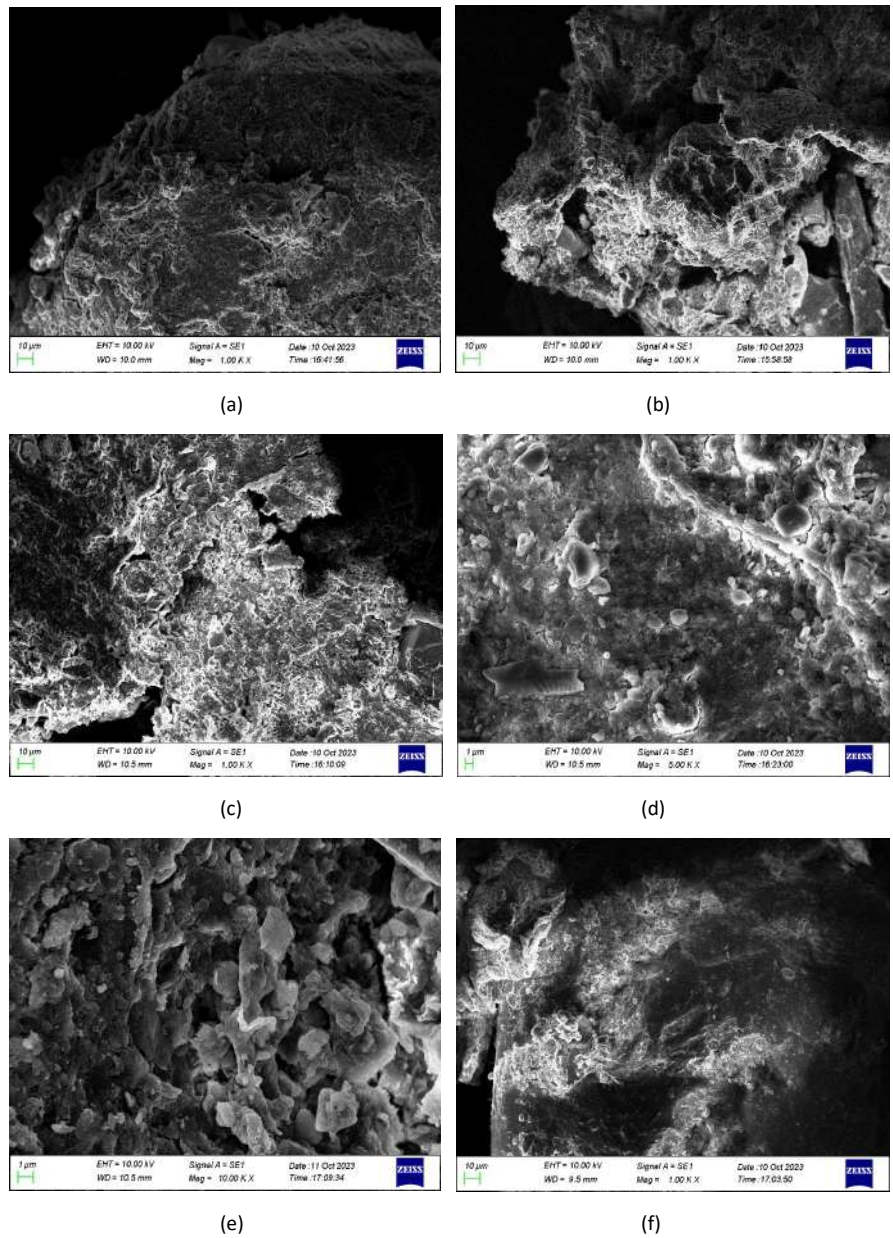

Fig. 10: Scanning Electron Microscopy images of C1 (a), C2 (b), C3 (c), C4 (d), C5 (e), Control (f).

Sadeeq 2019, Hameed et al. 2022, Wijerathna et al. 2024b). However, Kabasiita et al. (2022) have reported the FI and CI of MSW compost in Uganda, which ranged from 1.9-2.9 and 3.8 - 4.9, respectively. However, all the compost types generated in the present study had a greater FI, CI and compost class compared to those reported by Kabasiita et al. (2022), which may be attributed to the initial composition and MSW and the excellent performance of the inoculated consortium.

In the present study, the final compost quality of each treatment may have depended on the applied microbial consortia, which synergistically act with the existing environmental microbial community.

SEM results

Fig. 10 represents the SEM microstructural images of the prepared compost samples. The obtained SEM images represent the nature of the compost surface changes that occur when the solid waste degradation takes place by producing compost with inoculated five different consortia and a control. The formation of cavities and the destructed morphology represent the bacterial breakdown of solid waste into compost. The SEM images represent the surface and degradation of the MSW by inoculated microbial consortia.

Fig. 10 (a) represents the surface of the control sample, which indicates a dense, solid material, smooth and intact and

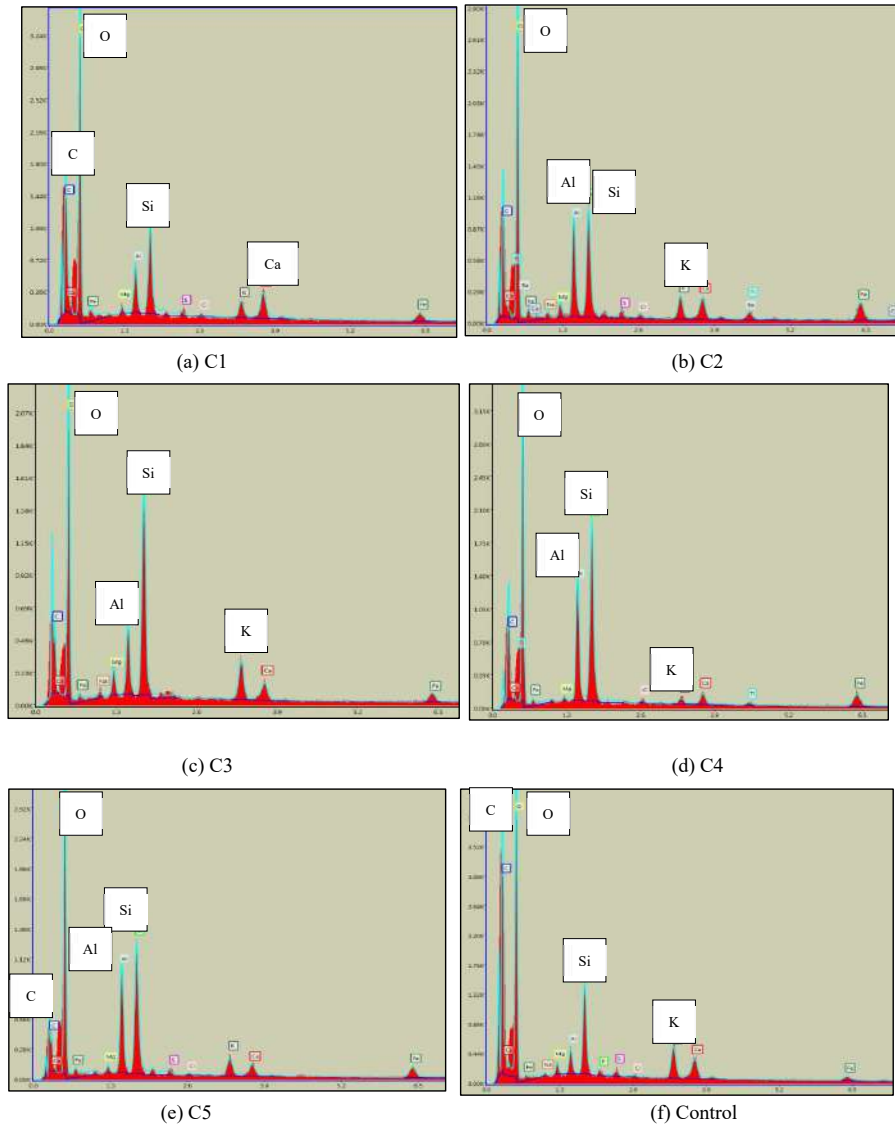

Fig. 11: Graphical representation of major elements in the compost samples from EDAX analysis.

compacted surface, indicating a relatively lower degradation of MSW. Compared to the control, the micrographs of all the bacteria-inoculated samples indicated more porous surface structures, indicating the faster degradation rate of solid waste by inoculated bacterial consortia. Furthermore, the C5 consortia inoculated sample (f) resulted from a noticeable surface degradation, demonstrating a very porous surface and a larger number of cavities represented by gaps and holes, a faster solid waste degradation and fragmentation of MSW. Porosity is an important factor of the compost, which is closely associated with the water-holding capacity of compost. A similar porosity pattern was recorded for the studies carried out by Pal & Singh (2024), and Arora & Kaur (2019 when MSW successfully converts to compost. Therefore, the microorganisms in the C5 compost may have synergistically interacted with environmental microbial communities to remarkably enhance the composting rate of MSW.

Manohara & Belagali (2017) have recorded a 41.51% carbon content in the compost samples collected from various composting sites in India when analyzed through EDX. The MSW compost includes various metals since municipal trash contains paints, electronic equipment, paper and plastic-based waste substances, chemicals, personal care products, and medications. The study revealed a decrease in these peaks as a result of a faster rate of breakdown that produced mature compost (Table 8).

Fig. 13 shows the compost specimens' XRD diffractograms. The existence of several important substances, such as the carbonate of calcium and silicon oxide, as well as minerals, such as quartz, calcite, and dolomite, was indicated by the strong peaks that appeared on the spectra of the XRD examination of compost material. The XRD pattern of immature compost is characterized by broad, diffuse peaks indicative of high amorphous content. In contrast, mature compost exhibits sharper, well-defined peaks corresponding to stable crystalline minerals, indicating a more advanced state of decomposition and stabilization (Benavente et al. 2018). In the XRD (Fig. 13), the control sample has given a large number of small peaks that are not sharp and well defined, indicating the dominance of immature compost, reflecting the presence of organic compounds that have not yet fully degraded.

However, the C5 consortia inoculated set-up exhibited several intense peaks, which are attributed to certain minerals (Calcite -$CaCO_3$, Quartz-SiO_2). Phosphates detected by XRD can serve as indicators of compost maturity, which is important for determining its suitability for agricultural use. Furthermore, as compost matures, the transformation of organic matter into humic substances and the precipitation of inorganic minerals become more pronounced, which is indicated by C5 compost. Sharma et al. (2019) have also noted several diffuse peaks indicative of high amorphous content in poorly matured compost produced in two different municipal solid waste dumpsites located in the cities of Solan and Mandi in Himachal Pradesh. However, the results of the C5 compost, which was produced from the novel bacterial consortia consisting of *Bacillus haynesii, Bacillus amyloliquefaciens, and Bacillus safensis*, have good agronomic value compared to the control and other consortia inoculated setups. The findings of the present study reveal the potential applicability of prepared C5 consortia as a greener solid waste management approach in the circular bio economy platform.

CONCLUSIONS

The seed germination assay confirmed the presence of phytotoxicity in control compost samples, whereas no phytotoxicity was recorded by the C5 compost sample,

Table 8: EDAX results of the analyzed compost samples.

C1		C2		C3		C4		C5		Control	
Element	Atomic %	Element	Atomic %	Element	Atomic %	Element	Atomic %	Element	Atomic %	Element	Atomic %
C	28.68	C	44.39	C	31.54	C	8.49	C	22.02	C	31.85
O	58.54	O	47.41	O	50.88	O	65.93	O	69.49	O	50.77
Mg	0.6	Na	0.18	Na	0.48	Mg	0.83	Na	0.77	Mg	0.29
Al	2.8	Mg	0.56	Mg	1.03	Al	8.81	Mg	2.76	Al	5.9
Si	4.53	Al	0.94	Al	4.97	Si	10.19	Al	5.41	Si	8.06
S	0.34	Si	3.03	Si	4.73	S	0.36	Si	8.37	Cl	0.2
Cl	0.11	P	0.07	S	0.38	Cl	0.15	K	3.73	K	0.4
K	1.17	S	0.23	Cl	0.25	K	1.91	Ca	1.85	Ca	0.82
Ca	2.2	Cl	0.06	K	1.36	Ca	1.37	Fe	1.59	Ti	0.21
Fe	1.04	K	1.54	Ca	1.52	Fe	1.97			Fe	1.51

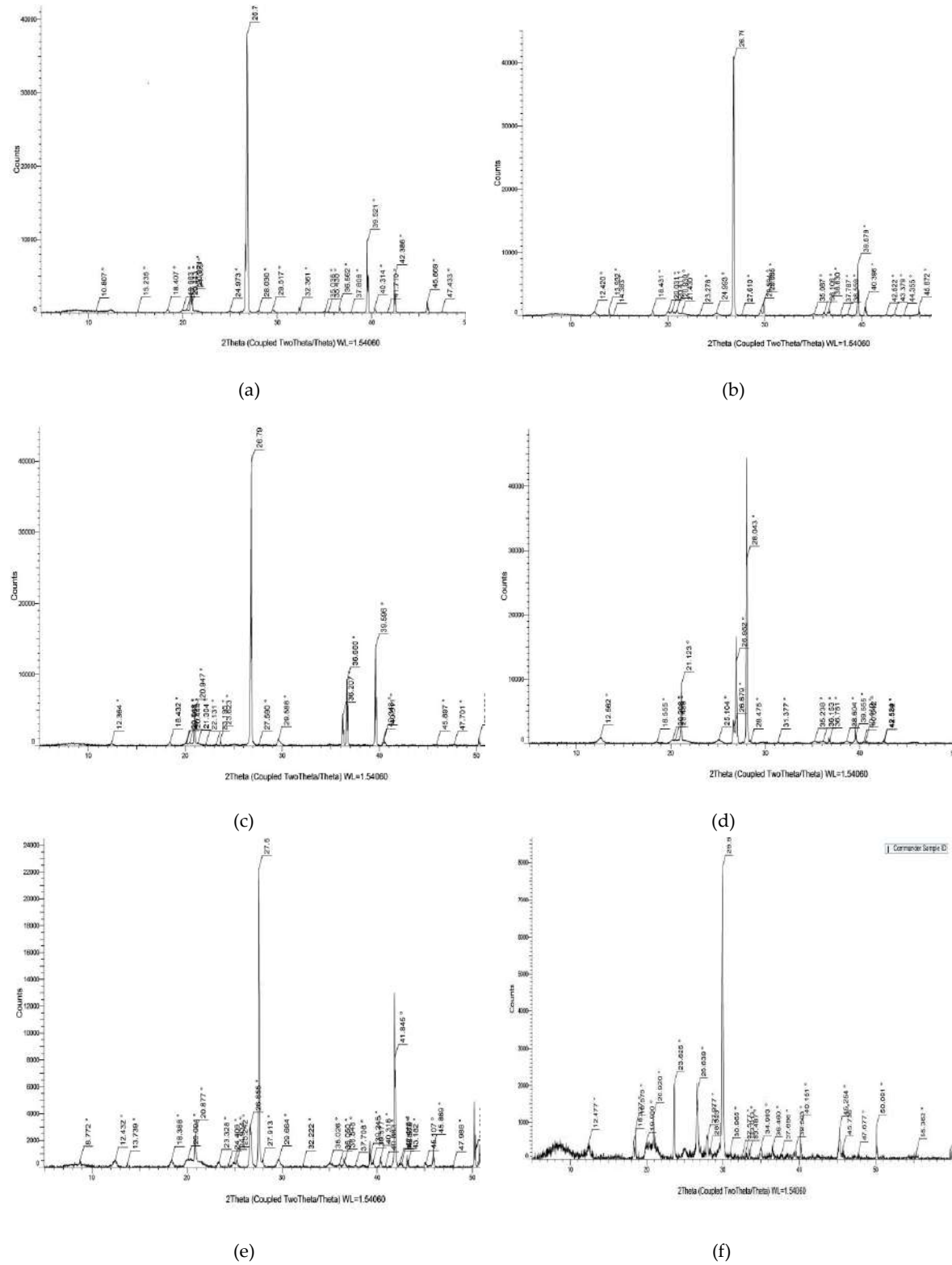

Fig. 13: XRD diffractograms of the compost samples (a) C1, (b) C2, (c) C3, (d) C4, (e) C5, (f) Control.

evidencing the potential applicability of C5 compost as a phyto nutrient. The indexing parameters and SEM, XRD and EDS spectrograms analysis confirmed the rapid waste degradation pattern and elemental composition alteration by the inoculated C5 consortium. The FI and CI values were also significantly high ($p<0.05$) in the C5 developed compost, showing a successful composting done by the C5 consortium. Based on the FI and CI, the resulting compost by the C5 consortium was categorized into to compost quality "A" category, which has high quality, whereas the control compost belonged to the compost quality "D" category, which has low quality. The results of this study evidence that the potential applicability of the C5 consortium as a rapid, greener waste management approach to produce high-quality compost from MSW.

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Air Aware IoT: Low-Cost Sensor Solutions for Urban Pollution Monitoring and Public Health

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ABSTRACT

Air pollution, especially in cities, has a considerable impact on human health and contributes to global morbidity and mortality rates. With urban populations increasing and public awareness of air quality being low, there is an urgent need for low-cost portable devices to monitor airborne contaminants in indoor and outdoor settings. This study presents the design and functionality of a low-cost, portable device capable of measuring major air quality parameters, such as gaseous pollutants (CO_2, O_3, TVOC, and $PM_{2.5}$) and physical indicators (i.e., temperature and humidity). The device connects various sensors to an ATmega microcontroller via a signal conditioning circuit, thereby solving current, format, and speed incompatibilities. The data processed by the microcontroller was sent to various devices using IoT technology. The device accurately measures ozone and $PM_{2.5}$, temperature, and humidity with precisions of ±5.02 $\mu g.m^{-3}$, ±7.94 $\mu g.m^{-3}$, ±0.67°C, and ±1.68%, respectively. The results demonstrate the dependability of the system for air quality monitoring, providing an affordable and accessible alternative for environmental surveillance. This innovation has the potential to raise public awareness and enable large-scale pollution monitoring, making it a useful tool for minimizing the negative consequences of air pollution on public health.

INTRODUCTION

Air pollution poses a significant threat to global public health, which is exacerbated by rapid urbanization and industrial growth (Kortoçi et al. 2022). Urban areas, characterized by dense populations, transportation networks, and industrial activities, are particularly vulnerable to elevated pollutant levels. Globally, air pollution contributes to over 7 million premature deaths annually due to respiratory diseases, cardiovascular disorders, and lung cancer, with 90% of the world's population exposed to pollutant concentrations exceeding the World Health Organization (WHO) guidelines (Jiang et al. 2016, Meo et al. 2021). These alarming statistics underscore the need for robust air quality monitoring systems to inform policy reforms and mitigate health risks (Perillo et al. 2022).

Existing regulatory monitoring networks, although critical, are often limited in spatial coverage and cost, restricting their deployment to specific urban zones (Baca-López et al. 2021). To address this gap, recent studies have emphasized the integration of low-cost sensors with traditional networks to enhance spatial resolution and data accessibility, particularly in underserved areas (Motlagh et al. 2021, Shindler 2021). However, conventional monitoring systems face challenges such as high power consumption, complex circuitry, and frequent calibration requirements, limiting their practicality for widespread deployment (Castell et al. 2013, Idrees et al. 2018).

Among these pollutants, particulate matter ($PM_{2.5}$) and ground-level ozone (O_3) are particularly hazardous because they can penetrate deep into the respiratory system and exacerbate conditions such as asthma and cardiovascular disease (Meo et al. 2021). $PM_{2.5}$ exposure is linked to elevated mortality rates, especially among vulnerable populations such as pregnant women and the elderly, whereas tropospheric ozone formed through reactions between vehicle emissions and sunlight poses severe risks in both indoor and outdoor environments (Cao & Thompson 2016). These health impacts necessitate affordable and portable devices capable of real-time multi-parameter monitoring to complement the existing infrastructure.

Recent advancements in IoT-enabled devices have shown great potential for enhancing air quality monitoring. However, many systems remain confined by low sensor precision, single-parameter concentration, and reliance on bulky or resource-bound hardware. For instance, wireless networks using ATmega328P microcontrollers have been deployed for indoor air quality monitoring but face memory constraints (Abraham & Li 2014), whereas mobile applications for outdoor pollution tracking often lack precision (Kodali & Sarjerao 2018). More recent advances, such as the IoT-based APM box, which incorporates machine learning to improve the calibration of low-cost sensors such as MQ-7 and MQ-131, provide greater pollutant measurement reliability (Rathnayake et al. 2024). Bidirectional Long Short-Term Memory (Bi-LSTM) models optimized with metaheuristic algorithms such as the Osprey Optimization Algorithm (OOA) have shown promising results for accurate $PM_{2.5}$ concentration estimation when paired with meteorological data (Saminathan & Malathy 2024). However, extensive calibration and environmental validation are required to ensure the wider applicability of these technologies.

This study aimed to design and validate a low-cost, portable, multi-sensor IoT device for real-time air quality monitoring in urban areas. To achieve this, a compact system was developed using a Raspberry Pi and an ATmega microcontroller to measure particulate matter ($PM_{2.5}$), ozone (O_3), total volatile organic compounds (TVOC), carbon dioxide (CO_2), temperature, and humidity. Although the primary emphasis of the system is on effective hardware integration, it presents a practical and scalable solution by strategically combining multiple low-cost sensors into a compact, portable, and IoT-enabled device. A custom signal conditioning circuit was implemented to address sensor incompatibilities, and Python-based software with IoT connectivity enabled real-time data visualization on both local and remote devices. Its novelty lies in the synergistic integration of diverse sensors for comprehensive air quality monitoring, real-time data transmission for accessible urban deployment, and adaptability for use across various environmental settings. This approach offers a scalable and cost-effective solution to address spatial coverage limitations in urban air quality monitoring, making it particularly suitable for city-wide deployment and community-based environmental tracking.

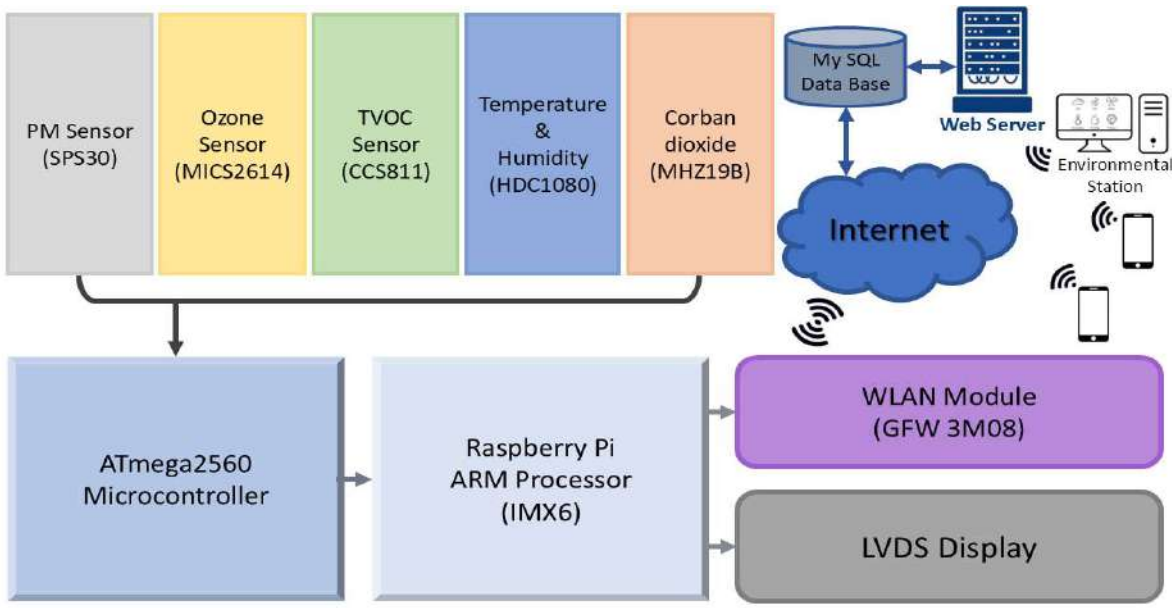

Fig. 1: Block diagram of a Portable device for measuring indoor and outdoor Air pollutants.

MATERIALS AND METHODS

Instrumentation

The hardware architecture of the system includes a processing unit with a Raspberry Pi, an ATmega microcontroller, and other critical components, as shown in Fig. 1. The sensors used in the sensing unit are listed in Table 1. Fig. 2 depicts the physical implementation of the portable device, emphasizing its compact and modular architecture. Fig. 3 depicts the data transmission process from the device to IoT-enabled platforms, highlighting their seamless connectivity and real-time monitoring capabilities.

To allow portability and outdoor use, the system was powered by a 5V USB supply, typically via a rechargeable power bank or wall adapter. It consumes approximately 350-400 mA during continuous operation, thereby enabling several hours of uninterrupted monitoring. As shown in Fig. 2, all the internal components are housed within a compact plastic enclosure. Although not industrially ruggedized, the casing provides basic protection against dust, light moisture, and handling, making it suitable for short-term urban and semi-outdoor deployments.

The sensing unit of the system incorporates various sensors, including the MICS 2614 ozone sensor, SPS30 particulate matter sensor, CCS811 TVOC sensor, MHZ19B CO_2 sensor, and HDC1080 temperature and humidity sensor. The system processing layer comprises an ATmega microcontroller(Al-Kofahi et al. 2019) and a Raspberry Pi (Vivek et al. 2017). Using these protocols, the ATmega microcontroller reads the sensor data through the signal conditioning circuit and processes the information.

The Raspberry Pi can run a software stack comprising an operating system, web server, database, and programming language. It has an execution speed of up to 1.2 GHz and can run operating systems such as Android and Linux. The communication layer of the system contains a WLAN module linked to the processor to transmit the sensor data to various IoT-enabled devices. The data analysis layer of the system displays the environmental parameters on a low-voltage differential signaling (LVDS) monitor and stores them in a database.

Most sensors used in this system provide factory-calibrated digital outputs, eliminating the need for manual calibration. The system uses temperature compensation and CRC-based checksum verification to achieve real-time software-level validation to guarantee data integrity, as shown in the software flow diagram (Fig. 4). The performance of the prototype was evaluated by placing it at a site where an Andhra Pradesh Pollution Control Board (APPCB) monitoring station was already in place. The

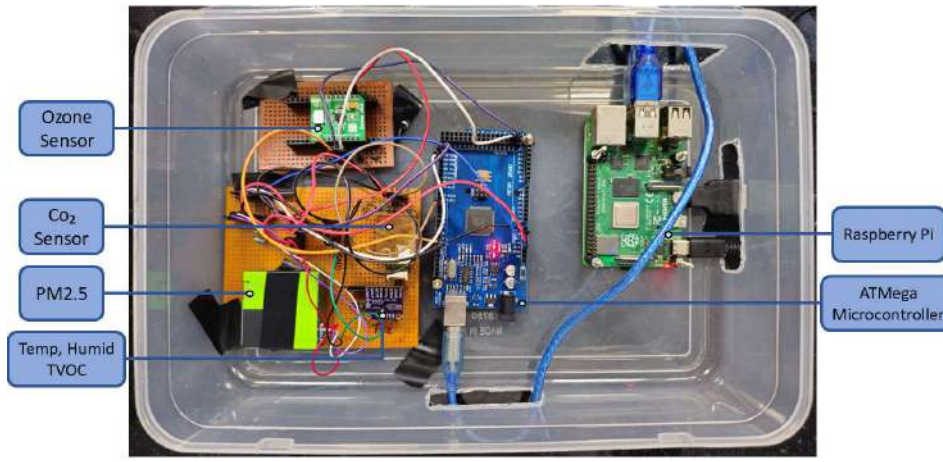

Fig. 2: Hardware Setup for a Portable Environmental Monitoring System.

Table 1: Sensors used in the sensing unit.

Sensor Name	Measured Parameter	Range	Accuracy
SPS30	Particulate Matter	0 to 1000 $\mu g.m^{-3}$	$\pm 10\ \mu g.m^{-3}$
MiCS2614	Ozone	10 to 1000 ppb	--
CCS811	TVOC	0 to 1187 ppb	±15%
MHZ19B	CO_2	0 to 5000 ppm	±50 ppm + 3% of reading
HDC1080	Temperature & Humidity	-40°C to 125°C, 0% to 100%	±0.2°C, ±2%

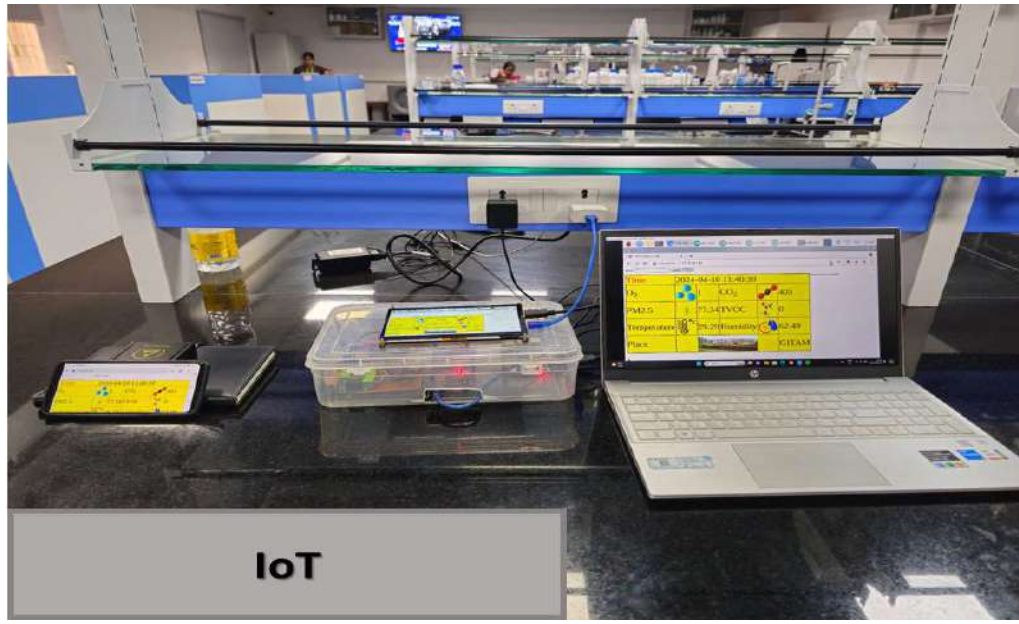

Fig. 3: Mechanism of Data Transmission to IoT Platforms.

APPCB reference values for that location were compared with the data collected by the device over 24 h. The accuracy of the system was robustly validated at the field level using statistical metrics such as Mean Absolute Error (MAE), Root Mean Square Error (RMSE), and Mean Bias (MB) to assess the results.

Interfacing of Particulate Matter Sensor

The SPS30 optical sensor is crucial for the precise measurement of 2.5-micrometre-diameter particles ($PM_{2.5}$) by leveraging laser scattering technology. Renowned for its stability and contamination resistance, the sensor features a built-in fan for controlled airflow regulated through an internal feedback loop. Airborne Particles passing through a laser beam generate light scattering, which is processed by algorithms on the SPS30 microcontroller, yielding an accurate mass concentration output. This comprehensive integration ensures robust performance under diverse environmental conditions.

Interfacing of Ozone Sensor

To monitor ozone concentrations, the MICS-2614, a MOS-type sensor, was employed with a measuring range of 10–1000 ppb. The sensor module integrates heating circuits along its edges and a sensing material. The heating circuit, powered by a voltage source, warms the sensing material by connecting the heater to the supply voltage through a resistor to form a voltage divider circuit. The MCP 3201 (ADC) operates within a voltage range of 2.7–5.5V and employs a successive approximation register (SAR) architecture. Communication between the microcontroller and ADC was facilitated using the SPI protocol. Eq. (1) is used to derive the ozone concentration from the measured ADC data.

$$\text{Ozone concentration [in ppb]} = 10^{\left(\frac{2566-(\text{ADC value}-584)}{1283}\right)} + 1 \quad ...(1)$$

Interfacing of TVOC Sensor

The CCS811 sensor, designed for measuring TVOC concentrations, integrates a microcontroller and an analog-to-digital converter. The analog output from the internal ADC was converted into a digital signal and processed by the microcontroller. Interfacing with the host microcontroller is established through the SDA and SCL pins, and operating within a current range of up to 30mA and a voltage range of 1.8 to 3.6 volts, the sensor maintains energy efficiency, consuming only 46mW.

Interfacing of Carbon Dioxide Sensor

An MHZ19B non-dispersive infrared (NDIR) sensor was used to monitor the ambient CO_2 concentration. The system consists of an infrared source, optical filter, detector, and gas chamber. The emitted infrared light closely matches the absorption band of CO_2, facilitating the accurate identification of CO_2 molecules. As light traverses the gas chamber, CO_2 molecules absorb specific wavelengths, and the detector measures the unabsorbed light and converts it into voltage. CO_2 concentration was gauged by analyzing the

on and off times of the PWM output connected to a PWM-compatible GPIO pin on the microcontroller. Additionally, the sensor was configured in the UART mode, which was connected to the transmitter and receiver pins of the controller. The on-time and off-time of the signal, extracted from the PWM output, were then substituted into Eq. (2) to derive the carbon dioxide concentration in parts per million (ppm). The integration of the PWM and UART modes enhanced the precision of the CO_2 measurement.

$$CO_2\ [\text{in ppm}] = \frac{Maximum\ detection\ range \times (T_{ON} - Cycle\ Start\ high\text{-}level\ output)}{T_{ON}+T_{OFF} - Cycle\ Start\ high\text{-}level\ output - Cycle\ End\ level\ output} \quad \ldots(2)$$

Interfacing of Temperature and Humidity Sensor

The HDC1080 sensor, connected to the microcontroller through the I2C protocol for atmospheric temperature and relative humidity measurements, operates as a digital moisture and temperature sensor. Notably, this factory-calibrated sensor eliminates the need for calibration by the user. The sensor boasts high precision, with ±2% accuracy for relative humidity and ±0.2°C for temperature.

Software Implementation

The software is responsible for controlling the different hardware devices in the system. The basic concept of embedded software is to control the operation of a group of hardware components without sacrificing their purpose or efficiency. The Raspberry Pi supports various software utilities, such as an operating system, web server, database, and scripting language for web development. In the present system, a Linux operating system is used, an Apache HTTP server is used to develop a web server, MySQL is used for database management, and the PHP scripting language is used for web development. The software implementation in this study was divided into two sections: the first was the Embedded C programming language, which was used to connect the sensors to an ATmega microcontroller. In the second section, the software stack, which runs on the Raspberry Pi, stores and distributes the data among the IoT-enabled devices. Fig. 4 shows the software implementation flowchart. The graphical user interface was built by creating PHP-scripted web pages that displayed the current environmental data and previous data stored in the database.

RESULTS AND DISCUSSION

Monitoring Locations and System Deployment

The system was engineered to assess air pollutant concentrations across seasons and explore variations in environmental conditions. Measurements at four locations in Visakhapatnam were compared with the Andhra Pradesh Pollution Control Board (APPCB) standards.

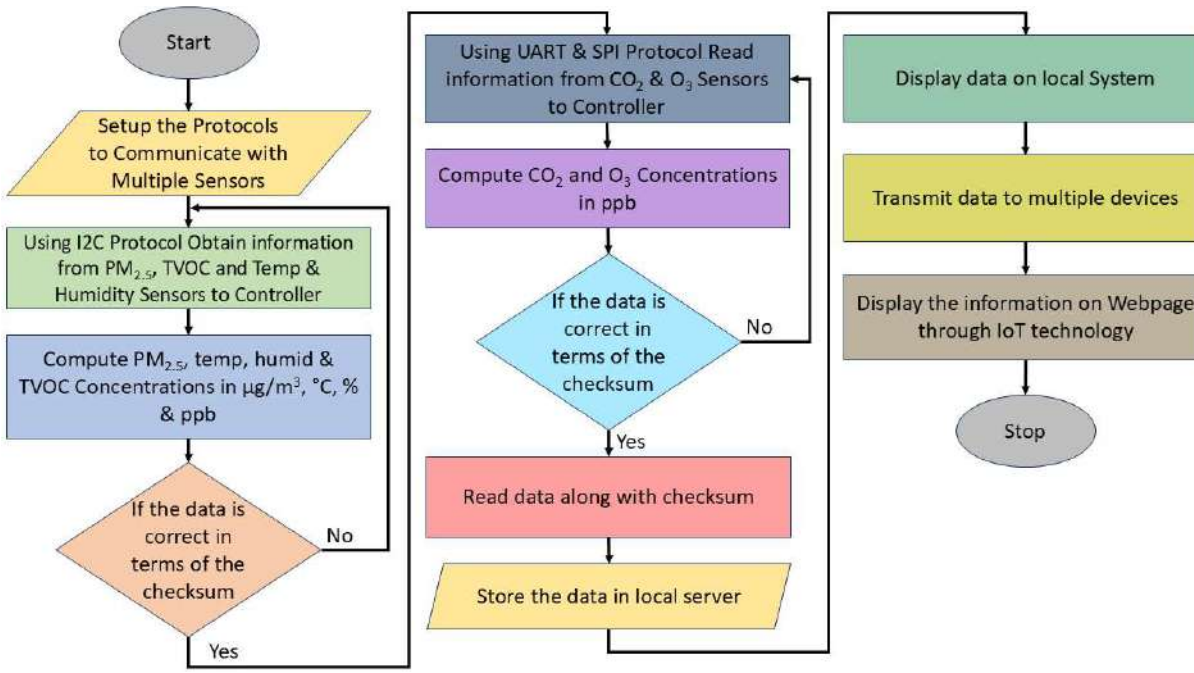

Fig. 4: Software flow diagram showing sensor communication protocols, temperature compensation, checksum validation, and IoT-based data transmission.

Continuous 24-hour measurements illuminated pollutant fluctuations in GVMC (Greater Visakhapatnam Municipal Corporation) (high population), PORT (industrial), NAD (Naval Armament Depot) (traffic), and GITAM (Gandhi Institute of Technology and Management) (green – Control Zone / Vegetative Zone). Fig. 5 illustrates the placement of the monitoring device at the GVMC and NAD locations. The system effectively measures O_3, $PM_{2.5}$, CO_2, TVOC, temperature, and humidity. The system allows for a rapid response to spikes in pollution levels. By monitoring these key pollutants and environmental conditions, the system provides valuable data for decision-makers to implement targeted interventions to improve the air quality in Visakhapatnam. This proactive approach can help mitigate the health risks associated with poor air quality and contribute to a healthier environment for urban residents. Furthermore, the collected data can be used to track trends over time and assess the effectiveness of the implemented interventions. This extensive monitoring system is essential for establishing a sustainable and healthy future for Visakhapatnam.

Sampling and Data Collection

The prototype was deployed for 24 h at each of the four monitoring sites, GVMC, PORT, NAD, and GITAM, during each of the four seasons: summer, monsoon, autumn, and winter. This resulted in a total of 96 h of monitoring per location. Environmental data was recorded at 30-second intervals, yielding approximately 11,520 readings per sensor per site (96 h × 60 min × 2 readings per min). Data integrity was ensured through checksum validation during the data acquisition process. No additional filtering or smoothing techniques were used. The validated data were then aggregated into hourly averages to align with the standard reporting intervals, enabling seasonal trend analysis and comparison with the APPCB reference data. A summary of the seasonal deployment, sampling strategy, and preprocessing steps is provided in Table 2.

Quantitative Error Analysis

The accuracy of the developed system was evaluated across various monitoring sites using statistical metrics, including the Mean Absolute Error (MAE), Root Mean Square Error (RMSE), and Mean Bias (MB). As shown in Table 3, the average values of O_3, $PM_{2.5}$, Temperature, and Humidity remained within acceptable limits, confirming the system's capability of consistent air quality monitoring. MAE values across pollutants were uniformly low, with minimal RMSE and bias, reflecting robust and precise sensor performance.

Fig. 5: The system was placed at NAD and GVMC locations in Visakhapatnam.

Table 2: Summary of Sampling and Pre-processing.

Location	Seasons Covered	Monitoring Duration	Sampling Frequency	Total Readings (per sensor)	Data Aggregation	Pre-processing Techniques
GVMC PORT NAD GITAM	Summer, Monsoon, Autumn, Winter	384 hours (24 h x 4 Seasons x 4 Locations)	Every 30 seconds	11,520	Hourly averages	Checksum validation only

Table 3: Average MAE, RMSE, and MB values for O_3 and $PM_{2.5}$, Temperature, and Humidity across all monitoring locations.

Location	MAE				RMSE				MB			
	O_3	$PM_{2.5}$	Temp.	Humidity	O_3	$PM_{2.5}$	Temp.	Humidity	O_3	$PM_{2.5}$	Temp.	Humidity
GVMC	0.719	2.480	0.540	0.342	0.636	3.667	0.595	0.055	-0.636	-3.667	-0.190	0.050
PORT	1.091	3.500	0.312	0.772	0.273	6.500	0.370	0.487	0.092	-6.500	0.145	-0.487
NAD	0.758	1.973	0.367	0.312	0.607	1.857	0.530	0.387	-0.607	-0.357	-0.380	-0.387
GITAM	1.134	2.417	0.382	1.080	0.878	1.885	0.448	0.715	0.633	-1.115	-0.412	0.440

Locations such as GVMC and NAD exhibited balanced metrics across all parameters, whereas PORT recorded higher $PM_{2.5}$ error, suggesting the presence of localized variability. Despite these differences, the system consistently produced accurate and reproducible measurements at all sites, demonstrating its suitability as a low-cost and scalable solution for urban environmental monitoring.

Correlation Analysis and Observations

Ozone dynamics: A spatial and temporal analysis: Throughout this study, the prototype was strategically positioned at four distinct locations, each corresponding to a unique season. Continuous measurements were meticulously conducted for a comprehensive 24-hour period. The collected data were then meticulously compared with the benchmarks established by the Andhra Pradesh Pollution Control Board (APPCB), India.

Higher O_3 levels were recorded at the three sampling sites: GVMC, PORT, and NAD. Although the values were higher than those in other seasons, they were within the limits, as sampling was done during the COVID-19 pandemic, when human and industrial activities were restricted. Higher concentrations of ozone can be attributed to localized pollutant emissions (Mohtar et al. 2018), whereas lower concentrations at GITAM are attributed to the dispersion of pollutants due to sea breezes (Latif et al. 2012). Although O_3 is not a primary pollutant, its formation is triggered by oxides of nitrogen and hydrocarbons when they react with sunlight. Owing to the tropical zone, the O_3 concentrations were recorded to be as high as 60 $\mu g.m^{-3}$. Nitrogen dioxide is an essential factor that influences the daily variations in O_3. The higher concentrations of O_3 during the late afternoon hours are attributed to the long daylight hours (Lv et al. 2022).

Fig. 6 presents Pearson's correlation analysis, highlighting linear positive relationships ranging from moderate to very high between the measured and standard ozone levels across the four locations and seasons. The heat map visually

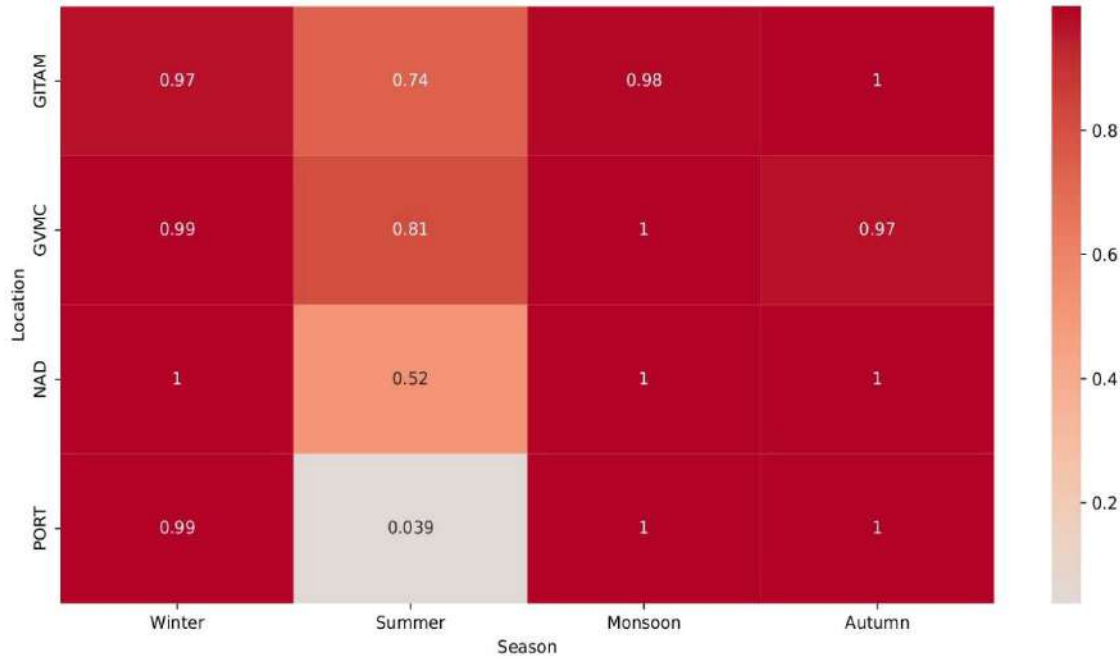

Fig. 6: Pearson Correlation of Seasonal Ozone Levels in Four Urban Locations.

depicts the correlation coefficients, showcasing the degree of correlation between the concentrations recorded by the developed system and the APPCB standards. The color intensity reflects the strength of these correlations, with values close to 1 indicating a strong positive relationship. This graph offers a concise overview of the seasonal consistency and reliability of the ozone measurements at each location.

The analysis revealed a consistently high correlation across most seasons and locations, indicating a strong alignment between the measured and standard O_3 levels. During the winter season, the correlations at all test locations were exceptionally high, ranging from 0.97 to 1. This suggests that the O_3 levels were almost perfectly aligned with the standards, reflecting reliable monitoring and stable environmental conditions. GVMC and NAD had reasonable correlations of 0.81 and 0.52, respectively, but the PORT location had a significantly low correlation of only 0.039. This rapid decline is most likely due to measurement errors. The monsoon season returned to significant correlations across all locations, with values approaching or equal to one, indicating that the measurements captured O_3 levels well during this period. This suggests that the accuracy of the measurements improved during the monsoon season, possibly because of less variability in the environmental factors. Overall, the data indicate that O_3 levels are more reliably captured during the monsoon season than during other times of the year.

$PM_{2.5}$ dynamics: A spatial and temporal analysis: The peak emission of particulate matter was noted between 6 AM and 12 PM and 12 PM and 6 PM near the GVMC during the Monsoon season. This diurnal variation is due to human activities, specifically vehicular movement by the population to work and businesses. $PM_{2.5}$ formed due to combustion is more hazardous as it can reach the lungs and bloodstream owing to its smaller dimensions. Changes in human activities have a significant impact on the differences in the concentration of air pollutants at various times of the day.

Fig. 7 visually represents the results of the bivariate correlation analysis, illustrating the dispersion between the concentrations measured by the developed system and the standard set by the APPCB. The Pearson correlation coefficient (r) values for the GVMC area ranged from 0.98 (winter) to 0.81 (autumn), indicating a robust and positive linear relationship. The PORT area coefficients varied from 0.92 (winter and monsoon) to 0.79 (summer and autumn), indicating a substantial correlation. NAD area exhibited correlations ranging from 0.98 (winter) to 0.92 (monsoon), underlining a commendable association. Similarly, the GITAM area demonstrated strong correlations, ranging from 0.99 (winter) to 0.89 (summer). These correlations demonstrate the effectiveness of the system in accurately capturing variations in $PM_{2.5}$ concentrations across different seasons and locations.

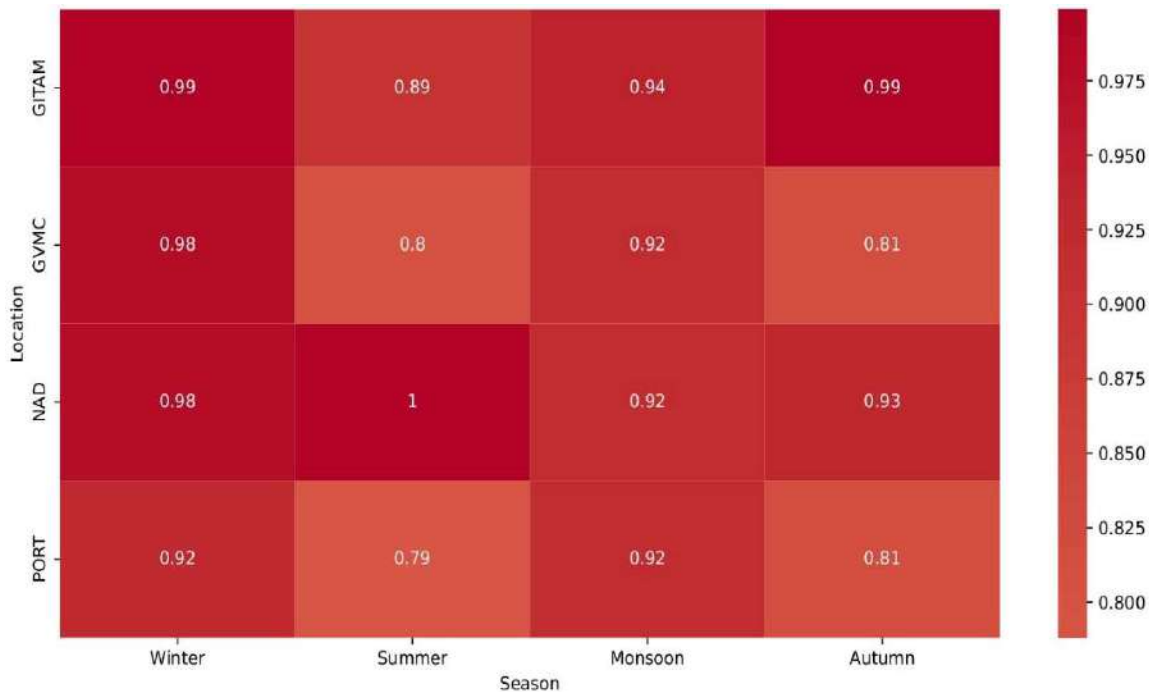

Fig. 7: Pearson Correlation of Seasonal $PM_{2.5}$ Levels in Four Urban Locations.

Temperature and humidity dynamics: A spatial and temporal analysis: GITAM had the lowest winter temperatures, most likely because of its green surroundings, which encourage cooling via flora. In contrast, the highest temperatures were recorded in the PORT region, which was ascribed to industrial activity that generated significant heat. Summer temperatures have increased in the GVMC, a densely populated area with significant urban heat island effects. During the monsoon, PORT and NAD had the highest temperatures, which were affected by industrial pollutants from PORT and heavy traffic on NAD. In the fall, the PORT area had the greatest and lowest temperatures, reflecting industrial heat during the day and cooling influences near the seaside at night. The average temperature concentration over seasons was compared to APPCB standard values, with an accuracy of ±0.67°C.

During winter, GVMC had elevated relative humidity owing to high human density and water vapor emissions,

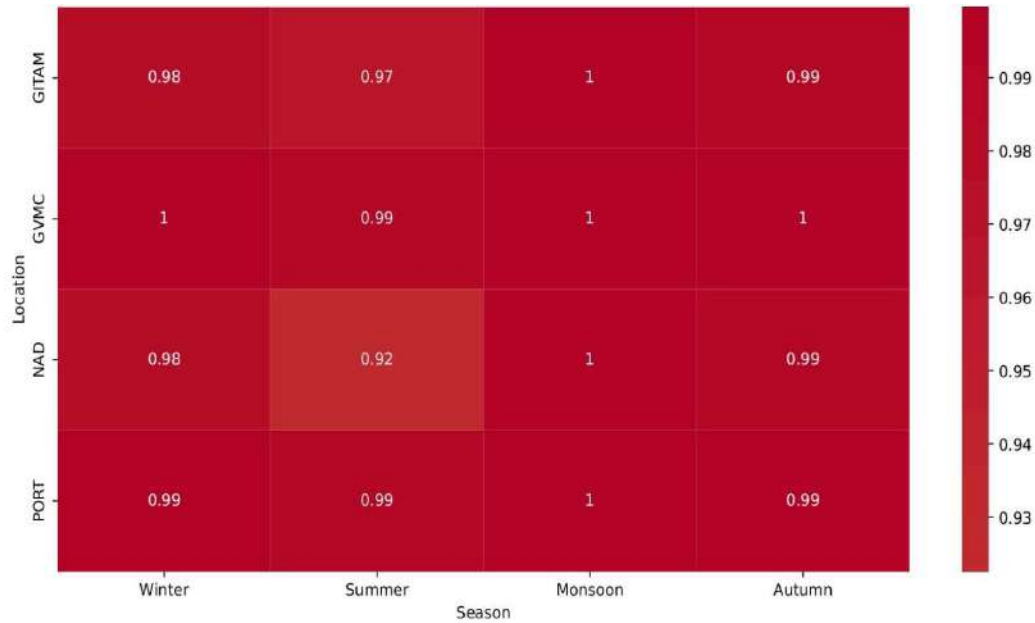

Fig. 8: Pearson Correlation of Seasonal Temperature Levels in Four Urban Locations.

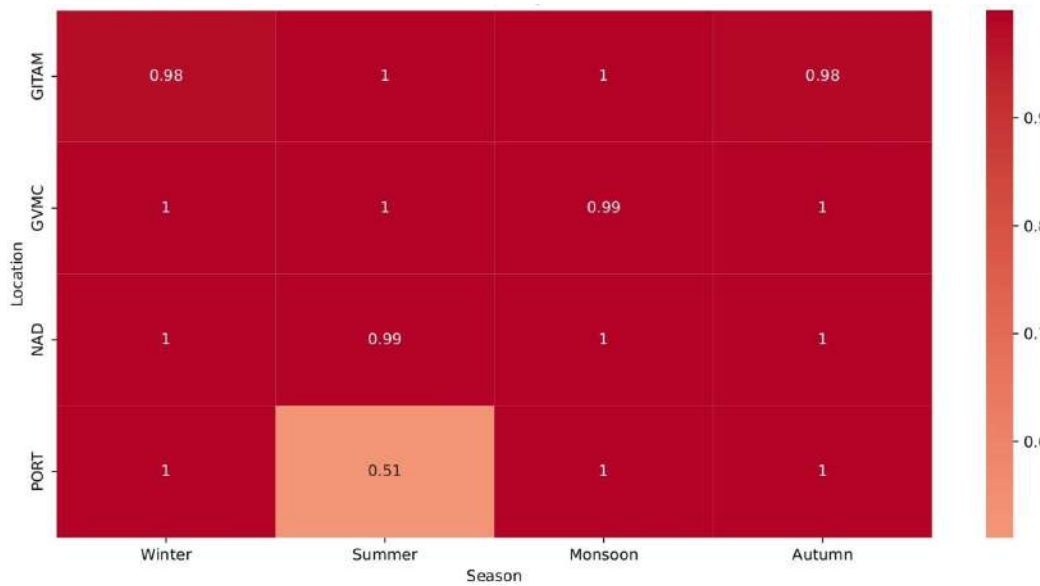

Fig. 9: Pearson Correlation of Seasonal Humidity Levels in Four Urban Locations.

whereas PORT had the lowest values, most likely because industrial heat lowered moisture. In summer, PORT had the highest humidity owing to its coastal vicinity and industrial cooling, whereas GITAM had the lowest humidity owing to its verdant surroundings and less human activity. During the monsoon, GVMC had the highest humidity, which was caused by rainfall and urban moisture retention, whereas PORT had the lowest humidity, presumably due to industrial pollutants. In autumn, GVMC again had the highest humidity owing to urban density and adjacent water sources, whereas PORT had the lowest owing to industrial heat. Across all seasons, the six-hour average humidity was compared to the APPCB requirements and achieved ±1.68% accuracy.

Figs. 8 and 9 visually portray the outcomes of the bivariate correlation analysis, showcasing the dispersion between the concentrations measured by the developed system and the standards set by the APPCB. Bivariate correlation analysis revealed strong positive relationships between the concentrations measured by the developed system and the standards set by the APPCB across all locations and seasons. In the GVMC area, the coefficients were consistently high, ranging from 0.99 to 1.00, indicating a robust correlation. Similarly, in the PORT area, the coefficients ranged from 0.99 to 1.00, indicating a strong and consistent positive correlation. In the NAD area, the coefficients showed a strong positive correlation ranging from 0.92 to 1.00. The GITAM area exhibited strong positive correlations, with coefficients ranging from 0.97–1.00 across different seasons. These findings emphasize the reliability and accuracy of the developed system for measuring pollutant concentrations under diverse environmental conditions.

In the GVMC area, a consistently strong correlation was observed across all seasons, with coefficients ranging from 0.99 to 1.00, indicating a robust agreement between the measured and standard values. The PORT area displayed a slightly lower correlation in summer (0.51), but in other seasons, it exhibited a strong correlation (1.00). Similarly, the NAD area maintained a robust correlation (1.00) across all the seasons. In the GITAM area, while maintaining a generally strong correlation, a minor decrease was observed in winter and autumn (0.98). These findings emphasize the reliability of the measurements of the developed system, which closely align with the established standards.

Seasonal Variation of Pollutants

Pollutant concentrations were recorded to be higher during the monsoon and autumn owing to adverse meteorological conditions that would enhance the accumulation of pollutants, which can be aggravated by increased humidity (Johnson 2022, Wang et al. 2022). Previous studies have also reported that lower temperatures and wind speeds enhance particulate matter concentrations, as the meteorological conditions tend to trap these pollutants, inhibiting their outward transportation. Furthermore, higher humidity and lower temperatures favor the conversion of semi-volatile species to the aerosol phase, leading to higher concentrations of particulate matter. Our study was in line with reports confirming higher concentrations of pollutants during the monsoon and winter, along with O_3, since the city's average temperatures during these seasons were the same as in other seasons.

CONCLUSION AND FUTURE WORKS

The current work presents the development of a compact air quality monitoring system built on a single-board computer and an ATmega microcontroller. The system integrates intelligent sensors to monitor O_3, $PM_{2.5}$, CO_2, TVOC, temperature, and relative humidity in both indoor and outdoor environments. Designed to be portable and cost-effective, the device operates with a 30-second sampling interval, enabling high-frequency data collection. Its IoT-enabled architecture supports real-time data transmission and remote access, making it suitable for individual use as well as community-scale monitoring. The low overall cost of the device enhances its scalability, allowing for broader deployment and improved spatial coverage in air quality assessments.

The system demonstrated consistent performance, with measurement accuracy validated against data from the Andhra Pradesh Pollution Control Board (APPCB). The observed accuracies for ozone, $PM_{2.5}$, temperature, and humidity sensors were ±5.02 $\mu g.m^{-3}$, ±7.94 $\mu g.m^{-3}$, ±0.67°C, and ±1.68%, respectively. Seasonal and spatial variations in pollutant levels were evident, with $PM_{2.5}$ concentrations notably higher during the monsoon season and peak traffic hours, highlighting the influence of both meteorological and anthropogenic factors.

While the system exhibited strong performance, certain limitations exist. The sensor accuracy under extreme temperature and humidity conditions was not extensively evaluated, and its long-term stability over several months of deployment is yet to be established. Future improvements may include weatherproofing to support extended outdoor use, integration of solar power for off-grid functionality, and cloud-based storage solutions to enable large-scale data aggregation and analysis. Additionally, the onboard Raspberry Pi presents opportunities to implement lightweight prediction algorithms capable of forecasting meteorological conditions or pollutant concentrations based on historical data. Such features could support early warning systems and enhance decision-making in environmental management.

The technology also shows significant potential for citizen science initiatives and urban-scale applications aimed at facilitating real-time, data-driven air quality interventions.

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Spatio-Temporal Assessment of Groundwater Quality in the Town of Moundou in South-Western Chad

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ABSTRACT

Assessment of groundwater quality is becoming essential for effective resource management. In this study, we conducted a seasonal assessment of groundwater quality, hydrogeochemical processes, and statistical analyses in the city of Moundou. A total of 62 groundwater samples were taken in the 27 districts of the city, in August 2022 (rainy season) and May 2023 (dry season), respectively. From a geochemical point of view, the results highlighted two dominant geochemical facies in both the dry and wet seasons: the calcium-magnesium bicarbonate facies (Ca-Mg-HCO_3) and the sodium-potassium bicarbonate facies (Na-K-HCO_3). The multivariate analysis showed that the mineralisation of gypsum ($CaSO_4 \cdot 2H_2O$), $CaCl_2$ salts, silicates, carbonates and the decomposition of organic matter are the main processes affecting the quality of Moundou's water. The physico-chemical results show that of the parameters monitored, only pH, iron and ammonium do not comply with the WHO standard, and almost 89% of the sites sampled are considered acceptable according to QWI values. Only the sites in the north-east of the city showed poor water quality during the rainy season. On the whole, this water is of better quality for irrigation.

INTRODUCTION

The degradation of groundwater quality worldwide has intensified in recent years due to uncontrolled industrial discharges, excessive use of chemical fertilisers in agriculture and inefficient management of water resources. (Khaldi et al. 2018). These factors alter the chemical composition of water, making it unsuitable for its intended uses. (Naima et al. 2022). Water resources, whether groundwater or surface water, are used by humans for a wide range of purposes (Ahoussi et al. 2010). The chemical composition of water from the natural environment varies considerably. It is influenced by the geological nature of the soil from which it comes, as well as by the reactive substances in contact during its flow. The presence of suspended and dissolved matter, whether of mineral or organic origin, affects the quality of groundwater, both quantitatively and qualitatively (Ngouala Mabonzo 2020). In developing countries, groundwater is a key resource for supplying people with drinking water. They generally offer satisfactory quality at a low cost, unlike surface water, which is expensive to treat and often inaccessible to nations with fragile economies, as is the case in many African countries (Yao et al. 2016).

In many parts of the world, particularly in developing countries, access to water is now essential for public health and socio-economic growth (Kettab et al. 2008). Of all the freshwater on Earth (2.53%), only 0.63% is groundwater. Groundwater is the world's main source of drinking water, as it is generally better protected from

pollution than surface water (Edith et al. 2023). Groundwater quality can deteriorate when it is exposed to harmful or even toxic substances as a result of human activities. Although essential to life, water can also become a vector for water-borne diseases due to its contamination by various mineral and organic wastes, as well as by excrement (Ndahama et al. 2014). +-In Chad, the expansion of major urban centres is leading to the establishment of new neighbourhoods in areas that are sometimes unsuitable for housing. At the same time, the water supply network is not expanding at the same pace as this growth, and the few neighbourhoods that are served suffer frequent interruptions. Faced with this situation, the most affluent households invest in human-powered boreholes, whose water quality is not systematically checked by qualified experts.

On the other hand, the most precarious households, located far from these boreholes, are resigned to using water from traditional wells (Mahamat et al. 2015). For several decades now, water quality has been a major issue, contributing to the spread of diarrhoeal diseases. In Chad, this problem affects almost the entire country, with greater intensity in the Saharan and Sahelian regions. To meet their water needs, whether domestic, agricultural or for livestock, people draw on traditional and modern boreholes, as well as rivers and ponds (Kriga et al. 2016).

Like most towns in Chad, the city of Moundou has a low drinking water supply. This situation can be explained on the one hand by strong urbanisation and exponential population growth. On the other hand, the drinking water supply network, with a coverage rate of 47%, is not keeping up with the high rate of urbanisation. In addition, the network is outdated, leading to water leaks. In addition, residents of neighbourhoods not served by the current network obtain their water directly from the river or from the surface water table, via boreholes and private wells. However, due to a lack of sanitation, these sources are often contaminated by the infiltration of wastewater, black water and other pollutants, leading to cases of water-borne diseases among the population (Urbaplan 2008). Despite this high anthropogenic pressure on water, very few studies have looked at the quality of groundwater in the town of Moundou. The only studies identified in the literature are those by Bandé (2016) and Schneider & Wolff (1992), which looked at hydrochemical facies, among other things. No study has examined the spatial and seasonal variation in water quality in the town of Moundou. This study was initiated to fill this information gap, which is crucial for the integrated management of the city's water resources. The aim of the study is therefore to assess the spatial and seasonal distribution of water quality in Moundou. This will involve determining the hydrochemical facies of the water, assessing the spatial and seasonal variability of the physico-chemical parameters and, finally, the potential uses (drinking water and water for agriculture).

MATERIALS AND METHODS

Study Area

Moundou is located in south-western Chad, in the Logone Occidental region, of which it is the capital. The country's second most populous city after N'Djaména, Moundou is considered to be the economic capital of Chad, due to the presence of numerous industries, particularly agri-food and brewing. It lies between 8˚33' and 8˚36' North and 16˚03' and 16˚06' East (Djako 2018). Moundou lies between 382 and 439 metres above sea level (Schneider & Wolff 1992). Demographic growth and accelerated urbanisation are leading to increasing pressure on water resources, with risks of pollution and overexploitation of groundwater (Mahamat et al. 2015). Moundou is located in the Sudanian climate zone, characterised by two contrasting seasons (the dry season and the rainy season). The rainy season runs from May to October, with average annual rainfall of between 900 and 1,200 mm, while the dry season extends from November to April and is dominated by the harmattan, a dry, dusty wind from the Sahara (Djebe et al. 2022). Temperatures are high, averaging between 25 and 35˚C, with peaks reaching 40˚C during periods of intense heat (Khaldi et al. 2018). The Moundou region belongs to the geological formations of the Terminal Continental, characterised by varied sedimentary deposits composed mainly of obliquely stratified sands and sandstones, multicoloured clays and kaolin and gravel and conglomerate lenses, bearing witness to ancient fluvial environments (Schneider & Wolff 1992). Underlying these formations is the Precambrian crystalline and metamorphic basement, which forms an impermeable barrier limiting the storage of groundwater (Mathieu 1980). Two types of aquifer dominate the hydrogeology of Moundou. The Continental Terminal water table, which is shallow (between 5 and 15 metres), is rapidly recharged in the rainy season and is highly vulnerable to anthropogenic pollution (latrines, waste water, solid waste, etc.). The Precambrian basement aquifers, which are continuous and located in rock fractures, are deep (between 50 and 150 metres) and generally of better quality, but require costly drilling (Schneider & Wolff 1992). Groundwater supply is therefore based on the joint exploitation of these aquifers, with disparities in quality and availability. The Logone River, which is the main watercourse and a source of supply for a number of uses (domestic, agricultural and industrial); Lake Wey and Lake Taba, located to the west and east of the town respectively, which contribute to groundwater recharge by infiltration

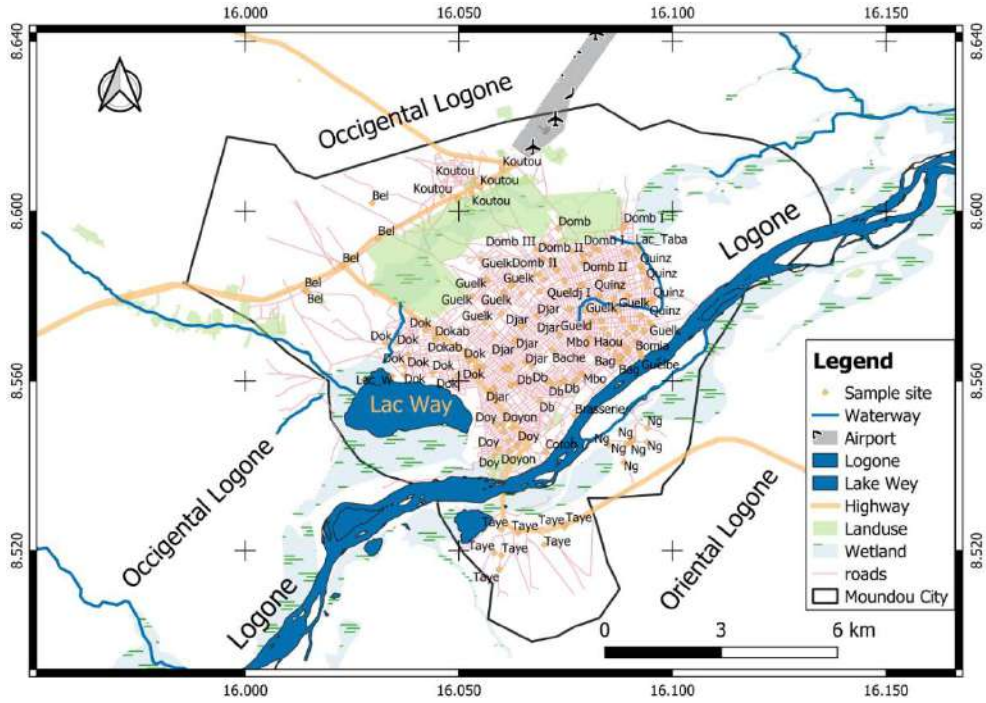


Fig. 1: Map of the study area and sampling points.

during flood periods; Lake Taba and the numerous temporary pools, which form during the rainy season and can influence the recharge of surface water (Djebe et al. 2022).

Sample Collection and Laboratory Analysis

A total of 62 groundwater samples from wells and boreholes were collected during the dry and rainy seasons. These samples were analysed at the Laboratoire National des Eaux (LNE) to assess their quality. The analyses focused on chemical parameters such as Na^+, K^+, Mg^{2+}, Ca^{2+}, HCO_3^-, SO_4^{2-}, NO_3^- and Cl^-. As soon as they were collected, the samples were placed in a refrigerated cooler and transported to the analysis laboratory. Samples were taken in the 27 districts of the town of Moundou, as shown in Fig. 1, which shows the geographical distribution of the collection points. Physical parameters, including pH, temperature, conductivity and turbidity, were measured directly on site using a multiparameter. The colour of the water was determined in accordance with French standard NF T 90-034. As regards chemical analyses, the concentrations of Fe^{2+}, Na^+, Cl^-, SO_4^{2-}, F^-, HCO_3^-, NO_3^- and NO_2^- were assessed in a specially equipped laboratory (LNE) in N'djaména, the Chadian capital. These analyses were carried out using a spectrophotometer, in accordance with the standard methods recommended by the French standards AFNOR.

Hydrochemistry of Groundwater

The Piper diagram is a widely used approach for identifying the hydrochemical type and facies of groundwater (Ousmane et al. 2022). According to this classification, groundwater is divided into main groups according to the dominant cations and anions: Mg^{2+}, Ca^{2+}, Na^+ and K^+; SO_4^{2-}, HCO_3^-, Cl^-. This diagram thus provides a better understanding of mineralisation processes and the geochemical evolution of groundwater. The factors influencing the chemical composition of groundwater will be analysed using the semi-logarithmic diagrams developed by Gibbs and McIntyre (1970). These diagrams represent the relationship between TDS and the ratios $Cl/(Cl+HCO_3)$ for anions, and $Na/(Na+Ca)$ for cations. According to this approach, the main mechanisms controlling the hydrochemical characteristics of groundwater can be grouped into three dominant categories: dominance of water-rock interaction, influence of precipitation, and impact of evaporation (Ngouala Mabonzo 2020). Interpretation of these diagrams provides a better understanding of the geochemical processes behind groundwater mineralisation.

Multivariate Statistical Analysis

For the study of groundwater in the city of Moundou, multivariate statistical analyses were performed to examine the interactions between the different hydrochemical

Table 1: Standard values, weights and relative weights of hydrochemical parameters (WHO 2011).

Hydrochemical parameters	Unit	Standard value (WHO 2011)	Weight [Wi]	Relative weight [Wi']
pH	-	6,5 - 8,5	4	0,085
Conductivity (CE)	$\mu S.cm^{-1}$	2500	5	0,106
TDS (Dissolved Solids)	$mg.L^{-1}$	500	5	0,106
Calcium (Ca^{2+})	$mg.L^{-1}$	100	2	0,043
Magnesium (Mg^{2+})	$Mg.L^{-1}$	50	2	0,043
Sodium (Na^{+})	$mg.L^{-1}$	200	4	0,085
Potassium (K^{+})	$mg.L^{-1}$	12	4	0,085
Bicarbonates (HCO_3^{-})	$mg.L^{-1}$	500	4	0,085
Chlorides (Cl^{-})	$mg.L^{-1}$	250	4	0,085
Sulphates (SO_4^{2-})	$mg.L^{-1}$	250	2	0,043
Nitrates (NO_3^{-})	$mg.L^{-1}$	50	5	0,106
Iron (Fe^{2+})	$mg.L^{-1}$	0,3	3	0,064
Ammonium (NH_4^{+})	$mg.L^{-1}$	0,5	3	0,064
			47	

parameters (Amroune 2018). In addition, the Pearson correlation matrix was used to identify relationships between hydrochemical parameters. A correlation coefficient between 0.5 and 0.7 was interpreted as a moderate relationship, while a value greater than 0.7 was considered highly significant (Bourjila 2023). Principal Component Analysis (PCA) was implemented to reduce the dimensionality of the data and identify the main sources of influence of groundwater hydrochemical parameters (Soro et al. 2019). When calculating the PCA, a rotation of the principal components was carried out using the Varimax method, allowing better interpretation of the results (Hassan & Firat Ersoy 2022).

Water Quality for Drinking

The Water Quality Index (WQI) is an essential tool for the overall assessment of the quality of groundwater intended for human consumption (Chaima 2024). It is calculated by assigning weights, ranging from 2 to 5, to the various hydrochemical parameters, according to their influence on water quality. First, the relative weights (Wi) were determined using equation (1), making it possible to quantify the impact of each parameter on water classification.

$$\mathrm{Wi} = \frac{wi}{\sum_1^n wi} \quad \ldots(1)$$

The weights assigned and the relative weights of each parameter are listed in Table 1 (Soro et al. 2022). In the second phase, the quality index (Qi) for each hydrochemical parameter was determined by calculating the ratio between the measured value and the reference standard established by WHO (2011), in accordance with equation (2).

$$Q_i = \frac{C_i}{S_i} \times 100 \quad \ldots(2)$$

Ci: The measured value of the parameters, Si: standard value of the parameters

In the final step, the water quality sub-index and the WQI are obtained using equations (3) and (4)

$$\boldsymbol{SL_I = Q_i x W_i} \quad \ldots(3)$$

$$\mathbf{WQI} = \sum_1^n \boldsymbol{SLi} \quad \ldots(4)$$

After calculating the WQI, Table 2 is used to classify the different types of groundwater quality.

To determine the areas where the risk of water pollution is high, the groundwater quality indices (WQI) calculated at each site were used as input data for extrapolating the WQI in the non-sampled areas using the inverse distance interpolation (IDW) method.

Water Quality for Agriculture

To assess the suitability of groundwater for agricultural purposes, Wilcox diagrams have been drawn using the value of the sodium absorption ratio (SAR) as a function of EC (Wilcox 1955). The SAR can be calculated by equation (5).

$$\boldsymbol{SAR} = \frac{\boldsymbol{Na^+}}{\sqrt{\frac{\boldsymbol{Mg^{2+} + Ca^{2+}}}{\boldsymbol{2}}}} \quad \ldots(5)$$

Table 2: WQI water quality index classification.

Classification	Groundwater quality
WQI <25	Excellent water
25 < WQI < 50	Good water
50 < WQI < 75	Poor water
75 < WQI < 100	Very poor water
WQI > 100	Unsuitable water

Table 3: The quality of groundwater for irrigation has been classified into four categories (Fallahati et al. 2020).

Class	Water quality for irrigation
C1S1	Sweet
C1S2 C2S2 C2S1	A little salty
C1S3 C2S3 C3S1 C3S2 C3S3	Salty
C1S4 C2S4 C3S4 C4S4 C4S3 C4S2 C4S1	Very salty

The groundwater quality for irrigation was categorized into four classes, as presented in Table 3.

RESULTS AND DISCUSSION

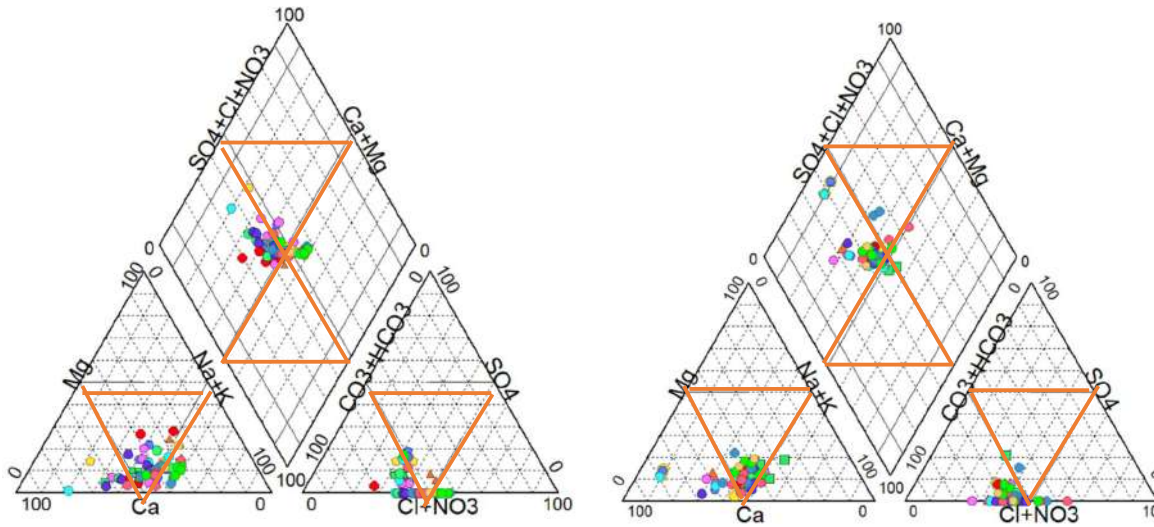
Global Statistics of Physicochemical Analyses

The overall characteristics of the hydrogeochemical variables used in this study concern the minimum-maximum values, the mean, the standard deviation and the coefficient of variation (Table 4). The average contents in the rainy season and in the dry season were compared with the admissible values for drinking water proposed by the WHO (2011). The results show that the pH of the groundwater of the city of Moundou was acidic, with an average of 5.14 in the rainy season and 5.77 in the dry season, i.e., an overall average of 5.45, which is lower than the limit recommended by the WHO (2011). The low conductivity values recorded suggest that the waters of this city are weakly mineralized. The average value of electrical conductivity in the rainy season (427 $\mu S.cm^{-1}$) is significantly higher than that in the dry season (262 $\mu S.cm^{-1}$), which corroborates well with the fact that the average value of most ions measured in the rainy season is also higher than that measured in the dry season. This seems contradictory because of the dilution phenomena that take place in the rainy season. This phenomenon could be explained by anthropogenic pollution sources, such as wastewater or agricultural activities, that contribute to an increase in ions in groundwater during the rainy season following infiltration and runoff. These waters are also soft and very aggressive, whatever the season. Of all the parameters monitored, only iron and ammonium are higher than the WHO standard. Iron would come from rocks, such as silicates, oxides, hydroxides, carbonates and sulfides or from organic matter, while ammonium has a purely organic origin. In the rainy season, in addition to temperature and pH, which have their standard deviation significantly lower than the mean, most of the monitored parameters have their standard deviation close to or higher than the mean, suggesting that all parameters except temperature and pH vary greatly from one sampling site to another. The same observations were made in the dry season, except that nitrate and ammonium did not experience a strong spatial variation. This low spatial variation of nitrate and ammonium indicates

Table 4: Statistical characteristics of physicochemical analyses (August 2022 and May 2023).

	Moyenne			**Écart**-type			Minimum			Maximum			WHO
	RS	DS	AN	RS	DS	AN	RS	DS	AN	RS	DS	AN	
pH	5.14	5.77	5.45	0.632	0.441	0.629	3.26	4.32	3.26	6.30	6.47	6.47	6.5–8.5
CE [$\mu S.cm^{-1}$]	427	262	345	572	266	452	14.5	15.5	14.5	3670	1000	3670	750
TDS [$mg.L^{-1}$]	213	152	182	287	176	239	7.27	3.86	3.86	1840	961	1840	500
Temp [°C]	29.2	30.0	29.6	1.21	1.60	1.46	26.2	25.1	25.1	33.0	33.7	33.7	-
Turb [NTU]	2.14	1.01	1.58	3.65	0.601	2.66	0.100	0.100	0.100	26.0	4.00	26.0	5
$CaCO_3$ [$mg.L^{-1}$]	43.6	31.0	37.3	54.2	39.0	47.5	4.00	0.400	0.400	300	300	300	-
Ca^{2+} [$mg.L^{-1}$]	12.9	9.58	11.3	14.8	10.6	12.9	1.40	0.100	0.100	80.0	80.0	80.0	75
Mg^{2+} [$mg.L^{-1}$]	2.74	1.71	2.23	4.48	3.20	3.91	0.01	0.00	0.00	24.3	24.3	24.3	30
K^+ [$mg.L^{-1}$]	1.64	1.05	1.35	2.05	1.13	1.67	0.100	0.00	0.00	14.0	7.00	14.0	30
Na^+ [$mg.L^{-1}$]	16.4	9.30	12.8	17.8	10.1	14.8	0.400	0.200	0.200	89.0	66.0	89.0	200
HCO_3^- [$mg.L^{-1}$]	53.2	35.3	44.2	66.4	34.8	53.6	3.70	0.500	0.500	366	244	366	500
Cl^- [$mg.L^{-1}$]	14.2	10.3	12.3	16.2	12.2	14.4	0.800	0.200	0.200	100	90.0	100	250
SO_4^{2-} [$mg.L^{-1}$]	4.73	2.66	3.70	7.71	8.80	8.30	0.00	0.00	0.00	36.0	63.0	63.0	250
NO_3^- [$mg.L^{-1}$]	12.7	6.32	9.50	15.9	4.93	12.1	0.660	0.200	0.200	88.0	37.0	88.0	45
Fe^{2+} [$mg.L^{-1}$]	0.0271	0.143	0.08	0.052	0.271	0.195	0.00	0.00	0.00	0.220	1.00	1.00	0.01
NH_4^+ [$mg.L^{-1}$]	1.82	0.740	1.29	8.29	0.248	5.92	0.110	0.110	0.110	66.0	1.12	66.0	0.25

DS = Dry season, RS = Rainy season, AM = Annual mean, Temp = température, Turb = turbidity



(a) Rainy season (b) : Dry season

Fig. 2: Piper diagram for the rainy and dry seasons.

that runoff plays an important role in the contamination of groundwater with nitrogen in this city.

Geochemical Facies of Groundwater

Piper's diagram allows us to characterize the chemical composition of the water by representing the distribution of the main cations (Ca^{2+}, Mg^{2+}, Na^{+}, K^{+}) and anions (SO_4^{2-}, Cl^{-}, HCO_3^{-}, etc.) (Piper 1944). As the 62 water points were sampled during both the wet and dry seasons, it makes sense to describe them by season. The geochemical facies given by Piper's diagrams are shown in Fig. 2a and 2b, which represent Piper's diagrams for the rainy and dry seasons, respectively. There was no significant difference between the two seasons, suggesting that season does not influence water facies in Moundou. In the lower left-hand section of each diagram, the dominant cations in all samples are calcium, followed by magnesium; while the anions, in the lower right-hand section, are dominated by HCO_3^{-} and sulfates (SO_4^{2-}), with a non-negligible contribution from chlorides (Cl^{-}). This results in two types of geochemical facies dominated by the calcium-magnesium bicarbonate facies (Ca-Mg-HCO_3), generally observed in recent, relatively undeveloped groundwater resulting from infiltration of precipitation and interaction with carbonate and silicate rocks (Olivier 2015), and the sodium-potassium bicarbonate facies (Na-K-HCO_3), which results mainly from chemical alteration processes of silicate rock minerals. A slight scattering of points suggests mixed influences, with possible anthropogenic input (urban and agricultural runoff) (Edmunds & Smedley 2000). These hydrochemical facies obtained by the diagram software reflect the local homogeneity of the geology of the town of Moundou, as indicated by Bandé (2016). Aquifers in sedimentary environments are more or less continuous, unlike those in basement environments, where several studies have revealed the discontinuous nature of the latter environment; this is linked to the polyphasic alteration that occurs there (Mfonka et al. 2021). These results are comparable to those obtained by Mfonka et al. (2024) at N'Djaména in Chad.

Groundwater Type and Facies Control Factors

The Gibbs diagram is a tool for identifying the dominant processes that control groundwater chemistry, as a function of total salinity (TDS - Total Dissolved Solids) and ionic ratios (Meryem 2021). He distinguishes three major processes, namely Precipitation Dominance (Influence of precipitation), Rock Dominance (Water-rock interaction) and Evaporation Dominance (Concentration by evaporation). The diagram in Fig. 3 (a) shows that the majority of points are located in the 'Rock Dominance' zone, with a high concentration which reflects the influence of rock alteration on groundwater chemistry; the presence of Na^{+} and Ca^{2+} suggests mineral alteration processes such as the dissolution of carbonates (calcite, dolomite) and silicates (feldspars) and finally a small proportion of samples are located in the 'Precipitation Dominance' zones showing that precipitation has a relatively limited influence on water chemistry. The diagram in Fig. 3 (b) shows a high concentration of all points in the 'Rock Dominance' zone, confirming that water-rock interaction is the main factor influencing the chemical composition of Moundou groundwater. The Gibbs diagram for the dry season in Fig. 4 (a) and (b) shows a similarity

to that for the rainy season, but there is a slight scattering of points in both the cation and anion diagrams. This phenomenon can be attributed to an increased concentration of dissolved salts due to evaporation, leading to a change in ionic ratios and greater variability in dissolution and mineral precipitation processes, linked to fluctuations in water table levels and greater mineralisation of groundwater. This result is consistent with that of Bouchagoura (2019). This analysis confirms the major influence of water-rock interactions on groundwater mineralisation, with a possible impact of precipitation and evaporation depending on the season

Multivariate Statistical Analysis (Correlation Analysis and Principal Component Analysis)

Correlation Analysis

The correlation matrix obtained from the rainy and dry season data is presented in Table 5. All the major ions showed a strong correlation with each other in both the rainy and dry seasons. Indeed, Ca^{2+} and Mg^{2+} correlated strongly with $CaCO_3$ (0.99 and 0.95, respectively), confirming their role in water hardness due to the dissolution of carbonates (calcite, dolomite). HCO_3- is also correlated with $CaCO_3$ (1.00), reinforcing the hypothesis of carbonate alteration as a major source of ions. Na^+ and Cl- show a strong positive correlation (0.96), suggesting a common origin, possibly the alteration of evaporitic rocks (halite) or anthropogenic inputs (saline intrusion, pollution). Sulphate (SO_4^{2-}) correlates well with Na^+ (0.86) and Cl- (0.77), suggesting a common source, such as dissolution of gypsum or other sulphide minerals. NO_3- was moderately correlated with CE/TDS (0.36), suggesting a possible influence of precipitation and infiltration containing nitrates of agricultural or domestic origin. In the dry season, iron showed no strong correlation with the other parameters, indicating that it could be due to reducing conditions (dissolution of iron oxides in oxygen-poor environments). In the rainy season, however,

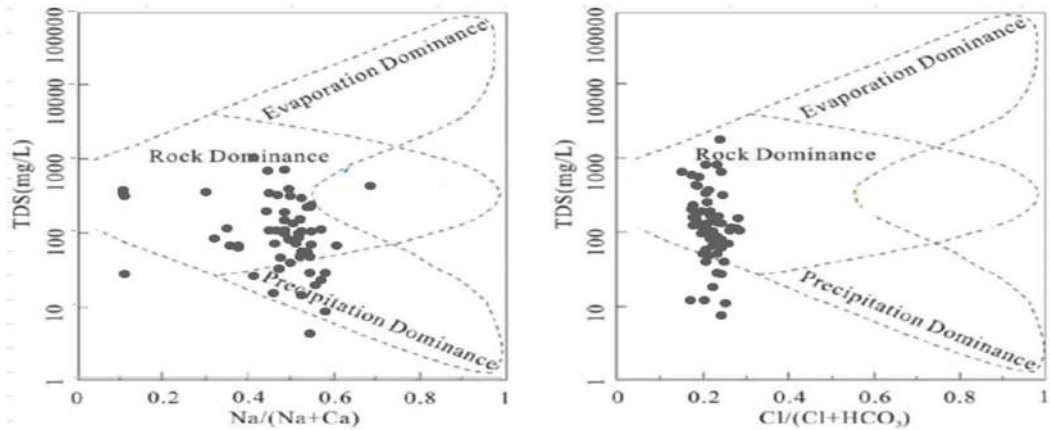


Fig. 3: Gibbs diagram for the rainy season.

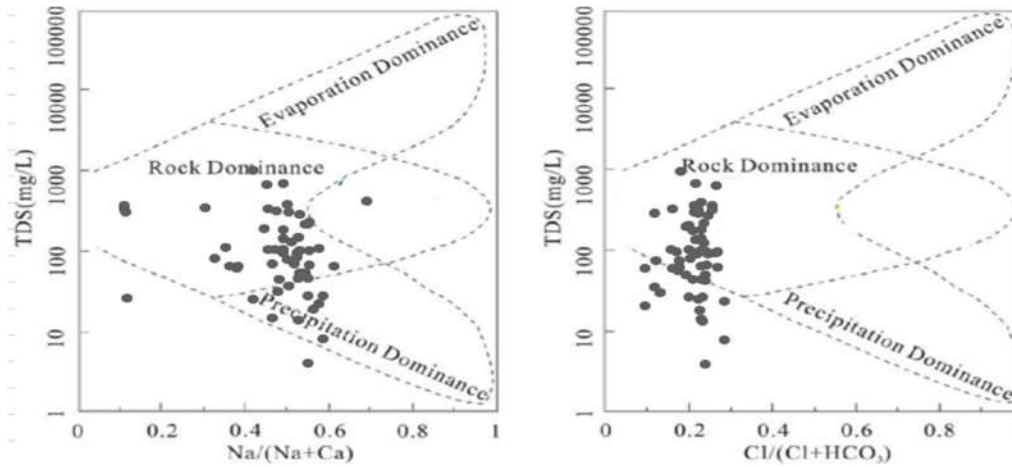


Fig. 4: Gibbs diagram for the dry season.

Table 5: Correlation matrix of physicochemical parameters in rainy seasons (SP) and dry seasons (SS).

SP	pH	CE	TDS	Temp	Turb	$CaCO_3$	Ca^{2+}	Mg^{2+}	K^+	Na^+	HCO_3^-	Cl^-	SO_4^{2-}	NO_3^-	Fe^{2+}	NH_4^+
pH	1															
CE	0,25	1,00														
TDS	0,25	1,00	1,00													
Temp	-0,37	-0,28	-0,28	1,00												
Turb	0,20	0,00	-0,01	-0,11	1,00											
CaCO3	0,20	0,32	0,32	-0,07	0,28	1,00										
Ca2+	0,21	0,29	0,29	-0,06	0,36	0,99	1,00									
Mg2+	0,16	0,36	0,36	-0,09	0,08	0,95	0,90	1,00								
K+	0,16	0,21	0,21	-0,06	0,10	0,55	0,55	0,52	1,00							
Na+	0,15	0,32	0,32	-0,02	0,21	0,97	0,95	0,94	0,58	1,00						
HCO3-	0,20	0,32	0,32	-0,07	0,28	1,00	0,99	0,95	0,56	0,97	1,00					
Cl-	0,17	0,32	0,31	-0,02	0,23	0,99	0,98	0,94	0,53	0,96	0,99	1,00				
SO42-	0,13	0,18	0,17	-0,01	0,16	0,79	0,79	0,75	0,63	0,86	0,79	0,77	1,00			
NO3-	0,07	0,36	0,36	0,05	0,23	0,87	0,86	0,84	0,50	0,92	0,87	0,88	0,68	1,00		
Fe2+	-0,04	0,09	0,09	0,25	-0,03	0,18	0,17	0,18	0,08	0,24	0,18	0,19	0,09	0,35	1,00	
NH4+	-0,21	-0,05	-0,05	0,13	-0,01	-0,05	-0,05	-0,05	-0,03	-0,04	-0,05	-0,03	-0,08	-0,02	-0,06	1,00

SS	pH	CE	TDS	Temp	Turb	CaCO3	Ca2+	Mg2+	K+	Na+	HCO3-	Cl-	SO42-	NO3-	Fe2+	NH4+
pH	1,00															
CE	0,24	1,00														
TDS	0,21	0,92	1,00													
Temp	-0,34	-0,13	-0,18	1,00												
Turb	0,13	0,33	0,37	-0,05	1,00											
CaCO3	0,27	0,48	0,45	-0,11	0,59	1,00										
Ca2+	0,29	0,48	0,45	-0,10	0,58	0,99	1,00									
Mg2+	0,23	0,47	0,44	-0,13	0,59	0,97	0,94	1,00								
K+	0,27	0,60	0,63	-0,15	0,42	0,87	0,86	0,85	1,00							
Na+	0,31	0,48	0,40	-0,13	0,45	0,93	0,92	0,92	0,91	1,00						
HCO3-	0,31	0,50	0,45	-0,13	0,50	0,98	0,98	0,93	0,90	0,97	1,00					
Cl-	0,29	0,49	0,44	-0,14	0,55	0,99	0,98	0,96	0,89	0,96	0,98	1,00				
SO42-	0,19	0,49	0,44	-0,08	0,59	0,93	0,89	0,96	0,81	0,90	0,88	0,91	1,00			
NO3-	0,31	0,42	0,44	-0,06	0,56	0,88	0,88	0,84	0,83	0,83	0,86	0,85	0,79	1,00		
Fe2+	0,03	0,23	0,28	-0,20	0,27	-0,12	-0,09	-0,17	-0,20	-0,24	-0,17	-0,16	-0,11	-0,14	1,00	
NH4+	-0,03	0,13	0,16	-0,12	-0,11	0,28	0,31	0,21	0,30	0,28	0,29	0,31	0,22	0,26	0,07	1,00

it was positively correlated with nitrate, indicating that some of the iron comes from anthropogenic activities. Ammonium (NH_4^+) is weakly correlated with the other parameters, which may indicate a local origin linked to organic sources (agricultural or domestic pollution or decomposition of organic matter). In the dry season, electrical conductivity (EC), total dissolved solids (TDS) and turbidity showed moderate positive correlation with all major ions, which was not the case in the wet season due to dilution.

Principal Component Analysis (PCA)

Principal component analyses were carried out to determine the relationship between physico-chemical variables and potential sources of groundwater pollution in the dry and rainy seasons. Concerning the number of principal components to be retained, we chose the eigenvalue analysis method (Table 6); thus, the factorial axes with eigenvalues of less than 1 were eliminated, which made it possible to retain the first four principal components in both the wet and dry seasons (Table 6). In the rainy season, the four

Table 6: Contributions of the components.

	Rainy season				Dry season			
	PC1	PC2	PC3	PC4	PC1	PC2	PC3	PC4
Eigenvalue	7.80	1.75	1.14	1.09	7.46	1.48	1.42	1.31
% variance	51.98	11.65	7.59	7.25	49.72	9.85	9.48	8.76
pH	0.11	0.72	0.22	0.18	0.23	0.82	0.21	-0.04
CE	0.34	0.5	0.09	-0.5	0.49	-0.03	0.07	0.4
Temp	0	-0.82	0.18	0.03	-0.02	-0.83	0.2	-0.12
Turb	0.22	0.15	0.01	0.82	0.07	0	0.73	0.48
CaCO3	0.98	0.09	0.06	0.08	0.96	0.12	-0.05	0
Ca2+	0.96	0.09	0.06	0.17	0.91	0.13	-0.07	0.02
Mg2+	0.94	0.09	0.05	-0.11	0.91	0.07	-0.01	-0.04
K+	0.63	0.11	-0.06	-0.06	0.87	0.07	-0.08	-0.1
Na+	0.98	0.03	0.09	0.01	0.95	0.09	-0.05	-0.13
HCO3-	0.98	0.09	0.06	0.08	0.96	0.12	-0.06	-0.07
Cl-	0.97	0.04	0.06	0.04	0.94	0.13	-0.11	-0.03
SO42-	0.84	0.02	-0.01	0.07	0.77	-0.09	-0.01	0.12
NO3-	0.91	-0.06	0.19	-0.03	0.64	0.16	0.17	-0.31
Fe2+	0.2	-0.34	0.72	-0.2	-0.23	0.11	-0.01	0.85
NH4+	0.04	-0.35	-0.69	-0.14	0.2	0.02	-0.86	0.19

principal components explained 78.5% of the total variance. The first principal component (PC1) explained 51.98% of the total variability and was positively correlated with all the major elements. The correlation matrix also showed a strong correlation between all major elements. Thus, PC1 reflects mineralization of natural origin (water-casement contact). The inclusion of nitrates in this batch indicates that PC1 also highlights mineralization of anthropogenic origin. The second principal component (PC2) explains 11.65% of the total variability. PC2 correlates positively with pH and electrical conductivity (EC) and negatively with temperature, highlighting the influence of climatic conditions on the mineralisation process. With 7.59% of the total variance explained, the third principal component (PC3) is strongly and positively correlated with iron and ammonium. This component seems to highlight the process of decomposition of organic matter. The fourth principal component (PC4), associated negatively with electrical conductivity and positively with turbidity, highlighting the dilution process resulting from the infiltration of rain water.

In the dry season, the four principal components explained 77.81% of the total variance (Table 6). The first principal component is positively correlated with all the major ions and electrical conductivity. PC1 explains the same processes in the wet and dry seasons. The absence of a strong correlation observed between major ions and electrical conductivity in the rainy season is linked to the dilution phenomenon. The second principal component (PC2) is positively related to pH and negatively related to temperature. PC2 also explains the same processes in the wet and dry seasons. PC3 is strongly and negatively correlated with ammonium and positively with turbidity, indicating pollution of organic origin. CP4 is positively associated with iron and turbidity, suggesting that iron is the cause of water turbidity.

Groundwater Quality Assessment

Water Quality for Drinking

Calculation of the WQI of 62 samples for the rainy and dry seasons made it possible to express the proportions of 5 classes of groundwater quality, as shown in Fig. 5 (a) and (b). Fig. 5 (a) shows a marked deterioration in water quality in the rainy season at certain points, such as the Lac Taba carré borehole, where the water was non-drinkable (1.61%); the Guelkoura 1 carré 4 well and the Guelbé Résidence Sœur borehole, where the water was of mediocre quality (3.23%); poor quality water was observed at the 15 ans II carré 3 well, the Doyon carré 2 well and the Mbomia carré 2 borehole (4.84%). 48.39 of % samples were of excellent quality, and 41.94% of the samples were of good quality. Unlike the rainy season, the dry season (Fig. 5 (b)) shows a reduction in pollution, as no water point was found to be either undrinkable or of poor quality. The percentage of poor quality water (4.84%) fell to 3.23%. More than half (54.84%) of the samples had excellent water quality, and

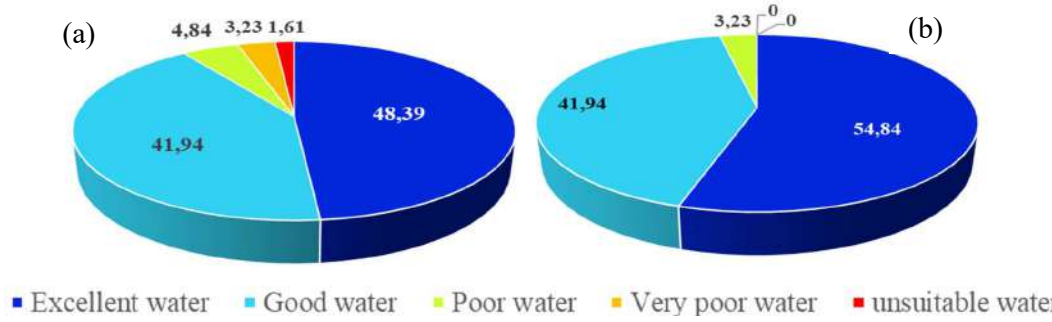


Fig. 5: Proportions of groundwater quality classes for the rainy season (a) and the dry season (b).

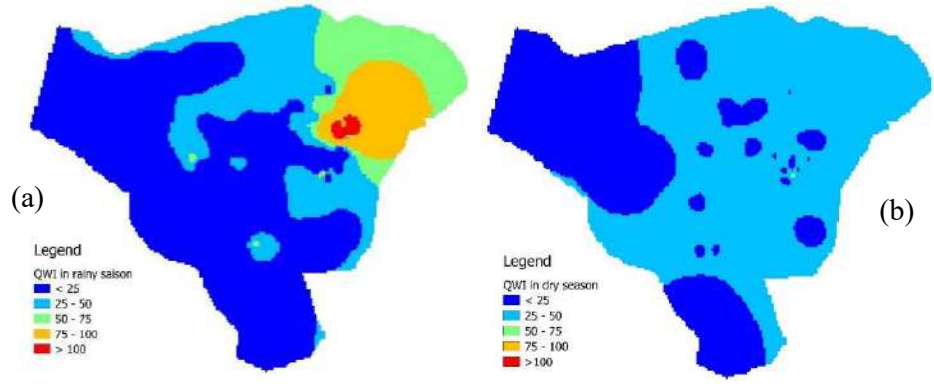


Fig. 6: WQI spatial distribution map of groundwater for the rainy season (a) and the dry season (b).

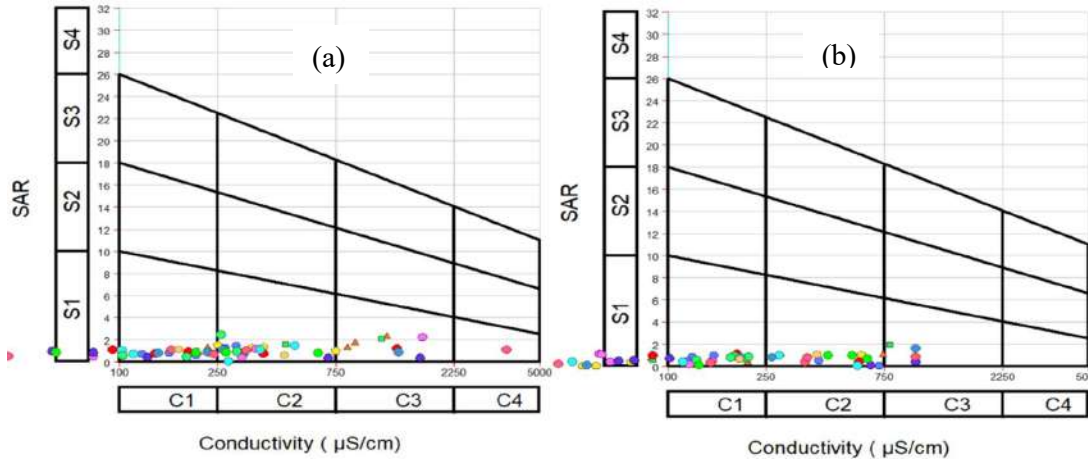


Fig. 7: Wilcox diagram for the rainy season (a) and the dry season (b).

41.84% of the samples had good water quality. Fig. 6 (a) and (b) for the rainy and dry seasons, respectively, show the spatial distribution of water quality indices on the map of the town of Moundou.

Water Quality for Irrigation

Overall, the distribution of points shown in Fig. 7 for the wet season (a) and dry season (b) shows that the majority of points are found in zones C1-S1 and C2-S1, suggesting low to medium salinity with a low Sodicity Index (SAR). Very few points appear in zones C3 and C4, indicating a low accumulation of dissolved salts. Finally, no points are located in zones S3 and S4, confirming a low risk of soil sodification. In the Wilcox diagram in Fig. 7 for the rainy season, the majority of samples are located in zones C1-S1 and C2-S1, indicating that rainfall dilutes the salinity of the

groundwater. The electrical conductivity (EC) is lower due to the freshwater input. One sample is located in zone C4S1, indicating high salinity, and a few samples are located in zone C3S1, reflecting medium salinity. In the dry season, a similar distribution of points is observed, but the electrical conductivity appears to be slightly higher, with the majority of points in zones C1S1 and C2S1, indicating a higher concentration of dissolved salts due to evaporation. A few points are also found in zone C3S1, suggesting average salinity. Nevertheless, the water is generally of good quality for irrigation, with no significant risk of sodification.

CONCLUSIONS

This study assessed the quality of groundwater in the town of Moundou according to seasonal variations and identified the main natural and anthropogenic influences on its chemical composition. Analysis of the hydrochemical facies revealed the predominance of calcium and magnesium bicarbonate facies (Ca-Mg-HCO_3), reflecting an interaction between seepage water and carbonate and silicate rocks. The sodium and potassium bicarbonate facies (Na-K-HCO_3) was also identified, resulting from alteration processes of silicate minerals. These facies were observed during the two seasons studied, with a slight dispersion in the dry season attributed to evaporation. Multivariate statistical analyses revealed sources of contamination of natural origin, linked to the weathering of rocks, as well as anthropogenic sources, notably industrial waste, fertilisers, pesticides, wastewater and metal carcasses. Assessment of drinking water quality revealed that pollution is more marked in the rainy season due to infiltration of run-off water, while in the dry season, quality improves with an increase in the number of samples showing good to excellent water quality. With regard to the suitability of groundwater for irrigation, the Wilcox diagram showed low to medium salinity, making the water suitable for irrigation in both seasons. However, vigilance is still required during the rainy season because of the increased risk of contamination. This study highlights the importance of rigorous monitoring of groundwater quality, particularly in urban areas, in order to prevent pollution risks and ensure sustainable management of water resources. Further investigations, including isotope studies and hydrogeological modelling, could provide a better understanding of the evolution of aquifers and the associated pollution mechanisms.

In light of the results of our study, we suggest that the local authorities in the town of Moundou extend the public drinking water distribution network, which currently has insufficient coverage (47%), in order to limit the population's reliance on polluted surface water. In addition, the authorities must ban groundwater abstraction for drinking water supply in the north-east of Moundou, identified as the area most vulnerable to pollution. Set up a permanent groundwater quality monitoring programme, particularly in densely urbanised areas, as well as an awareness-raising programme.

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Deciphering the Constraints Perceived by Farmers in the Adaptation of Climate-Resilient Technologies in the NICRA Village of Jharsuguda District in Odisha, India: RBQ and Kendall's Coefficient of Concordance Approach

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ABSTRACT

Climate change poses significant challenges to agriculture, necessitating the adoption of climate-resilient technology. Rising global temperatures, a direct consequence of climate change, negatively impact agricultural productivity, threaten farmers' livelihoods, and affect food availability. The studies suggest that this warming trend could lead to a 4.5-9% drop in crop yields, depending on how severe and widespread the temperature rise is. Since agriculture contributes approximately 17.4% to India's GDP, this decline in production could cost the economy up to 1.5% of its GDP each year. Thus, adopting climate-resilient agricultural technologies has become crucial in the current agro-ecological context. However, various socio-personal, financial, and technological constraints hinder their adoption. With this background, the current study was undertaken using an ex-post facto research design to uncover and quantify the constraints faced by the National Innovations in Climate Resilient Agriculture (NICRA) farmers in the Jharsuguda district of Odisha, India. The study identified these constraints using the Rank-Based Quotient (RBQ) method and ranking. The findings revealed that the strongest socio-personal constraint was a preference for conventional practices (RBQ = 84.44%). The lack of credit or capital (RBQ = 89.44%) ranked the highest among financial constraints. Among the technological constraints is the difficulty in implementing climate-resilient technologies (RBQ = 87.78%). The Rank-Based Quotient (RBQ) method, supplemented by Kendall's coefficient of concordance [W = 0.64, χ^2 (2) = 77.00, $p < 0.001$], indicated a strong consensus in the rankings. Data was collected from 60 NICRA farmers through structured interviews. The results highlight the need for targeted interventions, such as enhanced training, financial support, mechanization access, and timely input supply, to promote climate-resilient agriculture. Addressing these impediments will facilitate sustainable and adaptive farming systems in the region.

INTRODUCTION

Climate change poses a significant threat to agriculture and food security, making it a key priority under the United Nations Millennium Development Goals (Vinaya & Shivamurthy 2021). Its effects are felt worldwide, but countries like India, where agriculture sustains most of the population, are particularly vulnerable. Rising global temperatures directly result from climate change, harming agricultural productivity and jeopardizing farmers' livelihoods and food availability (Thakor & Joshi 2022). The increasing frequency and intensity of extreme weather events, such as droughts and floods, have profound implications for food security and the livelihoods of millions of farmers worldwide. Growing urbanization and industrial activity, coupled with climate change, have resulted in alarming levels of water shortages globally. Nearly one-fifth of the world's population lives in areas with

water scarcity (Kohli & Grover 2024). Risk can be minimized through structural and non-structural interventions, mass empowerment, planning strategies, and advanced awareness (Mallik et al. 2023b).

India has experienced notable climatic shifts over the past century, including a temperature rise of approximately 0.7°C, leading to more unpredictable and severe weather patterns. The Indian monsoon, which is crucial for the country's agriculture, has exhibited significant changes, with a gradual decline in rainfall over central India since the 1950s and a threefold increase in widespread extreme rainfall events between 1950 and 2015 (Harikrishna et al. 2021). Farmers perceive climate variability and identify increasing temperatures. Rain delays increase soil dryness, which is a critical factor affecting cultivation (Ratakonda et al. 2024). Rising temperatures are expected to have a significant impact on agriculture in India. According to the IPCC (2007), since 2020 temperatures have increased by 0.5-1.2°C, 0.88-3.16°C by 2050, and 1.56-5.44°C by 2080. Studies suggest that this warming trend could lead to a 4.5-9% drop in crop yields, depending on the severity and extent of the temperature rise (Naik et al. 2025). Since agriculture contributes about 17.4% to India's GDP, this decline in production could cost the economy up to 1.5% of its GDP each year. Recognizing the seriousness of the issue, the Indian government has placed greater emphasis on research and development to help farmers adapt to climate change and protect agricultural productivity (NICRA 2021).

India's National Action Plan on Climate Change has identified agriculture as one of the eight key national missions, aiming to sustain food production amidst climate change while promoting adaptation and mitigation strategies. In response, the Indian Council of Agricultural Research (ICAR) launched the National Initiative on Climate Resilient Agriculture (NICRA) in 2011, which was later renamed the National Innovations in Climate Resilient Agriculture under the XII Five-Year Plan (Rehman et al. 2021). NICRA focuses on enhancing agricultural resilience through research, technology demonstration, financial support, and capacity building. Although the initiative has introduced climate-resilient technologies in project villages, their adoption remains limited, reducing overall climate resilience. Recognizing these challenges, this study aims to analyze the constraints hindering farmers' adoption of climate-resilient technologies and explore strategies to improve their implementation (Rao et al. 2016).

Interconnected behavioral, economic, and institutional barriers constrain the adoption of climate-resilient agricultural technologies. Farmers' deep-rooted resistance to abandoning traditional practices emerges as the most persistent obstacle, often outweighing climate vulnerability perceptions (Jasna et al. 2015). This behavioral inertia interacts with structural challenges, including acute shortages of specialized farm implements, skilled labor deficits, and prohibitive upfront costs for essential infrastructure, such as drip irrigation and water storage systems (Mohokar et al. 2019). Region-specific studies reveal how these barriers manifest differently across agroecological zones in India. For example, pest outbreaks and flood vulnerabilities dominate in deltaic regions (Majumder et al. 2020), whereas semi-arid zones face compounded stresses from erratic input markets and institutional failures (Naik et al. 2022). Critically, extension system weaknesses appear universal, with poor Custom Hiring Center operations, untimely subsidy disbursements, and training programs that fail to account for farmers' literacy levels and technical comprehension (Shende et al. 2023). The constraints demonstrate spatial heterogeneity, with non-NICRA villages experiencing more severe knowledge and resource gaps (Acharitha et al. 2022), indicating that place-specific policy responses are necessary. Emerging evidence highlights how cultural perceptions and risk aversion mediate adoption decisions, requiring nuanced behavioral interventions alongside technological solutions (Shanabhoga et al. 2023). Therefore, a variety of adaptation strategies to mitigate the negative effects of climate change and maintain livelihoods are urgently needed (FAO 2009). Adaptation in the agriculture sector means addressing the negative impacts of climate change and making use of the opportunities that often come with a changing climate (Loria & Bhardwaj 2016). The existing literature has documented various adoption constraints; however, a critical methodological gap remains in systematically quantifying and prioritizing these barriers using robust statistical approaches. The proposed study addresses this gap by employing the Rank-Based Quotient (RBQ) and Kendall's Coefficient of Concordance to empirically rank the constraints faced by farmers in the NICRA villages of the Jharsuguda district of Odisha, thereby enabling targeted policy interventions for accelerated technology adoption.

Despite extensive studies on the constraints faced by farmers in adopting climate-resilient technologies, limited research has focused specifically on NICRA-adopted villages in Jharsuguda, Odisha. Most studies have addressed general challenges at the national or state level, but an in-depth, localized analysis of socio-personal, institutional, and technological constraints in NICRA villages in Jharsuguda is lacking. Furthermore, the role of extension services and government support in overcoming these constraints remains to be explored. Hence, the present study aims to document the constraints faced by farmers from the NICRA-adopted village during the adoption process of climate-resilient technologies

in the Jharsuguda district of Odisha. The study's findings are based on a small sample (n = 60) from a single NICRA village, limiting generalizability. As a single researcher, in social science studies, results are shaped by human behavior and localized interactions, making broad abstraction difficult. Future research with larger multisite samples is recommended.

MATERIALS AND METHODS

The research was conducted using an ex-post facto research design (Das et al. 2024). The Jharsuguda district of Odisha was purposively selected as the locale for this study. Farmers in the Jharsuguda district of Odisha face several climate-induced challenges that impact food production. These challenges include early season droughts in rainfed areas, untimely and unseasonal rainfall affecting both rainfed and irrigated lands, industrial accidents, and extreme events such as heatwaves, droughts and floods. To address these issues, the Krishi Vigyan Kendra (KVK) in Jharsuguda implemented the National Innovations in Climate Resilient Agriculture (NICRA) project in the villages of Bhoimunda and Tharkaspur, Odisha. Purposive and random sampling techniques were used in this study. From the Jharsuguda block, one village, Tharkaspur, was selected purposively as the location where the NICRA project was functioning. The respondents were selected through simple random sampling, resulting in a sample size of 60 respondents from the NICRA village.

A structured and pre-tested interview schedule was developed for the study. It was used to assess the socioeconomic profile of the respondents, along with identifying and quantifying the constraints faced by farmers. These constraints were categorized into the socio-personal, financial, and technological domains. The conceptual model of this study is illustrated in Fig. 1.

Each constraint was assessed based on the hindrance experienced during adaptation. The constraints were quantified using a three-point continuum scale of "severe," "moderate," and "low" constraints with scores assigned as 3, 2, and 1, respectively. The Rank-Based Quotient (RBQ) method was used to systematically compile, organize, and analyze the collected data. This approach involved ranking the identified constraints based on respondents' feedback and calculating the RBQ using the formula (Sabarathnam, 1988).

Constraints with higher RBQ scores were considered more significant, reflecting the severity assigned by the respondents. Rank-Based Quotient (RBQ) was calculated as follows:

$$RBQ = \frac{\sum_{i=1}^{n} fi(n+1-i)\times 100}{N\times n} \quad \ldots(1)$$

Where f_i = frequency of the respondent for the i^{th} rank of the problem

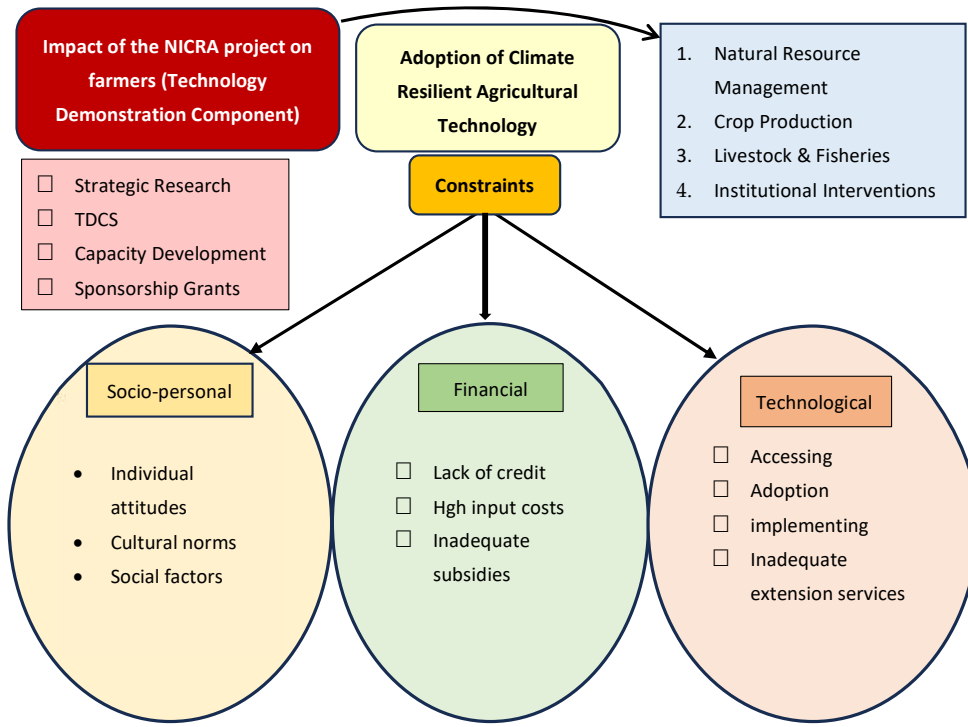

Fig. 1: Conceptual model of the research.

N = total number of respondents

n = Number of ranks

In addition, a comparison of various broad constraints used in the specific study was performed using Kendall's coefficient of concordance approach (Kendall & Smith 1939). As the ranks were tied between judges in the present study, the following formula was used to measure correction factors between tied ranks (Mallik et al. 2023a):

$$W = \frac{12 \sum_{i=1}^{n} (R_i - \bar{R})^2}{m^2 (n^3 - n)} \quad \ldots(2)$$

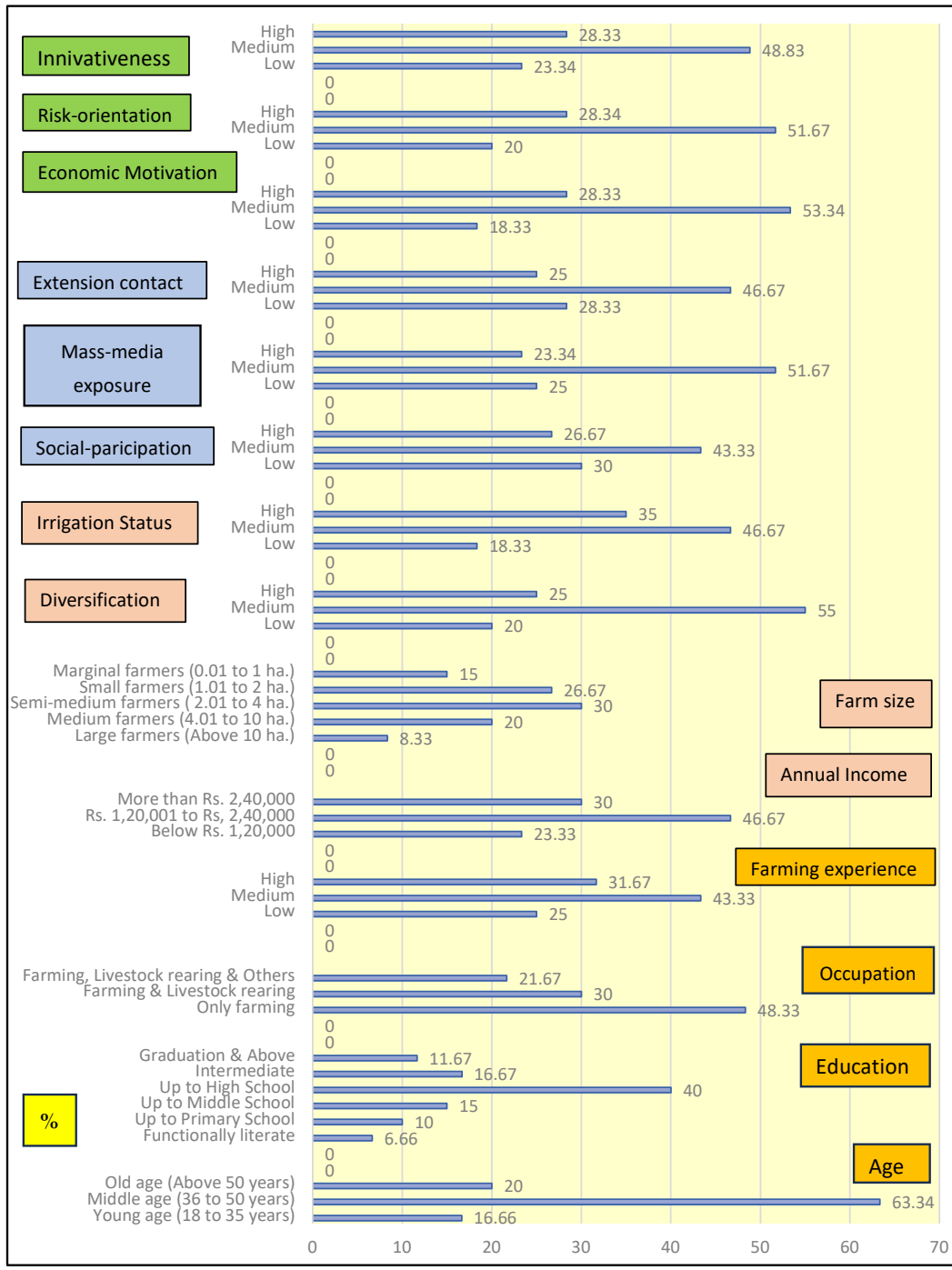

Fig. 2: Socio-economic profile analysis of Farmers from the NICRA Adopted Village (n=60).

m = number of respondents (farmers) = 60

n = number of items (constraint categories) = 3

Ri = total rank sum for the i^{th} category

= mean of the rank sums

Kendall's coefficient of concordance (Kendall's W) was used to evaluate the agreement or consistency between different judges or respondents to certain objects, ranging from 0 to 1, where zero denotes the nonexistence of agreement between judges and 1 denotes perfect agreement (Kendall & Smith, 1939). To validate the ranking consistency, Kendall's Coefficient of Concordance (W) was applied. Asymptotic significance tests are agreed upon assuming a large sample, while Monte Carlo significance offers robust validation for smaller samples through random resampling. Together, these confirm the statistical reliability of the constraint rankings.

RESULTS AND DISCUSSION

A detailed analysis of the socio-economic profile is illustrated in Fig. 2, revealing low levels of innovativeness (48.83%), risk orientation (51.67%), and economic motivation (53.34%), which hinder technology adoption. Limited extension contact (46.67%) and social participation (51.67%) further restrict knowledge dissemination in the region. Irrigation access

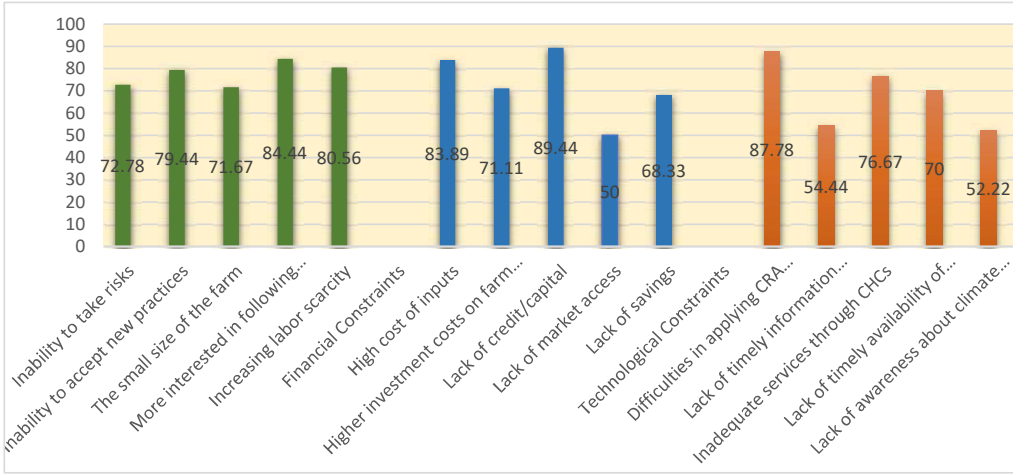

Fig. 3: The bar diagram illustrating the constraints based on their RBQ% score.

Table 1: Rank-Based Quotient (RBQ) Scores and Rankings for Constraints in the Adoption of Climate-Resilient Technologies by NICRA Farmers [Sample size (n)=60; *f* = Frequency, %= Percentage].

Constraint Socio-personal Constraints	Severe *f* [%]	Moderate *f* [%]	Lower *f* [%]	RBQ [%]	Rank
Inability to take risks	21 (35.00%)	29 (48.34%)	10 (16.66%)	72.78	IV
Inability to accept new practices	33 (55.00%)	17 (28.33%)	10 (16.66%)	79.44	III
The small size of the farm	25 (41.67%)	19 (31.67%)	16 (26.67%)	71.67	V
More interested in following conventional practices	36 (60.00%)	20 (33.33%)	4 (06.67%)	84.44	I
Increasing labor scarcity	35 (58.34%)	15 (25.00%)	10 (16.66%)	80.56	II
Financial Constraints					
High cost of inputs	33 (55.00%)	25 (41.66%)	2 (03.34%)	83.89	II
Higher investment costs for farm implements	24 (40.00%)	20 (33.33%)	16 (26.67%)	71.11	III
Lack of credit/capital	41 (68.33%)	19 (31.66%)	0 (00.00%)	89.44	I
Lack of market access	7 (11.67%)	16 (26.67%)	37 (61.66%)	50.00	V
Lack of savings	14 (23.33%)	35 (58.34%)	11 (18.34%)	68.33	IV
Technological Constraints					
Difficulties in applying CRA technologies at the farm level	44 (73.33%)	10 (16.66%)	6 (10.00%)	87.78	I
Lack of timely information related to CRA technologies	7 (11.67%)	24 (40.00%)	29 (48.33%)	54.44	IV
Inadequate services through CHCs	29 (48.33%)	20 (33.34%)	11 (18.33%)	76.67	II
Lack of timely availability of improved seed	22 (36.67%)	22 (36.67%)	16 (26.67%)	70.00	III
Lack of awareness about climate change	6 (10.00%)	22 (36.67%)	32 (53.33%)	52.22	V

(46.67%) was found to be inadequate, posing significant challenges in coping with climate variability. The study also observed low levels of diversification (55%), suggesting that most farmers rely on a limited range of crops and income sources, which increases their vulnerability to climate shocks. Most farmers had semi-medium landholdings (30%) and annual incomes between Rs. 1,20,001 and Rs. 2,40,000 (46.67%), and medium farming experience (43.33%). Education levels were mostly intermediate (16.67%), while the majority were young farmers (63.34%), indicating potential for future skill development. Farm size and behavioral variables suggest a trend wherein marginal and small farmers show disproportionately lower risk orientation and innovation levels. This implies that resource-constrained groups are not only financially vulnerable but also behaviorally hesitant, which collectively limits their responsiveness to climate-smart interventions. Conversely, semi-medium landholders, who form the majority (30%), tend to exhibit medium-to high-risk orientation, indicating a greater potential for technology adoption if appropriately supported. Understanding these factors is crucial for designing targeted interventions, addressing financial and technological constraints, and ensuring the successful adoption of climate-resilient agriculture within the NICRA framework. Collecting farmer-specific data helps identify region-specific challenges, enabling the design of targeted interventions that address financial constraints through institutional credit and subsidies, enhance extension services to improve awareness, and promote risk-taking behavior through demonstration programs. Therefore, integrating socioeconomic profile analysis with the constraints perceived by farmers is vital for ensuring the successful implementation of climate-resilient agriculture under the NICRA framework in the study area. Similar findings have been confirmed by Pise et al. (2018), Babu (2019), Pabba et al. (2021), Singh et al. (2022), and Naik et al. (2025) in their respective studies.

Table 2 presents the statistical analysis of the RBQ scores across socio-personal, financial, and technological constraints, revealing notable differences in the degree of variability perceived by respondents. Among the three categories, socio-personal constraints exhibited the highest level of consensus, as reflected by a low standard deviation (5.41) and a narrow RBQ range (12.77%). This suggests that challenges such as a preference for conventional practices, labor scarcity, and resistance to new methods are uniformly experienced and recognized by farmers. The relatively consistent responses within this category may be attributed to the shared cultural norms and behavioral tendencies prevalent in the study area. In contrast, financial and technological constraints showed greater variability, with standard deviations of 15.35 and 15.03, respectively. The financial constraint category, with the widest range (39.44%), indicates that experiences with issues such as credit access, input costs, and savings differed significantly among farmers, likely influenced by factors such as landholding size, income level, and prior access to institutional support.

Table 2: RBQ Variability and Dispersion Table by Constraint Category.

Category	Mean RBQ [%]	Standard Deviation	Minimum RBQ [%]	Maximum RBQ [%]	Range	No. of Constraints
Financial	72.55	15.35	50.00	89.44	39.44	5
Socio-personal	77.78	5.41	71.67	84.44	12.77	5
Technological	68.22	15.03	52.22	87.78	35.56	5

Red = Very high constraint (RBQ > 85%), Orange = High constraint (RBQ 75–85%), Yellow = Moderate constraint (RBQ 60–75%), Green = Low constraint (RBQ < 60%)

Constraints	RBQ Score (%)				
	Q1	Q2	Q3	Q4	Q5
Socio-Person					
Financial					
Technological					

Fig. 4: Composite Heat Map Depicting the Severity of Constraints in the Adoption of Climate-Resilient Technologies among NICRA Farmers.

Similarly, technological constraints, with a range of 35.56%, reflect uneven exposure and access to innovations such as Custom Hiring Centers and improved seed varieties. These variations indicate systemic disparities in service delivery and institutional outreach under the NICRA framework.

Table 1 shows the RBQ scores and ranks of the constraints. Fig. 3 illustrates a bar diagram of the constraints faced by the respondents, whereas Figs. 5,6, and 7 depict radar charts for each category of constraints, namely, socio-personal, financial, and technological constraints. As is evident from Table 1 and Figs. 3 and 4, among the socio-personal constraints faced by farmers in the selected NICRA villages, a strong preference for conventional practices (RBQ = 84.44%) was the most significant barrier. Farmers are reluctant to adopt new practices because of their deep-rooted belief in traditional methods. The second major constraint was the increasing scarcity of labor (RBQ = 80.56%), primarily due to a shift in the younger population toward industrial employment rather than in agriculture. As Jharsuguda is an industrially developed district, many farmers have diversified their income sources, contributing to the decline in the availability of agricultural labor. The inability to adopt new agricultural practices (RBQ = 79.44%) ranked third, largely influenced by climatic uncertainties such as droughts and erratic rainfall patterns, which created apprehensions regarding the viability of the new technique. The inability to take risks (RBQ = 72.78%) and small farm size (RBQ = 71.67%) were ranked fourth and fifth, respectively. Land fragmentation over generations has reduced farm sizes, making the large-scale adoption of climate-resilient agricultural technologies challenging. These findings are in alignment with previous studies by Mohokar et al. (2019), Acharitha et al. (2022), and Naik et al. (2022).

To address socio-personal constraints, regular training programs on climate-resilient technologies should be conducted to build confidence among farmers, particularly among smallholders hesitant to take risks. Large-scale demonstrations of successful climate-resilient practices can motivate farmers to transition from conventional to adaptive techniques. Additionally, policy interventions should focus on incentivizing agricultural labor to mitigate workforce shortages and ensure that youth remain engaged in farming activities.

As presented in Table 1 and Fig. 6, financial constraints are significant barriers to the adoption of climate-resilient agricultural technologies. The lack of credit or capital (RBQ = 89.44%) was identified as the most critical financial

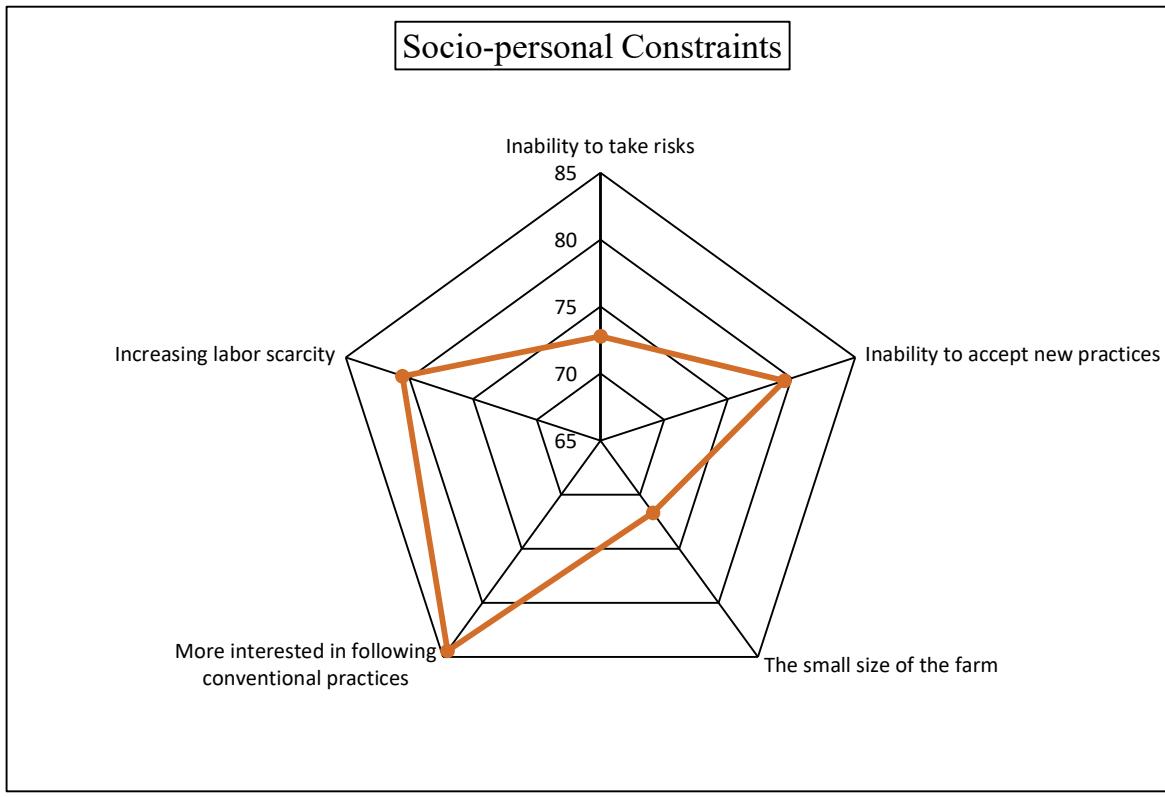

Fig. 5: Radar chart depicting the RBQ% in the context of socio-personal constraints faced by the respondents in the study area.

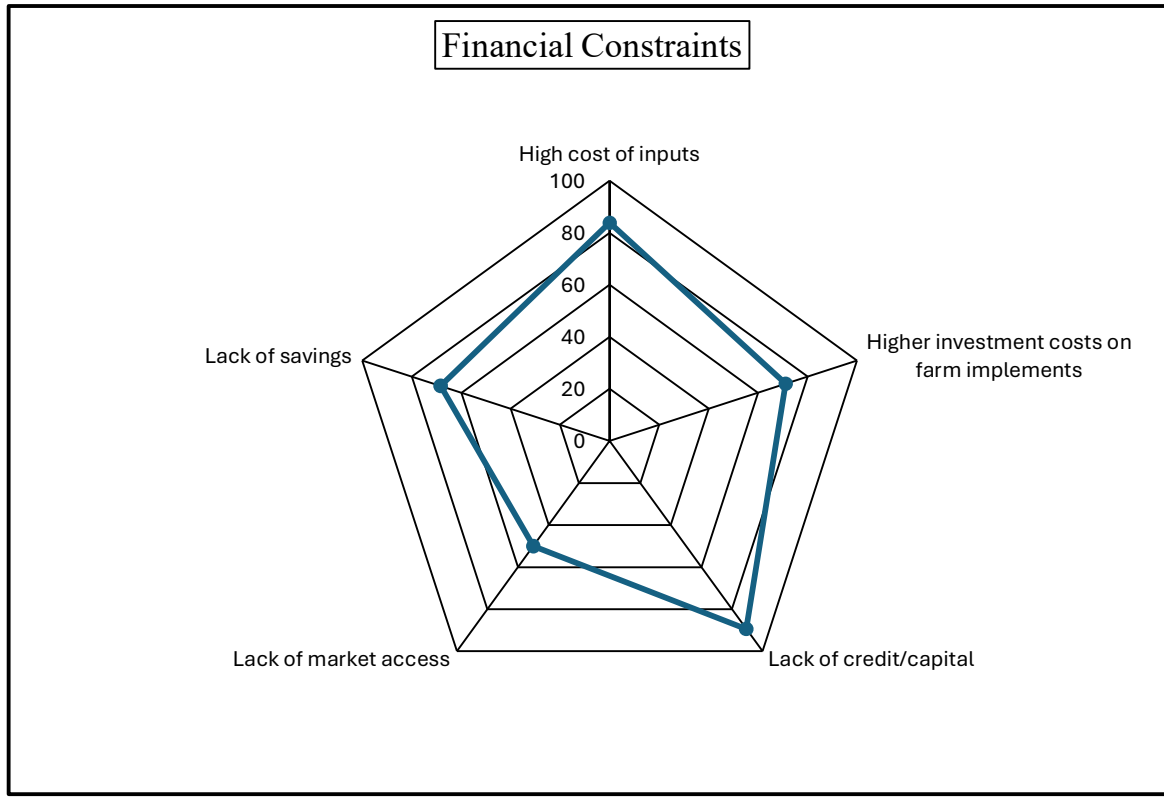

Fig. 6: Radar chart depicting the RBQ% in the context of financial constraints faced by the respondents in the study area.

limitation for the farmers. Most farmers are small and marginal, with limited access to institutional financial support and inadequate financial literacy, further restricting their ability to invest in climate-resilient technologies. The high cost of agricultural inputs (RBQ = 83.89%) was ranked second, as the escalating prices of seeds, fertilizers, and pesticides made technology adoption financially burdensome. The third major constraint was the high investment cost for farm implements (RBQ = 71.11%), as expensive machinery such as rotavators, planters, and seed drills remained out of reach for many farmers despite subsidy programs. The lack of savings (RBQ = 68.33%) was another notable constraint, as most farmers relied solely on agriculture for income, leaving little scope for investment in climate adaptation. Lastly, limited market access (RBQ = 50.00%) was ranked as the least significant financial constraint, although it still posed challenges for farmers in selling their produce at competitive prices. These findings align with those of Jasna et al. (2015), Pabba et al. (2022), Shanabhoga et al. (2023), and Shende et al. (2023).

Strengthening institutional financial support and enhancing farmers' financial literacy should improve their access to credit and capital. Implementing subsidy programs that specifically target small and marginal farmers can help make agricultural inputs more affordable to them. Establishing Custom Hiring Centers (CHCs) for mechanization at subsidized rates can be undertaken, thus allowing smallholders access to expensive farm implements. Promoting savings and cooperative banking models tailored to farmers' needs can enhance financial security. Improving market linkages and developing infrastructure to help farmers obtain better prices for their produce can also ensure economic viability.

Table 1 and Fig. 7 highlight that among the technological constraints, the most pressing issue was the difficulty in implementing climate-resilient technologies at the farm level (RBQ = 87.78%). Farmers find it challenging to adopt these technologies without adequate financial and technical assistance. Additionally, the lack of coordination among farmers in collective decision-making and resource sharing further hindered implementation. Inadequate services from Custom Hiring Centers (CHCs) (RBQ = 76.67%) ranked second most significant constraint, as farmers expressed dissatisfaction with the limited availability of mechanization services during peak agricultural seasons. The third constraint was the lack of timely availability of improved seeds (RBQ = 70.00%), which created difficulties in adopting drought-resistant and climate-resilient varieties. The lack

of timely information on climate-resilient agricultural technologies (RBQ = 54.44%) was ranked fourth, as poor access to extension services and inadequate information dissemination made it difficult for farmers to effectively plan climate-smart practices. Finally, the lack of awareness about climate change (RBQ = 52.22%) was ranked as the least severe constraint; however, it still highlighted the need for more effective extension strategies to educate farmers about the long-term benefits of climate-resilient practices. These findings corroborate those of Babu (2019), Majumdar et al. (2020), Naik et al. (2022), and Acharitha et al. (2022).

Government agencies and agricultural extension services should intensify farmer training on climate-resilient technologies to overcome technological constraints by emphasizing practical demonstrations and field trials. Strengthening CHCs by ensuring the timely availability of farm machinery and expanding their reach to remote areas can enhance mechanization. Establishing seed banks at the village level and ensuring the timely distribution of improved seeds can facilitate adoption. Additionally, leveraging digital platforms to provide real-time weather and market advisories can enhance farmers' decision-making and resilience to climate uncertainties.

Table 3: Calculation of Average Rank and Total Rank of Constraint Category.

Constraint Category	Kendall's W mean value
Socio-personal	1.2
Financial	2.0
Technological	2.8

For further analysis, Kendall's coefficient of concordance was used. Table 2 depicts Kendall's W mean value calculations for each constraint category, in Table 3. In Fig. 7, the average ranks were assigned as follows: socio-personal constraints at 1.2, financial constraints at 2.0, and technological constraints at 2.8. These yield total rank sums of 72, 120, and 168, respectively, with a mean total rank of 120. For example, based on the assumed data and the computed W of 0.64. Fig. 8 illustrates a radar chart showing the comparative ranking of various constraints using Kendall's W mean value. Table 3 depicts a detailed analysis of Kendall's coefficient of concordance.

From Tables 3 and 4, it is evident that Kendall's W value of 0.64 (on a scale of 0 to 1) indicates a strong agreement among the 60 respondents in their rankings of the three conditions. This suggests that the respondents

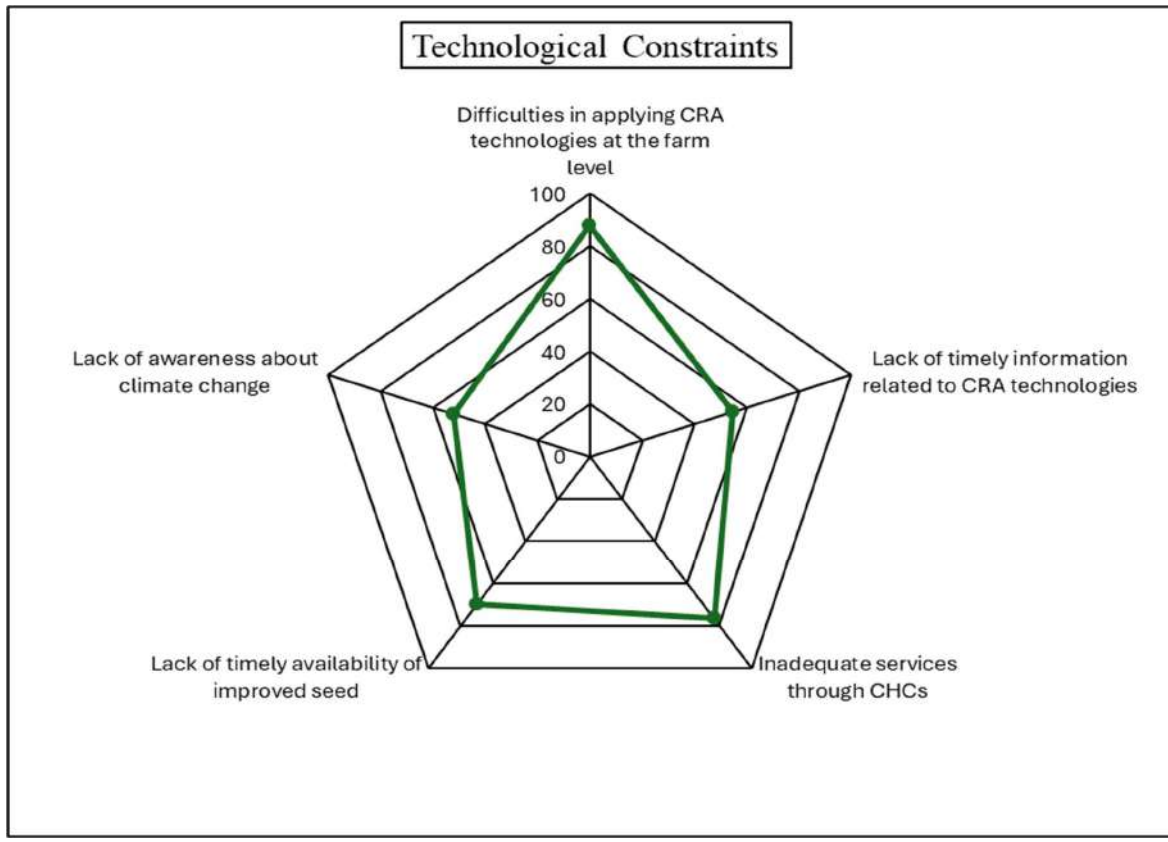

Fig. 7: Radar chart depicting the RBQ% in the context of technological constraints faced by the respondents in the study area.

consistently ordered the conditions similarly. The Friedman test (used for comparing rankings across multiple conditions) yielded a chi-square statistic of 77.00 with 2 degrees of freedom (degrees of freedom = k−1, where k = 3 conditions). Both the asymptotic (theoretical) p-values were .000, confirming that the observed agreement was highly statistically significant ($p < .001$). This implies that the likelihood of such an agreement occurring by chance is virtually zero. There was a strong, statistically significant agreement among respondents in their rankings of the three conditions. The consistency in rankings led to detectable differences between the conditions (Friedman test) and a high concordance measure (Kendall's W). This result is robust across theoretical and simulation-based methods.

The Kendall's W value of 0.64 (indicating strong agreement) and the highly significant chi-square statistic (χ^2 (2) = 77.00, $p < .001$) validate the Rank-Based Quotient (RBQ) method used in this study. Both the Asymptotic Significance and Monte Carlo Significance values were 0.000, reinforcing the robustness of the test results. This statistically confirms that the constraint rankings reported in this study, particularly the prominence of financial and socio-personal barriers, were consistently perceived across the respondent groups, thereby enhancing the credibility and reliability of the RBQ-derived conclusions. These results confirm that farmers consistently ranked constraints similarly, and a "preference for conventional practices" (RBQ = 84.44%) emerged as the top socio-personal barrier, reflecting a shared perception of the challenges. The low p-values (< .001) confirm that the observed rankings are statistically significant and not due to chance, reinforcing the reliability of the RBQ outcomes. In the socio-personal domain, the top constraint was the preference for conventional practices (RBQ = 84.44%), supported by Kendall's W, highlighting a strong consensus among farmers. This finding aligns with the cultural inertia and risk aversion common in agrarian communities. Additionally, labor scarcity (RBQ = 80.56%) and the inability to adopt new practices (RBQ = 79.44%) further underscore systemic issues that require interventions such as training and mechanization, including the establishment of Custom Hiring Centers (CHCs). In terms of financial constraints, the "lack of credit/capital" (RBQ = 89.44%) and "high input costs" (RBQ = 83.89%) are particularly critical, reinforcing the urgency of addressing these financial barriers through improved institutional credit and targeted subsidy programs. Regarding technological constraints, the difficulty in implementing CRA technologies at the farm level (RBQ = 87.78%) and inadequate CHC services (RBQ = 76.67%) were highlighted. The large effect size (W = 0.64) supports the need for focused policy measures regarding practical training and infrastructure improvements. The strengths of this analysis lie in its methodological rigor,

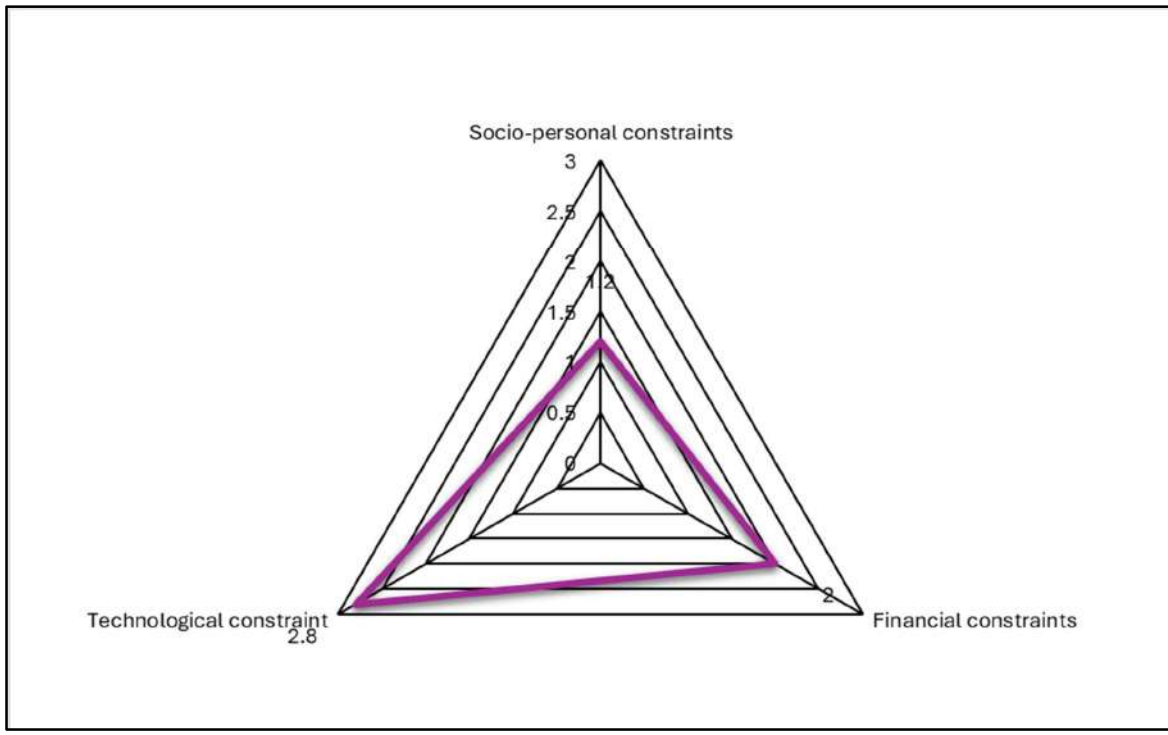

Fig. 8: Radar chart depicting the comparative ranking of various constraints through Kendall's W mean value.

Table 4: Test for Kendall's Coefficient of Concordance (Kendall's W).

Test for Kendall's Coefficient of Concordance (Kendall's W)	Values
N (Number of respondents)	60
Kendall's W	0.64
Chi-Square	77.00
Degrees of Freedom	2
Asymptotic Significance	.000
Monte Carlo Significance	.000
99% Confidence Interval (CI) Lower Bound	.000
99% Confidence Interval (CI) Upper Bound	.000

combining the RBQ to rank constraints with Kendall's W to measure consensus, thus providing a robust mixed-methods approach. This interpretation is aligned with that of Mallick et al. (2023a).

The statistical significance of the constraints, particularly financial barriers, justifies recommendations for enhanced credit access, subsidies, and CHC expansion. Moreover, the local specificity of the study, with a focus on Jharsuguda, fills a critical gap in localized NICRA research and emphasizes the need for region-specific interventions. Areas for further exploration include obtaining qualitative insights to understand why farmers resist change, conducting temporal analysis by repeating the study post-intervention, and testing Kendall's W in other NICRA districts to determine the broader generalizability of these findings. The statistical analysis demonstrated Kendall's W = 0.64 and χ^2 (2) = 77.00, $p < .001$, which strengthens the credibility by demonstrating that the constraints identified through the RBQ are both consistent and statistically significant. This dual validation supports targeted interventions, such as financial literacy programs, scalable demonstrations of climate-resilient technologies, and strengthening CHCs and seed distribution systems to enhance the adoption of climate-resilient technologies in Jharsuguda. The interpretation is aligned with the approach used by Mallick et al. (2023b).

Discussion

Regular training programs and large-scale demonstrations of climate-resilient technologies should be conducted to enhance farmers' knowledge and encourage adoption, particularly among small and marginal farmers who are hesitant to take risks. Extension personnel and scientists must play active roles in these initiatives. Expanding Custom Hiring Centers (CHCs) in NICRA villages can help address labor shortages and improve farm efficiency. Community-managed CHCs should ensure timely access to farm machinery, with revenue reinvested in maintenance and expansion. Popularizing water-saving techniques, such as low-cost rainwater harvesting structures, poly-mulching, and zero-energy cooling systems for vegetable storage, can improve resource efficiency and enhance resilience to erratic rainfall. Village-level seed banks should be established to provide high-quality and drought-tolerant seed varieties at affordable rates. This will ensure that farmers have reliable access to improved crop varieties suited to changing climatic conditions. Encouraging integrated farming systems (IFS), mushroom and marigold cultivation, poultry, and small-scale fisheries can improve farmers' livelihoods and their resilience. Financial support and follow-up interventions are required to sustain these activities. Although methodologically robust, the present study is limited by its quantitative orientation. Future research should adopt a mixed-methods approach to capture the nuanced behavioral and experiential dimensions of constraint perception. Qualitative interviews and focus groups can yield a deeper understanding of farmer psychology, risk aversion, and systemic trust issues regarding the adoption of climate-resilient agricultural technologies. Climate-resilient technology adoption should be demand-driven, with real-time problem-solving demonstrations and timely availability of critical inputs to ensure effective farm-level implementation. If implemented effectively, these interventions can enhance farmers' adaptive capacity and contribute to sustainable agricultural development. Table 5 shows the policy matrix highlighting major

Table 5: Policy Matrix Highlighting Major Constraints in the Adoption of Climate-Resilient Technologies

Constraint	Recommended Action	Implementing Agency
Lack of credit/capital	Expand access to institutional credit through SHGs, cooperative banks, and KCC schemes.	NABARD, Regional Rural Banks, State Agri Dept
Difficulty in applying CRA technologies	Conduct on-farm demonstrations and provide technical training through KVKs and CHCs	ICAR-KVKs, ATMA, Panchayat CHCs
High cost of agricultural inputs	Offer input subsidies and bulk procurement schemes for quality seeds and inputs.	Department of Agriculture, State Govt
Preference for conventional practices	Promote behavioral change via farmer field schools, progressive farmer exposure visits.	ICAR, State Extension Services
Labor scarcity	Strengthen mechanization support through well-equipped and locally managed CHCs	NICRA-VCRMC, State Agri Department, MANREGA

constraints, suggested actions concerning the constraints, and the implementing agency.

CONCLUSIONS

This study comprehensively analyzed the socio-personal, financial, and technological constraints affecting the adoption of climate-resilient agricultural technologies by farmers in the NICRA villages of Jharsuguda district, Odisha. To translate the findings into actionable insights, this study recommends a multi-pronged policy approach. Socio-personal barriers can be addressed through farmer-led knowledge platforms, participatory extension, and risk-mitigation training. Financial bottlenecks require expanded institutional credit schemes, targeted subsidies for inputs, and revolving funds for local Custom Hiring Centers (CHCs). On the technological front, enhancing the CHC infrastructure, establishing seed banks, and introducing mobile-based agro-advisory services are imperative. To build long-term resilience, future strategies must embed climate-smart practices into local development planning, encourage youth engagement in agri-innovation, and promote community-based adaptation. Scaling up successful interventions through digital platforms, village-level cooperatives, and decentralized climate services can further enhance their reach and impact. These findings provide a policy roadmap for institutional actors seeking to mainstream adaptive agriculture in vulnerable areas.

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Bacterial Diversity in Oil Field Environments and Evaluation of Their Ability to Biosynthesize Silver Nanoparticles (AgNPs)

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ABSTRACT

Bacteria isolated from oil reservoir environments possess unique enzymes that allow them to adapt to extreme environments, making them ideal candidates for producing high-value nanomaterials that can be used in various fields. In the present study, eight samples were collected from Badra and Ahdab oil fields in Iraq, the bacteria isolated and identified based on the *16SrRNA* gene, and the isolates were screened for the synthesis of silver nanoparticles (AgNPs). The characteristics of the AgNPs were analyzed using UV-Vis spectroscopy, FTIR, XRD, FE-SEM, zeta potential measurements, and dynamic light scattering (DLS). The results indicated the dominance of Gram-positive bacteria, with a percentage of 18 (72%). Genetic identification revealed that the bacteria were under 6 genera and 16 species; these genera include *Enterococcus*, *Priestia*, *Enterobacter*, *Acinetobacter*, *Flavobacterium*, and *Bacillus*. Seven new strains have been deposited in GenBank. The results of screening isolates for synthesized AgNPs showed high efficiency of a novel strain, *Bacillus halotolerans* strain AhWM4, with the maximum absorption peak at 430 nm. The average size of AgNPs using XRD, FE-SEM, and TEM was (31.3, 27.0, and 42.1) nm, respectively. Dynamic light scattering (DLS) measurements showed a wide dispersion with an effective diameter of 57.1 nm; the X-ray diffraction (XRD) spectrum matched the crystalline nature of the AgNPs. It also showed high stability, with a zeta potential of -42.3 mV. AgNPs have attracted considerable attention due to their staggering potential for a wide range of commercial and environmental applications.

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INTRODUCTION

The biosynthesis of silver nanoparticles (AgNPs) has been explored in various environments, but using bacteria isolated from harsh conditions such as oil reservoirs-characterised by a lack of oxygen, high temperatures, and extreme pressures-represents a significant innovation. These extreme environments challenge most forms of life and provide new insights into how microorganisms may enhance industrial processes, particularly in nanotechnology production. In 1926, Bastin discovered sulphur-reducing bacteria such as Desulfoplanes, Desulfovermiculus, and Desulfotignum within harsh environments (Kadnikov et al. 2023). The study of microbial communities in oil reservoir environments has since opened a new chapter in our understanding of these ecosystems. Subsequent research has revealed diverse microorganisms inhabiting oil reservoirs, including those found in crude oil, formation water, rocks, and organic matter, particularly in the oil–water transition zone (OWTZ), which is conducive to microbial growth and oil decomposition (Rajbongshi and Gogoi 2021).

The positive effects of these microbial communities can be harnessed by investigating their activities, metabolic processes, and capacity to produce bioproducts such as surfactants, biopolymers, solvents, acids, and gases (Augustine 2023). Much current research focuses on oil-contaminated environments, where

Alishewanella jeotgali, Bacillus cereus, and Pseudomonas stutzeri have likely adapted to harsh conditions and developed significant enzyme systems of interest for both environmental and commercial applications (AL-assdy et al. 2024, AL-Shami et al. 2023, Alyousif et al. 2022, Aboud et al. 2021, Hamzah et al. 2020a). Among these applications, the biosynthesis of AgNPs is gaining attention due to its environmentally friendly, energy-efficient, cost-effective, and rapid nature compared with traditional physical and chemical methods (Yali et al. 2023).

Furthermore, AgNPs have diverse applications in medicine, food technology, and environmental science (Arshad et al. 2024). The degradation of mixed polluted compounds such as oil spills, heavy metals, herbicides, particulate matter, pesticides, fertilisers, toxic gases, sewage, and industrial effluents (Singh et al. 2020) can be performed effectively using nanocatalysts (Astruc 2020). Recent studies indicate a direct relationship between nitrate reduction by microorganisms and their ability to synthesise AgNPs. Evidence suggests that the biological reduction of nitrate may release free electrons, which can subsequently reduce silver ions (Ag^+) to form AgNPs. This mechanism highlights the capacity of bacteria to transform toxic metals into non-toxic nanoforms and produce AgNPs with unique properties (Rose et al. 2023). The connection between nitrate reduction and nanoparticle synthesis underscores the importance of studying this process to better understand the biological mechanisms involved in AgNP biosynthesis.

Nanoparticles can be produced by extracellular or intracellular bacteria, often involving organic molecules such as proteins, sugars, and enzymes, which are believed to be crucial in reducing metal ions and forming nanoparticles through oxidation–reduction reactions (John et al. 2022). Despite significant advances in nanoparticle biosynthesis, gaps remain in our understanding of how microorganisms convert metals into nanoparticles, particularly in oilfield environments (Bharose et al. 2024). Characterising the bacterial community structure of produced water from oil fields presents significant challenges, including the difficulty of cultivating bacteria using conventional methods, which rely mainly on culture-based approaches rather than molecular analyses (Ziganshina et al. 2023). Therefore, DNA sequencing approaches, particularly targeting the 16S rRNA gene, have greatly enhanced our understanding of these microbial communities and their composition (Gao et al. 2024).

Research has revealed a remarkable diversity of bacterial species inhabiting oil reservoirs, with hundreds recorded and several species identified for their high capacity to produce

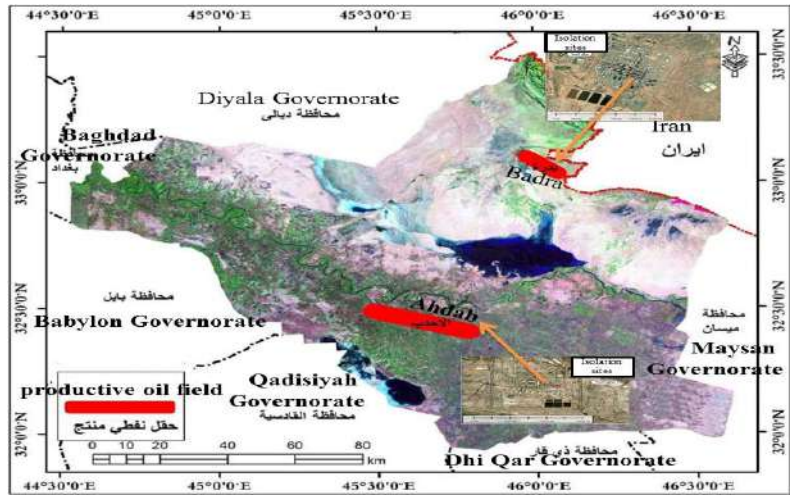

Fig. 1: Sample collection sites.

Table 1: Physicochemical properties of the produced water samples obtained from Badra and Ahdab oil fields.

Samples	Oil in water ppm	TSS ppm	pH	Total Iron ppm	Hydrogen sulfide ppm	Chloride ppm
BD- s 1	85	20.0	6.6	0.69	371.96	21423
BD- s 2	92	15.0	6.8	0.78	400	19080
Ah- s1	36.94	37.0	6.85	0.10	406	130804.81
Ah- s2	34.15	42.0	6.87	0.10	377	143362.65

BD = Badra, Ah = Ahdab, s = sites, TSS = Total Suspended Solids

AgNPs, paving the way for new insights in biological research and nanotechnology. In the Badra and Ahdab oil fields, the existence of bacterial communities has not yet been explored, and no data are available. Hence, the present study aims to isolate and identify bacteria from the reservoirs of these oil fields in Wasit Governorate, Iraq, and determine the most efficient isolates for synthesising AgNPs, while characterising the produced nanoparticles using different techniques.

MATERIALS AND METHODS

Sample Collection

Eight samples of produced water were collected from separator tanks of the Badra and Ahdab oil fields between October and December 2022. The Badra oil field is located in the northeast of Wasit Governorate in Iraq at approximately 33.037°N and 46.056°E. The depth of oil reservoirs was between 4700 and 6200 m below ground, depending on primary production of oil, while in the Ahdab oil field, located to the west of Wasit at approximately 32.424°N and 45.714°E, the oil reservoirs were between 2300 and 3500 m deep, extracting oil through flooding with water for secondary production Fig. 1. The samples were collected in sterile glass containers and transported to the laboratory immediately, keep at room temperature until used. Daily reports from the oil field laboratories were used to determine the physical and chemical properties of the samples Table 1.

Culturing of Bacteria

To isolate the indigenous bacteria, 5mL of each sample was inoculated into a 250mL Erlenmeyer flask containing 95 mL of nutrient broth. The flasks were incubated in a shaker at 35°C and 120 rpm for 48 h After the incubation period, 1 mL from each flask was serially diluted and spread using an L-shaped glass spreader onto the center of sterilized nutrient agar and MacConkey agar plates and incubated aerobically at 35°C for 24 h. Distinct colonies were isolated, and the subculture process was conducted three times to ensure the purity of the colonies. Additionally, the Gram stain protocol was employed for microscopic analysis.

Molecular Identification of Bacteria

Each isolate was activated for 24 h at 35°C in nutrient broth medium. An Eppendorf tube containing 1.5 ml of culture was centrifuged for two minutes at 13,000 rpm. After discarding the supernatant, DNA was extracted from the cells by the Presto™ Mini gDNA kit according to the manufacturer's instructions (Geneaid, Taiwan). A 1% agarose gel was used to prove the extracted gene. Identification of the isolated bacteria was performed using a 1500-bp-long *16S rRNA* gene. Polymerase chain reaction (PCR) was used for amplification, using the universal forward primer 27F 5'-AGAGTTTGATCCTGGCTCAG-3' and the reverse primer 1492R 5'GGTTACCTTGTTACGACTTR-3'. The AccuPower® PCR PreMix kit prepares the reaction mixture according to the manufacturer's instructions. The program of the thermal cycle was as follows: initial denaturation at 95°C for 5min., followed by denaturation with 30 cycles of 95°C for 30 sec., primer annealing at 52°C for 45sec., extension at 72°C for 1.5min., and final extension at 72°C for 10 min. (Alyousif et al. 2020). To verify the amplification of the target gene, the fragment of amplified DNA was separated using an electrophoresis device on a 1.5% (w/v) agarose gel stained with 0.5 µl of ethidium bromide to a concentration of 0.5 µg/mL (Lee et al. 2012). The amplified and labeled DNA tubes were sent to Macrogen Laboratories in South Korea for sequence analysis. The results were compared with the NCBI website using BLAST to identify the closest matches to the bacterial isolate. The phylogenetic tree was constructed using the neighbor-joining (NJ) technique in MEGA 11, with 1000 primers incorporated to ensure robustness (Burghal et al. 2021).

Screening the Ability of Bacterial Isolates to Synthesize AgNPs

All bacterial isolates were screened for the synthesis of AgNPs by the nitrate reduction test, which was conducted according to Bhusal & Muriana (2021). The positive isolates were selected for screened on nutrient agar supplemented with 1 mM $AgNO_3$, and the bacterial isolates were inoculated on the plates, incubated at 35°C for 24 to 72 h. The growth of bacteria indicated a positive result; the isolates that gave a positive result were chosen for the final screening, which was carried out to select the most efficient isolates for synthesizing AgNPs, based on changes in the color of the reaction mixture, following the method described by Bhusal & Muriana (2021) with some modifications. Selected bacterial strains were cultured in a 250 mL Erlenmeyer flask, incubated in a shaker at 35°C with a speed of 180 rpm for 24 h. After incubation, the cultures were centrifuged at 6000 rpm for 20 min. The resulting supernatant was then filtered using a Millipore filter with a pore diameter of 0.45 µm. Next, the reaction mixture was prepared by mixing equal volumes 1:1 (v/v) of the supernatant and a 1mM $AgNO_3$ solution in an aluminum-coated flask to prevent oxidation. The mixture was then incubated in a shaker at 180 rpm and 35°C for 72 to 120 h. The results were evaluated by observing the color change of the reaction mixture from yellow to dark brown compared with the control (nutrient broth with $AgNO_3$ solution). After the incubation period, to confirm the presence of AgNPs in

the reaction mixture, 2 mL of each mixture that had changed color to brown was taken and poured into a quartz cuvette. The absorbance was measured in the range of (200-800) nm using the UV-1800 double-beam spectrophotometer (Shimadzu, Japan).

Purification of AgNPs

After measuring the absorbance, the reaction mixture that achieved the highest absorbance peak was placed in test tubes in a centrifuge at 6000 rpm for 20 min, the supernatant was discarded, and the remaining precipitate was washed with deionized distilled water. This step was repeated three times until a clear and colorless solution was obtained. In the final step of centrifugation, the precipitate containing AgNPs was placed in a clean watch glass in an electric oven at 50°C to dry and remove excess water. The resulting powder was then collected in Eppendorf tubes covered with aluminum foil (AL-Shami et al. 2023).

Characterization of AgNPs Synthesized

The *Bacillus halotolerans* strain AhWM4 that gave the highest peak in the UV-visible spectrophotometer was characterized by an infrared spectrometer (Shimadzu, Japan). An X-ray diffractometer (PANalytical, Netherlands) is employed to ascertain the crystalline characteristics of AgNPs and to compute their size utilizing the Debye-Scherrer equation (Saleh & Alwan 2020). The detailed structure of the AgNPs was studied using a field emission scanning electron microscope (FE-SEM) and transmission electron microscope (TEM) (Hitachi, Japan), while the elemental composition was determined with energy dispersive X-ray spectroscopy (EDX). A zeta potential device (HORIBA Scientific SZ-100, Japan) was used to assess the surface charges of AgNPs, and dynamic light scattering (DLS) was employed to analyze the particle size and distribution in the colloidal solutions (Pallavi et al. 2022).

RESULTS AND DISCUSSION

Isolation of Bacteria

Twenty-five strains were isolated from the produced water of the Badra and Ahdab oil fields. 15 (60%) isolates from the Badra oil field and 10 (40%) from Ahdab, according to the protocol of Gram stain. In the Badra oil field, 4 (26.7%) isolates were Gram-negative, and 11 (73.3%) were Gram-positive, while in Ahdab, the numbers of Gram-negative and positive isolates were 3 (30%) and 7 (70%), respectively, as Fig. 2.

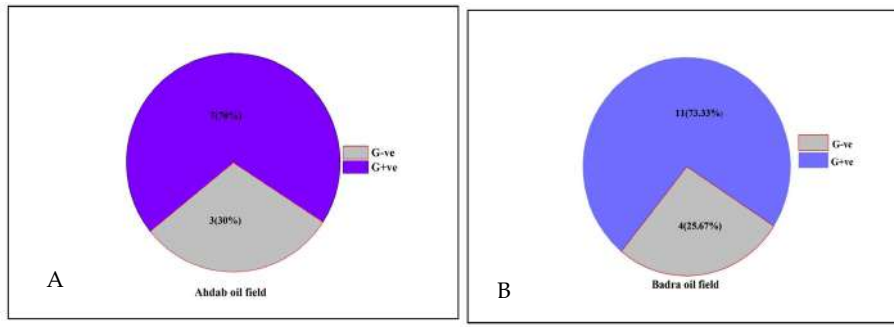

Fig. 2: Total percentage of Gram-positive and Gram-negative bacteria in produced water samples, A Ahdab oil field and B Badra oil field.

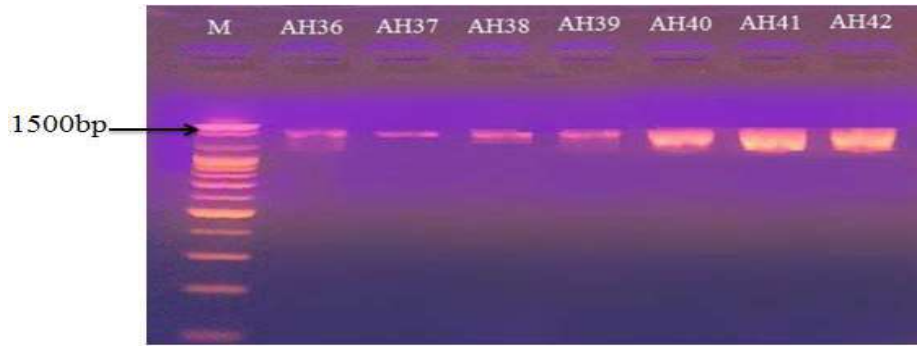

Fig. 3: Electrophoretic analysis demonstrating the amplified *16S rRNA* gene products derived from genomic DNA of some bacterial isolates. M. Ladder, AH36-AH42 AH code of bacterial isolates.

Table 2: Bacterial community of produced water samples that were identified by the *16S rRNA* gene (Ah Isolation symbol before molecular identification, BD= Badra oil field, Ah= Ahdab oil field).

Site	Accession no. reference strain	Isolates code	Cloest species	Match ratio with reference strain %
BD- s1	AH42	OM033656.1	Enterobacter hormaechei	100 %
	AH45	MN208152.1	*Enterobacter cloacae*	100 %
	AH14	OP852942.1	*Bacillus paramycoides*	98 %
	AH38	MN049561.1	*Acinetobacter calcoaceticus*	99 %
	AH36	KX950679.1	*Bacillus cereus strain MBGIPS 9*	100 %
	AH37	MN847602.1	*Bacillus velezensis*	99 %
	AH31	KF051091.1	*Flavobacterium columnare*	100 %
	AH18	FJ573187.1	*Bacillus cereus strain AK1871*	100 %
BD- s2	AH46	MT000148.1	*Enterococcus faecium*	100 %
	AH19	KY362201.1	*Bacillus amyloliquefaciens*	99 %
	AH30	KC790242.1	Bacillus anthracis	100 %
	AH11	MK547152.1	Bacillus tropicus	100 %
	AH10	MZ618716.1	Bacillus subtilis	100 %
	AH21	ON510000.1	Bacillus anthracis	100 %
	AH20	KR999903.1	Priestia flexa	99 %
Ah- s1	AH47	MN658825.1	*Enterobacter hormaechei*	100 %
	AH39	OP288194.1	*Enterobacter hormaechei*	100 %
	AH41	MT783973.1	*Bacillus cereus*	100 %
	AH40	MH050744.1	*Enterobacter cloacae*	100 %
Ah- s2	AH4	OK298998.1	*Bacillus rugosus*	99 %
	AH5	OQ152470.1	*Bacillus toyonensis*	100 %
	AH15	KX268482.1	*Bacillus cereus*	100 %
	AH3	MT539995.1	*Bacillus subtilis*	100 %
	AH12	LK392517.1	*Bacillus cereus strain ISU-02*	%100
	AH13	MT539148.1	*Bacillus halotolerans*	99 %

Genetic Identification of Bacterial Isolates

The *16S rRNA* gene was amplified to identify the bacterial isolates using the polymerase chain reaction (PCR) technique. Agarose gels 1% (w/v) displaying PCR-amplified 16S rDNA gene fragments. About 1,500 bp of amplified fragments were obtained for every isolate, determined based on the DNA marker (100 bp) (Fig. 3).

The BLAST tool was used to analyze and compare the DNA sequencing results of the bacterial isolates with those of the reference strains found in the GenBank database. The DNA sequencing results of most of the studied strains showed a 100% similarity in gene sequences compared to the reference strains, as shown in Table 2. However, some isolated strains displayed variations in certain nitrogenous bases relative to the reference strains available in GenBank. Seven new strains were recorded in the GenBank database, with unique accession numbers as illustrated in Table 3.

Table 3: The new bacterial strains that were deposited in the NCBI GenBank database

Name of the new strain in GenBank	Accession no.
Bacillus paramycoides strain AhWM5	PP559174.1
Acinetobacter calcoaceticus strain AWMBI11	PP564426.1
Bacillus velezensis strain AWMBI10	PP564424.1
Bacillus amyloliquefaciens strain AhWM6	PP559314.1
Priestia flexa strain AhWM7	PP559502.1
Bacillus rugosus strain AhWM2	PP558901.1
Bacillus halotolerans strain AhWM4	PP559020.1

The results of the phylogenetic tree also confirmed a clear genetic proximity between the studied isolates and the reference strains, as shown in Fig. 4.

DNA sequencing of the bacterial community of produced water samples revealed that bacterial isolates were under six different bacterial genera and belonged to sixteen species. The genera were *Enterococcus*, *Priestia, Enterobacter, Acinetobacter, Flavobacterium, and Bacillus*. Among them, the genus Bacillus was the most prevalent, 16 (64%), followed by *Enterobacter*, 5 (20%). For the species, *Bacillus cereus* was the most frequently recorded, 5 (20%), followed by *Enterobacter hormaechei*, 3 (12%) (Fig. 5).

Screening of Bacterial Isolates for the Ability to Synthesize AgNPs

All bacterial isolates were tested for the synthesis of AgNPs. The screening results by the nitrate reductase enzyme test showed that 18 of the isolates gave positive results. Only these isolates were screened on nutrient agar supplemented with 1mM $AgNO_3$, and the results revealed that only 11

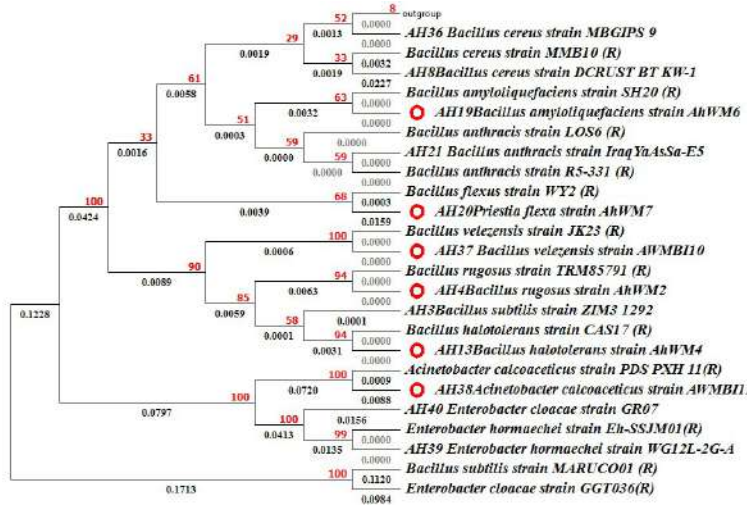

Fig. 4: A phylogenetic tree that shows the evolutionary relationships among bacterial strains of different species and their reference strains (R).

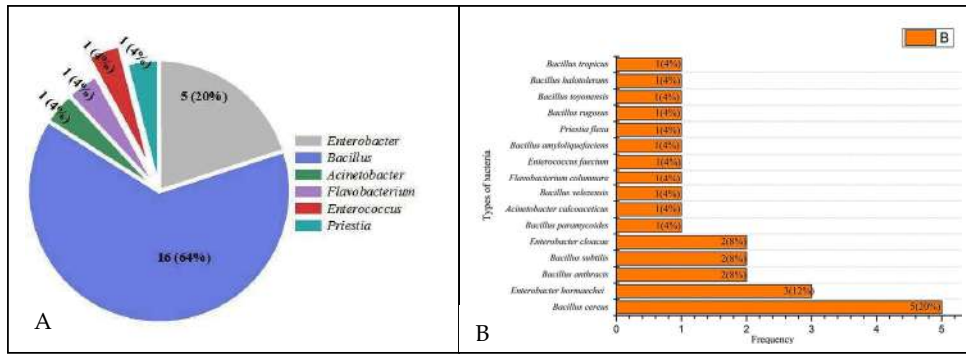

Fig. 5: Frequency of bacterial isolates in samples. (A). Percentage of genera (B). Percentage of species.

Fig. 6: Screening of bacterial isolates based on color change of the mixture: (AH). Isolates. (C). Control.

showed growth. These isolates were tested for a color change, as the supernatant of bacterial cultures treated with 1 mM $AgNO_3$. Only 2 strains, *Bacillus halotolerans* strain AhWM4 and *Bacillus rugosus* strain AhWM2, showed extracellular formation of AgNPs, which was initially validated by observing the color change of the supernatants from light yellow to brown compared to the control after the incubation period, indicating the formation of AgNPs (Fig. 6).

The UV-vis spectrophotometer was used to detect the biosynthesis of AgNPs after the color change of the mixture reaction of two strains, AH 13 and AH 4. *Bacillus halotolerans* strain AhWM4 and *Bacillus rugosus* strain AhWM2. The results indicated a maximum absorption peak of 0.47 λmax at 430 nm and 0.09 λmax at a wavelength of 440 nm, using the supernatant for both strains, respectively, indicating the synthesis of AgNPs with different optical properties (Fig. 7).

Characterization of AgNPs Synthesized by *Bacillus halotolerans* Strain AhWM4

Fourier transform infrared spectroscopy (FTIR): The FTIR spectra were used to identify the various functional groups of the synthesized AgNPs by Bacillus halotolerans strain AhWM4 that contributed to stability and the reduction of precursors to NPs. The results displayed peaks at specific wavelengths, specifically around 13495, 12935, and 1552 cm^{-1}. These peaks correspond to O-H stretching from hydrogen-bonded alcohols and phenolic vibrations, as well as C-H bonds in alkanes and C=O bonds in carbonaceous compounds, ketones, aldehydes, and carboxylic acids. N-H bonds in amides and secondary amines. The results further indicated a shift and a decrease in the intensity of some bands in the spectrum of the AgNPs formed during the reduction process. Notably, the peaks at 3214.19 cm^{-1}, 1647.21 cm^{-1}, and 1554.86 cm^{-1} showed a decrease in intensity and a shift in position (Fig. 8).

The X-ray diffraction (XRD) analysis: The crystallinity of AgNPs synthesized by the B. halotolerans strain AhWM4 was examined using XRD analysis. Five distinct diffraction peaks were observed at 2θ values of 38.27˚, 45.94˚, 55.59˚, 67.42˚, and 76.94˚. These peaks correspond to the silver crystal planes (200, 103, 006, 112, and 201), closely matching the previously cited reference (JCPDS: 00-041-1402). Additionally, the X-ray diffraction (XRD) pattern exhibited three extra peaks at 2θ values of 27.83˚, 32.32˚, and 57.61˚, which could be associated with the crude coating agents (Fig. 9). According to Scherrer's the average crystallite size of the AgNPs was calculated to be 31.3 nm.

Electron microscopy (FE-SEM): According to the Field Emission Scanning Electron Microscopy (FE-SEM) and Transmission Electron Microscopy (TEM) images, the particles synthesized by the B. halotolerans strain AhWM4 were spherical or sub-spherical and exhibited a polydisperse nature. The average sizes of the nanoparticles were between 27.0 nm and 42.1 nm, respectively, with no signs of agglomeration (Fig. 10).

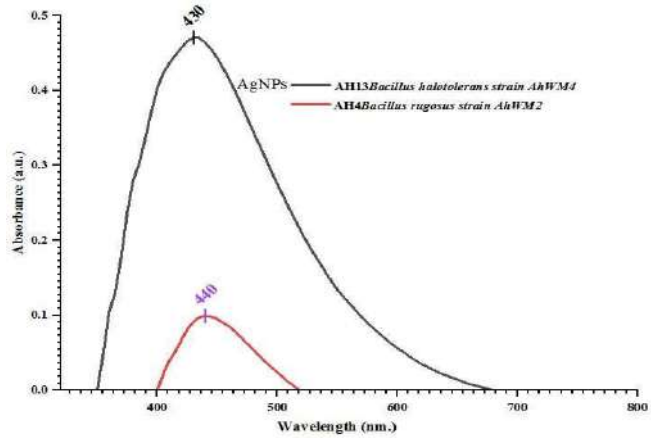

Fig. 7: UV-vis spectra of AgNPs biosynthesized by bacterial strains after color change in the reaction mixture.

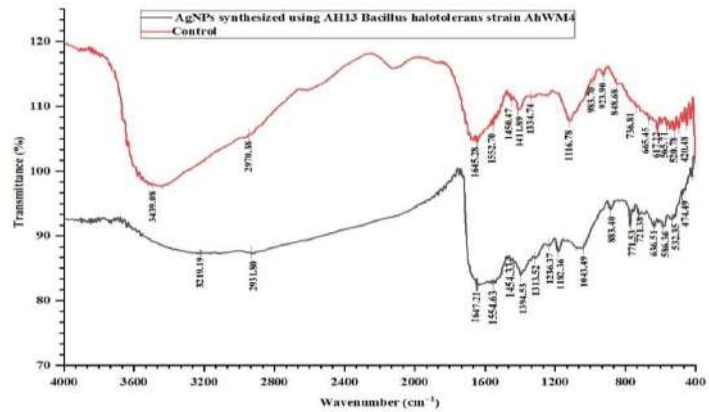

Fig. 8: FTIR spectra of control and Ag NPs synthesized by *B. halotolerans* strain AhWM4.

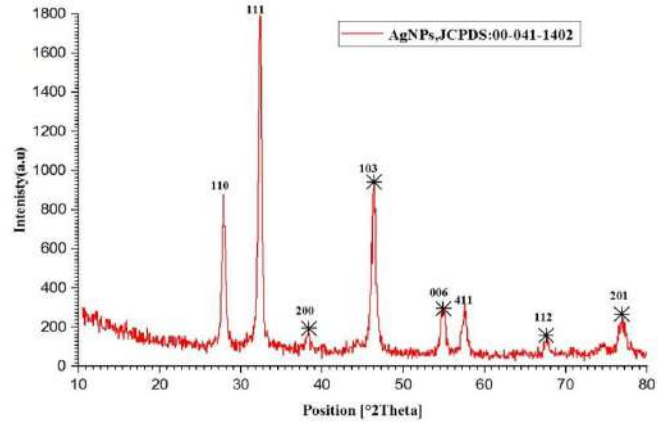

Fig. 9: XRD spectra of AgNPs synthesized by *B. halotolerans* strain AhWM4.

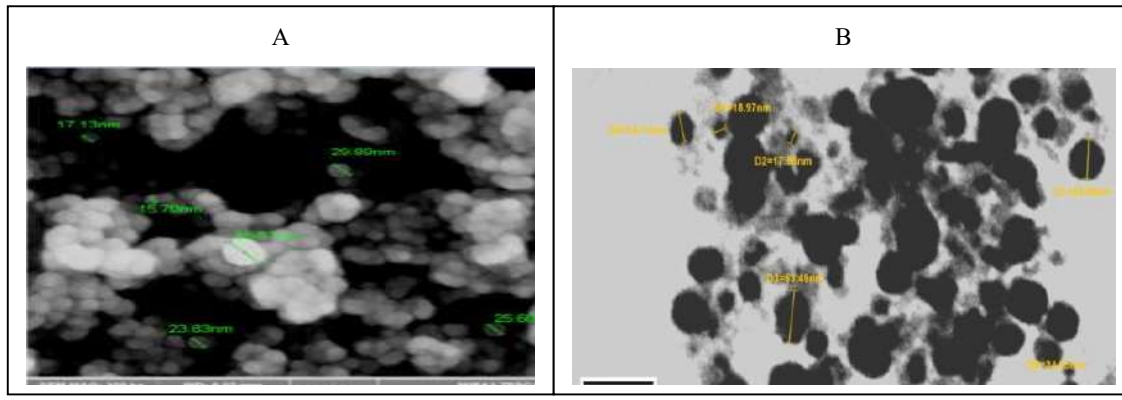

Fig. 10: Characteristic of AgNPs synthesized using *B. halotolerans* strain AhWM4: (A). FE-SEM (B). TEM.

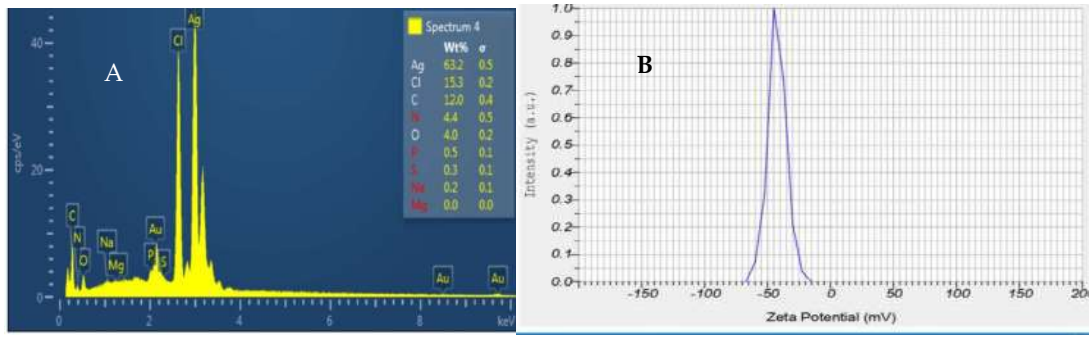

Fig. 11: Characteristics of AgNPs synthesized using *B. halotolerans* strain AhWM4. (A). EDX analysis. (B). Zeta potential value.

Energy dispersive X-ray spectroscopy (EDX) and zeta potential: The EDX analysis revealed that the elemental composition of the nanoparticles showed a significant peak at 3.0 keV, corresponding to an atomic percentage of 63.2%, indicating silver as the primary element. Additional peaks for carbon (C), oxygen (O), gold (Au), and nitrogen (N) are also observed throughout the scanning range (0–3 keV). Still, these elements are present at very low atomic percentages (Fig.11). Zeta potential analysis was conducted to evaluate the electrostatic stability of the synthesized AgNPs. The zeta potential of AgNPs was measured at -42.3 mV, indicating their superior stability Fig.11.

Dynamic light scattering (DLS): Dynamic light scattering (DLS) was employed to assess the nanoparticles' hydrodynamic diameter and aggregation in colloidal solutions. The average diameter was found to be 123 nm;

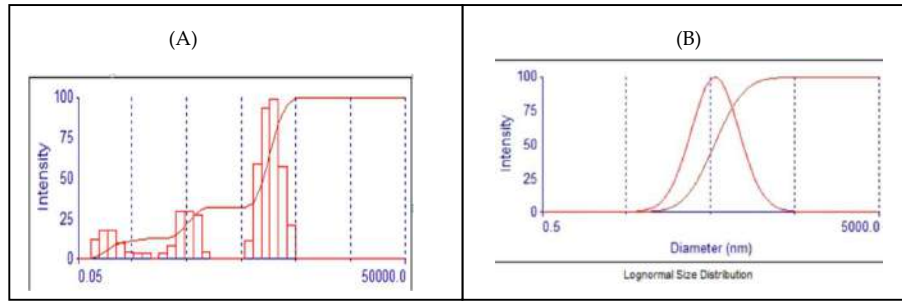

Fig. 12: Characteristic of AgNPs synthesized using the *B. halotolerans* strain AhWM4 through (A). Lognormal Distribution Multimodal Size Distribution (B). Multimodal Size Distribution.

most of the synthesized AgNPs had an effective diameter of 57.1 nm and a polydispersity index (PI) of 0.52 (Fig. 12).

DISCUSSION

The current study demonstrated a remarkable diversity of bacterial species in produced water in the Badra and Ahdab oil fields. The number of bacterial isolates in the Badra oil field was higher than in Ahdab; this may be due to differences in the physical and chemical properties of the two fields (Table 1). Other factors, such as geographical location, age of the oilfield, depth, and method of oil production, may also contribute. Al-shami et al. (2022) reported that oil reservoir environments show a great diversity of bacterial communities, influenced by a number of factors despite their harsh environmental conditions.

The results showed that the number of Gram-positive isolates in both fields was higher than that of Gram-negative isolates. This is consistent with the study of Vitaly et al. (2023). Gram-positive bacteria have a thick peptidoglycan wall, spores, enzymes, and metabolic pathways that enable them to degrade the complex hydrocarbons present in oil and use them as an energy source, giving them additional protection and a high ability to withstand harsh conditions (Huang et al. 2023). All bacterial isolates showed successful amplification of the 16S rRNA gene with fragments approximately 1,500 bp in size; these results were compatible with previous studies (Halami et al. 2024, AL-Zaidi et al. 2023, Hamzah et al. 2020b). The current study showed a clear dominance of the genus *Bacillus*, which is consistent with previous studies (Alyousif et al. 2020, Al-Tamimi et al. 2019). *Bacillus* is known for its ability to adapt to harsh conditions. Its cell wall is composed of peptidoglycan compounds, teichoic acid, and meso-diaminopimelic acid, which enhance its rigidity. Endospores play a crucial role in the persistence of this genus in these environments (Blanco Crivelli et al. 2024). These bacteria possess specific metabolic pathways that enable them to produce substances that inhibit other bacteria, giving them a competitive advantage in their living environment and making them a dominant genus in hydrocarbon-contaminated environments (Aboud et al. 2021). Through sequencing of the 16S rRNA gene, seven unique bacterial strains from the current investigation were registered in the NCBI GenBank database with specific accession codes. These bacterial isolates' DNA sequences may alter as a result of exposure to mutagens; the cause of mutation may be attributed to differences in ecological systems and exposure to mutagens, including chemicals and radiation. Several new bacterial strains isolated from Iraqi oil field environments have been reported (AL-assdy et al. 2025, Al-Shami et al. 2022, Alyousif et al. 2020).

In the present study, all bacterial isolates from the produced water of oil fields were screened for the synthesis of AgNPs. The bacterial isolates gave positive results for the nitrate reduction test and the ability to grow in the presence of 1 mM $AgNO_3$. Nitrate can be converted to nitrite by the NR enzyme, an NADH-dependent enzyme that takes up two electrons released by NADH oxidation. Released electrons can also reduce silver ions (Ag^+) to AgNPs. Metal nanotization by microbes is an essential part of geocycles and occurs as a response to heavy metal stress in harsh environments, resulting in adaptive and protective mechanisms for microbes against metal toxicity (Ghosh et al. 2021). AgNPs were synthesized using various methods, including bioreducing agents (Prodjosantoso et al. 2025). The results showed the ability of two bacterial isolate strains, *Bacillus halotolerans* strain AhWM4 and *Bacillus rugosus* strain AhWM2, to cause a colour change in the reaction mixture from light yellow to brown compared to the control. This change is an initial and visible indication of AgNP production, which occurs due to surface plasmon resonance when interacting with light. The strain *B. halotolerans* AhWM4 revealed a maximum absorption peak. Various factors, including nanoparticle size and shape, influence differences in electron density. Smaller nanoparticles may exhibit a different colour than larger ones due to variations in light interaction and electron distribution. Many previous

studies have also reported this phenomenon (Alfryyan et al. 2022, Haji et al. 2022).

UV–Vis, SEM, TEM, EDX, XRD, FTIR, zeta potential, and DLS analyses were used to characterise biosynthesised AgNPs from the bacterial strain *B. halotolerans* AhWM4. FTIR spectroscopy results showed that the bacterial supernatant contains compounds capable of reducing Ag^+ ions and acting as natural capping agents. Functional groups such as ketones, aldehydes, carboxylic acids, and amides indicate the presence of a wide range of organic compounds, including enzymes, proteins, amino acids, and carbohydrates, which may play a significant role in silver ion reduction and AgNP formation (Boldt et al. 2023, Al-Timimi et al. 2016). AgNPs exhibited a consistent crystalline structure, as shown by XRD peaks; the pattern matched the JCPDS database standard, demonstrating AgNP purity and hexagonal form (Lakhan et al. 2020). According to the Debye–Scherrer equation, broad peaks indicate small particles with an average crystallographic size of 31.33 nm. Prominent peaks indicate that bacterial extract biomolecules stabilise AgNPs. These results are similar to those reported in previous studies (Ghiuta et al. 2021, Thirumagal & Jeyakumari 2020).

The results are consistent with most previous research, which demonstrated the spherical shape of AgNPs synthesised by different *Bacillus* species, as shown by FE-SEM and TEM, with some variation in particle size (Alsamhary 2020). These differences may be due to factors such as metal ion concentration, reaction mixture temperature, duration, and pH (Marooufpour et al. 2019). AgNPs showed minimal agglomeration; this can be attributed to biomolecules that stabilise AgNPs and prevent aggregation (Khorrami et al. 2018). The EDX pattern showed a prominent silver peak with an atomic fraction of 63.2% at 3 keV, consistent with the standard silver peak associated with surface plasmon resonance. Peaks corresponding to carbon, oxygen, and nitrogen were also observed, with low atomic ratios detected in the scanning range of 0–3 keV. This indicates that biological molecules, enzymes, and proteins were associated with AgNP biosynthesis (Eid et al. 2024).

Zeta potential and DLS analyses characterise nanoparticles by particle size and surface charge. Zeta potential measurements were conducted to assess colloidal stability, with a value of −42.3 mV indicating high stability (Saied et al. 2024). Previously reported zeta potential values include −30, −34.1, −33.9, and −21.4 mV. The negative surface charge of silver nanoparticles induces electrostatic repulsion, stabilising the particles and inhibiting agglomeration (Solís-Sandí et al. 2023). The carboxyl (COOH) groups in the bacterial extract are responsible for the negative charge, as confirmed by FTIR analysis (Illanes Tormena et al. 2024). DLS measures the hydrodynamic radius of nanoparticles, which is influenced by particle size, conjugated molecules, sugars, proteins, and random or Brownian motion in solution.

CONCLUSIONS

Numerous factors influence the high diversity of bacterial communities that are found in produced water oil fields, despite their extreme environmental circumstances. Gram-positive bacteria outnumbered Gram-negative bacteria in these environments. *Bacillus* spp. was the most prevalent, with the predominant species of Bacillus cereus. The novel strain of *B. halotolerans* AhWM4 was the most effective and powerful in synthesizing AgNPs with distinct properties, an average diameter within the nanoscale range, spherical shape, and high stability in aqueous solutions, which makes it eligible for use in many medical, pharmaceutical, and environmental applications for treating various types of pollutants in the water and soil environment.

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Development of Zinc Oxide-Sawdust Composite Adsorbent for Methylene Blue Removal: Synthesis, Characterization and Adsorption Mechanism

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ABSTRACT

The natural world provides sawdust, also known as wood shavings, which is a reasonably abundant and affordable lignocellulosic compound. It is a waste product of agriculture and industry that is abundant and has disposal issues. A zinc oxide nanocomposite based on sawdust (ZnO@SD) was synthesized to efficiently remove the dye methylene blue (MB) from aqueous solutions. FTIR, SEM, TEM, and XRD analyses were used to characterize the freshly made sawdust material. Batch optimization of adsorption experimental parameters, including initial dye concentration, contact time, solution pH, temperature, and adsorbent dosage used, to achieve the maximum removal of MB dye from wastewater. For an initial MB dye concentration of 50 mg/L, the ideal parameters for the maximum removal of MB dye from aqueous solution were determined to be: 40 mg of adsorbent, 80 minutes of contact time, a pH of 6.0, and 25°C. The models that best fit the examined experimental data were the Freundlich and pseudo-second order. For the removal of MB dye, the experimental adsorption capacity of ZnO@SD was found to be 372.5 mg/g. According to the obtained results, the sawdust composite was thought to be a low-cost and efficient adsorbent for dye removal.

INTRODUCTION

All living organisms depend upon water for their survival and growth. Nowadays, the improved lifestyle, industrialization, as well as urbanization cause water contamination at a higher rate. Water contamination has worsened due to a number of factors, such as the discharge of agricultural as well as domestic waste and the presence of huge industrial residues in water bodies. Urbanization and industrialization have a significant impact on water quality because their effluents are highly contaminated with both organic and inorganic pollutants. As the industrial sector has grown, including rubber, wood, textile, and dye industries, the amount of untreated wastewater that is released into streams and water bodies has also increased (Yadav et al. 2023).

Among the pollutants found in wastewater, dyes in particular can cause major health issues for individuals because of their carcinogenicity, mutagenicity, and toxicity, even at lower concentrations. These pollutants include organic dyes, such as methylene blue (MB), which is present in high concentrations in wastewater from a variety of industries, such as the production of paper, textiles, paint, and wool (Prodjosantoso et al. 2025). Methylene blue is a synthetic dye that is highly soluble and one of the most widely used dyestuffs. The MB dyestuff contributes to increased ecosystem pollution and has detrimental effects on microbes, humans, and animals (Khan et al. 2022). As it causes a negative impact on the process of photosynthesis, the chemical and biological oxygen demands, and the quantities

of oxygen required, it impacts the entire aquatic environment and harms human health (Ahamad & Nasar 2024).

Furthermore, because of their chemical stability, they are extremely resistant to light, aerobic digestion, as well as oxidizing agents (Bo et al. 2021). As a result, wastewater treatment is crucial, and a variety of technologies, including biochemical degradation, photocatalytic, electrocoagulation, oxidative, and adsorption methods, have been used for dye removal (Pimental et al. 2023, Dutta et al. 2021). The majority of these techniques have demonstrated their suitability in treating dye-contaminated water at laboratory scales, but they have limitations when it comes to large-scale relevance because they are expensive, require a lot of energy and time, and produce large amounts of secondary sludge (Bulgariu et al. 2019, Yadav et al. 2023).

Therefore, choosing a suitable approach has always been difficult. Among the various methods, the adsorption technique has drawn a lot of interest because it is less harmful, more affordable, simpler, more flexible, and less sensitive to harmful pollutants. Additionally, this method offers adsorbent recovery and minimal sludge generation and disposal issues (Yadav et al. 2024a).

One of the recently utilised adsorbents is bio-waste and its transformation into valuable materials for specific uses. As a bio-waste from industry and agriculture, sawdust is a cheap and plentiful lignocellulosic material. In addition to different extractives (acids, soluble sugars, resins, waxes, oils, etc.), its primary constituents are cellulose, lignin, and hemicellulose. Sawdust modification enhances its ability to absorb contaminants. Therefore, various materials, such as organic compounds, basic solutions, or acidic solutions, can be used to modify it. In order to remove dyes from wastewater, metal oxides have been widely employed as promising adsorbents. Zinc oxide nanoparticles are used extensively in gas sensors, energy storage, and solar cells. Due to its high adsorptive capacity for a variety of pollutants, ZnO NPs have attracted a lot of attention in wastewater treatment (Cao et al. 2024, Yadav et al. 2024b).

According to numerous studies, ZnO NPs have shown promise as adsorbents for organic dyes (Sayed et al. 2024). A study conducted by Zafar et al. (2019) found that the Langmuir adsorption model and the pseudo-second-order kinetic model were used to fit the adsorption of amaranth (AM) and methyl orange (MO) onto ZnO NPs. A pH of 6.0 was found to yield the highest adsorption capacities. Zhang et al. (2016) reported that ZnO NPs demonstrated higher adsorption ability towards anionic dyes such as Congo red (CR) and acid fuchsin (AF), as well as the cationic dye malachite green (MG). For CR, AF, and MG, the maximum adsorption capacities were 1554 mg/g, 3307 mg/g, and 2963 mg/g, respectively. The present study aims to synthesise ZnO NPs distributed in AC made from wood sawdust (ZnO@SD composite) for MB dye adsorption. The parameters influencing the adsorption performance, including solution pH, contact duration, adsorbent dosage, and initial dye concentration, were thoroughly examined. Kinetic, isotherm, and thermodynamic properties were also examined.

MATERIALS AND METHODS

SD was used as a precursor after being gathered from a nearby sawmill. Distilled water was used to wash away any contaminants that may have adhered to the wood sawdust. It was then ground and sieved after being dried for 24 hours at 100°C in an oven. Direct pyrolysis was used to produce biochar from SD, and a muffle furnace was used to carry out the pyrolysis process. About 20 g of SD was placed in a crucible with a lid, heated for two hours at 600°C in a muffle furnace, and then allowed to cool at room temperature. After removing ash and inorganic salts with dilute HCl, the resulting biochar was cleaned with deionised water and dried at 100°C.

$ZnCl_2$ was used to chemically activate the biochar, producing activated carbon. The impregnation ratio of $ZnCl_2$ to biochar was 1:1. After dissolving 10 g of $ZnCl_2$ in 150 mL of distilled water, 10 g of biochar was added to the mixture. The mixture was allowed to stand at room temperature for six hours. After filtering the liquid portion, the remaining solids were dried in a hot oven set to 110°C for approximately 24 hours. About 10 g of impregnated biochar was placed in a lidded crucible. ZnBC (zinc biochar) was the name given to the activated carbon produced under the optimised operating conditions. Finally, the biochar was washed with dilute HCl and then with distilled water until the pH of the filtrate reached a constant value. After drying, the sample was stored in a tightly sealed container for dye treatment. (Nirmaladevi & Palanisamy 2021).

SEM–EDS and TEM were used to examine the composite's morphological characteristics. XRD was used to determine the composite's crystalline structure. FTIR (Fourier transform infrared) spectroscopy was used to identify the composite's basic functional groups.

The adsorption models were examined using methylene blue (MB), a model organic pollutant obtained from the Merck Group. A stock solution (1000 mg L^{-1}) was prepared by dissolving 1000 mg of MB in 1.0 L of distilled water. MB solutions were prepared at concentrations of 100, 150, 200, 250, and 300 mg L^{-1}. Using 0.010 M NaOH and/or HCl solutions, the pH values of the MB solutions were adjusted to 2, 4, 6, 8, and 10. A temperature-controlled shaker bath was

used to hold containers containing the mixture of adsorbent particles and adsorbate molecules at 25 °C. The following formula was used to determine the MB adsorption capacity (q_e) from the residual MB concentration:

$$\text{Adsorption efficiency (\%)} = \frac{Ci-Ce}{Ci} \times 100$$

$$\text{Adsorption capacity } (q_e) = \frac{Ci-Ce}{m} \times v$$

Where V is the volume of the solution, and m is the amount of composite used.

When the adsorption tends to equilibrium, the adsorption capacity and the remaining MB concentration are indicated by q_e and C_e, respectively".

RESULTS AND DISCUSSION

To determine which functional groups were present on the adsorbent's surface, FTIR spectroscopy was employed (Fig.1). Cellulose and hemicellulose appear to be the primary constituents in the FTIR spectra shown in Fig. 1. The FTIR spectrum revealed key functional groups in the adsorbent material. A strong absorption peak at 3354 cm^{-1} corresponds to O–H stretching vibrations of hydroxyl groups. The peak at 1745 cm^{-1} is attributed to C=O stretching from carboxylic groups in hemicellulose. A distinct band at 1516 cm^{-1} arises from –CH bending vibrations in cellulose, while the absorption at 1392 cm^{-1} is associated with C–O stretching and CH/OH bending in hemicellulose. The peak at 1039 cm^{-1} represents C–O, C–O–H, C–O–C, and ring vibrations in cellulose and hemicellulose. Finally, the band at 677 cm^{-1} suggests aromatic bending modes in cellulose (Mosoarca et al. 2021).

The XRD results for ZnO@SD are displayed in Fig. 2. Diffraction peaks were observed at 32.74°, 33.49°, 36.57°, and 55.87°, according to the XRD pattern analysis. The FWHM (full width at half maximum) data reported in the corresponding journals showed that the crystalline peaks of ZnO nanoparticles matched the JCPDS card in the 36–1451 range. Upon closer examination, ZnO exhibited comparable properties and behaviour in the range of $20° < 2\theta < 80°$. In addition to being pure and devoid of impurities, the synthesised nanopowder showed no distinctive XRD peaks other than the observed ZnO peaks (Aigbe & Kavaz 2021).

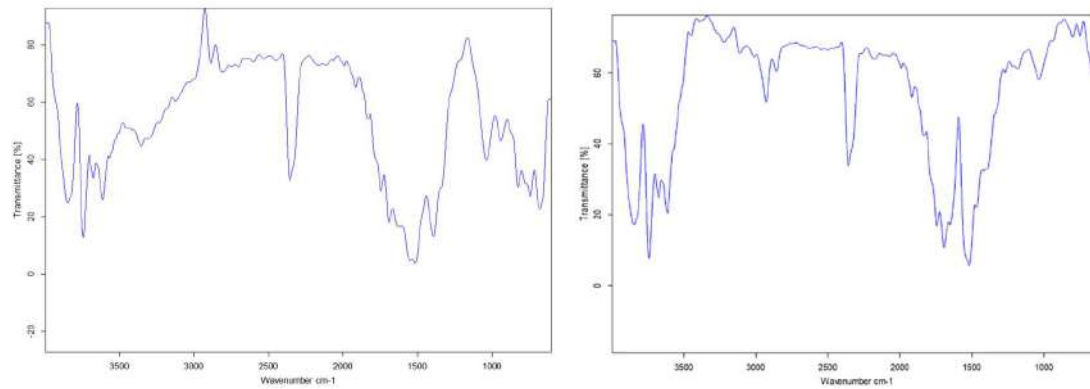

Fig. 1: FTIR spectrum of ZnO@SD before and after adsorption.

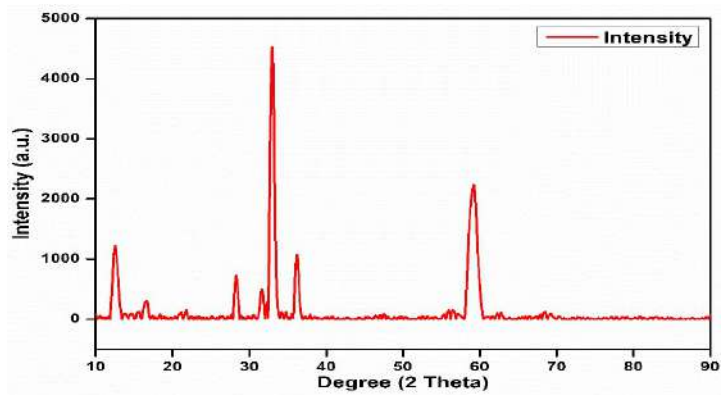

Fig. 2: XRD spectrum of ZnO@SD.

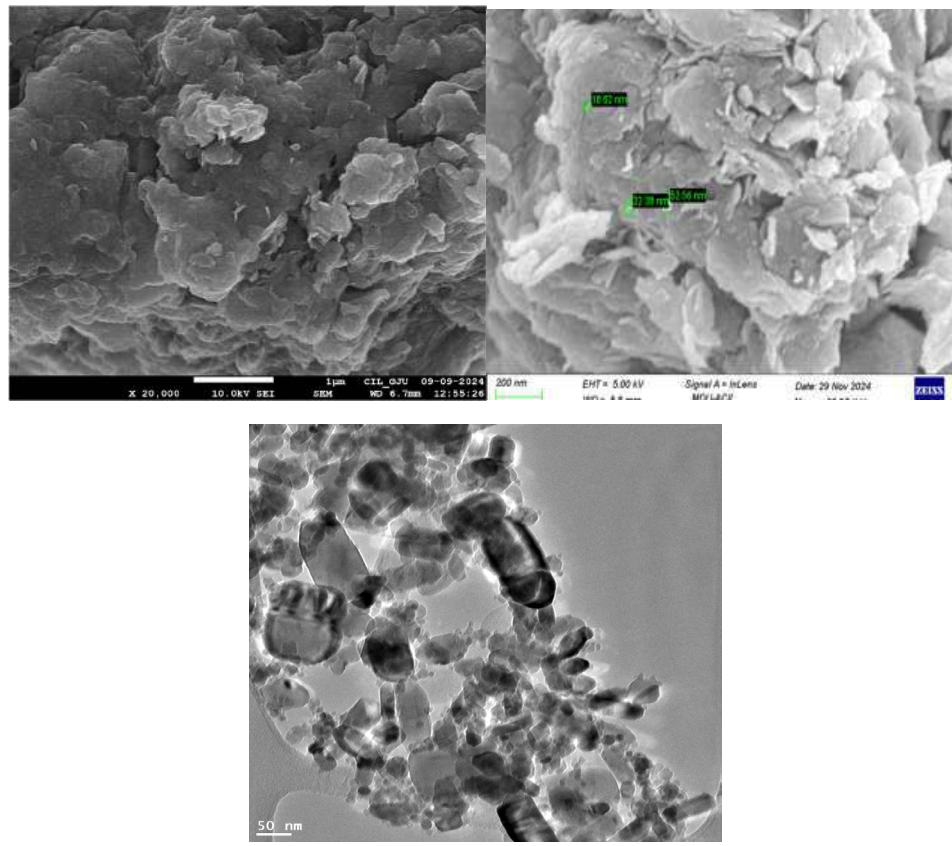

Fig. 3: SEM micrograph before and after MB adsorption, and TEM of ZnO@SD.

Fig. 3 displays ZnO@SD SEM images, which depict that the composite was found to have fewer uniform structures. In general, it can be stated that the addition of carbonized-sawdust composite altered the nanoparticles' surface morphology. The surface, which had a uniform structure and fewer cracks, became uneven. The nano-crystallites with large surface area and a strong propensity to aggregate, although the composite showed these aggregations more clearly (Aigbe et al. 2021). Multiple layers of particle arrangements are also visible, which raises the possibility of a high adsorption capacity.

The TEM image shows the porosity and surface texture of the adsorbent. It exhibited spherical surface coverage with rough, cracked surfaces and minuscule cavities. The morphology of the particles determines their suitability for adsorption. The image shows considerable roughness and a crisp texture that could enhance adsorption, and several pores are clearly visible on the surface. Extended fibrous particles and well-formed pores appear to result from the porous structure. The elemental composition of ZnO@SD nanoparticles is displayed in the EDX spectroscopy pattern in Fig. 4, which shows comparable peaks for each of the characterized samples. The patterns indicated that ZnO@SD nanoparticles had no elemental impurities and were primarily composed of Zn (8.1%), O (45.3%), and C (46.6%). The majority of organic contaminants in the sawdust biomass sample appear to have been broken down by carbonization. The EDX composition of ZnO@SD, synthesized and after MB adsorption, is displayed in Fig. 4, where Zn and O were the main compositional elements, with atomic percentages of C 73.3%, O 21.2%, and Zn 1.6%.

The adsorption of MB dye onto ZnO@SD was highly dependent on the composite's surface charge distribution and the pH of the solution. The pH of the solution affected the electrostatic interactions, which could be either repulsive or attractive, between the active sites on the surface of the composite and the MB dye species. The effect of changing the solution pH on MB dye adsorption is depicted in Fig. 5. It was found that the percentage of MB dye ions sorbed onto ZnO@SD increased significantly as the solution pH was changed from 2.0 to 12.0, with pH 6.0 showing the highest percentage removal of MB dye ions.

Since surface sites on ZnO@SD suggest the presence of significantly more negatively charged groups at low pH,

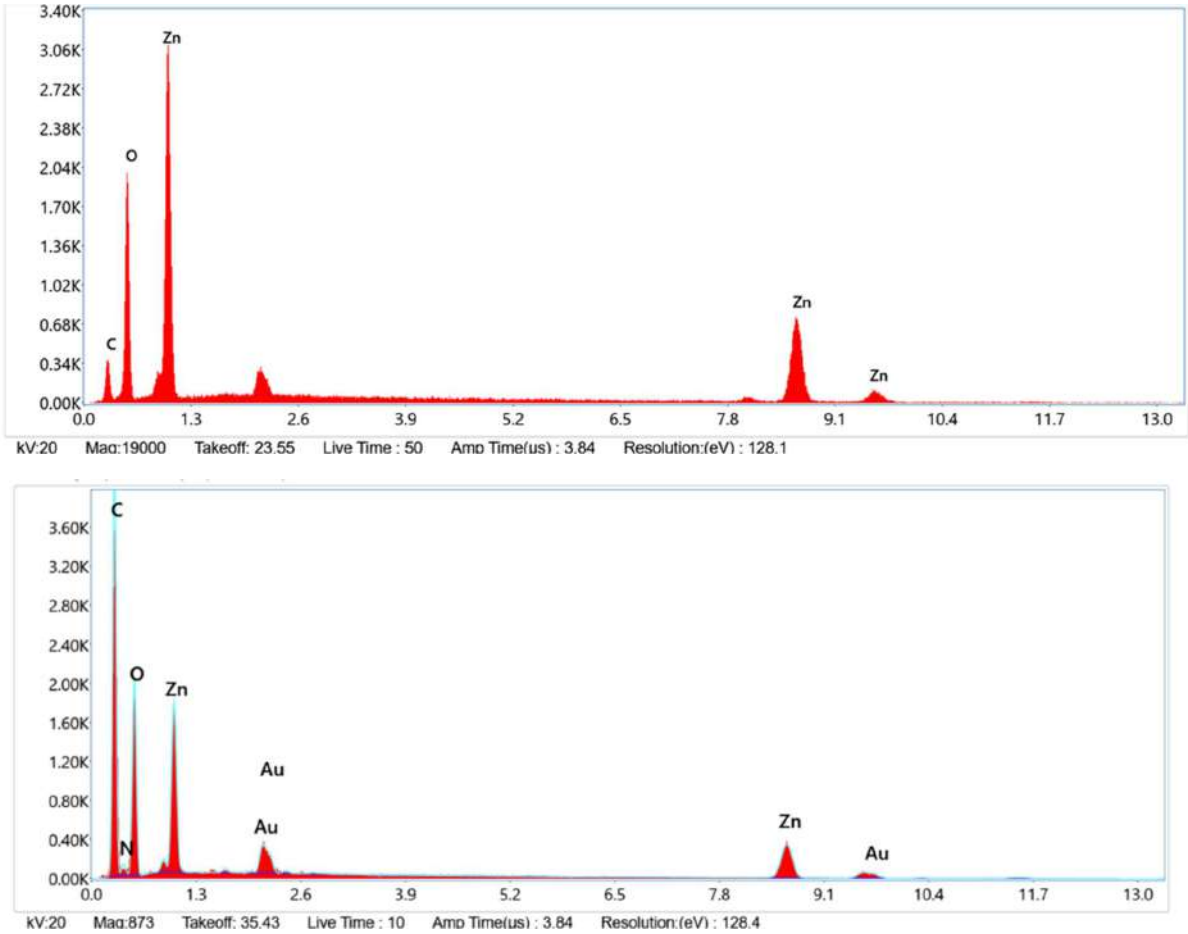

Fig. 4: EDS spectrum of ZnO@SD before and after adsorption of MB.

the lower percentage of MB dye adsorbed onto ZnO@SD at pH 2 was attributed to electrostatic repulsion between the negative surface charges and the cationic, undissociated MB species. Additionally, the percentage of MB dye removed at acidic pH decreased due to increased competition between MB^+ ions and H^+/H_3O^+ ions for biochar adsorption sites. At low pH values, interactions such as hydrogen bonding, π-π or π^+-π interactions, and pore diffusion were responsible for the adsorption of MB dye onto ZnO@SD (Phuong et al. 2019).

The improved percentage of MB dye removal at higher pH was attributed to electrostatic attraction between the dominant MB dye species and the increasingly negative surface charges as the solution pH increased. It has been reported that raising the pH of the solution increases the number of hydroxyl (-OH) and carboxylate ($-COO^-$) groups on the ZnO@SD surface, which in turn increases the number of negatively charged sites (Phuong et al. 2019). Studies by Phuong et al. (2019) and Al-Ghouti & Al-Absi (2020) revealed a similar pattern. The primary sorption mechanisms of MB dye at acidic and basic pH were hypothesized to be electrostatic interactions and hydrogen bonding (Al-Ghouti & Al-Absi 2020, Eldeeb et al. 2024).

Since it regulates the adsorption performance in the efficient removal of contaminants, the adsorbent dosage is an important parameter. The experiment used doses of ZnO@SD ranging from 10 mg to 80 mg. When the adsorbent dosage is increased from 10 mg to 70 mg, the percentage removal using ZnO@SD improves from 74.5 to 96.3% at equilibria (Fig. 5). Increased surface area and more binding sites for adsorption are the causes of the sorbate's constant improvement in percentage uptake with adsorbent dose. Nevertheless, an opposing trend in adsorption capacity was noted, as shown in Fig 5. The maximum adsorption capacity of 372.5 $mg.g^{-1}$ for M.B. by the adsorbent was noted at the lowest adsorbent dose. In light of this, it is important to note that adsorbent particles interact through amalgamation and aggregation at higher doses, resulting in a decrease in effective surface area per unit weight of adsorbent. It could

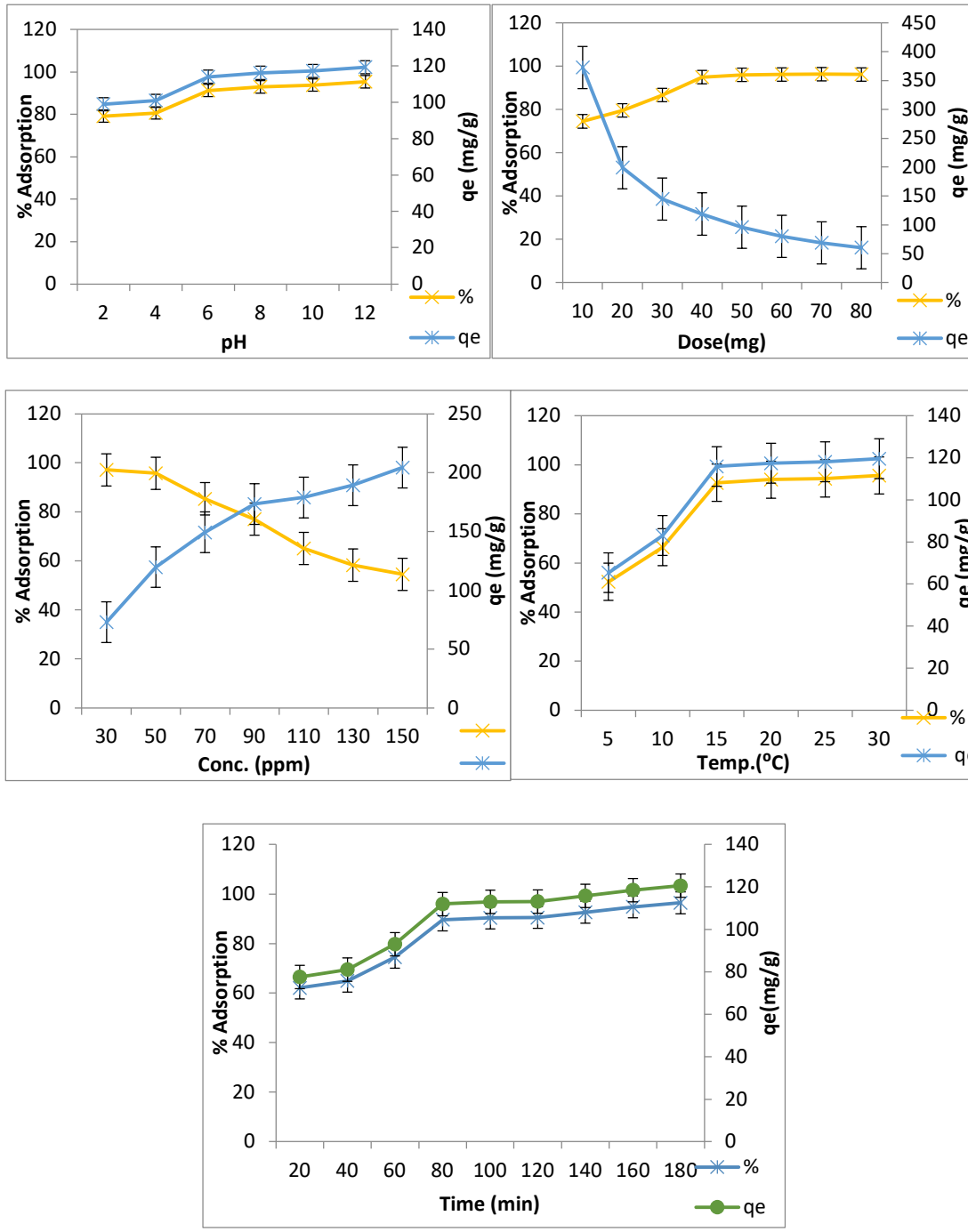

Fig. 5: Effect of pH of solution, adsorbent dosage, initial dye concentration, temperature and time on removal %age and adsorption capacity.

be explained by an increase in the adsorbent's surface area and an enhancement in accessibility of more active surface sites on the sorbent (Garg et al. 2004).

On the other hand, as the dose is increased, the adsorption capacity continuously and significantly decreases. Adsorbent particle interactions, such as agglomeration and aggregation, cause the adsorption capacity to decrease as the sorbent dose increases. As a result, the adsorbents' total surface area per unit weight decreases.

Methylene Blue (MB) dye adsorption tests were performed at various contact durations ranging from 20 to 180 min with an initial MB concentration of 50 ppm to

examine the adsorption kinetics. The effects of contact time on adsorption efficiency were examined at 298 K, 40 mg, and 6 pH. As contact time increased to 80 minutes removal %age rose to 89.56%, and the adsorption finally reached equilibrium. Fig. 5 shows how the initial MB concentration affected adsorption efficiency. There were sporadic variations in the curves of different starting concentrations. As the MB concentration ranged from 10 to 150 mg.L^{-1}, the highest adsorption efficiency of 97.13% at 30 ppm was noted.

The persistent active sites found it difficult to bind the increasing MB molecules from the bulk solution, even though there were enough active sites to explain the results at low initial MB dye concentrations. At higher methylene blue (MB) concentrations, increased electrostatic repulsion between dye cations reduced the adsorption efficiency. However, elevating the initial MB concentration also enhanced the adsorption capacity of the ZnO@SD composite. This occurred because a higher dye concentration provided a stronger driving force, helping to overcome mass transfer resistance between the aqueous phase and the adsorbent surface.(Hassaan et al. 2023).

Adsorption isotherm modeling can be used to describe the interaction of dye molecules with the sorbent at solid/liquid interfaces. Three isotherm models—the Langmuir, Freundlich, and Temkin models—were applied to the experimental data of dye adsorption at different dye concentrations (10–150 mg.L^{-1}). The Langmuir isotherm model is based on the assumption that adsorption occurs in a monolayer, where dye molecules bind to a finite number of identical and energetically equivalent active sites on the adsorbent surface. This model also presumes that no interactions occur between adsorbed molecules (Langmuir 1916). In contrast, the Freundlich isotherm model is an empirical equation that describes multilayer adsorption on a heterogeneous surface with non-uniform binding sites. Unlike the Langmuir model, it does not assume a saturation limit, implying an infinite number of active sites with varying adsorption energies (Freundlich 1906). According to the Temkin model, the adsorbate's heat of adsorption decreases linearly with monolayer coverage, and the surface has an even distribution of active binding sites (Temkin & Pyzhev 1940). The models' linear plots are displayed in Fig. 6. Table 1 summarizes the calculated regression coefficients and

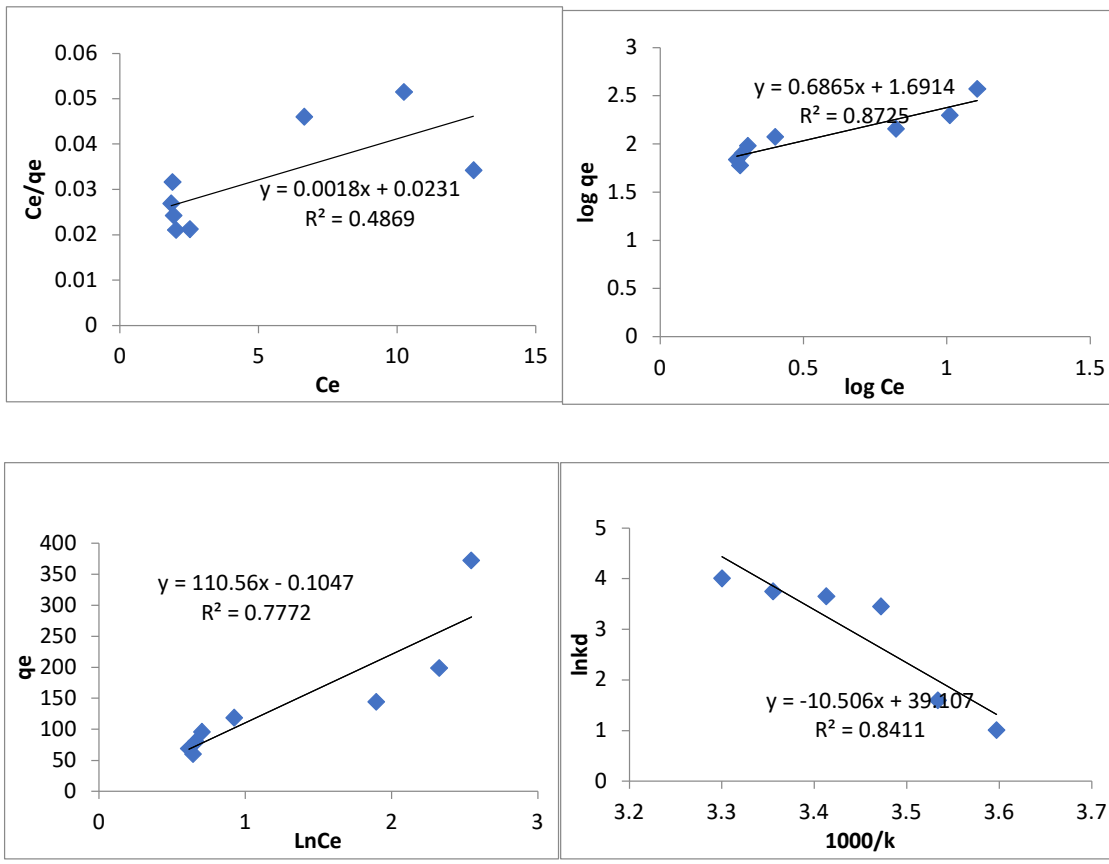

Fig. 6: Isotherms: Langmuir, Freundlich and Temkin, and Vant Hoff plot.

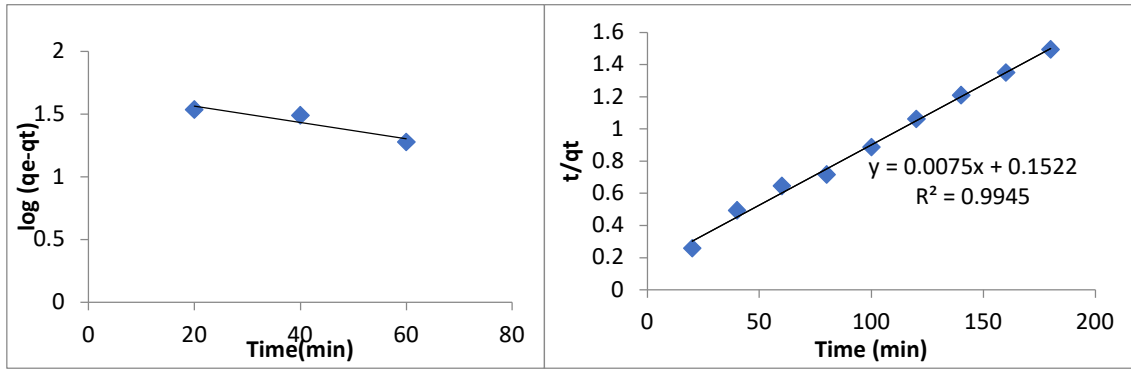

Fig. 7: Kinetics study.

isotherm parameters for each model using the corresponding linear plots. Each model's regression coefficient and isotherm parameters were used to determine its applicability to the dye adsorption experiments. The comparison revealed that the Freundlich model best described the MB dye adsorption process, indicating that MB dye molecules were adsorbed in multiple layers onto the heterogeneous surface-active sites.

For MB dye, the Freundlich model's R^2 was determined to be 0.87. The fact that the 1/n (slope) value ranged from 0 to 1, or 0.686 for MB, suggests that adsorption was favorable and that the dye molecules chemically bonded to the adsorbent surface, indicating chemisorption. A 1/n value less than 1 indicates chemisorption on the adsorbent's heterogeneous surface. The MB dye's R^L value was found to be 0.206 ($0 < R^L < 1$), confirming a favorable adsorption process. It was observed that MB dye had a maximum monolayer adsorption capacity (q_m) of 555.5 $mg.g^{-1}$, while the experimental value was 372.5 $mg.g^{-1}$. Consequently, the chemisorption of dye molecules onto ZnO@SD was validated by the isotherm models.

Temperature ranges of 278–303 K were examined because thermodynamic research offers more details regarding the adsorption mechanism of ZnO@SD on MB. The van't Hoff equation was used to calculate all thermodynamic parameters, including enthalpy (ΔH), entropy change (ΔS), and the standard Gibbs free energy (ΔG), which showed how the equilibrium constant depended on temperature. The equilibrium constant (K) was determined from the adsorption isotherm data using the following equation:

$K = q_e/C_e$, while the positive value of ΔS° (0.325 $kJ.moL^{-1}$ K^{-1}) indicated that the randomness at the solid/solution interface increased, and the positive value of enthalpy ΔH° (87.29 $kJ.moL^{-1}$), one of the thermodynamic parameters shown in Table 1, confirmed that the adsorption of MB onto ZnO@SD was endothermic in nature. This was because the adsorbate ions lost less transitional energy than the water molecules displaced by the adsorbate species. The adsorption mechanism of MB onto the ZnO@SD nanocomposite was a favorable, spontaneous process, as indicated by the negative values of ΔG°. Enthalpy values $\Delta H° < 84$ $kJ.moL^{-1}$ indicate physisorption, while values between 84 and 420 $kJ.moL^{-1}$ indicate chemisorption. The observed value of 87.29 $kJ.moL^{-1}$ suggests chemisorption, placing it on the borderline between physisorption and chemisorption. Multilayer adsorption or pore-filling mechanisms (physisorption) may be possible, along with the potential

Table 1: Isotherms, thermodynamics and kinetics.

	Parameters	Values
Langmuir	q_m	555.5
	B	0.077
	R_L	0.206
	R^2	0.48
Freundlich	1/n	0.686
	K_f	48.97
	R^2	0.87
Temkin	A_T	1.00
	R^2	0.77
Thermodynamics	ΔS°	0.325
	ΔH°	87.29
	ΔG° 278K	-1.67
	ΔG°283K	-3.27
	ΔG°288K	-4.87
	ΔG°293K	-6.47
	ΔG°298K	-8.07
	ΔG°303K	-9.67
PFO	q_e	1.01
	K1	3.89
	R^2	0.87
PSO	q_e	133.33
	K2	0.00037
	R^2	0.99

involvement of strong chemical interactions (ligand exchange, complexation) supporting chemisorption. The influence of temperature on the adsorption mechanism is noted, as higher ΔH° values often correlate with chemisorption but may also reflect high-energy physical adsorption. Consequently, the results suggest that chemical interaction may be the primary mechanism underlying the sorption of MB onto ZnO@SD.

Pseudo-first-order rate kinetics and pseudo-second-order rate kinetics models were used to further study the adsorption phenomenon, and Table 1 summarizes the parameters that were obtained. Table 1 shows that the kinetics of MB adsorption onto ZnO@SD nanocomposite are described by the pseudo-second order rate kinetics model. In comparison to the pseudo-first-order model, the linear regression values derived from this model yielded a higher correlation coefficient (R^2) of >0.99. The model's suitability for describing the adsorption process is further supported by the observation of linear curves in Fig. 7, which also shows fitment to pseudo-second order. It's possible that the methylene blue interaction with the adsorption site was chemical in nature because the adsorption process adheres to the pseudo-second-order model.

It was proposed that chemical adsorption played a role in achieving the highest MB adsorption from simulated water, given the scale used in the linearized form of the pseudo-second-order model. Pseudo-second-order kinetics was also used to model the early stage of kinetic experimental data to verify this (Fig. 7). Based on this evidence, the adsorption kinetics of MB onto ZnO@SD nanocomposite are primarily diffusion-based mechanisms. During a rate-limiting mechanistic step, various adsorption sites on a homogeneous solid substrate randomly collide with one another and diffuse through the adsorbent pore size.

CONCLUSIONS

Sawdust is a cheap and plentiful lignocellulosic material that is a bio-waste product of industry and agriculture. In this study, mixed sawdust was used as the raw material to produce ZnO@SD adsorbent. ZnO@SD has a network structure and works well as an adsorbent for cationic dyes because it has a lot of amino, hydroxyl, and carboxyl groups. ZnO@SD can be used to adsorb cationic dye MB in water and has a favorable separation capacity, high removal rate, and good adsorption capacity. The aforementioned processes are well-fitted by the Freundlich adsorption isotherm and the pseudo-second-order kinetic model. ZnO@SD's easy preparation method and superior adsorption capabilities. This work offers useful ZnO@SD adsorbents for the elimination of cationic dyes from wastewater. It is a low-cost bio-waste sawdust, which is a sustainable, scalable, and environmentally friendly method that can show great promise for a variety of additional uses in the future.

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Heavy Metals Removal from Polluted Water by Cement Kiln Dust

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ABSTRACT

Two laboratory experiments were conducted in the Desert Studies Center laboratories during the spring season of 2024 to evaluate the efficiency of Cement Kiln Dust (CKD) in removing lead and cadmium from polluted water. The first experiment represents the thermally isotropic adsorption of heavy metals on the cement kiln dust (CKD) surface. The experiment included three diameters of CKD particles, i.e., 0.3, 1.18, and 2 mm, treated with four concentrations of cadmium and lead, namely 20, 40, 80, and 160 mg.L^{-1} for each element (cadmium and lead). The amount of the adsorbed metals on the surface of CKD was calculated according to the Langmuir equation. In contrast, the second experiment represents the effect of contact time between the heavy metals and CKD particles for the same aforementioned diameters and concentrations. The results of the isothermal adsorption experiment showed that the adsorbed amount increased with increasing the added concentrations of heavy metals. The adsorbed quantity of cadmium is superior to that of lead. Also, the adsorption capacity of cadmium was higher compared to the lead adsorption capacity. In this context, the adsorption capacity reached 2880.00 and 2735.58 mg.kg^{-1} for cadmium and lead, respectively. Regarding the second experiment, the results showed that the amount of cadmium and lead adsorbed on the CKD particle's surface increased with time, where the highest amount of cadmium and lead adsorption was 39.94 and 34.93%, respectively, for shaking of 4 h. It is recommended to apply the experiment in real-world projects.

INTRODUCTION

Water pollution is considered one of the major global problems because it poses adverse economic impacts to life, exposes health to danger, and hinders the development of industrial and agricultural activity. Under an insufficient water supply, economic activities may be hampered or even halted (Yang et al. 2024). Excessive heavy metal concentrations in polluted water can indisputably pose a threat to ecological and human health risks by deteriorating freshwater resources such as rivers and streams (Hanif et al. 2025). Consequently, this restricts their use for agricultural, domestic, and industrial purposes (Kumar et al. 2022). Saeed et al. (2024) proposed using some techniques to mitigate the hazards of pollutants in the future. Universally, the agriculture sector consumes over 70% of global freshwater withdrawals. Therefore, wastewater reuse is a reliable and practical way to cope with freshwater scarcity and to provide a sustainable water source for the agricultural sector. However, the high cost of treating polluted water has caused many countries to discharge the polluted water without treatment into freshwater sources such as rivers, lakes, and streams, resulting in adverse effects on human, animal, and plant life. A rapid increase in urbanization and the industrial revolution has recently been attributed to an increase in the production of different kinds of contaminants, such as air pollutants, solid waste, and production of wastewater. Cement industry factories are the largest contributors to the production of these pollutants (Majeed et al. 2021). The cement factories discharge approximately 11

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million tons/year of solid waste (Millati et al. 2019). Cement Kiln Dust (CKD) represents the largest by-product of cement factory solid waste and is classified as a non-hazardous by-product (Seo et al. 2019). Previous studies indicated that the cement kiln dust consists of quartz, sodium chloride, and small amounts of gypsum, in addition to containing quantities of coagulants such as CaO, MgO, Al_2O_3, and Fe_2O_3, which make it a good adsorbent material (Rimal et al. 2019). Due to its low cost compared to lime and other coagulants and adsorbents, it could reduce the cost of waste treatment with the same performance (Kiran et al. 2019). Also, due to its availability and eco-friendliness, CKD has recently been used in polluted water treatment. Due to unmanaged anthropogenic activities, toxic heavy metals, such as lead (Pb) and cadmium (Cd), are continuously accumulating in agricultural soils. Plants mainly absorb heavy metals from the contaminated soil solution by roots, resulting in their accumulation in plant parts and transmission through the food chain to humans, posing potential health risks for consumers (Garg et al. 2014). It has become essential to remove heavy metals from water, especially wastewater (Al-Fahdawy et al. 2024a). Furthermore, the efficiency of using Injana clay formation for cadmium sorption was investigated by Ouda et al. (2024). It was concluded that the cadmium concentration was removed within the first 10 minutes of the experiments. The adsorption efficiency was found to

Table 1: The specification of Cement Kiln Dust.

Z	Symbol	Element	Norm Int.	Concentration	Abs. Error
11	Na_2O	Sodium	68.3702	2.082	0.027
12	MgO	Magnesium	167.9553	1.314	0.009
13	Al_2O_0	Aluminum	762.0226	2.028	0.005
14	SiO_2	Silicon	5572.71	7.406	0.008
15	P_2O_5	Phosphorus	47.7422	0.0313	0.0011
16	SO_3	Sulfur	12699.73	4.824	0.004
17	CL	Chlorine	2742.246	0.2004	0.0003
19	K_2O	Potassium	276.6975	0.8989	0.0043
20	CaO	Calcium	15123.35	52.12	0.04
22	TiO_2	Titanium	42.4222	0.2168	0.0034
23	V_2O_5	Vanadium	4.0752	0.014	0.0017
24	Cr_2O_3	Chromium	7.4002	0.00368	0.00024
25	MnO	Manganese	159.4424	0.1436	0.001
26	Fe_2O_3	Iron	5108.417	3.35	0.004
27	CoO	Cobalt	0.00000	0.00039	0.0000
28	NiO	Nickel	19.3438	0.00473	0.00017
29	CuO	Copper	20.6685	0.00412	0.00013
30	ZnO	Zinc	47.0666	0.00711	0.0001
31	Ga	Gallium	2.1121	0.0002	0.00007
32	Ge	Germanium	0.00000	0.00005	0.0
33	As_2O_3	Arsenic	21.0834	0.00152	0.00016
34	Se	Selenium	3.3357	0.00016	0.00003
35	Br	Bromine	63.4311	0.00287	0.00004
37	Rb_2O	Rubidium	180.7971	0.005031	0.00004
38	SrO	Strontium	5320.73	0.1524	0.0002
39	Y	Yttrium	19.6576	0.00046	0.00003
40	ZrO_2	Zirconium	12.3474	0.00601	0.00038
41	Nb_2O_5	Niobium	0.00000	0.00014	0.0
42	Mo	Molybdenum	3.2408	0.00084	0.00007
47	Ag	Silver	0.00000	0.0002	0.0
48	Cd	Cadmium	0.7683	0.0002	0.0
50	SnO_2	Tin	5.65900	0.00091	0.00009
51	Sb_2O_5	Antimony	3.7924	0.00072	0.0001
52	Te	Tellurium	4.71830	0.0003	0.0
53	I	Iodine	2.5906	0.0002	0.00007
55	Cs	Cesium	0.00000	0.0004	0.0
56	Ba	Barium	31.2651	0.02821	0.00098

increase as the temperature increased. The urgent need to understand human exposure to heavy metals in contaminated soils and to prevent the great threat resulting from human and environmental exposure to these metals is due to the considerable threats caused by heavy metal pollution to marine and terrestrial environments (Ekere et al. 2020, Al-Fahdawy et al. 2024b). The extensive contamination of soils with Cd and Pb, especially in agricultural soils, illustrates the need to highlight the risks caused by heavy metal accumulation (Zhao et al. 2022). The agricultural areas surrounding the Kubaisa cement factory were reported to be affected by the dust produced from the cement factory, which has an impact on the agricultural area by heavy metals (lead and cobalt) (Gharbi et al. 2024). Therefore, the current study was conducted to evaluate the efficiency of Cement Kiln Dust (CKD) in removing lead and cadmium from polluted water.

MATERIALS AND METHODS

A laboratory experiment was conducted at Anbar University, Desert Studies Center, during the spring season of 2024. In this study, two heavy metals were selected, namely Cd (II) and Pb (II). The selected heavy metals were prepared from their salts ($PbCl_2$ and $CdCl_2$) with a concentration of 1000 $mg.L^{-1}$ by dissolving 2.51 and 2.099 g in one litre of distilled water for lead and cadmium, respectively. The quantity of each heavy metal was calculated by dividing the atomic weight of each element by the required concentration, divided by the total atomic weight. On this basis, the above concentration was prepared. Due to its availability, cement kiln dust (CKD) was used in this study as an adsorbent. The CKD by-product was brought from the Kabaisa Cement Factory, located west of Ramadi city. The analysis was conducted in the Center of Desert Studies Lab – University of Anbar. The specification of the used by-product is listed in Table 1. Thermal isotropic adsorption experiment of heavy metals on the adsorbent material.

One gram of cement kiln dust (CKD) was weighed with different diameters, namely 0.3, 1.18, and 2 mm, using sieves. Four concentrations, i.e., 20, 40, 80, and 160 $mg.L^{-1}$ of each considered heavy metal (cadmium and lead), were added to each diameter. The mixture of CKD and heavy metal solution was placed in a flask and shaken for 2 h. The mixture was filtered using Whatman 42 filter paper. The output extract was collected using plastic containers to estimate the concentrations of heavy metals (cadmium and lead) in the resulting extract using an atomic absorption device. The amount of these adsorbed metals on the treatment material (CKD) was calculated and subjected to the Langmuir equation to describe the adsorption. The Langmuir equation, derived by Langmuir (1918), was used to describe the adsorption of heavy metals lead and cadmium. The linear equation is:

$$C/X = \frac{1}{kXm} + \frac{1}{Xm}C \quad \text{... (1)}$$

Where: C is the concentration of heavy metals in the equilibrium solution ($mg.L^{-1}$), X is the amount of heavy metals adsorbed on the material surfaces ($mg.kg^{-1}$), Xm is the maximum adsorption, and K is the bonding energy.

Effect of the Contact Time of the Treatment Material Used on the Concentration of Heavy Metals

One gram of cement kiln dust was taken (which is the by-product of the Kabaisa Cement Factory), with different diameters of 0.3, 1.18, and 2 mm, and mixed with different concentrations of 20, 40, 80, and 160 $mg.L^{-1}$ of heavy metals (lead and cadmium). The mixture was placed in tightly closed plastic containers. The containers were subjected to different shaking times, namely 0.5, 2, and 4 h for each element separately. After the shaking process was completed, the mixture was filtered using filter paper. The collected extract was kept in plastic containers and transferred to the laboratory to determine the heavy metal concentration using atomic absorption.

RESULTS AND DISCUSSION

Determining the thermally isotropic adsorption curves for heavy metals (cadmium and lead) on the used surface particles of CKD, when different concentrations of heavy metals were applied to the water, provided a comprehensive understanding of the nature of the reaction and the adsorbed amount (Fig. 1). It is noted that the adsorbed amount of cadmium and lead on the used surface particles of CKD increased with increasing the applied concentration of heavy metals in water (20, 40, 80, and 160 $mg.L^{-1}$).

Also, it can be seen from Fig. 1 that the adsorbed cadmium is superior to that of lead, since the amount adsorbed depends mainly on the nature of the adsorbed material. Moreover, the adsorption of cadmium and lead is similar to a C-type isotherm curve, which indicates the mechanism of partition adsorption, where the adsorbed material is distributed between the separation surface of the solid and liquid phase without any connection occurring between the adsorbed material and the adsorption surface (Sposito 2008). The adsorption capacity of cadmium was higher compared to the adsorption capacity of lead, as the adsorption capacity of cadmium reached 2880.00 $mg.kg^{-1}$, while the adsorption capacity of lead reached 2735.58 $mg.kg^{-1}$. Regarding the thermally isotropic adsorption, the values of Langmuir equation parameters shown in Fig. 2 and listed in Table 3 show that the highest adsorption observed

for lead reached 3333.333 mg.kg⁻¹ with a binding capacity of 0.0245 L.mg⁻¹, which is superior to that of cadmium, which had an adsorption of 2000 mg.kg⁻¹ and a binding capacity of 0.0105 L.mg⁻¹. The bond strength of lead on the surface of the adsorbent may be due to lower hydration energy, which makes it less strongly bound to water molecules and potentially more available for adsorption onto the CKD surface (Alghamdi et al. 2019). However, the coefficient of determination (R^2) for the simple linear regression for cadmium is higher than the coefficient of determination for lead, with values reaching 0.9968 and 0.9738, respectively. The superiority of the coefficient of determination for cadmium over lead may be due to the smaller ionic radius of cadmium, which gives it the ability to fit easily into smaller pores or spaces on the adsorbent surface compared to lead.

Effect of the Contact Time of CKD Particles on the Concentration of Cadmium (20 mg.L⁻¹)

Fig. 3 shows the effect of contact time on cadmium adsorption for different shaking times. The results show that different cadmium concentrations affect the cement kiln dust adsorption efficiency, which indicates that CKD

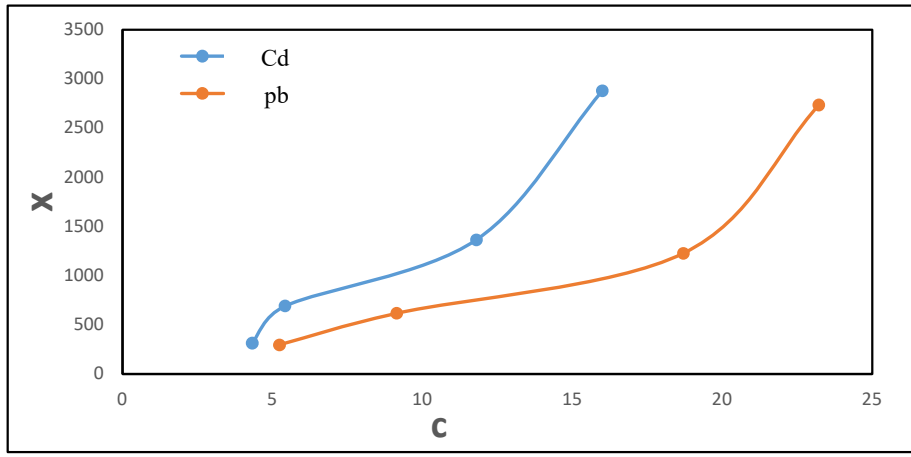

Fig. 1: The relationship between the concentration of the equilibrium solution and the adsorbed amount.

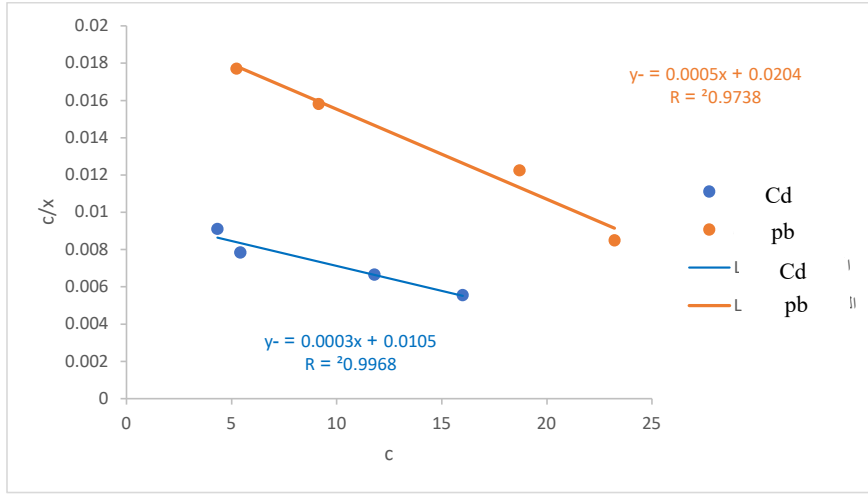

Fig. 2: Thermal isotropic adsorption according to the Langmuir equation for the adsorption of some heavy metals on the adsorbent material.

Table 2: Parameters of thermal isotropic adsorption of selected heavy metals on the adsorbent materials

Adsorbent material	Heavy metals	Maximum adsorption [mg.kg^{-1}]	Binding energy [L.mg^{-1}]
CKD	Cd	2000.000	0.0245
	Pb	3333.333	0.0105

may have an effect in regulating the cadmium concentration in the medium (Naushad et al. 2019). The CKD adsorption efficiency depends on several factors, such as pH, the chemical composition of CKD, and time (Elbaz et al. 2019). With increasing the CKD particle diameter from 0.3 to 2.0 mm, the adsorption shows increasing efficiency but only up to a certain point. After that, the adsorption starts to reach a saturation state. In this regard, the highest amount of cadmium adsorption reached 566.31 $\mu g.g^{-1}$ with a removal rate of 28.32% at a concentration of 20 $mg.L^{-1}$ of cadmium, 4 h of shaking time, and 2 mm diameter of CKD particles. The cadmium adsorption decreased to 409 $\mu g.g^{-1}$ with a removal rate of 20.45% at 4 h of shaking time and 0.3 mm diameter of CKD particles. Decreasing the adsorption when the diameter of the CKD particles decreased to 0.3 mm may be attributed to the aggregation of these particles, which limited pore accessibility, decreased effective surface area, kinetic limitations, and potential competitive adsorption. The combination of these factors could explain the decrease in cadmium adsorption to 409 $\mu g.g^{-1}$. Similarly, the adsorbed amount of cadmium decreased to 350.11 $\mu g.g^{-1}$ with a removal rate of 17.51% when the shaking time decreased to 0.5 h, and the diameter of CKD particles was 0.3 mm. The decrease in the adsorption rate compared to the aforementioned treatment may be due to the decrease in both CKD particle diameter and shaking time.

Concerning 1.18 mm and 4 h of shaking time, the cadmium adsorption slightly differs compared to 2 mm of CKD particles and 4 h of shaking time, where the adsorption reached 498.66 $\mu g.g^{-1}$ with a removal rate of 24.94% at 20 $\mu g.g^{-1}$ of cadmium concentration, 4 h of shaking time, and 1.18 mm diameter of CKD particles. Decreasing shaking time under the same CKD particle diameter (1.18 mm) led to a decrease in cadmium adsorption to 402.51 $\mu g.g^{-1}$ with a removal rate of 20.13%.

Effect of the Contact Time of CKD Particles on the Concentration of Cadmium (40 $mg.L^{-1}$)

Fig. 4 shows that increasing the concentration of cadmium from 20 $mg.L^{-1}$ to 40 $mg.L^{-1}$ in the solution led to an increase in the amount of adsorbed cadmium, where the

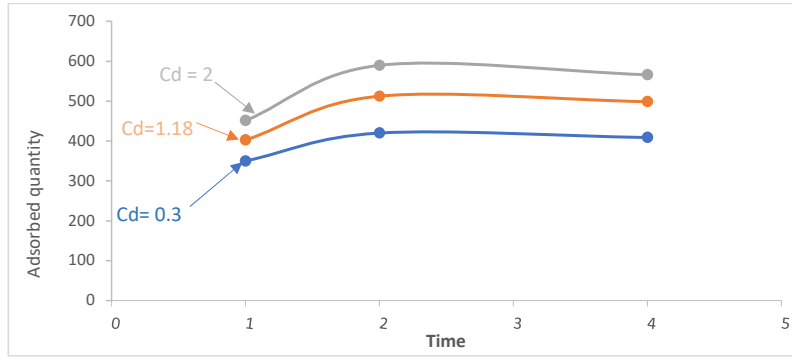

Fig. 3: Contact time for cadmium at a concentration of 20 and different diameters of CKD (0.3,1.18, and 2 mm).

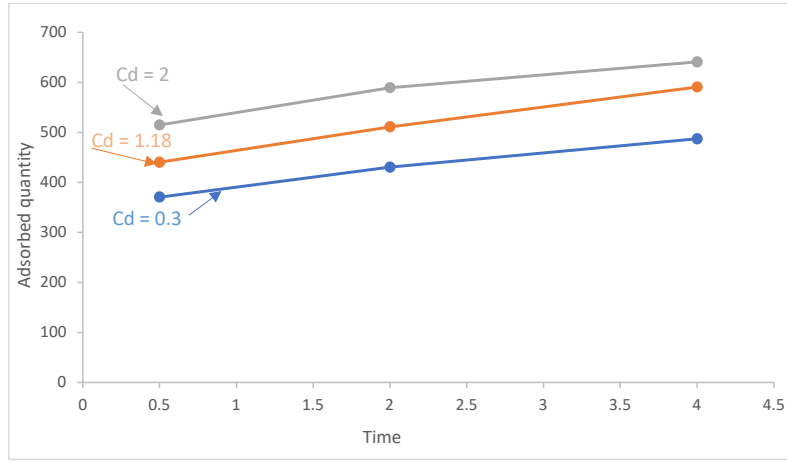

Fig. 4: Contact time for cadmium at a concentration of 40 and different diameters of CKD (0.3,1.18, and 2 mm).

line of adsorption became steeper and took a near-linear state. The steep behavior of cadmium adsorption (increases in the amount of adsorbed cadmium) can be attributed to the increased driving force for adsorption, availability of cadmium ions in the solution, and enhanced mass transfer (Basu et al. 2019). The combination of these three factors generates a linear relationship between contact time and the adsorbed cadmium.

The adsorbed cadmium increased with increasing shaking time. In this context, the highest amount of cadmium adsorption reached 640.65 μg.g^{-1} with a removal rate of 32.04% at 40 μg.g^{-1} of cadmium concentration, 4 h of shaking time, and 2 mm diameter of CKD particles. Decreasing the diameter of cement kiln dust particles led to a decrease in cadmium adsorption to 486.98 μg.g^{-1} with a removal rate of 24.35% at 4 h of shaking time and 0.3 mm diameter of CKD particles.

The reduction of cadmium adsorption might be attributed to the fact that during the shaking process, large molecules may maintain their structure more than small molecules that aggregate to form larger molecules (more than 2 mm), thus reducing the adsorption efficiency. The same pattern of decline was observed at 0.5 h of shaking time and 0.3 mm diameter of CKD particles, where the adsorbed cadmium reached 370.51 μg.g^{-1} with a removal rate of 18.53%. The decrease in cadmium adsorption was a result of the combination of reduced shaking time and the small diameter of the CKD particles.

Increasing both the shaking time and the diameter of cement particles (4 h and 1.18 mm) led again to an increase in the adsorption efficiency, where the adsorbed cadmium reached 590.76 μg.g^{-1} with a removal rate of 29.54%, while the adsorbed cadmium decreased to 370.51 μg.g^{-1} with a removal rate of 18.53 % at ½ h shaking time and 1.18 mm diameter of CKD particles.

Effect of the Contact Time of CKD Particles on the Concentration of Cadmium (80 mg.L^{-1})

Fig. 5 showed that increasing the cadmium concentration to 80 mg led to the affinity of the adsorption curves, indicating saturation of all sites on the surface of the adsorbent. The same trend was observed at 80 μg.g^{-1} concentration of cadmium, where the bigger particles were superior to the smaller particles (0.3 mm) in adsorption efficiency. In this sense, the adsorbed cadmium increased with increasing shaking time (Fig. 5). The highest amount of cadmium adsorption was 662.32 μg.g^{-1}, with a removal rate of 34.02%, observed at a concentration of 80 μg.g^{-1}, a shaking time of 4 h, and a 2 mm diameter of CKD particles. Also, the decrease in the diameter of cement kiln dust particles to 0.3 mm, with constant shaking time (4 h), led to a decrease in the efficiency of cadmium adsorption, where the adsorbed cadmium reached 560.64 μg.g^{-1} with a removal rate of 28.04%. As previously mentioned, decreasing the shaking time and cement kiln dust diameter led to a decrease in the adsorbed cadmium, where the adsorbed cadmium reached 410.60 μg.g^{-1} with a removal rate of 20.53% at 0.5 h of shaking time and a 0.3 mm diameter of CKD particles.

When the amount of adsorbed cadmium on the adsorbent reached equilibrium, the amount of adsorbed cadmium reached 560.64 μg.g^{-1} with a removal rate of 28.04%, at 80 μg.g^{-1} of cadmium concentration, 4 h of shaking time, and

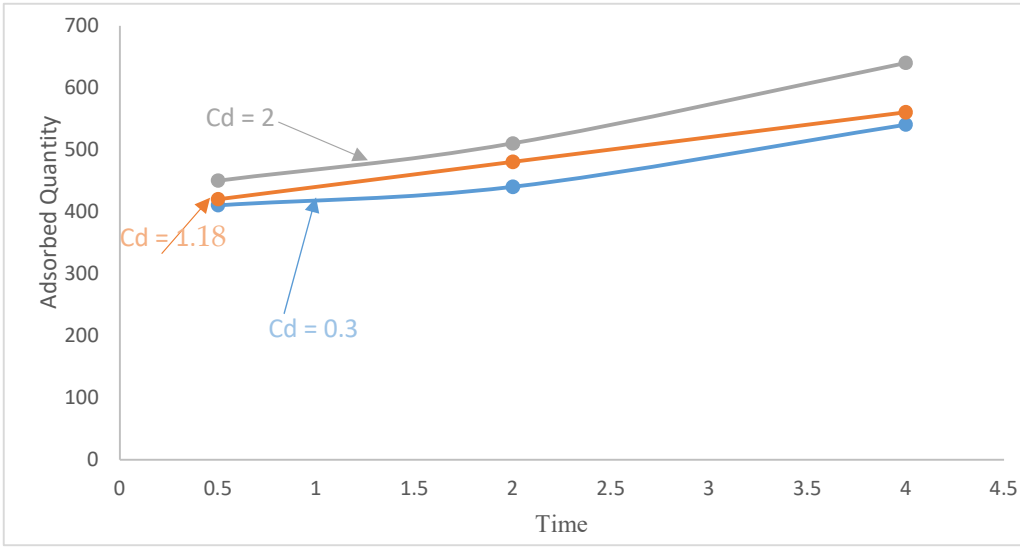

Fig. 5: Contact time for cadmium at a concentration of 80 and different diameters of CKD (0.3,1.18, and 2 mm).

1.18 mm diameter of CKD particles. The adsorbed cadmium decreased to 420.08 μg.g^{-1} when the shaking time decreased to half an h, with a removal rate of 21.01% under the same CKD particle diameter (1.18 mm).

Effect of the Contact Time of CKD Particles on the Concentration of Cadmium (160 mg.L^{-1})

The observed results showed an interaction between the treatments due to increasing the cadmium concentration to 160 μg.g^{-1}, which increased the adsorption speed at the beginning of the reaction time (first h) until most of the active sites on the surfaces of the adsorbent material were saturated (Fig. 6). After that, the reaction speed decreased. The continued closeness between the diameters of 0.3 and 1.18 mm may be due to them containing approximately the same number of active sites on the surfaces, due to the accumulation of cement particles with a diameter of 0.3 mm into diameters approaching 1.18 mm. Increasing cadmium to 160 μg.g^{-1} led to an increase in the amount of cadmium adsorbed (Fig. 6). In this context, the highest amount of cadmium adsorption reached 798.62 μg.g^{-1} with a removal rate of 39.94% at 160 μg.g^{-1} of cadmium concentration, 4 h of shaking time, and 2 mm diameter of CKD particles. The amount of adsorbed cadmium decreased to 609.72 μg.g^{-1} with a removal rate of 30.49% at 4 h of shaking time and 0.3 mm diameter of CKD particles. The same pattern of decline was observed at 0.5 h of shaking time and 0.3 mm diameter of CKD particles, where the adsorbed cadmium reached 501.10 μg.g^{-1} with a removal rate of 25.06%.

When the amount of adsorbed cadmium on the adsorbent reached equilibrium, the amount of adsorbed cadmium reached 630.12 μg.g^{-1} with a removal rate of 31.51%, at 160 μg.g^{-1} of cadmium concentration, 4 h of shaking time and 1.18 mm diameter of CKD particles, while the adsorbed cadmium

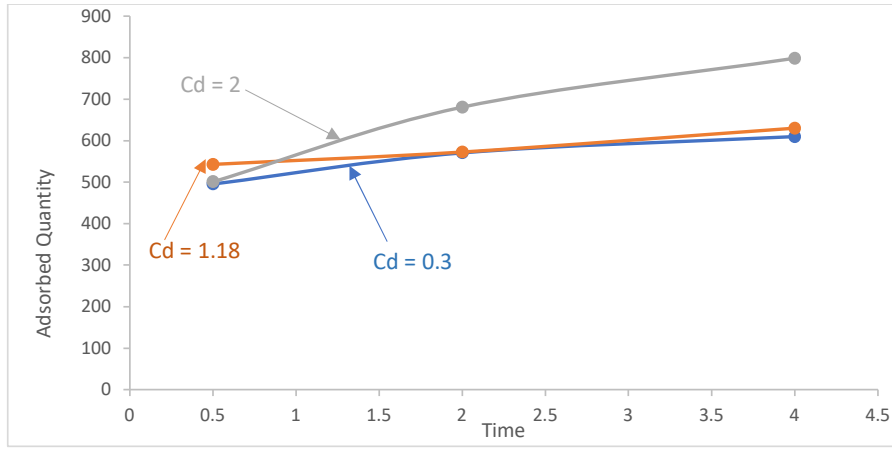

Fig. 6: Contact time for cadmium at a concentration of 160 and different diameters of CKD (0.3,1.18, and 2 mm).

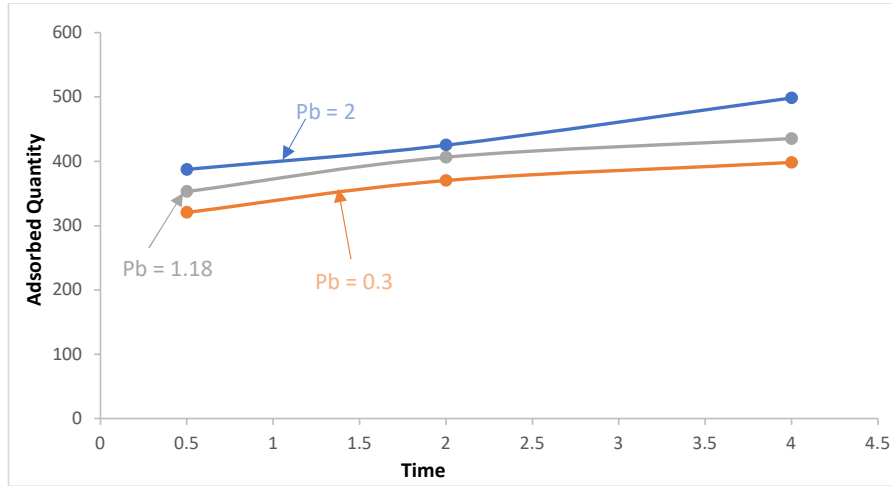

Fig. 7: Contact time for lead at a concentration of 20 and different diameters of CKD (0.3,1.18, and 2 mm).

decreased to 542.75 μg.g^{-1} with a removal rate of 27.14% at ½ h shaking time and 1.18 mm diameter of CKD particles.

Effect of the Contact Time of CKD Particles on the Concentration of Lead (20 mg.L^{-1})

Unlike cadmium, lead adsorption at a concentration of 20 mg.L^{-1} was lower (Fig. 7). Despite that, the results showed that the amount of adsorbed lead had almost a positive relationship with time (Fig. 7), where the highest amount of lead adsorption reached 498.68 μg.g^{-1} with a removal rate of 24.94% of the added amount at 20 μg.g^{-1} of lead, 4 h of shaking time, and 2 mm diameter of CKD particles. The adsorbed lead decreased to 387.30 μg.g^{-1} as a result of a decrease in the diameter of the CKD particles to 0.3 mm, with a removal rate of 19.36% at 4 h of shaking time. Increasing the diameter of the CKD particles to 1.18 mm, along with increasing the shaking time to 4 h, led to an increase in the amount of adsorbed lead up to 435.22 μg.g^{-1}, with the removal rate reaching 21.76%. The lowest amount of adsorbed lead was at a shaking time of 0.5 h, reaching 352.98 μg.g^{-1} with a removal rate of 17.64%.

As shown in Fig. 7, the amount of adsorbed lead on the adsorbent material increases with time. The highest amount of lead adsorption at a shaking time of 4 h and a diameter of 0.3 mm reached 398.24 μg.g^{-1}, with a removal rate of 19.91%, while the lowest removal rate was at a shaking time of 0.5 h, reaching 320.60 μg.g^{-1}, with a removal rate of 16.03%.

Effect of the Contact Time of CKD Particles on the Concentration of Lead (40 mg.L^{-1})

By increasing the concentration of lead to 40 mg.L^{-1}, the adsorption of lead on the surface of the adsorbent material increased (Fig. 8). However, this adsorption was still lower compared to the adsorption of cadmium under the same conditions. The superiority of cadmium over lead in the adsorption process on the CKD surface may be due to differences in their chemical properties, such as ionic radius, charge density, and lead ion binding affinity on the CKD surface. In this sense, the highest amount of lead adsorption at a shaking time of 4 h and a diameter of 2 mm was 540.61 μg.g^{-1}, with a removal rate of 27.03% of the added amount. The smallest amount of adsorbed lead was at a shaking time of 0.5 h, reaching 389.76 μg.g^{-1}, with a removal rate of 19.48%. Concerning the amount of lead adsorption at a shaking time of 4 h and a diameter of 1.18 mm, it was 498.65 μg.g^{-1}, with a removal rate of 24.93% of the added amount. The smallest amount of adsorbed lead was at a shaking time of 0.5 h, reaching 375.46 μg.g^{-1}, with a removal rate of 18.77% of the added amount.

Also, Fig. 8 shows that the adsorbed lead on the adsorbent material increased with time, where the highest amount of lead adsorption at a shaking time of 4 h and a diameter of 0.3 mm reached 400.31 μg.g^{-1}, with a removal rate up to 20.01%, while the lowest removal rate was at a shaking time of 1/2 h, reaching 351.45 μg.g^{-1}, with a removal rate of 17.57%.

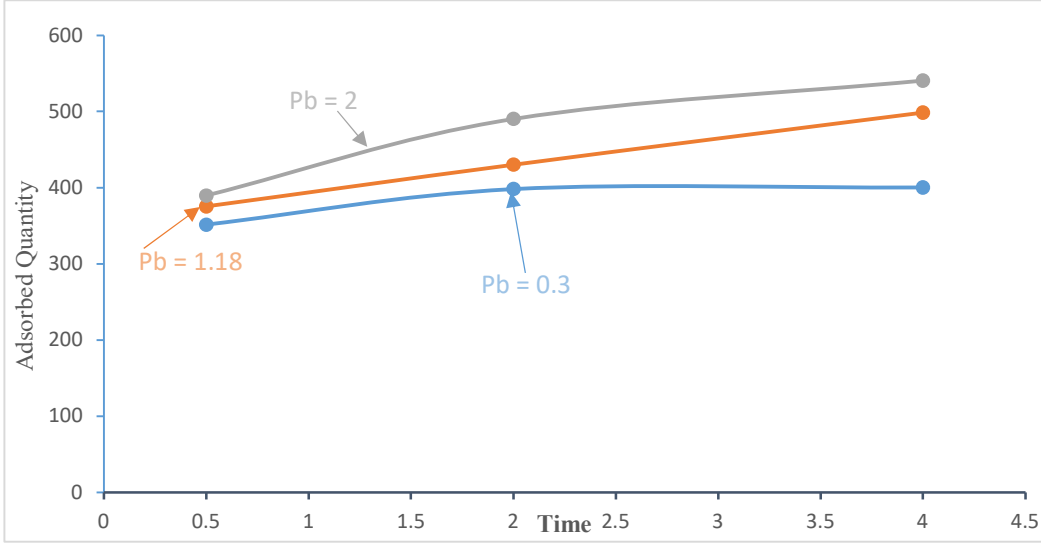

Fig. 8: Contact time for lead at a concentration of 40 and different diameters of CKD (0.3,1.18, and 2 mm).

Effect of the Contact Time of CKD Particles on the Concentration of Lead (80 mg.L^{-1})

Fig. 9 shows the high affinity in the adsorption process between diameters 1.18 and 2 mm. This is probably because larger diameters have a larger surface area for reaction, resulting in higher adsorption efficiency. Conversely, smaller diameters, such as 0.3 mm, may be subject to aggregation, which reduces the overall adsorption efficiency compared to larger diameters. Supporting this theory is the superiority of the diameter of 2 mm over other diameters (0.3 and 1.18 mm). It is noted from the Fig.9 that the amount of adsorbed lead on the adsorbent increases with increasing contact time, where the highest amount of adsorbed lead was observed at a contact time of 4 h and a diameter of 2 mm, reaching 624.40 µg.g^{-1}, with a removal rate up to 31.20, while the lowest amount of lead adsorption was 433.54 µg.g^{-1}at a contact time of 1/2 h, with a removal rate of 21.67%. The amount of adsorbed lead increased when the size of cement kiln dust particles and the shaking time increased. At a shaking time of 4 h and the diameter of 1.18 mm was 572.31 µg.g^{-1}, with a removal rate of 28.61% of the added amount. The smallest amount of adsorbed lead was 487.05 µg.g^{-1} at a shaking time of 1/2 h, with a removal rate of 24.35%.

Furthermore, Fig. 9 shows that the adsorbed lead on the adsorbent surface increased with time, where the amount of adsorbed lead increased from 401 µg.g^{-1} after two h to 486.34 µg.g^{-1} after four h of shaking time and at 0.3 mm of CKD particles, with a removal rate of up to 24.31%, while the lowest removal rate was at a shaking time of 0.5 h, reaching 401.2 µg.g^{-1}, with a removal rate of 20.06%.

Effect of the Contact Time of CKD Particles on the Concentration of Lead (160 mg.L^{-1})

Fig. 10 shows that the adsorption at diameters 0.3 and 1.18 mm was slightly different during the first 1.5 h from the start of the reaction. Later, the match between the 0.3 and 1.18 mm adsorption lines becomes perfect, highlighting the similarity of the adsorption on the adsorbent (0.3 and 1.18 mm). This is consistent with earlier observations indicating that small particles (0.3 mm) tend to aggregate, forming larger particles. Consequently, their behavior mimics that of larger particles.

Concerning the particles with a diameter of 2 mm, the adsorption continued to increase even after 4 h. The continuation of adsorption at a diameter of 2 mm and when adsorption lines emerge at diameters 0.3 and 1.18 mm supports the conclusion that larger diameters of cement kiln dust are more efficient in the adsorption process than smaller particles.

Regarding the adsorption efficiency, Fig. 10 shows that the adsorbed lead on the adsorbent material increases with time, where the highest amount of lead adsorption at a shaking time of 4 h and a diameter of 2 mm was 698.76 µg.g^{-1}, with a removal rate of up to 34.93%. The smallest adsorbed lead was observed at a shaking time of 0.5 h, which amounted to 488.64 µg.g^{-1}, with a removal rate of 24.43%. The adsorption increased again with increasing shaking time and diameter of cement particles, where at a shaking time of 4 h and a diameter of 1.18 mm, it reached 576.60 µg.g^{-1}, with a removal rate of 28.83%. The smallest amount of adsorbed lead was at a shaking time of 0.5 h, reaching 487.00 µg.g^{-1}, with a removal rate of 24.35%.

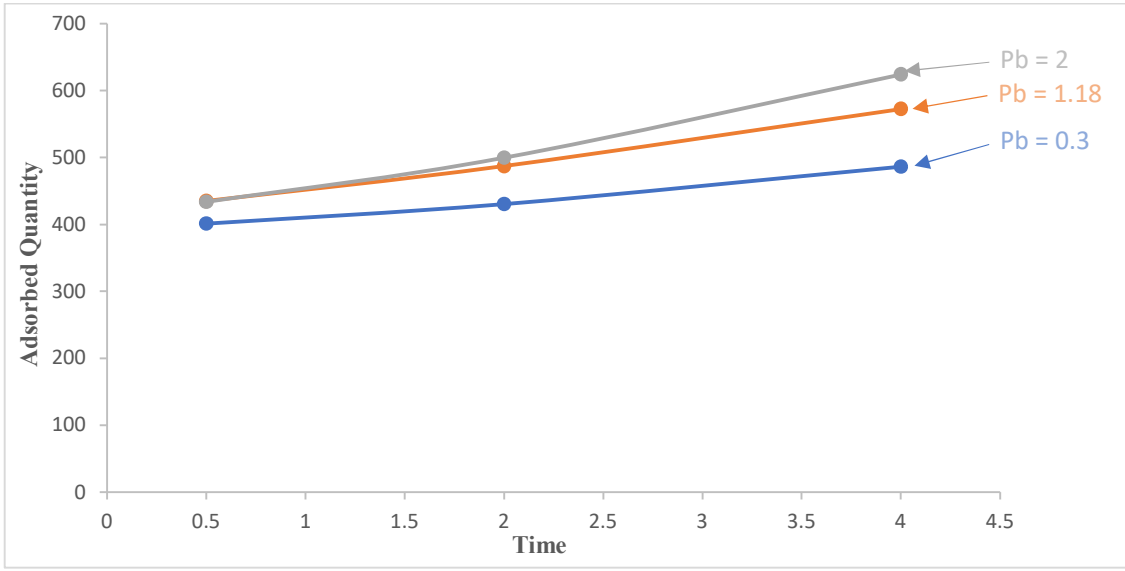

Fig. 9: Contact time for lead at a concentration of 80 and different diameters of CKD (0.3,1.18, and 2 mm).

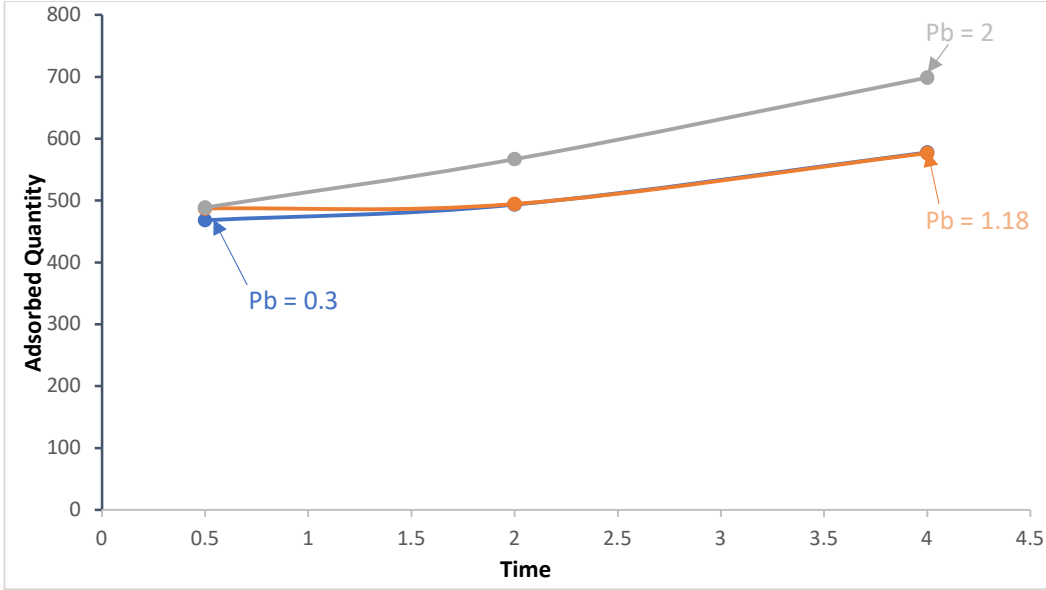

Fig. 10: Contact time for lead at a concentration of 160 and different diameters of CKD (0.3,1.18, and 2 mm).

It is noted from Fig. 10 that the amount of adsorbed lead on the adsorbent increases with time. The highest amount of lead adsorption at a shaking time of 4 h and a diameter of 0.3 mm reached 577.62 $\mu g.g^{-1}$, with a removal rate of 28.88%, while the lowest removal rate was observed at a shaking time of 1/2 h, reaching 468.00 $\mu g.g^{-1}$, with a removal rate reached 23.40%.

CONCLUSIONS

The current study found that the concentration of cadmium and lead ions had different adsorption efficiencies. The findings show that the adsorbent (CKD) absorbs the cadmium ions more efficiently than lead. Also, the study findings show that the bigger particles had bigger surfaces to absorb the polluted ions. In terms of concentration, the study shows that the adsorption efficiency increased with increasing concentration due to the ion availability in the polluted aqueous solution. Moreover, the study shows that small particles require a longer time to complete the adsorption process. Perhaps the reason for this is that small particles are more likely to collect than large particles. This technology can be used to remove heavy elements from sewage, and treatment systems can be created to get rid of these elements. There are future ideas for this work.

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Pharmaceutical Wastes as Emerging Groundwater Contaminants: A Review of Their Sources, Fate, Health Impacts, and Techniques for Analytical Detection and Treatment

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ABSTRACT

Pharmaceutical wastes are emerging as significant groundwater contaminants, raising concerns over their long-term impacts on public health and ecosystems. This review examines the major sources of contamination—such as wastewater treatment plants (WWTPs), septic systems, industrial discharges, and agricultural runoff—and explores how physicochemical properties and subsurface interactions influence their environmental fate. Although advanced analytical tools, such as liquid chromatography–tandem mass spectrometry (LC-MS/MS), have improved detection at trace levels, challenges in effective removal persist. Frequently detected contaminants include non-steroidal anti-inflammatory drugs (NSAIDs), antibiotics, antidepressants, and hormone-disrupting compounds, often present in concentrations ranging from nanograms to micrograms per litre. Promising treatment technologies, including advanced oxidation processes and activated carbon adsorption, are discussed alongside their limitations. This review highlights the urgent need for comprehensive monitoring programmes, cost-effective remediation methods, and further investigation into the chronic effects of low-dose pharmaceutical exposure.

INTRODUCTION

Pharmaceuticals are essential to modern medicine and are widely used in both human and veterinary healthcare for treating infections, alleviating pain, regulating hormones, and managing psychological conditions. Major classes include antibiotics, anti-inflammatories, hormones, β-blockers, and lipid regulators (Xiang et al. 2021, Boxall et al. 2012). However, the widespread use of these compounds, coupled with their partial metabolism and poor removal in conventional treatment systems, has resulted in their persistent accumulation in the environment-particularly in groundwater (Ortúzar et al. 2022).

Pharmaceutical residues reach groundwater through various pathways, including effluent discharge from wastewater treatment plants (WWTPs), leaking sewer systems, agricultural runoff, industrial effluents, landfill leachates, and improper drug disposal (Shaheen et al. 2022). Consequently, groundwater-an essential source of drinking water—is increasingly contaminated with a diverse array of pharmaceutical compounds, such as antibiotics, antidepressants, hormones, and anti-inflammatory agents (Morin-Crini et al. 2022, Nag et al. 2023).

Despite significant advancements in analytical detection-particularly through techniques such as liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS), the effective removal of pharmaceuticals remains a challenge. Most existing treatment systems are not designed to target such compounds, and their chronic, low-level presence poses potential long-term risks to ecosystems and human health. Furthermore, transformation products generated during degradation

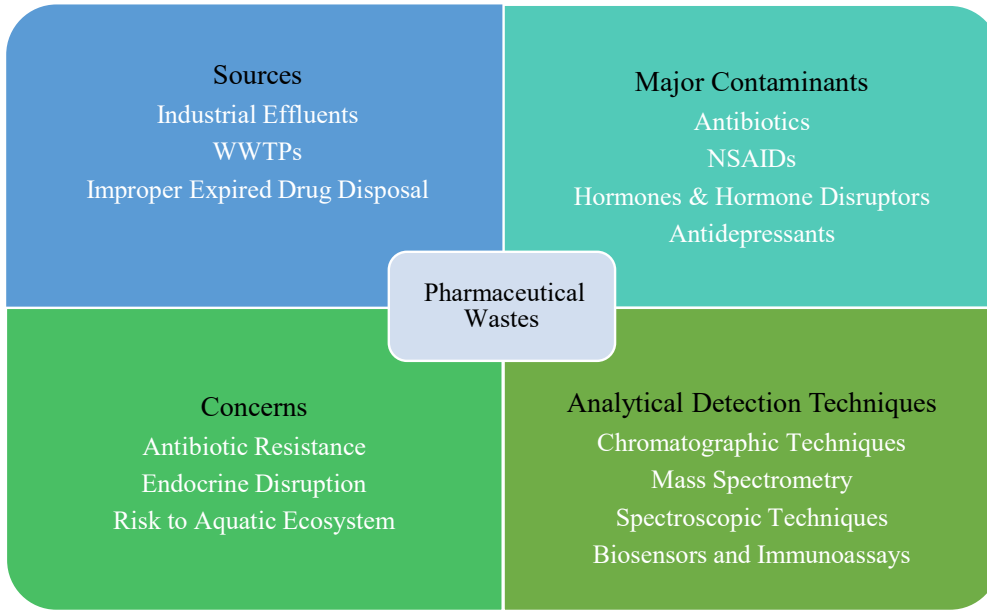

Fig. 1: Conceptual Overview of Pharmaceutical Wastes in the Environment.

processes may be equally or more toxic and persistent than the parent compounds.

Although awareness of pharmaceutical contamination is increasing, comprehensive reviews specifically focused on their occurrence and behaviour in groundwater systems remain limited. This review addresses that gap by synthesising current knowledge on the sources, environmental fate, health risks, detection methods, and treatment technologies related to pharmaceutical contaminants in groundwater. The review also highlights emerging research challenges and emphasises the need for improved regulatory and remediation frameworks.

MATERIALS AND METHODS

This review was conducted following PRISMA guidelines. Peer-reviewed studies published between 2005 and 2024 were retrieved from databases including Scopus, Web of Science, PubMed, MEDLINE, AGRIS, UGC CARE, and Reaxys.

Search Strategy

Search terms included combinations of "pharmaceutical contaminants," "groundwater pollution," "NSAIDs fate," "LC-MS/MS detection," and "treatment technologies," using Boolean operators.

Selection Criteria

Included studies focused on pharmaceutical contamination in groundwater, covering sources, fate, detection, health risks, and treatment. Excluded were non-peer-reviewed articles, surface water-only studies, or those lacking methodological rigor.

Screening and Data Extraction

Out of 450 initial articles, 400 remained after duplicates were removed. After abstract screening and full-text review, 92 studies were selected. Extracted data included compound types, concentrations, analytical methods, and treatment outcomes. Results were synthesized thematically and presented in tables where applicable.

OCCURRENCE AND SOURCES OF PHARMACEUTICAL CONTAMINANTS

Pharmaceutical residues are increasingly detected in groundwater globally, typically at concentrations ranging from nanograms to micrograms per litre. Their presence is primarily attributed to anthropogenic sources and the limited removal efficiency of wastewater treatment systems. Key contributors include municipal WWTPs, leaky sewer lines, industrial effluents, agricultural runoff, and improper drug disposal (Lapworth et al. 2012, Luo et al. 2014).

A nationwide study conducted in China investigated 35 pharmaceutical compounds in influent and effluent samples from 12 WWTPs across different cities. The results revealed significant regional variation, with WWTPs in northern cities exhibiting higher influent concentrations than those in the south. Caffeine was reported at the highest influent concentration, reaching up to 1775.98 $ng.L^{-1}$. Despite advanced infrastructure in some facilities, the overall

removal efficiency was low: only 14.3% of pharmaceuticals achieved a mean removal efficiency greater than 70%, while 51.4% were removed at rates below 30% (Liu et al. 2017). These findings highlight not only the prevalence of pharmaceutical pollutants in urban wastewater systems but also the inefficiency of existing treatment frameworks, especially in rapidly urbanising regions.

In contrast, many European countries have adopted tertiary treatment technologies such as ozonation, activated carbon adsorption, and membrane filtration-resulting in higher removal efficiencies (up to 80–95% for selected compounds). However, even these advanced systems are often ineffective at fully removing persistent pharmaceuticals such as carbamazepine and diclofenac (Zhang et al. 2008, Luo et al. 2014).

Pharmaceuticals also enter groundwater through leaky sewer systems, surface runoff, improper disposal, and agricultural practices. In urban areas, ageing infrastructure allows compounds such as oestrogens and NSAIDs to migrate into shallow aquifers, as documented in Kraków, Poland (Rusiniak et al. 2021). In rural areas, veterinary pharmaceuticals from manure, biosolids, and reclaimed water used in irrigation contribute to diffuse contamination (Kümmerer 2009). Industrial effluents represent another major source, particularly in regions with inadequate environmental regulation. For instance, elevated levels of ibuprofen and naproxen were detected in soil and groundwater near pharmaceutical manufacturing facilities in Pakistan (Ashfaq et al. 2017), while hospital-associated WWTPs in South Africa were found to discharge multiple pharmaceuticals, including antibiotics, -blockers, and antihistamines (Kanama et al. 2018). These examples illustrate that pharmaceutical contamination in groundwater is both widespread and multifactorial. Urban areas tend to be affected by centralised WWTP inefficiencies and infrastructure leaks, while rural and peri-urban zones are impacted more by agricultural runoff and decentralised disposal practices. Regional differences in regulation, treatment technology, and pharmaceutical consumption patterns further influence contaminant profiles.

FATE AND TRANSPORT IN GROUNDWATER

The behavior of pharmaceuticals in groundwater is governed by their physicochemical properties-such as solubility, polarity, molecular weight, and ionization potential, as well as site-specific environmental conditions. Hydrophilic

Table 1: Prevalence and levels of frequently identified pharmaceutical pollutants across various matrices in multiple countries (Alqarni 2024).

Pharmaceutical Contaminants	Classification	Occurrence	Concentration	Country	References
Ofloxacin and Ciprofloxacin	Antibiotics	WWTP influent	1000–2200 $ng.L^{-1}$	Italy	Verlicchi et al. 2012.
Tramadol, ofloxacin, gemfibrozil, atenolol, caffeine, and cetirizine	Analgesic, antibiotics, lipid regulator, β-blocker, stimulant and antihistamine	Irrigation water	1100–4400 $ng.L^{-1}$	Spain	García-Valverde et al. 2023.
Estrone, 17b-estradiol, estriol, 2-hydroxyestrone, 16α-hydroxyestrone, 4-hydroxyestrone, 2-hydroxyestradiol, 4- hydroxyestradiol, 17-epiestriol, 16 keto-estradiol, and 16-epiestriol	Steroidal hormones	WWTP (effluent & influent)	n.d.–62.9 $ng.L^{-1}$	China	Tang et al. 2020
		River water	n.d.–51.7 $ng.L^{-1}$		
Ketoprofen, diclofenac, and indomethacin	Analgesic/NSAIDs	Sludge	4.4–77 $ng.g^{-1}$	Japan	Matsuo et al. 2011.
Amoxicillin	Antibiotics	STP	172 $ng.L^{-1}$	India	Mutiyar & Mittal et al. 2013.
Atenolol, ciprofloxacin, sulfamethoxazole, ranitidine	β-blocker, Antibiotics and antihistamine	WWTP (effluent and influent)	34–3585 $ng.L^{-1}$	Saudi Arabia	Mostafa et al. 2023.

STP sewage treatment plant, n.d.: Not detected

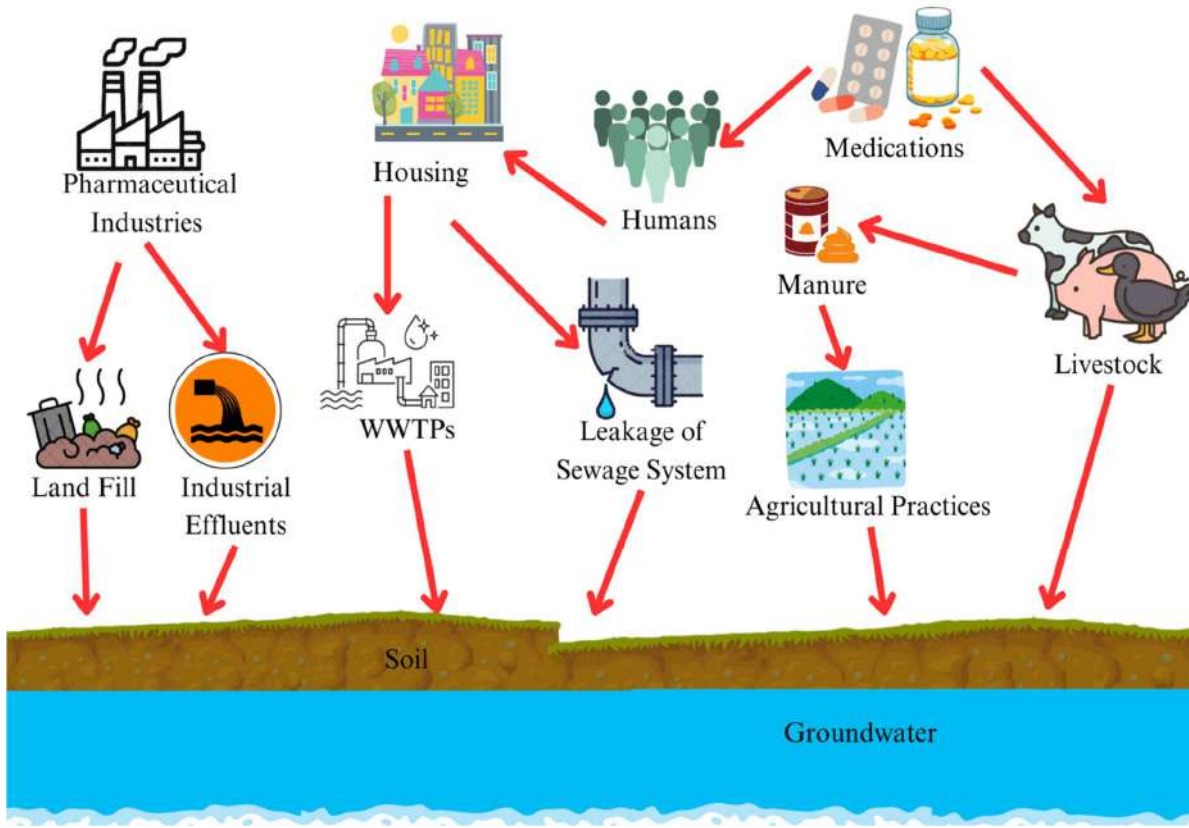

Fig. 2: Overview Schematic Representations of Various Sources of Pharmaceutical Contaminants in Groundwater.

compounds like acetaminophen and caffeine migrate more readily through aquifers, whereas hydrophobic or ionizable compounds, such as carbamazepine and diclofenac, tend to adsorb to organic matter, limiting their mobility but enhancing persistence (Lv et al. 2019, Clara et al. 2005).

Environmental factors, including redox conditions, pH, temperature, and microbial activity, influence degradation rates and transport dynamics (Jelic et al. 2011). Some pharmaceuticals persist in aquifers for extended periods, long after their initial release, reflecting their environmental stability (Lapworth et al. 2012).

Table 2: Different classes and nature of pharmaceutical drug (Chander et al. 2016).

Pharmaceutical classes	Pharmaceutical drugs	Nature of Pharmaceutical Drug
Anti-inflammatory	Aspirin	Hydrophilic
	Diclofenac	Varies
	Ibuprofen	Moderate hydrophobic
Antidepressants	Clofibric acid	Moderate hydrophobic
	Bezafibrate	Hydrophobic
	Fenofibric acid	Hydrophobic
Antiepileptics	Carbamazepine	Moderate hydrophobic
Antibiotics	Ciprofloxacin	Hydrophilic

Wastewater treatment efficiency also affects pharmaceutical transport. Advanced technologies like membrane bioreactors and sequencing batch reactors provide improved removal but still leave detectable residues of compounds such as ofloxacin and clarithromycin (Liu et al. 2017, Zhang et al. 2008).

These findings highlight the complexity of pharmaceutical behavior in subsurface environments and the need for refined models to assess long-term contaminant mobility and exposure risk (Ruhoy & Daughton 2008, Petrie et al. 2015).

Transformation Products and Metabolites

The degradation of pharmaceutical compounds in the environment often leads to the formation of transformation products (TPs), which may be more persistent, mobile, or toxic than their parent molecules (Kümmerer 2009). For instance, carbamazepine commonly transforms into carbamazepine-10,11-epoxide, a compound known for its environmental stability and comparable toxicity (Miao et al. 2005).

Transformation processes such as microbial metabolism, hydrolysis, photolysis, and partial oxidation produce by-products with altered physicochemical properties, often resulting in increased water solubility and reduced sorption to soils. These characteristics enhance their transport through aquifers, contributing to their widespread detection in groundwater (Patel et al. 2019).

Because treated wastewater and landfill leachates often contain both parent compounds and TPs, the continuous release of these mixtures results in pseudo-persistent environmental exposure. Even as individual compounds degrade, their sustained input maintains relatively stable concentrations in groundwater.

Despite their significance, TPs are frequently overlooked in environmental monitoring and regulation. This omission hampers risk assessments, as TPs can differ substantially in toxicity and behavior from their precursors. There is a critical need to expand monitoring frameworks and analytical protocols to specifically target TPs and develop tailored remediation strategies (Petrie et al. 2015).

PHARMACEUTICAL CONTAMINANTS (PCS)

Pharmaceuticals are categorised based on their mechanisms of action, chemical structure, interaction with biological systems, and therapeutic use. The primary classes of pharmaceutical contaminants in the environment include antibiotics, non-steroidal anti-inflammatory drugs (NSAIDs), hormonal agents (including endocrine disruptors), and antidepressants.

Antibiotics

Antibiotics are widely used in both human and veterinary medicine to prevent and treat bacterial infections. While these compounds have greatly advanced medical outcomes, their extensive and often indiscriminate use has led to their pervasive presence in aquatic environments, where they now represent a major class of environmental contaminants (Bilal et al. 2019, Gillings 2014). The most commonly detected antibiotic pollutants include fluoroquinolones, macrolides, tetracyclines, and sulphonamides (Sonkar et al. 2024).

These compounds enter the environment through multiple pathways, including domestic wastewater discharge, agricultural runoff, hospital effluents, and pharmaceutical manufacturing waste. Consequently, antibiotics have been detected at varying concentrations in seawater, surface water, groundwater, and even treated drinking water supplies (Bilal et al. 2019).

Their environmental presence poses significant risks to both aquatic and terrestrial ecosystems. In aquatic systems, antibiotics can adversely affect planktonic organisms, which occupy the foundational level of the food web. Exposure has been shown to disrupt their physiological, morphological, and behavioural functions, leading to shifts in interspecies dynamics and ecosystem structure (Gunathilaka et al. 2023). Similarly, in terrestrial environments, antibiotic residues in soil can alter microbial community composition, thereby disturbing nutrient cycling and ecosystem balance (Brooks et al. 2008).

One of the most pressing concerns associated with environmental antibiotic contamination is the proliferation of antibiotic-resistant bacteria, which poses a critical threat to both public and animal health (Gangar & Patra 2023, Gillings 2014). Resistant microorganisms have been detected in wild-caught marine and terrestrial species, including those intended for human consumption (Gillings 2014). In addition to fostering resistance, antibiotics can also be toxic to non-target species, further compromising ecosystem stability (Gangar & Patra 2023).

An emerging concern is the development of the so-called "plastisphere" microbial communities that colonise plastic debris in aquatic environments. These surfaces have been identified as vectors for antibiotic-resistant bacteria and resistance genes, thereby facilitating the spread of resistance across marine ecosystems (Andryukov et al. 2022).

Non-Steroidal Anti-Inflammatory Drugs (NSAIDs)

NSAIDs are among the most widely prescribed medications for pain, inflammation, and fever. Common examples include paracetamol, diclofenac, and ibuprofen, all of which have been frequently detected in aquatic environments (Parolini 2020). These compounds primarily enter water systems as excreted metabolites.

NSAIDs are known to bioaccumulate in aquatic organisms such as Mytilus galloprovincialis, leading to toxic effects such as oxidative stress, hepatotoxicity, immunosuppression, and developmental abnormalities (Elizalde-Velázquez & Gómez-Oliván 2020). Sublethal effects can occur even at environmentally relevant concentrations. For example, NSAID exposure has been linked to male-biased sex ratios and altered gene expression in zebrafish (Bereketoglu et al. 2020).

Hormones & Hormone Disruptors

Hormonal pharmaceuticals and endocrine-disrupting chemicals (EDCs) are exogenous compounds that mimic, block, or otherwise interfere with endogenous hormonal signaling pathways. These substances have been increasingly associated with adverse physiological and developmental effects in both aquatic organisms and humans (De Falco & Laforgia 2021, Streifer & Gore 2021).

In aquatic environments, EDCs have been shown to impair reproductive and behavioural functions in fish, potentially leading to long-term disruptions in population dynamics (Hotchkiss et al. 2008). Notably, several EDCs are capable of inducing epigenetic modifications, which may be heritable across generations, thereby compounding their ecological and health risks (Fudvoye et al. 2014). In humans, exposure to EDCs has been implicated in a range of health conditions, including infertility, thyroid disorders, precocious puberty, metabolic dysfunctions such as obesity and diabetes, and skeletal abnormalities (Pironti et al. 2021, Turan 2021). Moreover, these compounds may interfere with neurodevelopment, contributing to elevated risks of neurological and cognitive disorders (Streifer & Gore 2021).

Antidepressants

Antidepressants are pharmaceutical agents prescribed for the management of depression and other related psychiatric disorders. They are typically categorized into four main classes: tricyclic antidepressants (TCAs), monoamine oxidase inhibitors (MAOIs), selective serotonin reuptake inhibitors (SSRIs), and serotonin-norepinephrine reuptake inhibitors (SNRIs) (Carreno & Frazer 2022). In recent years, the widespread detection of these compounds-particularly SSRIs in aquatic environments has emerged as a significant ecological concern.

Experimental studies utilizing artificial stream mesocosms have demonstrated that environmentally relevant concentrations of fluoxetine and citalopram (20 $\mu g.L^{-1}$) can lead to measurable ecological disturbances. These include a reduction in gross primary productivity by up to 29% and a decline in biofilm community respiration by at least 43%. Additionally, treated streams exhibited earlier emergence of dipteran midges compared to control conditions (Richmond et al. 2016). These results suggest that even low-level concentrations of SSRIs can substantially alter aquatic ecosystem function, potentially disrupting trophic interactions and the life cycles of aquatic organisms.

IMPACTS OF PHARMACEUTICALS ON HUMAN HEALTH

Pharmaceutical contaminants in groundwater raise growing concerns for public health, particularly through chronic low-dose exposure via drinking water, food crops, and recreational use of groundwater-fed systems. While concentrations are generally in the $ng.L^{-1}$ to low $\mu g.L^{-1}$ range, certain compounds have been detected at levels exceeding risk thresholds in various studies (Lapworth et al. 2012).

Risk assessment frameworks commonly use the Hazard Quotient (HQ), Risk Quotient (RQ), and Excess Cancer Risk (ECR) to evaluate human exposure:

- HQ is the ratio of exposure concentration to a reference dose (RfD).
- RQ compares environmental concentrations to predicted no-effect concentrations (PNECs).
- ECR estimates lifetime cancer risk from chronic exposure to carcinogenic substances.

For instance, carbamazepine, frequently detected in groundwater across Europe and Asia, has been reported with HQ values approaching or exceeding 1, indicating potential chronic health risks (Mompelat et al. 2009). Similarly, diclofenac and sulfamethoxazole often present RQ values >1 in both WWTP effluents and adjacent aquifers, especially near urban or industrial hotspots (Rizzo et al. 2013).

Recent studies have shown that infants and immunocompromised individuals are particularly vulnerable to compounds with endocrine-disrupting potential, such as estrone and 17β-estradiol, found in some drinking water wells at levels exceeding 10 $ng.L^{-1}$ (Kleywegt et al. 2011). These concentrations, although low, are within ranges known to disrupt hormonal regulation during developmental windows of exposure (Sumpter & Johnson 2005).

A meta-analysis of global occurrence and risk studies (Patel et al. 2019) highlighted that at least 18 commonly detected pharmaceuticals in environmental waters exceed their Predicted No-Effect Concentration (PNEC) or acceptable daily intake (ADI) thresholds in localized scenarios. Among them, ofloxacin, clarithromycin, and erythromycin-H_2O were frequently identified as high-risk, particularly in areas with high WWTP effluent discharge and limited water reuse treatment infrastructure.

Moreover, many existing risk assessments fail to account for cumulative exposure, synergistic effects, or transformation products, which may enhance or compound toxicological effects. The limited scope of monitoring efforts-often focused solely on parent compounds-underestimates potential human health risks (Petrie et al. 2015).

Therefore, future assessments should incorporate multi-compound exposure models, population-specific vulnerability assessments, and expanded monitoring frameworks that include both parent pharmaceuticals and transformation products. This is especially critical in regions dependent on groundwater for potable use and lacking advanced treatment technologies.

ANALYTICAL TECHNIQUES FOR THE DETECTION OF PHARMACEUTICALS

The analysis of pharmaceutical residues in environmental matrices demands high sensitivity and specificity due to the low concentrations at which these compounds typically occur. Chromatographic methods, particularly when coupled with mass spectrometric detection, are extensively utilized for their ability to separate, detect, and quantify diverse pharmaceutical compounds across complex matrices.

Chromatographic Techniques

High-Performance Liquid Chromatography (HPLC): High-Performance Liquid Chromatography (HPLC) is a widely used technique in the analysis of pharmaceutical residues due to its high resolution, precision, and reproducibility. It separates analytes based on their polarity and interactions with the stationary and mobile phases. HPLC, when coupled with ultraviolet (UV), diode array detector (DAD), or mass spectrometry (MS) detectors, offers robust sensitivity and quantification. For instance, Petrović et al. (2013) identified 81 pharmaceuticals in Serbian surface waters using ultra-performance liquid chromatography with mass spectrometry (UPLC-MS), with ibuprofen concentrations up to 20.1 $\mu g.L^{-1}$. Similarly, Cai et al. (2014) reported carbamazepine at 38.24 $ng.L^{-1}$ in Beijing tap water using HPLC-MS/MS. However, matrix effects such as ion suppression may reduce accuracy; this can be mitigated through solid-phase microextraction (SPME) and micellar desorption (MD) (Padrón et al. 2009, Cahill et al. 2004).

Gas Chromatography (GC): Gas Chromatography (GC) is especially suitable for volatile and thermally stable pharmaceutical compounds. GC coupled with mass spectrometry (GC-MS) is commonly used for identifying analgesics, NSAIDs, and antibiotics. Farré et al. (2001) and Sadkowska et al. (2017) detected ibuprofen and diclofenac in river and wastewater samples using GC-MS techniques. Derivatization is often necessary for polar compounds, increasing sample preparation time and potential for analyte loss (Quintana et al. 2007).

Mass Spectrometry-Based Hyphenated Techniques

Liquid Chromatography-Mass Spectrometry (LC-MS): Liquid Chromatography–Mass Spectrometry (LC-MS) combines chromatographic separation with molecular detection, offering high sensitivity for a wide range of pharmaceutical compounds, both polar and non-polar. Detection limits typically range from 1 to 1000 $ng.L^{-1}$ depending on the sample matrix and instrument configuration (Ferrer & Thurman 2012). Nonetheless, matrix-induced ion suppression may impact quantitative accuracy, which necessitates the use of internal standards and thorough calibration procedures (Rodrigues et al. 2024).

Gas Chromatography-Mass Spectrometry (GC-MS): GC-MS is employed for thermally stable pharmaceuticals and is ideal for analyzing volatile residues. Reddersen & Heberer (2003) reported detection of 19 pharmaceutical compounds with GC-MS using selective ion monitoring (SIM), achieving Limits of detection (LODs) in the range of 1–10 $ng.L^{-1}$. While highly selective, GC-MS is limited by its requirement for analyte volatility or chemical derivatization.

Tandem Mass Spectrometry Coupled with Liquid Chromatography (LC-MS/MS): LC-MS/MS utilizes multiple reaction monitoring (MRM) to improve specificity and reduce background noise in complex matrices. Boix et al.

Table 3: Analytical approaches for detecting pharmaceutical compounds of different classes in various locations.

Class of Pharmaceutical	Pharmaceutical Compound	Concentration	Analytical Technique	Location	References
Antibiotic, Fluoroquinolone Antihistamine Beta -adrenoreceptor antagonist	Ciprofloxacin Enrofloxacin ofloxacin Cetirizine Metoprolol	1400 $\mu g.L^{-1}$ 210 $\mu g.L^{-1}$ 55 $\mu g.L^{-1}$ 2100 $\mu g.L^{-1}$ 4 $\mu g.L^{-1}$	LC-MS	Hyderabad, India	Fick et al. 2009
Anti-Epilepsy Antibiotic	Carbamazepine Sulfamethoxazole	26,329.6 $ng.L^{-1}$ 6968 $ng.L^{-1}$	UPLC - ESI-MS/MS	Eastern Cape Province, South Africa	Vumazonke et al. 2020
Anti-inflammatories	Naproxen Ibuprofen	0.16 $\mu g.L^{-1}$ 0.17 $\mu g.L^{-1}$	HPLC - PDA	Kwazulu-Natal Province, South Africa	Ngubane et al. 2019

LC-MS, Liquid Chromatography –Mass Spectrometry

UPLC- ESI-MS/MS, Ultra-Performance Liquid Chromatography Electrospray Ionization Tandem mass Spectrometry

HPLC – PDA, High-Performance Liquid Chromatography with a Photodiode Array Detector

(2014) and Gracia-Lor et al. (2009) successfully quantified a wide range of pharmaceuticals, including sulfamethoxazole and ketoprofen, in environmental waters at sub-ng.L^{-1} levels. Sample preparation typically includes solid-phase extraction (SPE), and method validation often requires the use of isotope-labeled internal standards.

Spectroscopic Techniques

UV-Visible Spectroscopy: UV-Vis spectroscopy is a cost-effective screening method based on the Beer–Lambert principle of light absorption. While useful for high-concentration assessments in relatively clean matrices, its applicability in complex environmental samples is restricted due to low specificity and interference from natural organic matter (Singh et al. 2021, Hussain et al. 2020).

Fluorescence Spectroscopy and Infrared (IR) Spectroscopy: Fluorescence spectroscopy, especially using excitation-emission matrix (EEM) analysis, offers high sensitivity for naturally fluorescent pharmaceuticals, although background signal interference may compromise accuracy (Jing et al. 2022). Infrared spectroscopy, particularly Fourier-transform IR (FTIR) and attenuated total reflectance (ATR) modes, is employed for functional group identification. However, spectral distortions due to water absorption present analytical challenges (Hahn et al. 2010, Goldberg & Chaffotte 2005).

Advanced and Emerging Techniques

Biosensors and Immunoassays: Biosensors and immunoassays represent innovative, high-throughput platforms for the rapid detection of pharmaceuticals in aquatic systems. Electrochemical and optical biosensors offer real-time monitoring with high specificity, while immunoassays provide robust sensitivity via antigen–antibody interactions (Sanvicens et al. 2011, Cháfer-Pericás et al. 2010). However, issues such as cross-reactivity, matrix effects, and limited standardization constrain their widespread implementation (Adrián et al. 2009, Petz 2009).

TREATMENT TECHNOLOGIES

Advanced Oxidation Processes

Advanced Oxidation Processes (AOPs) are highly effective technologies for degrading pharmaceutical contaminants in water. They function by generating hydroxyl radicals (•OH), which are extremely reactive species capable of breaking down complex organic pollutants into carbon dioxide and water (Stefan 2017, Wang & Xu 2012). These radicals are produced through mechanisms such as ultraviolet (UV) radiation, photocatalysis, ozonation, Fenton reactions, and electrochemical oxidation (Khader et al. 2024). AOPs are particularly effective in treating recalcitrant compounds like antibiotics and NSAIDs, which are often resistant to conventional biological processes (Michael et al. 2013, Zawadzki 2022). However, AOPs require significant energy input and chemical reagents, leading to high operational costs. Their efficiency can also be affected by pH, background matrix composition, and the presence of halide ions. In some cases, partial oxidation may result in the formation of toxic by-products, necessitating careful optimisation and monitoring (Taoufik et al. 2021, Stefan 2017).

Membrane Filtration

Membrane filtration is a pressure-driven separation technique that employs semi-permeable membranes to remove pharmaceuticals based on size exclusion and charge interactions. Four main types exist: microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO), with NF and RO being most effective for pharmaceutical removal due to their finer pore sizes (Scott 1995, Cevallos-Mendoza et al. 2022). Removal efficiencies for many pharmaceuticals often exceed 90%, with reports of up to 99% for certain compounds (Iqbal et al. 2023).

Table 4: Comparative evaluation of analytical techniques for pharmaceutical contaminant detection in environmental matrices.

Technique	LOD [ng.L^{-1}]	Sensitivity	Selectivity	Application	References
HPLC	50–100	Moderate	Moderate–High	Quantification of target drugs	Ahmed 2024, Cai et al. 2014
GC-MS	1–50	High	High	Volatile pharmaceuticals	Farré et al. 2001, Sadkowska et al. 2017
LC-MS/MS	<1–10	Very High	Very High	Multi-residue trace analysis	Boix et al. 2014, Gracia-Lor et al. 2009
UV-Vis	1000–5000	Low	Low	Screening of APIs	Singh et al. 2021
Fluorescence	<100	High	Moderate–High	Naturally fluorescent drugs	Jing et al. 2022
IR (FTIR, ATR)	500–2000	Moderate	Low–Moderate	Functional group ID	Hahn et al. 2010
Biosensors	<50	High	High	On-site screening	Sanvicens et al. 2011
Immunoassays	<10	High	High	Hormonal drugs, antibiotics	Cháfer-Pericás et al. 2010

Table 5: Comparative summary of pharmaceutical treatment technologies.

Technology	Removal Efficiency	Advantages	Limitations	Scalability	References
Advanced Oxidation Processes (AOPs)	70–100%	Effective for recalcitrant compounds, broad spectrum	High cost, toxic by-products, energy-intensive	Medium to High	Stefan 2017, Michael et al. 2013
Membrane Filtration (NF/RO)	90–99%	High selectivity, physical barrier	Membrane fouling, high energy demand	High	Scott, 1995, Iqbal et al. 2023
Activated Carbon Adsorption	50–95%	Cost-effective, high adsorption capacity	Competitive adsorption, regeneration costs	High	Wang et al. 2021, Zieliński et al. 2022

Despite high effectiveness, membrane fouling remains a significant limitation, reducing efficiency and increasing energy consumption. Additionally, not all pharmaceuticals are retained, and small polar compounds may pass through certain membranes (Rosman et al. 2018). Hybrid systems such as membrane bioreactors (MBRs), which combine biological degradation and physical filtration, are being explored to enhance treatment performance and operational efficiency (Hernando et al. 2007).

Activated Carbon Adsorption

Activated carbon adsorption is a well-established and effective method for removing pharmaceutical residues from aqueous media. The technique relies on physical and chemical interactions such as pore filling, hydrogen bonding, and electrostatic forces between pharmaceutical molecules and the high-surface-area carbon material (Wang et al. 2021). Adsorption capacity depends on pore structure, surface area, and the physicochemical properties of both the pharmaceutical and the carbon surface. Studies have shown that microporous carbons derived from sources such as pinecones and Butia capitata endocarp effectively adsorb ketoprofen, paracetamol, and other drugs (Kerkhoff et al. 2021, Yu et al. 2023). Isotherm models such as Langmuir, Freundlich, and Redlich–Peterson are used to describe adsorption behaviour (Delgado et al. 2019). Challenges include competitive adsorption from co-existing organics, expensive regeneration, and decreased performance over time due to fouling and pore blockage (Zieliński et al. 2022, Heusser et al. 2023).

CONCLUSIONS

Pharmaceuticals and their transformation products (TPs) persist in groundwater due to incomplete removal by conventional treatment technologies, particularly under real-world environmental conditions. Current research often overlooks key factors, such as mixture toxicity, chronic low-dose exposure, and site-specific hydrogeological variability limiting the effectiveness of risk assessments and management strategies. While analytical techniques have advanced, treatment technologies and regulatory frameworks remain fragmented and inadequate.

KEY RECOMMENDATIONS

1. Establishing unified regulatory standards for pharmaceuticals and TPs in groundwater,
2. Integrating multi-barrier treatment solutions-such as membrane processes integrated with advanced oxidation processes (AOPs)-that are both scalable and adaptable to real-world conditions,
3. Developing affordable, real-time monitoring tools and decentralized filtration systems,
4. Expanding environmental surveillance frameworks to include TPs, and
5. Incentivizing green drug design and responsible disposal through take-back programs.

A coordinated, cross-disciplinary approach is essential to advance regulatory policies, technological innovation, and research efforts for safeguarding groundwater quality.

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Application of Analytical Hierarchy Process (AHP) to Assess Bio and Thermal-Conversion Technology Options for Organic Solid Waste Management

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ABSTRACT

Indonesia is increasingly challenged by the management of organic solid waste, especially in Bali Province, where organic waste accounts for about 68% of the total municipal waste produced. The current waste management strategies mainly depend on landfilling and basic composting techniques, which are inadequate to mitigate the environmental and socio-economic effects. This research utilizes the Analytical Hierarchy Process (AHP) to systematically assess and prioritize eight bioconversion and thermal-conversion technologies for managing organic waste in Bali. The evaluation considers four main criteria, environmental, social, technical, and economic, along with their sub-criteria, based on expert opinions and literature review. The results reveal that bioconversion technologies, particularly composting, black soldier fly (BSF) processing, and eco-enzyme production, are the most appropriate choices, as they offer high community acceptance, reduced greenhouse gas emissions, and better compatibility with local waste characteristics and socio-economic conditions. Thermal technologies like incineration and gasification are less favored due to their higher environmental risks and capital expenses. The findings offer a comprehensive decision-support framework for policymakers and practitioners to create sustainable organic waste management strategies tailored to Indonesia's context.

INTRODUCTION

Managing organic waste effectively is an escalating environmental issue globally, especially in developing areas where organic materials make up most of the municipal solid waste. Despite this, such waste is frequently disposed of through unsustainable practices like landfilling, which exacerbates environmental harm and increases greenhouse gas (GHG) emissions (Yensukho et al. 2022, Gunamantha et al. 2023a). In Bali Province, Indonesia, organic waste accounts for about 68% of the municipal waste stream and is predominantly sent to landfills (Gunamantha et al. 2023c), highlighting the pressing need for sustainable processing technologies.

Recent studies indicate that selecting appropriate organic waste treatment technologies necessitates a balanced assessment of environmental, economic, technical, and social factors (Abu et al. 2021, Gunamantha et al. 2023b). Multi-Criteria Decision-Making (MCDM) tools, such as the Analytic Hierarchy Process (AHP), have been extensively used for this purpose. AHP is particularly effective in decision-making environments with varied and conflicting goals, as it enables structured comparisons among different options (Kurbatova & Abu-Qdais 2020, Siejka 2020, Sun et al. 2020).

Numerous studies have employed AHP to aid in solid waste management planning, including technology selection (Afrane et al. 2022, Agbejule et al. 2021), landfill siting (Mallick 2021), and system optimization (Apaydin & Akçay Han 2023, Fogarassy et al. 2022). For example, Afrane et al. (2022) utilized AHP-

TOPSIS to assess waste-to-energy options in Ghana, while Apaydin & Akçay Han (2023) applied AHP to evaluate collection methods within a zero-waste policy framework.

However, much of the existing research is narrowly focused on established solutions like composting or anaerobic digestion, often neglecting emerging alternatives such as black soldier fly (BSF) larvae bioconversion, eco-enzyme production, or newer thermal treatments like gasification and pyrolysis (Siciliano et al. 2021, Benny et al. 2023, Torres-Lozada et al. 2023). These technologies are gaining attention for their potential in circular economies and their adaptability to decentralized systems in resource-constrained settings (Abu-Qdais et al. 2025).

To address this gap, the current study uses AHP to evaluate a wider range of organic waste processing technologies tailored to Bali's socio-environmental context. These include composting, BSF, anaerobic digestion, eco-enzyme production, pyrolysis, gasification, incineration, and RDF (refuse-derived fuel) production. This approach allows for a systematic comparison based on environmental, social, technical, and economic criteria (Paul & Paul 2021, Abu et al. 2021).

The novelty of this research lies in its integrated evaluation of underutilized yet promising bioconversion and thermal conversion technologies through an AHP-based framework. By contextualizing these options within Bali's waste management priorities, this study offers actionable insights for stakeholders aiming to divert organic waste from landfills while promoting community involvement and income generation (Abu-Qdais et al. 2025, Gunamantha et al. 2023, 2023b, 2023c).

MATERIALS AND METHODS

Research Framework and Approach

The Analytical Hierarchy Process (AHP) was employed in this study as the primary method for evaluating and ranking organic waste treatment technologies in Bali Province, Indonesia. This multi-criteria decision-making (MCDM) approach was selected due to its effectiveness in handling complex decision problems that integrate both qualitative and quantitative factors, particularly in contexts where stakeholder preferences must be aligned with sustainability objectives.

The research framework consists of four main stages:

1. Identification of criteria and sub-criteria based on a comprehensive literature review and expert consultations.
2. Selection of alternative technologies, comprising both bioconversion and thermal-conversion options, chosen for their relevance to Indonesia's waste management challenges.
3. Pairwise comparisons and judgment elicitation from a panel of experts to evaluate the relative importance of criteria and alternatives.
4. Synthesis of priorities and consistency analysis to ensure the reliability of the results.

Selection of Criteria and Sub-Criteria

Four main criteria were used to evaluate the technologies: Environmental, Social, Technical, and Economic. These criteria reflect the priorities of sustainable waste management in tropical and developing regions and were aligned with previous AHP applications in the waste sector (Kurbatova & Abu-Qdais 2020, Siejka 2020, Sun et al. 2020).

Each main criterion was further broken down into sub-criteria to reflect specific dimensions relevant to the Bali context:

- Environmental: Greenhouse gas emissions, occupational and community health risks, and water/soil pollution.
- Social: Community acceptance, job creation, and responsible management.
- Technical: Feasibility and sustainability, energy recovery, material recovery, technological complexity.
- Economics: Capital investment, operational/maintenance costs, revenue potential.

Table 1 delineates each sub-criterion, illustrating how various aspects of sustainability—consistent with global indicators and adapted to local environmental and socio-economic contexts-are integrated into the analysis, as supported by previous studies (Kurbatova & Abu-Qdais 2020, Siejka 2020, Sun et al. 2020).

Selection of Alternative Technologies

Eight organic waste processing technologies were selected for evaluation:

- Bioconversion Technologies: Composting, Black Soldier Fly (BSF) processing, Anaerobic Digestion (AD), and Eco-Enzyme production.
- Thermal Conversion Technologies: Incineration, Gasification, Carbonization (slow pyrolysis), and Drying & Compaction (RDF production).

Table 2 enumerates the eight determined technological alternatives, along with concise descriptions of their primary products and roles in waste valorization. These technologies were chosen based on their relevance to Indonesia's organic waste profile, technical maturity, and

evidence from global case studies (Babalola 2015, Kurbatova & Abu-Qdais 2020, Sun et al. 2020, Gunamantha et al. 2023b).

Expert Panel Composition

The AHP analysis relied on expert judgments from six qualified professionals:

- Three academic experts from Universitas Pendidikan Ganesha.
- Three practitioners from government agencies overseeing waste management in Bali.

All experts had a minimum of 10 years of experience in solid waste management and demonstrated familiarity with AHP methodology, ensuring reliable and context-aware input.

Pairwise Comparisons and Data Collection

Experts participated in structured interviews and completed Saaty's pairwise comparison questionnaires to evaluate:

Table 1: main criteria, sub-criteria, and brief description.

No.	Criteria	Sub-criteria	Description
1.	Environmental	Global warming	Referring to the selected technology's ability to reduce greenhouse gas emissions and other pollutants.
		Occupational and public health	Referring to the selected technology's ability to reduce risks to the health of workers and the surrounding community.
		Water and soil pollution	Refers to the selected technology with the least environmental impact on water and soil
2.	Social	Community acceptability:	Referring to the selected technology that is accepted as appropriate, valid, or suitable by the community.
		Job creation	Referring to the selected technology's ability to generate the most employment opportunities.
		Responsible management group	Referring to the selected technology with a clear and accountable management structure.
3.	Technical	Feasibility and sustainability	Referring to the selected technology that can be practically implemented and sustainably operated.
		Energy recovery	Refers to the selected technology with the highest potential for energy production.
		Technological sophistication	Referring to the selected technology that is advanced and requires skilled human resources.
		Material recovery	Referring to the selected technology with the highest potential for material recovery.
4.	Economic	Investment cost	Referring to the selected technology with the lowest initial investment cost.
		Operation and maintenance cost	Referring to the selected technology with the lowest operational and maintenance expenses.
		Revenue	Referring to the selected technology with the highest potential revenue generation.

Table 2: Technology options.

No.	Conversion Method	Kinds of Technology	Description
1.	Thermal conversion	Drying and compaction	The main product of the compaction and drying process is RDF solid fuel, which is converted into electrical energy.
		Incineration	The main product of the incineration process is hot gas, which is converted into electrical energy.
		Gasification	The main product of the gasification process is gas fuel (syngas), which is converted into electrical energy.
		Carbonization (slow pyrolysis)	The main product of the slow pyrolysis process is biochar, which is used as a soil conditioner.
2.	Bioconversion	Anaerobic Digestion	The main products of the AD process are biogas, which is converted into electrical energy and digestate, which is stabilized into compost.
		Composting	The main product of the composting process is biogas.
		Black Soldier Fly (BSF) Process	The main products of the BSF process are larvae, which are used as animal feed and residues, which are stabilized into compost.
		Eco-enzyme Manufacturing Process	The main product of the eco-enzyme manufacturing process is a liquid that can be used as a disinfectant and a residue that is stabilized into compost.

- The relative importance of criteria and sub-criteria.
- The comparative performance of each alternative technology against each sub-criterion.

The geometric mean method was used to consolidate individual judgments into group consensus matrices (Saaty 1987).

Consistency and Validity Testing

Consistency Ratios (CRs) were calculated for all pairwise matrices. A CR of less than 0.1 was considered acceptable, following AHP standards. Validity checks covered:

- Comparisons among the main criteria
- Sub-criteria matrices
- Technology alternative evaluations

This step ensured that expert judgments were logically consistent and robust (Siejka 2020).

Priority Synthesis and Final Ranking

The Average Normalized Column (ANC) method was used to synthesize priorities through:

- Matrix normalization
- Local and global weight calculation
- Aggregation of weights to derive final technology rankings

This method maintains methodological transparency and supports reproducibility in technology evaluation.

Sensitivity Analysis

In this study, a sensitivity analysis was conducted to evaluate the robustness of the AHP results by assessing how variations in criteria weights affected the final ranking of waste treatment alternatives. This technique was considered essential in Multi-Criteria Decision Analysis (MCDA) for examining the stability of outcomes under different decision-making scenarios (Babalola 2015, Kurbatova & Abu-Qdais 2020, Sun et al. 2020). Multiple scenarios were explored by systematically altering the weights assigned to the four main criteria. In the first scenario, full weight (1.0) was assigned to one criterion while the others were given a weight of zero, resulting in four possible configurations. In the second scenario, equal weights (0.5) were allocated to two criteria, with the remaining two set to zero, generating six combinations. The third scenario involved assigning a weight of 0.33 to three criteria, while the fourth was assigned zero, producing four additional combinations. Finally, an equal weight of 0.25 was applied to all four criteria in a balanced scenario. This approach provides valuable insights into how shifting stakeholder priorities influence the selection of optimal organic waste processing technologies.

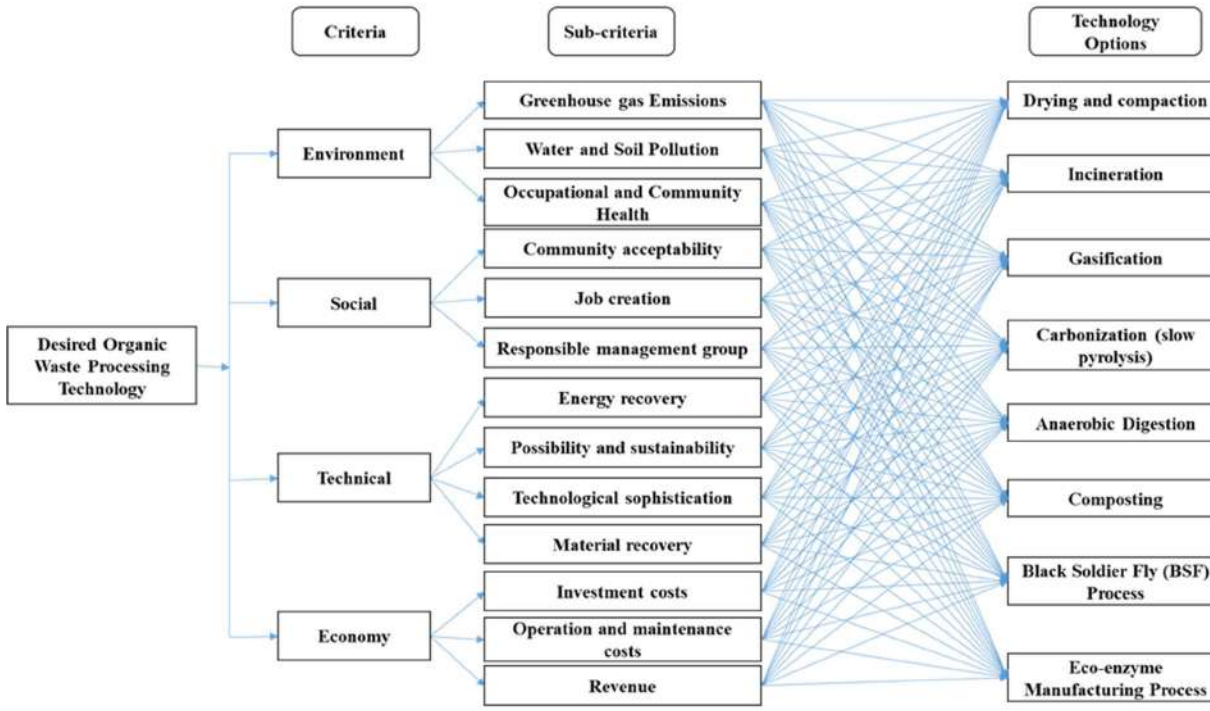

Fig. 1: Levels of criteria used in technological selection.

Table 3: Pairwise comparison matrix and weights for main criteria relative to objectives.

Criteria	Environment	Social	Economy	Technic	Weight	Consistency ratio
Environment	1.000	1.732	4.189	4.857	0.492	0.068147
Social	0.577	1.000	3.533	0.907	0.253	
Economy	0.239	0.283	1.000	1.089	0.107	
Technical	0.206	1.103	0.918	1.000	0.148	

RESULTS AND DISCUSSION

The process starts with establishing a structured hierarchy (Fig. 1) that begins with the goal and progresses through intermediate levels (criteria) down to the lowest level, which consists of a set of alternatives. This hierarchical framework is based on best practices from numerous previous AHP applications in solid waste and energy system planning (Siejka 2020, Sun et al. 2020, Agbejule et al. 2021, Afrane et al. 2022). The objective is to identify the most suitable technology for processing organic waste. To achieve this, a series of questions has been formulated at the first level: "How strong and significant is one criterion compared to other criteria in determining the choice of organic waste processing technology?" At the second level, the question is: "How strong and significant is one sub-criterion compared to other sub-criteria in relation to the main criterion?" Finally, at the third level, a matrix has been prepared to assess: "How strongly and significantly does one alternative compare to other alternatives in contributing to the sub-criteria?"

According to the combined assessments of experts, Environmental criteria were identified as the most crucial element in choosing a technology for organic waste treatment, carrying a weight of 0.492. This highlights the increasing concern about environmental effects, especially greenhouse gas (GHG) emissions, which play a major role in climate change in Indonesia (Gunamantha et al. 2023a). This concern is further emphasized by the impacts on climate, emissions from landfills, and health risks in areas with high population density or tourism (Yensukho et al. 2022, Gunamantha et al. 2023b). Subsequently, the Social (0.253), Technical (0.148), and Economic (0.107) criteria were prioritized, mirroring consistent trends observed in similar global settings (Kurbatova & Abu-Qdais 2020, Abu et al. 2021). Table 3 presents the results of the pairwise comparison matrix for the main criteria in relation to the primary objectives.

Table 4 presents the weights assigned to the sub-criteria under the environmental criterion, revealing that greenhouse gas emissions and occupational and community health are the top priorities, each with a weight of 0.353. These are followed by water and soil pollution, which hold a slightly lower weight of 0.294. This distribution indicates that the primary focus in selecting organic waste processing technologies is on minimizing emissions and protecting public and worker health. These findings align with those of Agbejule et al. (2021), who similarly ranked health impacts and greenhouse gas emissions as the most critical environmental concerns, with pollution potential receiving relatively less emphasis. This prioritization is also supported by broader literature highlighting the waste sector's significant contribution to air pollution and its implications for public health (Gunamantha et al. 2023b, Degefu & Asefa 2024, Saghi et al. 2024).

Table 5 shows that within the social criteria, community acceptability holds the highest weight (0.366), followed by job creation (0.337), and responsible management group (0.297). The prioritization of community acceptance reflects the importance of participatory approaches in improving the adoption of waste management technologies, as supported by Agbejule et al. (2021) and Saifi & Jha (2024). In the technical criteria (Table 6), feasibility and sustainability dominate with a weight of 0.455, indicating a strong preference for technologies that are easy to implement and maintain in localized settings. This is followed by energy recovery (0.270), material recovery (0.192), and technological sophistication (0.084), consistent with the emphasis on operational simplicity highlighted by Paul & Paul (2021). Table 7 presents the economic sub-criteria, where revenue generation ranks highest (0.403), followed by operational and maintenance costs (0.345), and investment costs (0.252). This contrasts with previous studies such as Agbejule et al. (2021), who identified investment costs as the most critical, and Qazi et al. (2018), who emphasized operational expenses.

Table 4: Pairwise comparison matrix and weights for environmental sub-criteria.

Sub-criteria	Greenhouse Gas Emissions	Water and Soil Pollution	Occupational and Community Health	Weight	Consistency ratio
Greenhouse Gas Emissions (GHG)	1.000	1.201	1.000	0.353	0
Water and Soil Pollution (WSP)	0.833	1.000	0.833	0.294	
Occupational and Community Health (OPH)	1.000	1.201	1.000	0.353	

Table 5: Pairwise comparison matrix and weights for social sub-criteria.

Sub-criteria	Community acceptability	Job creation	Responsible management group	Weight	Consistency ratio
Community acceptability (CA)	1.000	1.081	1.238	0.366	0
Job creation (JC)	0.925	1.000	1.132	0.337	
Responsible management group (RMG)	0.808	0.884	1.000	0.297	

Table 6: Pairwise comparison matrix and weights for technical sub-criteria.

Sub-criteria	Energy recovery	Feasibility and sustainability	Technological sophistication	Material recovery	Weight	Consistency ratio
Energy recovery (ER)	1.000	0.785	2.877	1.201	0.270	0.02637
Feasibility and sustainability (PS)	1.274	1.000	4.886	3.625	0.455	
Technological sophistication (TS)	0.348	0.205	1.000	0.354	0.084	
Material recovery (MR)	0.833	0.276	2.821	1.000	0.192	

Table 7: Pairwise comparison matrix and weights for economic sub-criteria.

Sub-criteria	Investment costs	Operation and maintenance costs	Revenue	Weight	Consistency ratio
Investment costs (IC)	1.000	0.450	0.964	0.252	0.09799
Operation and maintenance costs (OMC)	2.221	1.000	0.533	0.345	
Revenue (RV)	1.037	1.877	1.000	0.403	

The high priority given to revenue reflects growing interest in circular economy models and entrepreneurship in waste management, as echoed by Afrane et al. (2022) and Torres-Lozada et al. (2023).

Based on Tables 3 to 7, the final weights of all sub-criteria are consolidated and presented in Table 8. The results show that the three environmental sub-criteria rank first, second, and third overall. This outcome is expected, given that the environmental criterion received the highest overall weight compared to the other main criteria.

As shown in Table 9, bioconversion methods outperform thermal technologies under the environmental criterion. Black Soldier Fly (BSF) processing (0.215) and composting (0.213) receive the highest environmental weights, indicating their strong performance in reducing greenhouse gas emissions (GHG), minimizing occupational and public health (OPH) risks, and limiting water and soil pollution (WSP). These results align with evidence from field implementations in both urban and rural settings, which highlight the environmental benefits of BSF and composting methods (Dzepe et al. 2021, Fogarassy et al.

Table 8: Final calculation of sub-criteria.

Criteria	Weight of Criteria	Sub-criteria	Weight of sub-criteria	Weight Total of sub-criteria
Environment	0.492	Greenhouse Gas Emissions	0.353	0.174
		Water and Soil Pollution	0.294	0.145
		Occupational and Community Health	0.353	0.174
Social	0.253	Community acceptability	0.366	0.093
		Job creation	0.337	0.085
		Responsible management group	0.297	0.075
Technical	0.107	Energy recovery	0.270	0.029
		Feasibility and sustainability	0.455	0.049
		Technological sophistication	0.084	0.009
		Material recovery	0.192	0.021
Economy	0.148	Investment costs	0.252	0.037
		Operation and maintenance costs	0.345	0.051
		Revenue	0.403	0.060

2022, Madonsela et al. 2024). In contrast, incineration scores the lowest (0.037), reinforcing longstanding concerns about its contribution to air pollutants and toxic residue generation (Tait et al. 2020). Their systematic review found consistent associations between proximity to waste incinerators and elevated risks of respiratory problems, cancer, and adverse birth outcomes, emphasizing their potential public health hazards. These comparative scores support the prioritization of bioconversion technologies in environmentally sensitive waste management strategies.

Table 10 presents the relative weights of each technology option based on the social criteria. Among the alternatives, Composting ranks highest in overall social performance with a global weight of 0.226, driven by strong community acceptance (0.238) and high job creation potential (0.230). Eco-enzyme manufacturing follows with a global weight of 0.172, supported by balanced performance across all three sub-criteria, particularly in its ability to be managed by local groups (0.163). Black Soldier Fly (BSF) processing and drying, and compaction are tied at a global weight of 0.139, showing moderate community acceptability and organizational feasibility. In contrast, incineration and gasification are the lowest ranked (both at 0.068), indicating limited social acceptability and low employment creation, which aligns with previous findings that suggest low public support for high-tech and centralized waste solutions in community settings. These results underscore a strong preference for socially inclusive and participatory waste treatment methods that align with local capacities and community engagement.

As shown in Table 11, among all alternatives, incineration achieves the highest overall technical score (0.163), primarily due to its top ranking in feasibility and sustainability (0.243) and strong performance in technological sophistication (0.217), reflecting its advanced capabilities in high-energy recovery systems. Composting follows with a global weight of 0.139, supported by its high score in material recovery (0.210) and feasibility (0.164), making it well-suited for decentralized applications. Eco-enzyme production ranks third (0.132), showing balanced performance across feasibility (0.152) and material recovery (0.187). Technologies like carbonization and gasification also perform well in energy recovery but fall short in feasibility and sustainability, affecting their total technical weight. Meanwhile, BSF processing and anaerobic digestion show moderate scores, particularly in material recovery and feasibility, reinforcing their relevance in local, low-tech contexts. Drying and compaction, although technically consistent, rank lower overall due to relatively modest scores across all sub-criteria. In line with the previous studies (Sun

Table 9: Weight of each technology option relative to environmental sub-criteria.

Technology Options/Alternative	Environment (0.492)			Global Weight of Environment
	GHG (0.353)	OPH (0.294)	WSP (0.353)	
Drying and compaction	0.069	0.066	0.103	0.080
Incineration	0.032	0.031	0.046	0.037
Gasification	0.068	0.056	0.088	0.072
Carbonization (slow pyrolysis)	0.059	0.082	0.100	0.080
Anaerobic Digestion	0.108	0.112	0.130	0.117
Composting	0.258	0.173	0.201	0.213
Black Soldier Fly (BSF) Process	0.212	0.253	0.186	0.215
Eco-enzyme Manufacturing Process	0.194	0.227	0.146	0.187

Table 10: Weight of each technological option relative to sociocultural.

Technology Options/Alternative	Social (0.253)			Global Weight of Social
	CA (0.366)	JC (0.337)	RMG (0.297)	
Drying and compaction	0.112	0.196	0.109	0.139
Incineration	0.087	0.047	0.068	0.068
Gasification	0.062	0.057	0.087	0.068
Carbonization (slow pyrolysis)	0.073	0.107	0.128	0.101
Anaerobic Digestion	0.073	0.096	0.098	0.088
Composting	0.238	0.230	0.208	0.226
Black Soldier Fly (BSF) Process	0.143	0.134	0.140	0.139
Eco-enzyme Manufacturing Process	0.213	0.134	0.163	0.172

Table. 11: Weight of each technology option relative to technical sub-criteria.

Technology Options/Alternative	Technical (0.107)				Global Weight of Technical
	ER(0.270)	PS (0.455)	TS (0.084)	MR (0.192)	
Drying and compaction	0.123	0.118	0.076	0.117	0.116
Incineration	0.098	0.243	0.217	0.039	0.163
Gasification	0.214	0.059	0.178	0.066	0.112
Carbonization (slow pyrolysis)	0.223	0.055	0.215	0.096	0.122
Anaerobic Digestion	0.129	0.089	0.118	0.122	0.109
Composting	0.070	0.164	0.058	0.210	0.139
Black Soldier Fly (BSF) Process	0.065	0.120	0.069	0.162	0.109
Eco-enzyme Manufacturing Process	0.077	0.152	0.070	0.187	0.132

Table 12: Weight of each technology option relative to economic sub-criteria.

Technology Options/Alternative	Economy (0.148)			Global Weight of Economy
	IC (0.252)	OMC (0.345)	RV (0.403)	
Drying and compaction	0.056	0.063	0.105	0.078
Incineration	0.228	0.138	0.058	0.128
Gasification	0.171	0.134	0.100	0.130
Carbonization (slow pyrolysis)	0.212	0.115	0.148	0.153
Anaerobic Digestion	0.171	0.170	0.116	0.148
Composting	0.056	0.115	0.145	0.112
Black Soldier Fly (BSF) Process	0.059	0.127	0.180	0.131
Eco-enzyme Manufacturing Process	0.047	0.137	0.148	0.119

et al. 2020), these results highlight that while incineration excels in advanced technical metrics, simpler technologies like composting and BSF offer a better fit for practical, scalable, and sustainable applications, especially in resource-constrained or decentralized environments.

Table 12 outlines the economic outcomes of different technological options. Carbonization, or slow pyrolysis, stands out as the most economically beneficial, boasting the highest global economic weight (0.153) due to its significant revenue potential (0.148) and relatively favorable investment cost (0.212). Anaerobic digestion is a close second (0.148), showing a well-rounded performance across all sub-criteria. The Black Soldier Fly (BSF) process also demonstrates strong economic performance (0.131), driven by the highest revenue score (0.180), highlighting its considerable income-generating potential. Gasification (0.130) and incineration (0.128) produce moderate results but are limited by lower revenue contributions. Composting (0.112) and eco-enzyme manufacturing (0.119) show strengths in revenue generation with lower investment needs but are hindered by higher operational costs. Drying and compaction rank the lowest (0.078), indicating minimal economic attractiveness. Overall, the table emphasizes that revenue potential is the key economic factor, favoring technologies that enable marketable outputs and entrepreneurial opportunities.

Discussion

Analysis given in Table 13 reveals a distinct preference for bioconversion technologies, such as Composting, Black Soldier Fly (BSF) Processing, and Eco-Enzyme Production, due to their strong performance in environmental, social, and feasibility aspects. These technologies are well-aligned with Bali's sustainability goals, particularly in minimizing greenhouse gas emissions, enhancing health, and encouraging community involvement. Their decentralized and cost-effective nature makes them particularly suitable for both rural and urban areas in developing regions. This trend is consistent with global findings that highlight the importance of local engagement for effective and sustainable waste management solutions (Yan et al. 2020, Sunarti et al. 2024). On the other hand, thermal technologies like incineration and gasification, despite their technical capabilities and potential for energy recovery, are less preferred due to environmental concerns and limited public acceptance. Even mid-range options such as carbonization and anaerobic digestion show potential but lack the comprehensive benefits of the top three methods. Overall, the findings suggest a significant policy implication: future

Table. 13: Ranking results based on global weight.

Technology Options/Alternative	Global Weight of Environment (0.497)	Global Weight of Social (0.253)	Global Weight of Technical (0.107)	Global Weight of Economy (0.148)	Global Weight
Drying and compaction	0.080	0.139	0.116	0.078	0.099
Incineration	0.037	0.068	0.163	0.128	0.072
Gasification	0.072	0.068	0.112	0.130	0.084
Carbonization (slow pyrolysis)	0.080	0.101	0.122	0.153	0.101
Anaerobic Digestion	0.117	0.088	0.109	0.148	0.113
Composting	0.213	0.226	0.139	0.112	0.193
Black Soldier Fly (BSF) Process	0.215	0.139	0.109	0.131	0.172
Eco-enzyme Manufacturing Process	0.187	0.172	0.132	0.119	0.167

waste management strategies should focus on bioconversion pathways that combine environmental performance with social inclusivity and operational feasibility.

Based on the sensitivity analysis, Fig. 2 shows that under the first scenario-where one criterion is assigned full weight (1.0), and the remaining three are set to zero-pyrolysis experiences a notable increase in ranking, while the other technologies display both positive and negative shifts. When the environmental, social, or technical criteria individually receive full weight, composting consistently ranks highest. Conversely, when the economic criterion alone is emphasized, pyrolysis takes the top position. In the second scenario (Fig. 3), where pairs of criteria are equally weighted at 0.5, and the others are excluded, ranking fluctuations are observed across all technologies. Composting maintains the lead when combinations such as environmental-sociocultural, environmental-technical, sociocultural-technical, and sociocultural-economic are used. However, BSF becomes the top-ranked option under the environmental-economic pairing, while incineration takes the lead when technical and economic criteria are prioritized together.

In the third scenario (Fig. 4), when the environmental criterion is weighed at zero and the other three at 0.333 each, composting still emerges as the best choice across all combinations. In the fourth scenario (Fig. 5), where all main criteria are equally weighed at 0.25, composting continues to be the preferred option.

The sensitivity of the outcomes across the four scenarios reveals a consistent trend, where bioconversion-based processing technologies (such as composting, BSF, and eco-enzyme) are generally the most favourable options for handling organic waste. In contrast, gasification and other thermal conversion-based technologies are considered the least favourable choices. Although combinations of criteria weights can lead to countless scenarios, certain features distinctly demonstrate how results vary with changes in weights (Babalola 2015).

To select the appropriate technology, it is crucial to evaluate various criteria using a multi-criteria approach. The findings in this study illustrate how pairwise comparisons help establish priorities among criteria and processing technology options. The synthesis of priority criteria in

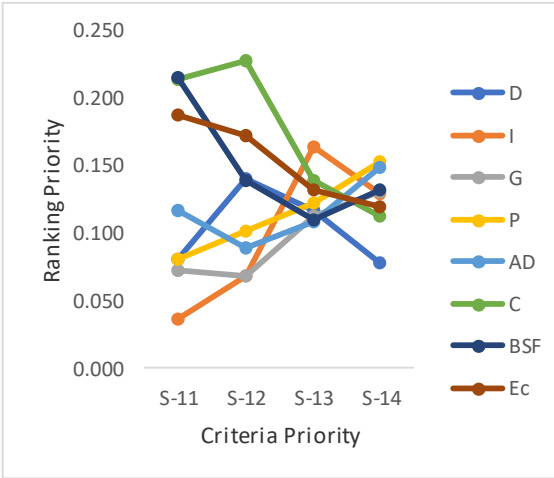

Fig. 2: Scenario I.

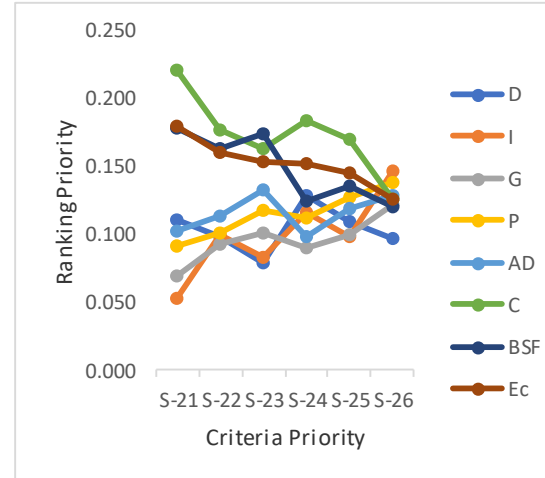

Fig. 3: Scenario II.

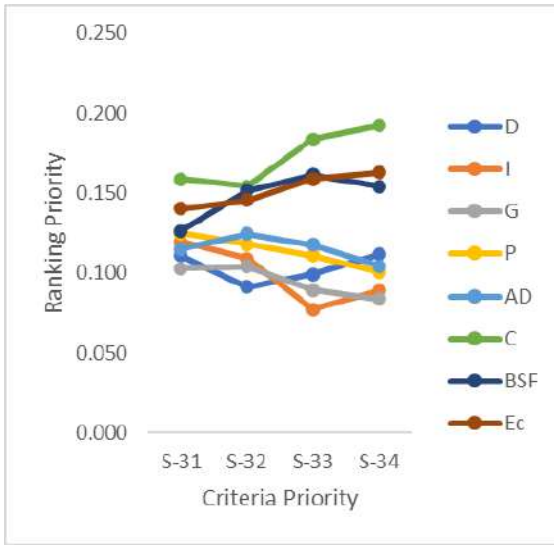

Fig. 4: Scenario III.

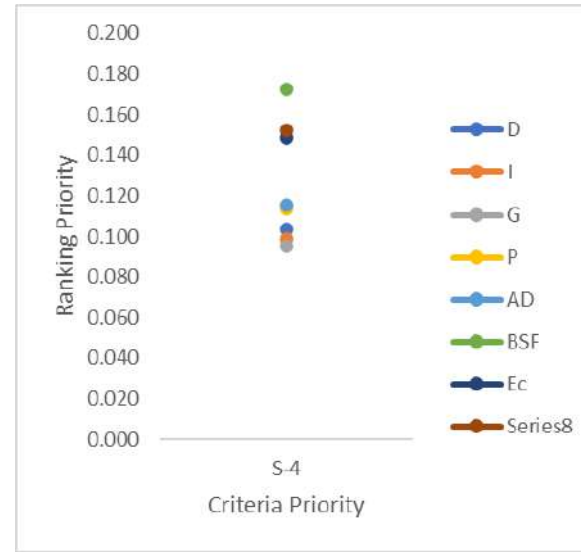

Fig. 5: Scenario IV.

relation to objectives and options, as shown in Table 13, indicates that composting is preferred across all four criteria. The preference for composting technology is largely due to its widespread current use, both individually and centrally. However, significant improvements are necessary, including the development of more centralized composting systems to enhance material recovery and community use of compost. Currently, compost is less favoured by farmers because its impact on plant productivity is not as strong as that of synthetic fertilizers. Therefore, enhancing the composting management system is essential.

Considering the continuous increase in waste production, particularly organic waste, it is imperative that composting technology accelerates the decomposition process beyond the capabilities of current methods. The existing composting capacities are markedly insufficient when compared to the rate of waste generation. Moreover, a significant proportion of compost products do not meet established quality standards, leading to diminished interest in their utilization. It is essential to enhance institutional and governance capacities to promote the intensification of composting within TPS3R, which currently predominates composting practices in Bali. Additionally, the integration of composting facility development into the regional development plan strategy is crucial. Furthermore, the establishment of an effective waste collection system is necessary to ensure the segregation of organic waste from other waste types.

Policy Implications

Furthermore, the results of this study align with various MCDM-based investigations in organic waste management, which underscore the significance of incorporating community-based composting and Black Soldier Fly (BSF) larvae cultivation within decentralized waste management systems. This strategy has demonstrated not only environmental sustainability but also efficacy in generating local employment opportunities (Paul & Paul 2021, Madonsela et al. 2024). Conversely, incentive policies such as tax breaks and grants for startups, coupled with capacity-building programs, can expedite technology adoption and secure sustained community support (Achmad et al. 2023). Additionally, the production of eco-enzymes-a straightforward yet highly participatory method-warrants further exploration, particularly for implementation in educational institutions, cooperatives, and rural areas as a direct community empowerment initiative. Cooperatives, especially those involved in environmental awareness and local economic development, offer structured networks and shared resources that can support the adoption of eco-enzyme production. This model of collaboration has proven effective, as seen in a community service program in Pojok Village, Kediri, East Java, Indonesia, where partnerships with the local waste bank successfully empowered residents-primarily housewives-through participatory training to turn organic waste into eco-enzyme products, leading to increased environmental awareness and waste reduction (Prodyanatasari et al. 2024).

Study Limitations

This study was conducted with input from six expert participants. Although their subject-matter expertise contributed to reliable and informed judgments, the

relatively small and homogeneous sample may limit the generalizability of the findings. Incorporating a broader panel of stakeholders-such as private sector representatives, community organizations, and policymakers-would provide a more comprehensive perspective on the feasibility and acceptance of each technology. Furthermore, the economic evaluation relied solely on expert scoring rather than empirical cost data or detailed cost-benefit analyses, which diminishes the precision of financial comparisons across alternatives. Future studies should incorporate real-world cost data and apply robust economic modeling techniques. Additionally, this research did not include a life cycle assessment (LCA), which is essential for evaluating the full environmental impacts of each treatment option. The absence of LCA, combined with the potential for bias in subjective expert scoring, underscores the need for more data-driven, multi-dimensional evaluation frameworks in future research.

CONCLUSIONS

This study identifies composting, Black Soldier Fly (BSF) processing, and eco-enzyme production as the most suitable organic waste treatment technologies for Bali Province. Bioconversion methods demonstrably outperform thermal alternatives across environmental, social, and economic dimensions. Composting consistently ranks highest due to its adaptability, community acceptance, and environmental benefits, while BSF processing offers significant revenue potential and job creation opportunities. Sensitivity analysis confirms the robustness of these rankings under various stakeholder priorities.

For policy implications, decentralized composting and BSF systems should be prioritized, supported by incentives, training, and pilot projects for eco-enzyme initiatives. Broader stakeholder inclusion, integration of life cycle assessment, and empirical cost analysis are recommended for future evaluations to strengthen the evidence base and guide sustainable waste management strategies.

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Environmental Remediation and Sustainable Approaches to Heavy Metal Removal: A Comprehensive Review of Biosorption Techniques

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ABSTRACT

Heavy metal-laden contaminated water poses a severe environmental threat due to its bioaccumulation tendency, persistent nature, and toxicity. Conventional wastewater treatment techniques, such as ion exchange, chemical precipitation, membrane separation, and electrocoagulation, can result in secondary pollutants, require high energy use, and involve high costs. This review discusses environmentally friendly methodologies for the removal of heavy metals from aqueous streams, and the biosorption process is a viable alternative to address sustainability concerns in conventional, energy-intensive industrial processes. The biosorption process utilises a wide range of natural biomasses, such as plants, fungi, algae, and bacteria, for the sequestration and removal of heavy metals from aqueous solutions. Biosorption is mediated through various mechanisms, including physical adsorption, ion exchange, complexation, precipitation, and intracellular transport. The effectiveness of a biosorbent is based on the efficiency of sequestration and removal of heavy metals under given conditions. Some important factors affecting this include pH, temperature, contact time, biomass loading, and initial heavy metal ion concentration in the solution. Additionally, the capacity for biosorption for regeneration and reuse increases its commercial viability. This work explored the sources of biosorbents and the driving forces that govern their biosorption efficiency. Furthermore, this study provides an in-depth discussion of the factors that affect the effectiveness of the process. It establishes a fundamental understanding of biosorption mechanisms and influencing factors, paving the way for future commercialisation of this promising technology.

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Note: From 2025, the journal has adopted the use of Article IDs in citations instead of traditional consecutive page numbers. Each article is now given individual page ranges starting from page 1.

INTRODUCTION

Environmental pollution due to heavy metals has multiplied from a regional problem to a worldwide one, exacerbated by rising urbanisation, industrialisation, and poor wastewater treatment. Water pollution, which may arise from an array of sources such as agriculture, wastewater, oil spills, and radioactive materials, is among the significant problems that civilisation is currently experiencing(Jahan & Singh 2023). Heavy metals (HMs) such as arsenic, copper, cadmium, chromium, lead, nickel, and zinc are persistent pollutants that accumulate in the environment, and industrial effluent in particular has the potential to be a substantial carrier of these pollutants (Aliyu et al. 2023). Because they contaminate the habitats of marine organisms, these heavy metals constitute a serious hazard to water bodies, including streams, lakes, and oceans (Jahan & Singh 2023). HMs bioaccumulate in the food chain, and people inadvertently endanger their health when they consume contaminated foods such as fish and vegetables. Owing to this threat, regulatory bodies worldwide have tightened permissible discharge limits and call for sustainable remediation alternatives.

Biosorption, as a biologically inspired technique, has garnered attention due to diverse biosorbents, ranging from microbial biomass to plant-based waste. However, in terms of data comparability, method standardisation, and clarity

about sorption mechanisms across various biosorbent types, the area is still fragmented despite a large number of investigations. To create systems that are adaptable, dependable, and economical for treating waters, more research and development of bioprocesses is necessary. Presenting the state of the art in biosorption research and contrasting findings from previous studies are the goals of this work.

The originality of this work lies in three main aspects:

Material Innovation: Modification (chemical/physical) enhances surface area and functional groups, thereby increasing metal-binding affinity. Modified selected biosorbents with their adsorption capacity are elaborated in the review.

Critical Analysis of Influencing Parameters: To assess how important aspects like pH, temperature, contact time, biomass dosage, and initial metal ion concentration affect biosorption performance. This work focuses on results from diverse investigations rather than presenting experimental data. Based on kinetic and isotherm models from the literature, the review also addresses the mechanisms at play, including complexation, ion exchange, physical adsorption, and others.

Sustainability Emphasis: The use of abundant, biodegradable biomass supports circular economy principles and promotes waste materials for environmental remediation.

Overall, the findings contribute to expanding the library of efficient biosorbents and offer a pathway for practical, sustainable heavy metal remediation technologies in developing regions.

BIOSORPTION INSIGHT

Plants and microorganisms produce many biomaterials in the form of biomass, which is utilised in the biological physicochemical process of biosorption to absorb or adsorb a target species, such as metal ions or dyes (Gadd 2009).

Biosorption using a biomaterial is a two-phase process: one for the mobile or liquid phase and one for the stationary phase, which in this case is biomass. The mobile phase is usually an aqueous solution of dissolved metal ions or dyes, known as sorbates. Algae, bacteria, fungi, and plants act as immobile or stationary phases of biosorbents in treatment processes. They act as effective biomass for removing heavy metals from aquatic systems (Apriani et al. 2024). Metal ions are mainly adsorbed through the cell walls of these biological sorbents (Ali Redha 2020). A variety of biosorbents, such as fungi and algae, consist of an extensive number of functional groups in their cell walls, which play an important role in the process of biosorption.

These functional groups include carboxyl (-COOH) groups found in proteins, fatty acids, and organic acids, esters (-O-) present in lipids, carbonyl groups (C=O), which can be either internal or terminal, as seen in ketones and polysaccharides, phosphate groups, ester linkages (RCOOR') found in lipids, alcohols, and carbohydrates, and hydroxyl (-OH) groups (Silva et al. 2018). Certain biosorbents possess functional groups, such as imidazole, amino, sulfonyl, sulphate, phenolic, thioether, and amide groups, which improve the adsorption procedure. Calculation using Equation 1 is carried out to find the percentage removal of metal during the biosorption process.

$$\%\text{Removal of Cr(VI)} = \frac{C_{initial} - C_{final}}{C_{initial}} \times 10 \quad \ldots (1)$$

The biosorption capacity and equilibrium time of the adsorption process were calculated using the following formulas:

$$Q = \frac{(C_o - C_e)\,V}{m} \quad \ldots(2)$$

Where

- C_f: Final concentration of Cr(VI) in solution ($mg.L^{-1}$)
- C_i: Initial concentration of Cr(VI) in solution ($mg.L^{-1}$)
- C_0: Initial concentration of Cr(VI) in solution ($mg.L^{-1}$)
- Ce: Concentration of Cr(VI) in solution at equilibrium ($mg.L^{-1}$)
- V: Volume of the solution (L)
- m: Mass of biomass (g)
- Q: Metal uptake or adsorption capacity ($mg.g^{-1}$)

(Singh et al. 2021).

The quantity of heavy metal ions that a biosorbent can adsorb or absorb (biosorbents) is directly proportional to its sorption capability (Zyoud et al. 2019).

MECHANISMS OF BIOSORPTION

The ability of biological materials to accumulate heavy metals from wastewater through either spontaneous or metabolically mediated uptake pathways (using ATP) is known as biosorption and bioaccumulation, respectively (Ahmed et al. 2022, Chugh et al. 2022). The biosorption mechanism is also influenced by the nature and number of binding/reactive sites, accessibility and availability of binding sites, and the affinity between the binding site of the biosorbent and the concerned metal ion, which are just a few of the properties that affect the mechanism of biosorption (Abdel-Aty et al. 2013). It can also occur as a result of certain types of inactive, non-living microbial biomass that can bind and concentrate heavy metals even from very dilute aqueous solutions.

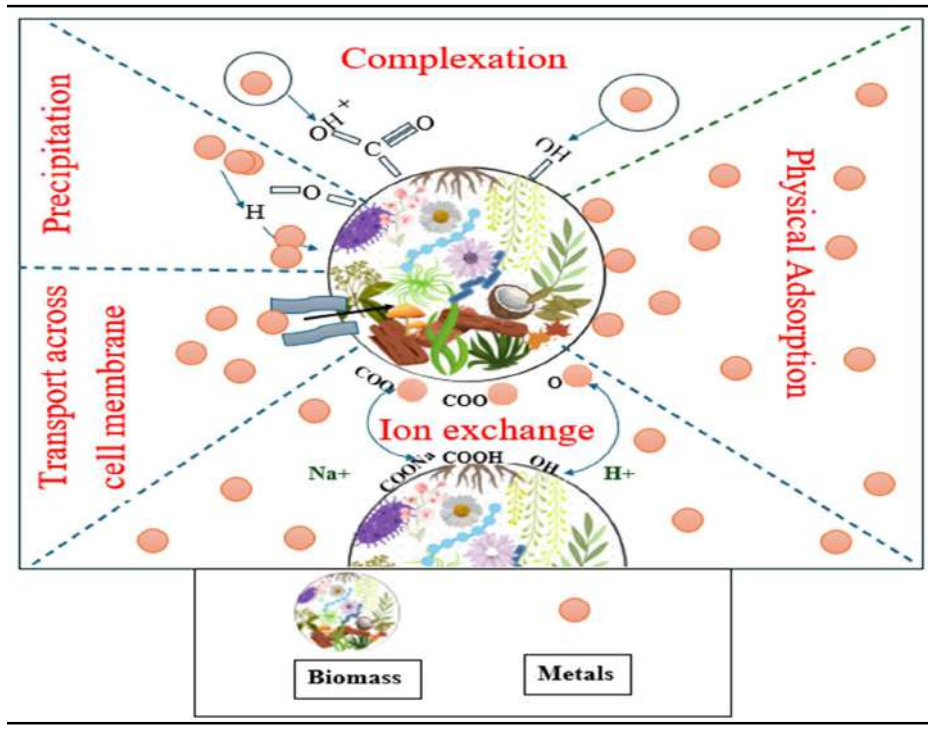

Fig. 1: Mechanism of biosorption.

Biosorption mechanisms include ion exchange, physical adsorption, chemical adsorption, precipitation, complexation, and electrostatic interactions (Javanbakht et al. 2014, Alavi & Zilouei 2014). In certain situations, many mechanisms may occur together in a multistage procedure (Noli et al. 2019). Biosorption efficiency and specificity depend on the type of biosorbent used and the physicochemical characteristics of the pollutants. Furthermore, because of the biological complexity of biosorbents, the majority of these processes are not completely known in detail.

Biosorption can be (i) metabolism-dependent or (ii) non-metabolism-dependent according to biomass activity. The non-metabolic mechanism is depicted in Fig. 1, which covers the mechanisms involved, such as intracellular accumulation, precipitation, ion exchange, and cell-surface adsorption/precipitation (Manmohan & Gajalakshmi 2024).

Precipitation and passage across the cell membrane are mechanisms of biosorption or bioaccumulation that are dependent on cell metabolism, whereas physical adsorption, ion exchange, precipitation, and complexation are mechanisms of biosorption that are independent of cell metabolism (Avanzi et al. 2014, Rene et al. 2017). Regarding the second criterion, transport across the cell membrane results in extracellular accumulation and precipitation, while ion exchange, complexation, physical adsorption, and precipitation result in cell surface sorption and precipitation. Precipitation, on the other hand, results in intracellular accumulation (Ali Redha 2020). Another fundamental mechanism observed in the majority of biosorption types is simple diffusion. The coordination, stereochemical, and chemical properties of the target metal, which comprise the ionic radii, ion mass, and oxidation state of the metal ion, as well as other variables, may play a role in regulating these processes (Kanamarlapudi et al. 2018).

Other parameters, such as concentration, time, pH, temperature, and the complex matrix of the solution containing the metal ion, depend on the operational conditions in which biosorption occurs. (Kanamarlapudi et al. 2018).

Physical Adsorption

Physical adsorption is one of the major biosorption processes, in which adsorbates attach to biosorbent surfaces by weak interactions such as van der Waals forces, dipole-dipole interactions, London dispersion forces, and hydrogen bonding. Such forces tend to interact with functional groups on the biosorbent surface, such as those found on cell walls. Physical adsorption can lead to the development of several layers of adsorbates on the biosorbent. Several factors impact the efficiency of physical adsorption, including biosorbent surface area, temperature, pressure, pore structure, and the nature of the adsorbate and adsorbent, and, in some instances, solution pH (Bashir et al. 2019). Research on Pb biosorption

using hami melon peels revealed that physical adsorption was highest in alkaline media. This resulted from the engagement of hydroxyl (-OH) and carboxyl (-COOH) groups on the surface of the biomass, which enhanced metal binding (Bashir et al. 2019). Similarly, pinewood biomass-derived biochars produced through hydrothermal liquefaction have been used to remove Pb from water, affirming the efficiency of physical adsorption in metal recovery processes. These results confirm the relevance of surface functional groups as well as environmental parameters to guarantee optimal physical adsorption for treating wastewater.

Ion Exchange

Ion exchange is an immobile solid material-bound, stoichiometric, and reversible chemical process whereby ions of an electrolyte solution or molten salt are exchanged with ions of like charge bound to an immobile, solid material, to achieve overall electroneutrality (Gadd 2009). The most widely used ion-exchange materials are synthetic. Organic resins, inorganic matrices, and advanced hybrid materials are the most widely used synthetic ion-exchange materials. For instance, Ganoderma lucidum fungus has been found to biosorb copper ions through ion exchange, as enabled by polysaccharide-rich cell walls of microorganisms such as bacteria and fungi, which allow counter-ion exchange (Ali Redha 2020).

Studies have proven that heavy metal ions like cadmium(II), copper(II), and zinc(II) are bound to brown algae, with the release of lighter ions such as sodium, potassium, magnesium, and calcium (Abdi & Kazemi 2015, Volesky and Holan, 1995). Similarly, rice straw, which is a non-metabolism-dependent biosorbent, removes cadmium(II) ions effectively by ion exchange of lighter ions. Solution pH plays an extremely crucial role in ion-exchange mechanisms. For instance, ion exchange is responsible for lead ion biosorption by hami melon peels at relatively low pH levels, while electrostatic interaction dominates at relatively high pH levels.

The interaction between lead ions and functional groups such as hydroxyl and carboxyl on biomass becomes increasingly prevalent (Bashir et al. 2019). Furthermore, cyanobacteria like Spirulina were able to biosorb copper(II), chromium(III), and cadmium(II) ions by means of ion exchange, facilitated by functional groups such as carboxyl, phosphate, and hydroxyl groups on their surface (Kanamarlapudi et al. 2018). Such findings exhibit the diversity of ion exchange in biosorption processes across different biosorbents and environmental conditions.

Complexation

Complexation is the process of electrostatic attraction or covalent bonding of metal ions and organic molecules that serve as ligands capable of donating electrons. Chelation is an advanced form of complexation where an organic ligand forms covalent bonding with the metal ion from multiple directions and results in a more stable resultant complex. Hard and Soft Acids and Bases (HSAB) theory predicts that elements be classified as hard or soft bases (mainly non-metals) and as hard or soft acids (mainly metals) is the premise upon which metal ions and organic ligands' affinity depends, and is the most crucial parameter in complexation.

For being of a soft acidic type, lead selectively covalent binds to biosorbent organic ligands that bear soft bases like nitrogen or sulphur. Moreover, borderline-classified ions like manganese, zinc, cadmium, as well as copper, also possess a high affinity to form compound's complexes with organic compounds that bear nitrogen or sulphur (Afolabi et al. 2021). But the availability and accessibility of that binding site that contains the base's donor atom matter to influence complexation. One can confirm that the biosorption mechanism is done by complexation or not by performing desorption experiments using energy dispersive X-ray analysis (EDX), transmission electron microscopy (TEM), and scanning electron microscopy (SEM) (Bhat et al. 2024).

Wastewater can be treated effectively using complexation to strip metal ions like Pb, Cu, Zn, and Cr. In undertaking this, it is important to take into consideration parameters like ligands, cost, toxicity, and possible environmental implications (Chai et al. 2021).

Precipitation

One of the metabolism-dependent processes in biosorption is precipitation, which is the formation of insoluble forms of metals as precipitates, however, precipitation can also lead to metabolism-independent biosorption. Precipitation results from the active defence process of microorganisms in metabolism-dependent biosorption upon exposure to toxic metal ions (Ali Redha 2020). Precipitation is triggered by the chemical interaction of metal ions with the functional groups of the cell wall of the biosorbent in metabolism-independent biosorption (Kanamarlapudi et al. 2018). Such processes might involve oxidation-reduction reactions.

Transport Across the Cell Membrane

Microorganisms are the only organisms that often exhibit the mechanism of heavy metal ion transport across cell membranes (Patil et al. 2024, Shafiq & Rehman 2024). This mechanism consists of two stages, the first is called independent binding metabolism and involves the binding of metal ions to binding sites on the microorganism's cell wall (Spain et al. 2021). The second stage is metabolism-

dependent intracellular uptake and involves the transport of metal ions into the cell through the cell membrane (Ali Redha 2020). This technique is analogous to how necessary metal ions are absorbed by cells. Heavy metal ions with charges and ionic radii comparable to those of essential metal ions have been reported to deceive cellular metal transport mechanisms.

TYPES OF BIOSORBENTS

Bacteria

Gram-positive and gram-negative bacteria are categorized according to cell wall thickness and composition, which is the primary distinction between the two types of bacteria(Zyoud et al. 2019). Thicker peptidoglycan layers linked by amino acid bridges were observed on the cell walls of gram-positive bacteria. Gram-positive bacteria contain polyalcohols by name, teichoic and teichuronic acids, which are connected by phosphodiester bonds and adhere to the peptidoglycan of the cell wall, therefore, they have a greater capacity to eliminate heavy metal cations because of their substantial electronegative charge density (Abdi & Kazemi 2015).

Functional groups, including oxygen, nitrogen, sulphur, or phosphorus, commonly facilitate heavy metal biosorption by bacteria. Using FTIR spectrum data, a finding on the biosorption of lead(II) ions by Aeromonas hydrophila revealed that the hydroxyl, sulphate, thiol, thioether, phosphate, phosphonate, carboxyl, and amine groups of the biosorbent surface exhibit higher removal capacity of lead(II) ions from aqueous medium. In a fairly recent study, the removal of Uranium (VI) biosorption onto Bacillus amyloliquefaciens was investigated. FTIR and XPS of Bacillus amyloliquefaciens suggest the presence of functional groups like -COOH, -OH, and -NH2 (Liu et al. 2019).

Algae

Micro-algae and macro-algae are two main phyla of algae. Macro-algae are multicellular marine algae (macroscopic) that can grow in saltwater and freshwaters (Pinto et al. 2023). Macro-algae can be differentiated into three divisions based on pigmentation: Chlorophyta (green macroalgae), Rhodophyta (red macroalgae), and Phaeophyta/Ochrophyta (brown macroalgae) (Handayani et al. 2023).

Micro-algae, on the other hand, are photosynthetic one-celled plants that can grow in surface water (Goswami et al. 2022). Micro-algae can be distinguished based on their morphology, color, and orientation of photosynthetic membranes (Fernández et al. 2018). Blue-green algae (cyanobacteria), green algae, diatoms, and golden algae further classify the microalgae (Rajkumar & Yaakob 2013).

Algae hold high biosorption capacity thanks to a cell wall structure that is made up of chitin, polysaccharides, proteins, and lipids (Ahmed et al. 2022). All these components have important functional groups that support biosorption. Algae contain oxygen, nitrogen, phosphorus, and sulphur as the major functional groups that support heavy metal adsorption on the algae surface.

When comparing different macroalgae, it was observed that brown algae exhibited high metal binding. Cellulose, alginic acid, polymers (such as mannuronic and guluronic acids) complexed with light metals (such as calcium, potassium, and sodium), and polysaccharides (such as fucoidan) make up the majority of cell walls. Fucoidan and alginate both can bind metals by ion exchange, alginate's primary binding sites for biosorption are carboxyl groups, followed by sulphate groups (Hamza et al. 2022). Proteins, which comprise functional groups such as amino, carboxyl, hydroxyl, and sulphate that aid in metal biosorption, constitute the majority of the material in the cell walls of green algae. However, despite having mostly cellulose in their cell walls, red algae can biosorb substances because they contain sulphated polysaccharides derived from galactans. The cell wall composition of microalgae varies slightly from that of other algae because they are primarily composed of polysaccharides, lipids, and proteins. These substances include carboxyl, hydroxyl, phosphate, and sulphate groups that give the surface of algae an overall negative net charge, which facilitates the biosorption of metal cations by counter-ion interactions.

Of all the different types of algae and biosorbents, brown algae have shown the highest capacity for biosorption of various metal ions. Its surface area to volume ratio is large. It's readily available, produces little sludge. Furthermore, biosorbed metals may be recovered and regenerated by brown algae. All things considered, algae are the most extensively used biosorbents due to their accessibility, comparatively low processing costs, and exceptional efficiency.

It was studied that the Blue-green algae, Spherical Anabaena, were efficient at extracting Pb (II) and Cd (II) from aqueous solutions (Ali Redha 2020). The results show that the algae can remove lead (II) and cadmium (II) with relatively high biosorption capacities, with estimated levels of 111.1 $mg.g^{-1}$ and 121.95 $mg.g^{-1}$, respectively (Abdel-Aty et al. 2013). The FTIR analysis of functional groups on algal surfaces indicates that amino, carboxyl, hydroxyl, and carbonyl groups bio-sequester metal ions (Abdel-Aty et al. 2013).

Fungi

Fungi possess a unique high cell wall composition, which

in turn shows the existence of a variety of functional groups that may affect the elimination of metals. Mannoproteins (glycoproteins), β-Glucans(polysaccharides), chitin (Polymer), cellulose, chitosan, α-glucans, galactomannans, galactosaminogalactan and pigments are components of fungal cell walls. Fungal biomass is more easily generated on a large scale using straightforward fermentation procedures at a relatively low cost, in contrast to yeast, algae, and plants. FTIR study on Mucor rouxii fungi suggests that the presence of amine and phosphate groups contributed most to the biosorption of Pb, Cd, Ni, and Zn ions (Ali Redha 2020).

Using Aspergillus niger fungi (isolated from electroplating industry effluent), 97% removal of Pb(II) ions was observed (Dhaka et al. 2025). Myconanotechnology" is a promising field that employs fungal biomass for the making of nanoparticles. Fungal species, including Penicillium, Aspergillus, Alternaria, Chrysosporium, Cladosporium, Candida, Cryptococcus, Neurospora, Fusarium, Trichoderma, Pleurotus, Agaricus, Coriolus, Ganoderma, Verticillium, etc., are broadly employed in nanoparticle synthesis (Meera et al. 2025).

Plants

Plants have been employed as biosorbents for the reuse and recycling of waste products from the food and agricultural sectors. Consequently, plant materials are inexpensive. The cell walls of plants contain a cellulosic matrix, which is known to have carboxylic or phenolic functional groups or cellulose-related components, such as lignin and hemicellulose, which are primarily responsible for the potential of plant biosorbents. The binding of a metal ion with a functional group causes biosorption, which removes the metal ions from the medium based on cation exchange between functional groups and metal ions(Abdel-Aty et al. 2013). It was discovered that the carboxyl, carbonyl, and hydroxyl groups promoted metal binding when Aloe vera waste was employed as a biosorbent to remove uranium and cadmium from water (Kapashi et al. 2019). Mango seed cover with kernels and jamun seed cover with kernels removed led to 94.85% and 92.78%, respectively (Pal et al. 2022).

Yeast

Yeast is a topic of interest in biosorption, as several studies have been successfully conducted (Danouche et al. 2021). The most significant commercial yeasts belong to the Saccharomyces genus, specifically baker's and brewer's yeasts. With basic growing media, they are non-pathogenic, simple to cultivate, and provide high biomass production. A literature review reveals that yeast biomass may be efficiently consumed as a biosorption agent for metals such as Au, Ag, Cd, Cu, Ni, Pb, Cr, and radioactive metals such as Th and U (Danouche et al. 2021). Yeasts such as Candida, Saccharomyces, and Pichia are effective biosorbents for metals like Pb, U, Hg, and Cd (Jamir et al. 2024). Most yeasts can be selective to one metal or can sorb a broad category of metals (Ahmed et al. 2022).

Moss and Lichens

Some lichen species, along with mosses, have been widely investigated for their ability to adsorb heavy metals from the environment (A et al. 2022). Lichens, which are composite plants made up of fungi and algae, are thought to be an indication of the quality of the environment because of their capacity to absorb and hold onto a wide range of pollutants, including heavy metals and radionuclides (Boruah et al. 2024). The batch approach was used to assess the capacity of lichen biomass (Xanthoparmelia conspersa) to remove mercury(II) ions from aqueous solutions via biosorption. Kinetic studies were carried out, which revealed that it followed second-order kinetics, whereas ΔG^o, ΔH^o, and ΔS^o indicated that the process was exothermic. Living lichens such as R. fraxinea accumulate metals across their surfaces. An investigation on seven different types of moss species growing in the area of the Bory Stobrawskie forest (southern Poland) was carried out in the laboratory. The study revealed that the sorption of Zinc (Zn) and Cadmium (Cd) depends on moss species, and the percentage sorption increases in the following order: P. commune < L. glaucum < Eurhynchium praelongum < T. tamtariscifolium ≤ D. scoparium ≤ P. schreberi < Sphagnum sp. (Kłos et al. 2014)

Comparison of the biosorption efficiency of different biosorbents shows that fungi and plants exhibit the highest efficiency, followed by bacteria and algae (Fig. 2).

A trend line graph (Fig. 3) indicates how biosorption efficiency has increased over the years for different biosorbents. The data imply that fungi and plants have exhibited the most consistent increase in efficiency, followed by bacteria and algae.

The scatter plot (Fig. 4) shows the relationship between pH levels and biosorption efficiency for different biosorbents. It highlights that fungi and plants exhibit higher biosorption efficiency across a wide pH range, while bacteria and algae show optimal performance in neutral to slightly acidic conditions.

The pie chart (Fig. 5) demonstrates the impact of different biosorbents on overall biosorption. Fungi (30%) and plants (28%) give the most, while bacteria (22%) and algae (20%) play slightly smaller roles.

Using a predictive regression model, the biosorption efficiency of fungi-the best-performing biosorbent-shows

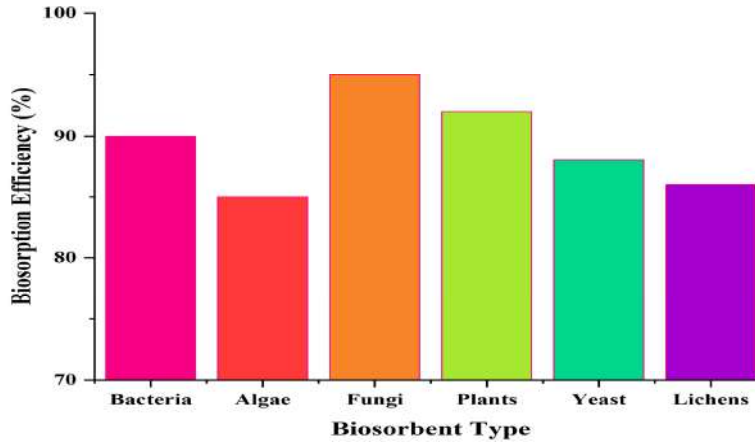

Fig. 2: Biosorbent type and its biosorption efficiency %.

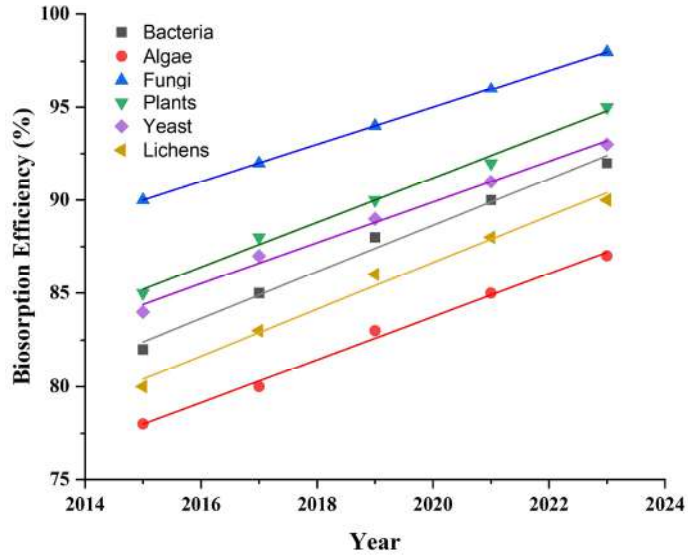

Fig. 3: Trend line graph indicating biosorption efficiency.

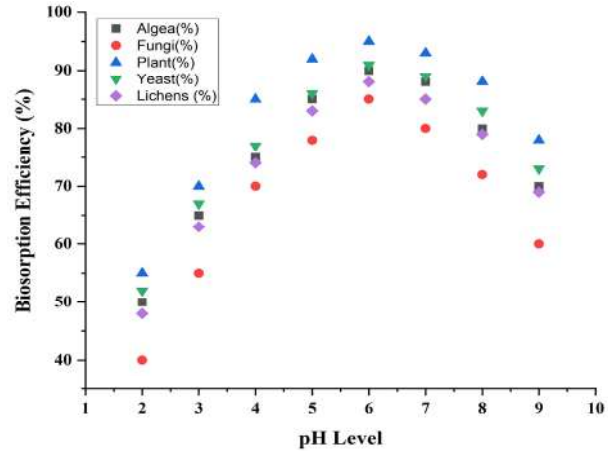

Fig. 4: Influence of pH on biosorption efficiency.

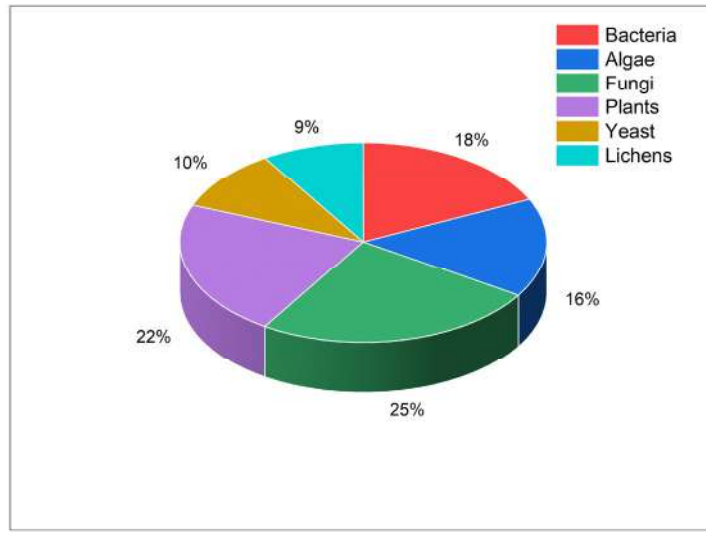

Fig. 5: Impact of different biosorbents on overall biosorption.

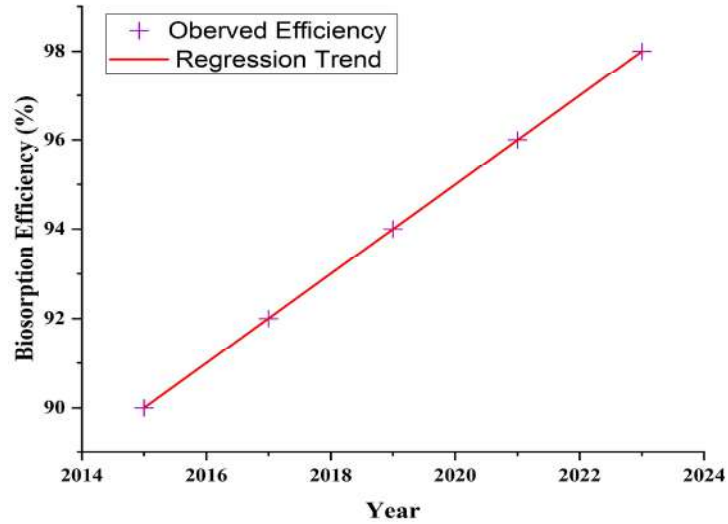

Fig. 6: Regression model to predict biosorption efficiency trends.

a rising tendency over time. Future developments in biosorption methods may further increase efficiency, as seen by the black regression line, which shows a consistent improvement (Fig. 6).

BIOSORPTION OF SELECTED HEAVY METALS

Heavy metals, including lead (Pb), chromium (Cr)(VI), arsenic (As), cadmium (Cd), and mercury (Hg), are non-threshold poisons that may cause harm even at low doses (Chen et al. 2023, Campbell 2023). These are toxic heavy metals (THMs) and are the most hazardous heavy metals. Owing to several industrial operations, as well as some natural operations, the environment is heavily contaminated. Elevated levels of heavy metals in many natural systems, including the atmosphere, pedosphere, hydrosphere, and biosphere, have recently emerged as a worldwide concern. THMs severely damage a wide range of plants, animals, and microbes (Gupta et al. 2022). Human exposure to THMs can cause serious health issues, impairments, and, in rare circumstances, death in certain extremities (Gupta 2024). Heavy metals must be eliminated from wastewater and effluents before their release into the environment, owing to their toxicity, and to ensure that they meet safe discharge regulations (Liberti 2024). The discharge limit set for the above-listed metals: Lead (Pb) = 0.01 $mg.L^{-1}$, Chromium (Cr) = 0.05 $mg.L^{-1}$, Arsenic (As) =0.01 $mg.L^{-1}$, Cadmium (Cd)=0.003 $mg.L^{-1}$, and Mercury (Hg)=0.006 $mg.L^{-1}$ (BIS 2012).

Biosorption of Arsenic (As)

Arsenic is a naturally occurring element that often exists in two forms: organic and inorganic. More harmful inorganic arsenic compounds are frequently present in food and water, and can seriously harm human health (Rahaman et al. 2021).

Industries such as mining, smelting, and pesticide production can release arsenic into the environment. (Bundschuh et al. 2021, Khatun & Intekhab 2022). This in turn contaminates nearby water bodies, enters the food chain, and causes toxicity in plants and animals (Verma & Prakash 2022). Table 1 depicts the potential of the different biosorbents.

It causes toxicity through several methods. Oxidative stress causes the body to produce reactive oxygen species (ROS), damaging lipids, proteins, and DNA (Hu et al. 2020, Kumar 2021). Arsenic exposure causes enzyme inhibition, resulting in cellular malfunction and death by inhibiting the vital enzymes required for DNA repair and cellular respiration. (Mukherjee & Valsala Gopalakrishnan 2023). Prolonged exposure to arsenic leads to epigenetic modifications changing gene expression without affecting the DNA sequence, which may result in cancer (Mukherjee & Valsala Gopalakrishnan 2023).

Acute exposure leads to gastrointestinal distress, hypertension, skin diseases, and headaches.

As has been extracted from aqueous solutions using blue pine, walnut shells, and chickpea test (Bibi et al. 2017). Blue-green algae and the cyanobacterium Spirulina sp. may be used to extract arsenic from polluted waterways. At pH 6, the highest sorption capacities of both dead and live spirulina were 402 and 525 $mg.g^{-1}$, respectively.

Biosorption of Cadmium (Cd)

Cadmium (Cd) is hazardous to both the environment and human beings. Cadmium, present in the atmosphere, water, and food, when exposed to low concentrations of Cd, causes serious health problems.

Table 1: Biosorption capacity of different biosorbents for the removal of Arsenic ions from aqueous media.

Adsorbent	Biosorbent type	Temperature [°C]	pH	Metal ion	% Adsorption OR Adsorption capacity [$mg.g^{-1}$]	Reference(s)
Bacillus thuringiensis strain WS3, *Pseudomonas stutzeri* strain WS9	Bacteria	37	7.0	As (III) As (V)	95%, 98%	Altowayti et al. (2020)
Bacillus sp. KL1,KL4, KL6	Bacteria	40	5.0	As(V)	77% , 91.66%, 88%	Taran et al. (2019)
Bosea sp. AS-1	Bacteria	35	7.0	As (III)	99%	Lu et al. (2018)
Chlorella vulgaris	Algae	2	6.0	As (III)	93%	Alharbi et al. (2023)
Ulva reticulata	Algae	-	4.0	As (V)	59.5%	Senthilkumar et al. (2020)
Chlorella vulgaris	Algae	50	6.0	As (V)	13 $mg.g^{-1}$	Ghayedi, Borazjani and Jafari (2019)
Indigenous fungi	Fungi	27±2	6.0	As	70 $mg.g^{-1}$	Jaiswal et al. (2018)
Aspergillus spp. APR-1 and APR-2	Fungi	40	2.0	As (III)	53.94%	Tanvi et al. (2020)
Talaromyces sp.	Fungi	30	6.0	(As)	70 %	Nam et al. (2019)
Mucor circinelloides	Fungi	25	6.0	(As)	29.4 $mg.g^{-1}$	Li et al. (2021)
Warnstorfia fluitans	moss	20	6.5	(As)	90%	Sandhi, Landberg and Greger (2018)
Saccharomyces cerevisiae	Yeast	35	5.0	As (III)	62.90 $mg.g^{-1}$	Wu et al. (2012)
Saccharomyces cerevisiae	Yeast	25	7.0	As (III) As (V)	66.2%, 15.8%	Hadiani et al. (2019)
Psychrotolerant *Yersinia* sp. strain	Yeast	30	7.0	As (III)	96%	Asadi Haris et al. (2018)
Bacillus thuringiensis	Yeast	37	7.0	As (III)	10.94 $mg.g^{-1}$	Altowayti et al. (2019)
Cassia fistula pods	Plant	30	6.0	As (III)	91%	Giri et al. (2022)
Leaves of *Tectona Lagerstroemia speciosa*	Plant	25	6.0	As(V)	94.6%	Verma and Singh (2019)

Table 2: Biosorption capacity of different biosorbents for the removal of Cadmium ions from aqueous media.

Adsorbent	Biosorbent type	Temperature [°C]	pH	% Adsorption OR Adsorption capacity [mg.g^{-1}]	Reference(s)
Bacillus subtilis	Bacteria	30	4.0	83.5%	Devatha (2020)
Pseudomonas fluorescens	Bacteria	32.7	6.01	90%	Rahman et al. (2022)
Bacillus subtilis	Bacteria	30	4.0	83.5%	Devatha (2020)
Pseudomonas fluorescens	Bacteria	32.7	6.01	90%	Rahman et al. (2022)
Halomonas BVR 1	Bacteria	-	8.0	12.023 mg.g^{-1}	Manasi et al. (2014)
Chlorella sp.	Algae	-	7.8–8.0.	59.67%	Mátyás et al. (2018)
Ulva lactuca sp.	Algae	25	5.0	43.12 mg.g^{-1}	Ghoneim et al. (2014)
Caulerpa fastigiata	Algae	25	5.5	92.01%	Sarada et al. (2014)
Sargassum polycystum	Algae	25	4.65	86.20 mg.g^{-1}	Jayakumar et al. (2022)
Phlebia brevispora	Fungi	25	7.0	91.6%	Sharma et al. (2020)
Fusarium solani	Fungi	28 ± 1	10.0	92.4%	Kumar et al. (2019)
Phanerochaete chrysosporium	Fungi	-	4.15	60%	Rudakiya et al. (2018)
Saccharomyces cerevisiae + Alg beads	Yeast	25	6.0	83%	Rivas et al. (2019)
Saccharomyces cerevisiae	Yeast	-	6.0	90%	Arora (2019)
Xanthate-modified baker's yeast	Yeast	46	8.0	239.80 mg.g^{-1}	Song et al. (2019)
Hovenia acerba	Plant	25 ± 2	3.0	56.99%	Pyrzynska (2019)
Murraya koengii	Plant	25	7.0	22.29 mg.g^{-1}	Mukherjee et al. (2020)
Poplar sawdust	Plant	-	5.0	49.32 mg.g^{-1}	Cheng et al. (2021)
Corn stalk	Plant	-	7.0	40 mg.g^{-1}	Chen et al. (2020)
Moringa oleifera (Moringa) seeds	Plant	20 ± 1	3.0	97%	Aziz et al. (2016)

Cd exposure through water, air, and soil results in Cd toxicity, which can affect the respiratory system, bones, kidneys, and reproduction, and, in some cases, can lead to cancer in humans. Low concentrations of Cd are highly toxic and carcinogenic to plants. (Hayat et al. 2018). Cadmium can bind to ligands such as cysteine, glutamate, histidine, and aspartate in the human body, which can result in iron deficiency (Burnase et al. 2022). Cd can induce hepatotoxicity when it binds to cysteine-rich proteins such as metallothionein in the liver. Mining, alloys, batteries, paint pigments, smelting, electroplating, and fertilizer sectors are sources of Cd. The biosorption capacities of several biosorbents investigated for the removal of Cd ions are compared in Table 2.

Biosorption of Chromium (Cr)

According to Chen et al. (2024), chromium (Cr) is a heavy metal that is toxic, carcinogenic, mutagenic, and teratogenic. Hexavalent and trivalent versions are the most common (Itankar & Patil 2021). The hexavalent form of Cr(VI), which is roughly 500–1000 times more dangerous than the trivalent form, is a major contaminant in both surface water and groundwater due to its high mobility and solubility in aqueous environments (Itankar & Patil 2022). It can damage the kidneys, liver, and stomach in addition to causing allergic reactions, respiratory problems, and compromised immune systems.

The primary industrial sources of chromium include foundries for iron and steel, electroplating, metallurgy, metal finishing, welding of alloys/steel, ceramic manufacturing, tanneries, textiles, leather tanning, and inorganic chemical facilities (Nilisha Itankar & Yogesh Patil 2022). Before releasing the effluent into the environment, the industry must eliminate all traces of chromium.

Experimental studies were carried out to demonstrate the ability of various biomasses to eliminate chromium from aqueous media, and the results are shown in Table 3, which contrasts the biosorption capacity of each biosorbent.

Biosorption of Lead (Pb)

Table 3: Biosorption capacity of different biosorbents for the removal of Chromium ions from aqueous media.

Adsorbent	Biosorbent type	Temperature [°C]	pH	Metal ion	% Adsorption OR Adsorption capacity [mg.g^{-1}]	Reference(s)
Chelatococcus daeguensis	Bacteria	50	7.0	Cr (VI)	88.89%	Fernández et al. (2018)
Klebsiella sp.	Bacteria	30	9.0	Cr (VI)	95%	Hossan et al. (2020)
Bacillus amyloliquefaciens	Bacteria	37	6.0	Cr (VI)	79.90%	Ramachandran et al. (2022)
Escherichia coli	Bacteria	37	-	Cr (III)	91.29%,	Wang et al. (2021a)
Rhizobium	Bacteria	28±1	-	Cr (III), Cr (VI)	76 %	Srinivas Ravi et al. (2022)
Spirulina platensis	Algae	60	1.0	Cr(III), Cr (VI)	82.5%	Nithya et al. (2019)
Durvillaea antarctica	Algae	45	2.0	Cr (VI)	66.6%	Al-Homaidan et al. (2018)
Scenedesmus quadricauda	Algae	5–35	2.0	Cr (VI)	100%	Daneshvar et al. (2019)
Sargassum filipendula	Algae	60	3.5	Cr (III)	67.5%	da Costa et al. (2022)
Green microalgae	Algae	80	3.0	Cr (VI)	99.75%	Indhumathi et al. (2014)
Aspergillus niger	Fungi	40	3.0	Cr (VI)	>99%	Chatterjee et al. (2020)
Rhizopus sp.	Fungi	30	2.0	Cr (VI)	95%	Espinoza-Sánchez et al. (2019)
Aspergillus flavus CR500	Fungi	20–40	(5.0–9.0)	Cr (VI)	89.1%	Kumar and& Dwivedi (2019)
Aspergillus niger	Fungal Biomass	25	3.0	Cr (VI)	90%	Kanamarlapudi et al. (2018)
Penicillium chrysogenum	Fungal Biomass	25	5.0	Cr (VI)	80%	Sheikhi and Rezaei (2021)
Penicillium sp.	Fungal Biomass	30	4.0	Cr (III)	75%	Sheikhi and Rezaei (2021)
Cladonia rangiferina	Lichen	25	2.0	Cr (VI)	92%	Pakade et al. (2019)
Sphagnum squarrosum	Moss	20	5.0	Cr (III)	75%	Pakadee et al. (2019)
Moss (*Sphagnum* spp.)	Moss	25	4.0	Cr (VI)	85.5%	Kabdaşlı and Tünay (2023)
Lichen (*Cladonia* spp.)	Lichen	28	6.0	Cr (III)	88.7%	Kabdaşlı and Tünay, (2023)
Saccharomyces cerevisiae	Dead yeast cells	25	4.0	Cr (VI)	85.4%	Saini et al. (2023)
Candida utilis	Live yeast	30	5.5	Cr (III)	92.3%	Saini et al. (2023)
Saccharomyces cerevisiae	Yeast cells	25	2.0	Cr (VI)	83.5%	Acharyya et al. (2023)
Saccharomyces cerevisiae	Immobilized yeast	30	4.0	Cr (III)	78.2%	Arrisujaya et al. (2023)
Kluyveromyces marxianus	Yeast biomass	25	3.0	Cr (VI)	88.0%	Arrisujaya et al. (2023)
Avocado seed	Plant	25	2.0	Cr (VI)	98.5	Sen (2023)
Wood apple Shell powder	Plant	30	4.0	Cr (VI)	99.8%	Itankar and Patil (2021)
Prosopis spicegera	Plant	-	2.0	Cr (VI)	97.69 %	Fernández et al. (2018)
Sargassum dentifolium	Plant	50	7.0	Cr (VI)	99.68%	Husien et al. (2019)

Lead(Pb) is emitted into the atmosphere by burning fossil fuels, lead compounds, automobile emissions, and companies that use lead (Violante et al. 2010). It usually combines with other elements to generate lead compounds. Lead sulphate, lead carbonates, and lead oxide are the products of the reaction of lead with air and water. Although lead is prevalent, human activity has been identified as the primary reason for rising lead levels in the environment (Hakeem 2015).

The estimated lead(II) ion concentration released in the environment from the battery sector is 5-66 $mg.L^{-1}$, the mining industry is 0.02-2.5 $mg.L^{-1}$, and the oil industry is 125-150 $mg.L^{-1}$ (Tasar et al. 2014). 173.8 Mt of Pb was released into the atmosphere between 1930 and 2010, with the majority coming from the manufacturing (26%), consumption (20%), and waste management and recycling (48%) phases. $PbSO_4$, PbO, Pb, and PbS were the primary species released, accounting for 61.2% of the total emissions (Hettiarachchi et al. 2024).

Animals and plants are considered to be fatally affected by lead. It results in several illnesses in humans, including anaemia, brain damage, mental deficiency, renal damage, encephalopathy, anorexia, cognitive impairment, behavioural problems, and vomiting (Singh et al. 2023). Pb may bioaccumulate in bones over more than 20 years and alter the cellular membrane permeability of organs and haemoglobin production in people when it binds to those enzymes (Singh et al. 2023). Pb (II) ions have a strong affinity for thio, oxo, and phosphate groups, which are found in a variety of enzymes and macromolecules in living organisms (Morozanu et al. 2017). Several experiments were carried out to assess the ability of various biosorbents to remove lead (II) ions from aqueous environments, a selection of these investigations, comparing the biosorption capacities of each, is shown in Table 4.

Biosorption of Mercury (Hg)

According to USEPA, Mercury is a ubiquitous contaminant, a global pollutant that is highly toxic and is readily gathered in the ecosystem (USEPA). It has been linked to public health catastrophes in Iran and Japan (Minamata Bay). Exposure to mercury vapours is the primary pathway affecting the human brain and lungs. Mercury poisoning primarily affects the nervous system, kidneys, and immune system. It also endangers aquatic and wild species (Ahmed et al. 2022).

It's salts, such as mercurous and mercuric salts, attack the gut lining and the kidneys. According to the WHO, the main sources of exposure include degassing of mercury from dental amalgam, consumption of contaminated fish, and other seafood. It enters by multiple pathways, both natural and anthropogenic, and contaminates the ecosystem's air, water, and soil. Natural forest fires, weathering of mercuriferous regions, degassing from surface water and the Earth's crust through volcanic eruptions, and biogenic emissions are all sources of naturally occurring mercury emissions into the environment(Gworek et al. 2020). Mercury pollution also originates from human activities, such as agriculture, battery

Table 4: Biosorption capacity of different biosorbents for the removal of Lead ions from aqueous media.

Adsorbent	Biosorbent type	Temperature [°C]	pH	% Adsorption OR Adsorption capacity [$mg.g^{-1}$]	Reference(s)
Bacillus licheniformis	Bacteria	30	6.0	98%	Wen et al. (2018)
Pseudomonas azotoformans	Bacteria	30	6.0	88.58%	Choińska-Pulit, Sobolczyk-Bednarek and Łaba, (2018)
Pannonibacter phragmitetus	Bacteria	30	6.0	49.79 $mg.g^{-1}$	Saravanan et al. (2021)RBTB
Ralstonia solanacearum	Bacteria	35	6.0	90%	Pugazhendhi et al. (2018)
Mixed- culture of algae	Algae	25 ± 2	6.0	95.43%	Mousavi et al. (2019)
Sargassum muticum	Algae	25	5.0	76 $mg.g^{-1}$	Hannachi & Hafidh, (2020)
Cladophora	Algae	-	4.0	20.56 $mg.g^{-1}$	Amro & Abhary (2019)
Talaromyces islandicus	Fungi	30	5.0	90.06%	Sharma et al. (2020)
Rhizopus arrhizus	Fungi	25	4.0	103.70 $mg.g^{-1}$	Senol et al. (2021)
Filamentous fungus	Fungi	28	5.0	53.7%	Wang et al. (2019a)
Phanerochaete chrysoporium	Fungi	-	5.5	75%	Zhao et al. (2020)
Sphagnum peat moss	Moss	26 ± 2	8.0	97.6%	Lubbad & Al-Batta, (2020)
Guar gum	Plant Material	-	5.0	83%	Mukherjee et al. (2018)
Arundinaria alpina	Plant Material	25	5.0	99.8%	Asrat et al. (2021)
Rice husk, wheat straw, and corncob	Plant Material	-	5.5	96.41%, 95.38%, 96.92%	Amen et al. (2020)
Phytolacca americana L.	Plant Material	25	6.0	93.29%	Wang et al. (2018)
Banana Peels	Plant Material	-	5.0	98.14 %	Afolabi et al. (2021)

Table 5: Biosorption capacity of different biosorbents for the removal of Mercury ions from aqueous media.

Adsorbent	Biosorbent type	Temperature [°C]	pH	% Adsorption OR Adsorption capacity [mg.g^{-1}]	Reference(s)
Pseudomonas putida	Bacteria	30	8.0	99.72%	Zhao et al. (2021)
Klebsiella sp. NT8 and *Bacillus* sp. NT10	Bacteria	25	6.0	2597.62 , 2617.23 mg.g^{-1}	Xia et al. (2020)
Live or dead biomass of *A. marina* SSS2	Bacteria	35	7.0	87%, 95%	Mukkata et al. (2019)
Chlorella vulgaris	Algae	35	6.0	95.5%	Kumar et al., (2020)
Ulva intestinalis, Ulva lactuca, Fucus spiralis, Fucus vesiculosus, Gracilaria sp., *Osmundea pinnatifida*	Algae	Room Temp.	8.5	87.2%, 94.0%, 88.0%, 93.0%, 86.0%, 87.7%	Fabre et al. (2020)
Ulva lactuca Linnaeus	Algae	22.2	4.0	96.1% ± 0.7	Çetintaş et al. (2022)
Sargassum crassifolium	Algae	Room Temp.	9.0	98%	Putri and Syafiqa, (2019)
F. velutipes	Fungi	Room Temp.	7.0	69.35%	Li et al. (2018)
Saccharomyces cerevisiae	Fungi	25	5.4	89%	Hadiani et al.(2018)
Penicillium sp.	Fungi	60	4–5.0	99.6%	Sánchez-Castellón et al. (2022)
Saccharomyces cerevisiae	Yeast	25	5.4	88.9%	Hadiani et al.(2018)
Roasted Date Pits	Plant material	25	6.0	95%	Al-Ghouti et al. (2019)
Pine biochar	Plant material	25	5.0	1641 mg.g^{-1}	Johs et al. 2019)
Coffee waste	Plant material	33	7.00	97%	Mora Alvarez et al. (2018)
Thymus schimperi	Plant material	25	7.0	90%	Geneti et al. (2022)

manufacturing, burning fossil fuels, mining and metallurgical processes, paint and chloralkali industries, and wood pulping. Other significant sources of mercury pollution include thermometers, electronic devices (LEDs, CFLs), wiring and control devices, the paper and pulp industries, and oil refining (Kumar et al. 2020).

In a recent study on Lentinus edodes, U. lactuca, and Typha domingensis, the mercury removal efficiency was found to be 100-337 mg.g^{-1} (Rani et al. 2021). According to research on biosorption, the hydrocolloid Gum Karaya had a maximum biosorption capacity of 62.5 mg.g^{-1} and suited the Langmuir isotherm ($R^2 > 0.999$)(Padil et al. 2021). Experimental results showed that algal biomass (Cristoseira baccata) removed 178 mg.g^{-1} of Hg^{2+} at 4.5 pH and 329 mg.g^{-1} at 6 pH. In batch mode, it was found that lichen biomass (Xanthoparmelia conspersa) removed 82.8 mg.L^{-1} of Hg^{2+} ions. In laboratory settings, Pseudomonas aeruginosa was found to exhibit a Hg^{2+} bioremediation capability of 62% (Tanwer et al. 2022). The different biosorption capacity of different biosorbents for the removal of Mercury ions from aqueous media is given in Table 5.

Fig. 7 shows the heat map that summarizes the function of different biosorbents towards different metals, displaying the percentage adsorption capacity. It demonstrates the biosorption efficiency of different biosorbents for several heavy metals. The darker shades suggest higher efficiency, with fungi consistently achieving the best across all metals, followed by plants, bacteria, and algae.

FACTORS AFFECTING BIOSORPTION CAPACITY

Numerous crucial operational and physicochemical factors regulate biosorption, such as pH, contact time, initial metal ion concentration, temperature, and biomass quantity. As covered below (Table 6), these have an impact on how metal ions and biosorbent functional groups interact.

Other Factors

Surface properties, including surface functional groups, surface area, pore size distribution, and particle size, all play an important role in determining biosorption efficiency. Functional groups involving carboxyl, hydroxyl, amino, and sulphuryl groups have been found to be active in metal ion binding, as evidenced using FTIR analysis in Chlorella vulgaris biomass for mercury removal. Increased surface areas and optimal pore sizes enhance metal ion capture, while smaller particle sizes improve the ratio of surface

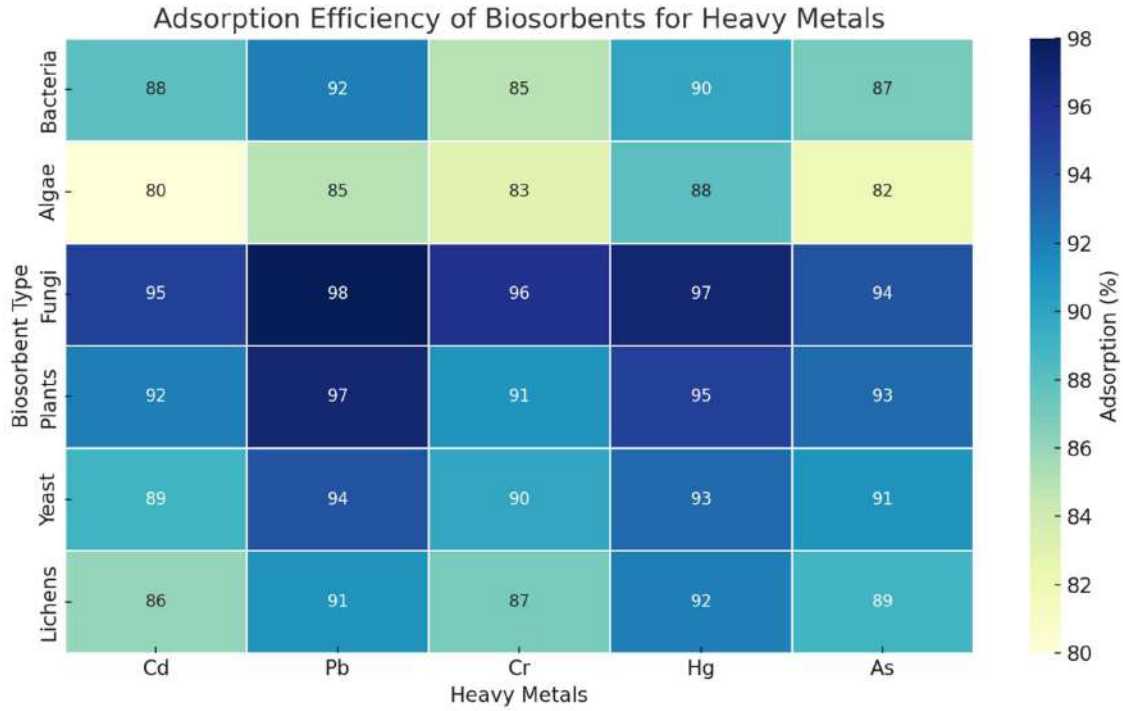

Fig. 7: Heavy metals adsorption by different biosorbents.

Table 6: Factors affecting biosorption.

Parameters	Effect on Biosorption	Optimal Range / Observation	Reference(s)
pH	Regulates metal ion speciation and charge on biosorbent surfaces. Protonation of binding sites at low pH decreases biosorption	Optimal range: 4–7, Precipitation at higher pH	Gadd (2009), Priya et al. (2022), Bilal et al.2018
Temperature	Affects biosorption kinetics and thermodynamics. Exothermic processes show decreased removal at higher temperatures	20–35°C stable for most biosorbents	Ali Redha (2020), Bilal et al. (2018)
Contact Time	Establishes the time required for equilibrium to be achieved. Greater contact enhances uptake up to the saturation point	60–120 minutes, not significantly altered after equilibrium	Nemeş and Bulgariu (2016)
Biomass Dose	Increases the number of binding sites present. Too high a dose causes site overlap or aggregation.	0.5–6.0 $g.L^{-1}$	Avanzi et al. (2014), Ali Redha (2020)
Initial Metal Concentration	Determines saturation level and helps assess biosorbent capacity	5–200 $mg.L^{-1}$	Al-Azzawi et al. (2013)
Other Factors	Surface area, Functional groups present on biomass, particle size, porous nature, agitation, and pre-treatment affect efficiency.	Modified biomass increases biosorption.	Kumar et al. (2020), Agoun & Avci (2024)

area to volume, further boosting sorption. Powdered forms excelled over dried leaves in extracting Zn, Pb, and Cu ions in a study involving lettuce leaves, indicating that particle size is crucial. The presence of more than one metal ion causes competition for binding sites and hence a reduction in sorption, depending on the affinity of each ion to the biosorbent. Agitation enhances the interaction between metal and biomass, but must be carefully controlled to avoid fracturing particles. Pre-treatment processes involving washing, modification by chemicals (acid/base/surfactant treatment), and physical treatment (drying and grinding) change surface properties and improve biosorption capacity. Chemically altered biosorbents have been found to exhibit significantly improved performance, for example, grafted copolymer-treated orange peel exhibited 4.2 to 16.5 times higher biosorption capacity for Pb^{2+}, Cd^{2+}, and Ni^{2+} ions as

compared to untreated material, demonstrating strongly the efficacy of modifying surfaces to improve metal capture in orange peel.

DESORPTION

Metals are precious, and desorbing agents help in the removal of biosorbed species (metal ions) from the biosorbent surfaces. The recovered metal species can be reused in the industry, and biosorbents can be reprocessed (Bayuo et al. 2024, Raji et al. 2023, Patil 2021).

Desorption is the step adopted by most researchers after biosorption. Desorption can be performed by removing the biosorbed metal ions utilizing a desorbing agent, which enables the reuse of the exhausted biosorbent (Bayuo et al. 2024, Calero et al. 2013). However, challenges related to the loss of biosorption efficiency after regeneration have not been thoroughly studied.

The following formula is used to compute the proportion of desorbed species:

$$\%\text{Desorption} = (q_{desorbed}/q_{adsorbed}) \times 100$$

Where:

- $q_{desorbed}$ = amount of substance desorbed (released) from the material (in appropriate units such as moles, grams, etc)
- $q_{adsorbed}$ = the amount of substance originally adsorbed onto the material

A high desorption percentage indicates a higher degree of biosorbent regeneration for future usage. This should be considered when selecting a biosorbent to increase sustainability.

Desorbing agents are typically categorized into three groups: chelating agents (EDTA, ethylenediaminetetraacetic acid), alkalis (sodium hydroxide, sodium hydrogen carbonate, potassium hydroxide, and hydrochloric acid), and acids (hydrochloric acid, sulphuric acid, nitric acid, and acetic acid) (Calero et al. 2013). In terms of speed and desorption %, acidic desorbing agents have been reported to be more effective than basic and neutral agents (Calero et al. 2013).As indicated in Table 7, Desorption efficiencies of heavy metals varied from 50.29% to as high as 99.99%, with EDTA and HCl proving to be most efficient. Reusability was in a range from 3 to 10 cycles, reflecting a high regeneration potential in various biosorbents such as cryogels and modified silicas. High desorption (>85%) was often found for Pb(II) as well as Cd(II) with potential recovery by low-concentration.

ANALYSIS OF BIOSORPTION

The biosorption performance of plants, fungi, algae, and bacteria for the removal of various heavy metals, such as As, Cd, Cr, Pb, and Hg, was studied. Biosorption exhibits good adsorption effectiveness, is economically viable, and is simple to use and operate (Torres 2020). Waste, such as agricultural residues, agro-industrial wastes, dairy waste, industrial waste, and leftover plant materials (such as peels, seeds, skin, shells, and stones) can be converted into biomass (Taneja et al. 2023). These wastes, which are viewed as

Table 7: Desorption Efficiency and Reuse Potential of Different Adsorbents in Heavy Metal Ion Recovery.

Heavy Metal	Type of absorbent	Desorbing agent	Desorption [%]	Reuse cycle	Reference(s)
Cr(VI) , Pb(II)	Groundnut husk	HCl and H_2SO_4	76.1, 82.1	5	Bayuo et al. (2020)
Pb(II)	Magnetic bentonite (M-B)	1.00 moL.L^{-1} $NaNO_3$	90.21	6	Zou et al.(2019)
Cu(II), Cd(II), Pb(II), Ni(II)	A-MIL-121	80 °C in water	90.0	10	Ji et al. (2021)
Zn (II)	Mango leaf powder	0.1 moL.L^{-1} HCl, 0.1 moL.L^{-1} HNO_3	94.7, 89.5	3	Kaushal (2023)
Cd (II)	Soil + humic acid	0.05 M EDTA	50.29	3	Zheng et al. (2022)
Cu(II)	Poly(vinyl imidazole) cryogel	0.1 M Na_2 EDTA and 1 M HCl	99.99	5	Zhong et al. (2021)
Hg(II)	Sulfhydryl-modified SiO_2 cryogel	1 M HCl	89.0	5	Zhu et al. (2021)
Cd(II) Pb(II)	MgO-SiO	0.05 M HCl	84.5, 89.9	-	Ciesielczyk, Bartczak and Jesionowski, (2016)
Pb(II)	Brown macroalga Sargassum ilicifolium	0.2 M HCl	88.0	3	Tabaraki et al. (2014)
Cd (II)	Araucaria heterophylla	2 N HCl	84.45	4	Sarada et al. (2017)
Cd (II)	Mealworm frass (MF).	0.02 M HCl	90.0	5	Kim et al. (2025)

trash, can be acquired without charge. In developing nations with limited technological advancements, biosorption techniques are highly beneficial. Another benefit of plant-based biosorbents is that they reduce the biological and/or chemical sludge (Hassan et al. 2020). In addition to providing the possibility of metal recovery for various industrial applications, several types of biosorbents have the ability to both desorb and regenerate, making their use compatible with sustainable development.

Much research has looked at the removal of one or two metals through biosorption, however, the results of these studies may not be applicable in real-world situations because contaminated water might contain a wide range of heavy metals. Additionally, organic pollutants may be present in water, which could alter the composition of metal ions and the biosorption process. One recommended step in biosorption research is to examine how lab results are applied to real contaminated water samples (Nathan et al. 2022).

For instance, it would not be easy to maximise the water pH in practical settings, despite it being the most important component. The biosorption capabilities of biosorbents measured for particular pH values in laboratory settings might not be accurate in real life because there are many other environmental factors that affect pH (Fertu et al. 2022). The intricacy and matrix structures of polluted water are typically not reflected in the tested water samples.

Furthermore, the removal of heavy metals is the primary goal of the majority of biosorption research, nevertheless, the removal of hazardous organic pollutants has not received much attention. Actually, many studies focus on removing heavy metals in their cationic state rather than their anionic form (e.g., $CrO4^{2-}$).

Early biosorbent saturation may be one of the drawbacks of biosorption processes, therefore, biosorbents need to be changed often. The recovered metal must be carefully disposed of or used when the saturated biosorbent is desorbed from the biosorbed metal. The efficiency of the recycled or repurposed biosorbent and the desorption procedure are also variable. According to the Scopus database, 16,202 publications on the subject of "biosorption" were released between 1970 and December 4, 2024. Unfortunately, companies have not yet brought biosorbents to the market (Gadd 2009). Industries often choose the use of oxidation-reduction methods, reverse osmosis, ion exchange, and chemical precipitation (Wang et al. 2021). Chemical precipitation is preferred because it is highly effective in removing a variety of heavy metals, even if it is not selective (Pohl 2020). However, this process generates a large amount of solid sludge, which is detrimental to the environment (Fei & Hu 2023).

Another challenge for the industrial use of biosorption is that existing methods, such as ion exchange and precipitation, are well-established in their processes and have proven to be suitable on a large scale. Industries may find it unsafe and unfeasible to adopt new procedures in place of established ones.

The fundamentals of the main factors affecting biosorption efficacy have been established, including the effects of different experimental settings, batch and continuous process operations, and the manner in which different kinds of biosorbents absorb metals. The next stage is to apply this economical and effective technique in a business context. Researchers should carry out further research for this reason. It is more difficult to scale up the continuous biosorption process to pilot or industrial scale due to the behavior of biosorbents in real industrial effluents.

CONCLUSION AND FUTURE PERSPECTIVES

A low-cost, sludge-free, and environmentally friendly substitute for traditional heavy metal cleanup techniques is biosorption. It works by complexation, ion exchange, and physical adsorption. Biosorbents with strong metal-binding capabilities include fungi and brown algae. However, despite extensive lab-scale research that frequently utilizes synthesis solutions, biosorption still faces critical challenges in real-world applications, such as variability in wastewater composition, biosorbent stability, and scalability.

To advance biosorption from experimental to practical application, the following research gaps and future directions must be addressed:

- **Real wastewater validation**: Evaluating the performance of biosorbents using real industrial effluents to reflect complex matrices.
- **Pilot-scale and commercial trials**: Demonstrating scalability, economic viability, and process reliability in real settings.
- **Long-term regeneration and reuse**: Evaluating the durability and reusability of biosorbents over multiple adsorption–desorption cycles.
- **Hybrid technology development**: Integrating biosorption with chemical, membrane, or advanced oxidation processes for enhanced performance.
- **Nanomaterial integration**: Improving surface characteristics, selectivity, and adsorption kinetics through nanoparticle functionalization.
- **Multi-metal and competitive ion studies**: Identifying biosorption behaviour in complex, multi-contaminant systems.

- **Standardization and regulatory frameworks**: Establishing guidelines and protocols to support biosorption as a dependable and acceptable technology.

In conclusion, biosorption has great potential as an eco-friendly and effective approach for heavy metal removal. Bridging the gap between laboratory research and field implementation requires interdisciplinary collaboration among researchers, industries, and policymakers to translate biosorption technology into sustainable environmental practice.

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Sustainable Corrosion Protection in Concrete Using Henna Coatings: An Environmentally Friendly Alternative to Zinc Coatings

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ABSTRACT

Corrosion of reinforcement bars in concrete compromises structural integrity and increases maintenance costs. This study investigates the effectiveness of organic (henna powder) and inorganic (zinc powder) corrosion inhibitor coatings in enhancing both corrosion resistance and bond strength retention of coated reinforcement bars embedded in concrete. The significance of the study lies in its approach to measuring the bond strength retention of coated reinforcement bars in a chloride-induced environment. To evaluate the corrosion mitigation and bond strength retention of the coated bars, cylindrical specimens of M20 grade of concrete were cast, having embedded coated and non-coated reinforcement bars having varying coating layers. Accelerated corrosion tests using a 3.5% NaCl solution were applied to cast specimens to simulate aggressive environmental conditions. Bond strength retention was assessed through pull-out tests in accordance with IS 2770: Part 1 (1967). Results showed that reinforcement bars with four coats of henna delayed corrosion initiation by up to 14,525 minutes (~10 days), compared to 6,132 minutes (~5 days) for uncoated bars, representing a 137% improvement in corrosion resistance at 20% corrosion levels. Zinc coatings improved corrosion resistance by up to 65% with four coats at 20% corrosion levels. In bond strength tests, uncoated samples exhibited a 42% reduction in bond strength at 20% corrosion, while henna-coated samples retained up to 90% of their original bond strength, significantly outperforming zinc-coated samples, which retained approximately 84%. The superior performance of henna coatings is attributed to the formation of a protective passive layer containing organic tannins and polyphenolic compounds such as lawsone. Unlike conventional admixture-based or epoxy-based corrosion inhibitors, which are either dispersed within the concrete matrix or applied externally to hardened surfaces, this study pioneers the direct application of henna as a coating on reinforcement bars-targeting corrosion mitigation precisely at the steel–concrete interface without compromising bond strength. These findings highlight the potential of organic inhibitors as cost-effective solutions and present a viable alternative to traditional epoxy-coating-based prevention methods for mitigating reinforcement corrosion while preserving bond strength, offering a promising approach for enhancing the durability of reinforced concrete structures. The study promotes the use of environmentally safe inhibitors to reduce the ecological footprint of reinforced concrete structures and supports the transition toward green and sustainable construction practices.

INTRODUCTION

Strength, durability and adaptability are the major factors driving reinforced cement concrete as one of the most widely used construction materials. The service life of reinforced concrete remains unhindered under ideal environmental conditions. However, in some cases, its performance is compromised due to factors such as inadequate design, poor construction practices, substandard material selection, or exposure to harsher environmental conditions than originally anticipated (Afandi et al. 2023). Among the many challenges faced by civil engineers, corrosion of reinforcement in concrete stands out as a significant concern, particularly when dealing with aging infrastructure. Addressing corrosion-related deterioration has become a crucial aspect of structural maintenance, creating a growing demand for

specialized rehabilitation techniques (Arslan et al. 2024). While this presents opportunities for experts in the field, it also poses substantial challenges for those responsible for maintaining and restoring affected structures.

The economic impact of corrosion is staggering. In the United States alone, it is estimated that corrosion-related damage to interstate highway bridges, primarily caused by deicing salts and marine exposure, amounts to approximately $150 billion. Additionally, difficulties in properly curing concrete further reduce the lifespan of reinforced concrete structures. In rapidly developing nations, cost-cutting measures in construction have led to lower-quality concrete and insufficient concrete cover over reinforcement, making structures more susceptible to carbonation-induced corrosion (Asaad et al. 2021, Bajaj & Khurpade 2024, Cai et al. 2022).

A more thought-provoking question might be: Why doesn't steel corrode in concrete more frequently? It is well known that mild steel and high-strength reinforcing bars are prone to rusting when exposed to air and moisture. Given that concrete is a porous material containing water, one might expect embedded steel to corrode easily. However, under normal conditions, this is not the case. The key reason behind steel's resistance to corrosion in concrete lies in the material's inherent alkalinity. Unlike acids, which actively corrode metals, alkaline environments often provide protection against corrosion. Concrete contains microscopic pores filled with high concentrations of soluble calcium, sodium, and potassium oxides. When mixed with water, these oxides form hydroxides, creating a highly alkaline environment with a pH ranging between 12 and 13. This alkaline condition plays a crucial role in the durability of reinforced concrete structures by influencing the composition of pore water and controlling the movement of ions and gases through the concrete matrix (Çöğürcü & Uzun 2022, Courard et al. 2014).

One of the most important protective mechanisms in reinforced concrete is the formation of a passive layer on the steel surface. This layer consists of a dense, impenetrable film, likely composed of metal oxides, hydroxides, and minerals derived from cement. Once fully developed, this passive layer significantly slows down the oxidation process, preventing corrosion. In fact, when properly maintained, this natural protective barrier can be more effective than artificial coatings like galvanization or fusion-bonded epoxy, which may degrade over time and expose the underlying steel to corrosion. However, this passivating environment is not always stable. Two primary mechanisms can disrupt it, leading to steel corrosion in concrete: carbonation and chloride attack. These processes alter the chemical balance within the concrete, weakening the protective layer and making the embedded steel vulnerable to corrosion. Understanding these degradation mechanisms is essential for developing strategies to enhance the longevity of reinforced concrete structures (Deliktas et al. 2024, Dybel & Walach 2017, El-Etre et al. 2005).

As infrastructure continues to age and new structures are built in increasingly demanding environments, addressing the issue of corrosion in reinforced concrete remains a critical priority for engineers worldwide.

Several factors have been researched that affect the corrosion of rebars embedded in concrete. The quality of concrete, environmental conditions, and specific construction practices carried out are key among these. It is the surface at the steel/concrete interface and its relationship with water content, pore structure, and components of the local environment that largely determine the onset and propagation of corrosion. In addition, the required critical chloride content for initiation of corrosion is not a fixed value and is affected by several variables, resulting in very complex predictions of corrosion rates (Fernando et al. 2023, Ye et al. 2020, Zhao & Luo 2024).

Different methods of protection for rebars have been developed and studied over the years to combat these issues. Some common methods include protective coatings and sacrificial anode or impressed current systems. The efficacy of these techniques has been measured through experimental studies that ascertain how these techniques can limit corrosion in a variety of concrete environments. For example, small-scale projects have widely used protective coatings, while for harsher environments, sacrificial anodes are suggested. In addition, corrosion inhibitors for concrete have been examined as a possible alternative way to improve the durability of reinforced structures. If applied properly, these inhibitors can have a massive effect on extending the lifespan of concrete constructions by reducing the corrosion rate to a low value. These coatings operate most effectively if the type of inhibitor used and its compatibility with the concrete mix design are taken into consideration. In determining the durability and structural integrity of reinforced concrete structures, one of the critical contributing factors is the bond strength of reinforcement bars in concrete. A few studies have also been conducted to evaluate the effect of corrosion inhibitors, carbonation, and other binder materials on bond strength (Julio et al. 2004, Suguma & Pyatina 2017, Yan et al. 2016). Traditional corrosion mitigation strategies, such as epoxy coatings or corrosion-inhibiting admixtures, face limitations such as poor bond strength retention or imprecise delivery to corrosion-prone areas. While plant-based corrosion inhibitors have been explored in various forms, their use has been mostly limited to admixture inclusion or surface application (Suguma & Pyatina 2017).

This study presents a novel strategy by applying henna powder directly onto the reinforcement bars as a protective coating. This direct-contact application is significant because it actively targets the corrosion initiation zone at the steel–concrete interface, potentially offering both improved corrosion resistance and bond retention, an area not sufficiently explored in existing literature.

For a better understanding of the utilization of corrosion inhibitors in concrete, an in-depth literature review was done, and the papers were analyzed from 2004 to 2024, as shown in Fig. 1. As per the database made from Google Scholar, the number of articles published on the use of corrosion inhibitors in concrete, year-wise wise was:

2004 – 2009: 9,543

2010 – 2014: 13,287

2015 – 2019: 16,785

2020 – to present: 21,124

As evident from these increasing numbers, it further highlights the importance of studying the effect of corrosion in concrete. The various keywords used to search the varied literature were “Corrosion Inhibitors”, “corrosion mitigation”, “corrosion”, “durability of concrete” and “inhibitors”.

The study of the various phytochemicals responsible for exhibiting corrosion inhibition properties in plant-based inhibitors when used in concrete is very important. Fig. 2 highlights the five key classes of phytochemicals, i.e., alkaloids, phenolic acids, carbohydrates, terpenoids, and lipids, which are responsible for exhibiting corrosion inhibition properties. Alkaloids such as glucosinolates and

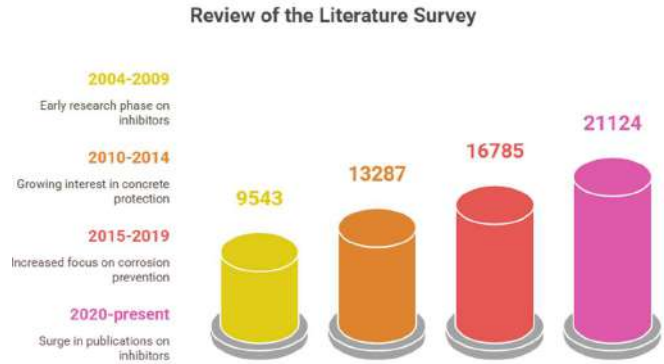

Fig. 1: Review of Literature Survey.

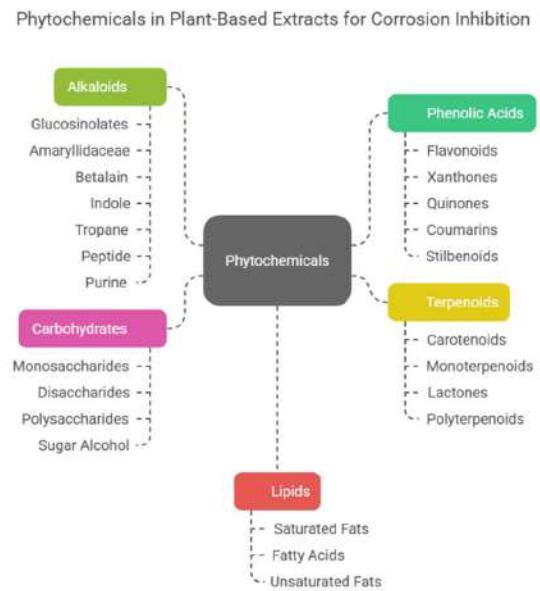

Fig. 2: Phytochemicals in Plant-Based Extracts for Corrosion Inhibition (Kadhim et al. 2021, Kumar et al. 2019, Kumar et al. 2025, Shunmuga & Ammasi 2023, Söylev & Richardson 2008).

indole are known for their bioactive properties, while phenolic acids like flavonoids and quinones are notable for their antioxidant and metal-chelating capabilities. Carbohydrates, including monosaccharides and polysaccharides, can form protective layers on metal surfaces. Terpenoids like carotenoids and monoterpenoids contribute to corrosion resistance through their hydrophobic characteristics. Lastly, lipids, including saturated and unsaturated fats, can create protective barriers that reduce moisture penetration. This comprehensive classification underscores the diverse chemical nature of plant extracts and their promising role in enhancing the durability of reinforced concrete and metal structures. Conventional corrosion inhibitors often involve synthetic or heavy metal-based compounds that pose disposal and leaching concerns. In contrast, natural inhibitors like henna, being biodegradable and non-toxic, present a viable green alternative. Their use aligns with global efforts to minimize environmental degradation in construction activities.

RESEARCH GAP AND RESEARCH SIGNIFICANCE

Although corrosion inhibitors have been widely used in previous studies, mainly as admixtures mixed into concrete or surface-applied on hardened structures, the direct application of plant-based inhibitors such as henna as a coating on reinforcement bars remains largely unexplored. Common methods include epoxy coatings, admixtures, and surface-applied treatments. While these methods improve corrosion resistance, they may compromise bond strength or incur high costs. Organic inhibitors such as plant extracts have shown potential in corrosion mitigation, but their direct application as coatings on reinforcement bars has not been explored extensively. Previous studies primarily focus on corrosion inhibitors mixed within concrete or applied on hardened concrete surfaces, but their performance as direct coatings on reinforcement bars has not been adequately investigated. Unlike epoxy coatings, which may act as a barrier to mechanical bonding, or admixtures that rely on diffusion throughout the concrete, henna, as a direct coating, combines physical barrier protection with chemical inhibition at the precise corrosion-prone interface-without negatively affecting bond strength.

This study introduces a novel approach of applying organic (henna powder) and inorganic (zinc powder) corrosion inhibitor coatings directly to reinforcement bars to simultaneously enhance corrosion resistance and preserve bond strength. The dual-benefit strategy, combined with quantitative performance data, addresses a critical gap in existing research and offers a practical solution for improving the durability of reinforced concrete structures. The application of organic (henna) and inorganic (zinc) inhibitor coatings directly on reinforcement bars represents a novel approach that addresses corrosion mitigation at its source-the steel–concrete interface. This research extends existing knowledge by demonstrating that these coatings significantly enhance corrosion resistance while also improving bond strength retention. This insight fills a research gap regarding the dual benefit of corrosion control and bond strength preservation.

Henna contains organic tannins and polyphenolic compounds such as lawsone (2-hydroxy-1,4-naphthoquinone) (Kamarska 2024, Kumar 2020). These compounds form a protective passive layer on the steel surface, which acts as a physical barrier, limiting the diffusion of chloride ions, oxygen, and moisture. These active constituents in henna have chelating properties that allow them to bond with metal ions. This forms a stable complex that reduces the reactivity of the metal surface, thus minimizing corrosion. The coatings were prepared by mixing used engine oil with the powdered inhibitors. The henna coating exhibits hydrophobic characteristics when mixed with a binder like engine oil. This property further reduces moisture ingress to the reinforcement surface, a critical factor in corrosion prevention.

Consequently, the primary objective of this study is to investigate the effectiveness of organic (henna powder) and inorganic (zinc powder) corrosion inhibitor coatings on reinforcement bars embedded in concrete (Kumar et al. 2024, Monticelli et al. 2000). The focus is to assess the ability of these coatings to mitigate corrosion in concrete using accelerated corrosion techniques, evaluate the influence of these coatings on bond strength retention under varying corrosion levels, and compare the performance of organic and inorganic coatings to identify the most effective solution for corrosion mitigation and bond strength retention in reinforced concrete structures.

MATERIALS AND METHODS

To conduct the research, two different corrosion inhibitors, i.e., organic and inorganic inhibitors, were used to provide coatings on the reinforcement bars of grade HYSD Fe 415. The organic inhibitor used in the study is Henna Powder, and the inorganic corrosion inhibitor used for the study is Zinc powder. Henna (Lawsonia inermis) used in this study is derived from plant sources and is biodegradable, non-toxic, and harvested with minimal environmental impact. This supports the circular economy and aligns with principles of green chemistry. Henna powder used in this study had a specific gravity of 0.95 and particle size between 75 to 150

microns (micro-level), ensuring a finer particle size which is required for better surface coverage and uniform application on the surface of the steel reinforcement. Henna powder was mixed with engine oil at a concentration ratio of 1:1 by weight to create a uniform paste. The pH of the Henna paste was maintained between 6.5 and 7.5 to optimize the stability of tannins and polyphenolic compounds, which are responsible for corrosion inhibition.

Before coating, the reinforcement bars were subjected to a standardized surface preparation protocol to ensure proper adhesion. The bars were first mechanically cleaned using a rotary wire brush to remove rust and other contaminants. After mechanical cleaning, the bars were degreased using acetone and wiped clean to remove any residual oils or dust to obtain a bright, clear surface. The prepared bars were then air-dried under ambient conditions for 1 h before coating was applied. The procedure followed for the surface preparation of the reinforcement bar for the application of coatings was as per IS 9077:1979. For samples with two coats, the total thickness was approximately 0.3 – 0.5 mm, while samples with four coats reached a total thickness of approximately 0.6 – 0.8 mm. The thickness was measured using a digital Vernier caliper at multiple points along the length of the bar and finally by taking the average of the readings obtained. The prepared paste was evenly applied to cleaned reinforcement bars using a brush. Each coating layer was air-dried for 24 h before applying the next layer to ensure proper adhesion. The corrosion protection mechanism of the henna coating is attributed to both physical barrier formation and chemical interaction with the steel surface. Henna paste, when applied as a coating, dries to form a dense, adherent layer on the reinforcement bar. This layer physically isolates the steel surface from aggressive agents such as chlorides, oxygen, and moisture, which are key factors in corrosion initiation.

Furthermore, henna contains bioactive compounds such as lawsone (2-hydroxy-1,4-naphthoquinone), flavonoids, and phenolic compounds. These components play a crucial role in corrosion inhibition through adsorption and film formation. The oxygen (O) and hydroxyl (–OH) groups in lawsone molecules have a strong affinity for metal surfaces. These functional groups form coordinate bonds with the iron (Fe) atoms on the steel bar. This adsorption process results in the formation of a stable, protective layer, reducing the anodic and cathodic reactions responsible for corrosion. Further, when henna is mixed with a binder agent, i.e., engine oil, it increases the hydrophobic characteristics of the paste, which repel moisture from the coated surface. This minimizes water penetration into the concrete-rebar interface, further reducing the risk of corrosion. The reinforcement sample of 16mm diameter and 500mm length was used for the study. A total of 45 reinforcement samples were used for the study and coated with 2 and 4 layers of the inhibitors. 45 reinforcement samples were divided into 5 batches, each containing 9 reinforcement samples. Batch 1 consisted of 9 samples having no coated reinforcement bars, Batch 2 contained 9 reinforcement samples coated with 2 coats of Henna powder, Batch 3 contained 9 reinforcement bars coated with 4 coats of Henna Powder, Batch 4 contained 9 reinforcement samples with 2 coats of Zinc powder, and Batch 5 contained 9 reinforcement samples of 4 coats of Zinc powder. Fig. 3 explains the coating treatments of the reinforcement samples.

Once the specimen was coated, its initial weight was recorded. Further, the test specimens to measure the bond strength of concrete were cast in accordance with IS 2770: Part 1(1967), with a single reinforcement bar embedded vertically along the central axis of the specimen. The grade of the concrete taken for the study was M20 grade mix, and its details are mentioned in Table 1. The reinforcement bar

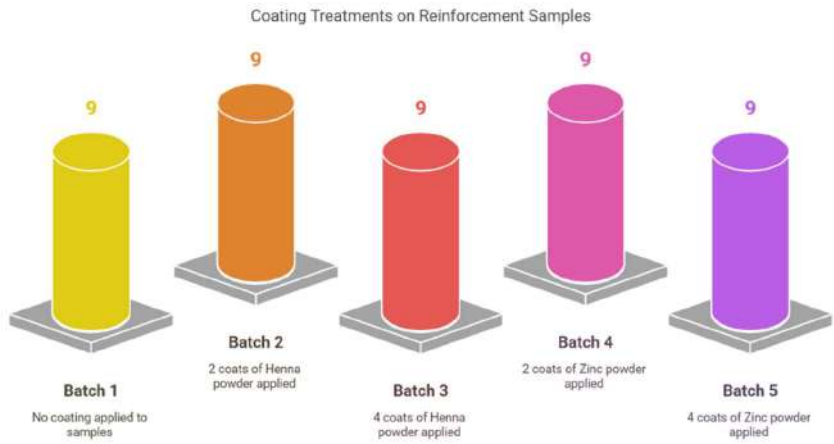

Fig. 3: Coating Treatment on Reinforcement Samples.

Table 1: Mix Proportioning.

Proportioning of Mix – M20 grade	
Grade of Concrete	M20
Proportioning	1:1.5:3
Water/Cement Ratio	0.5
Cement	320 $kg.m^{-3}$
Fine Aggregate	480 $kg.m^{-3}$
Coarse Aggregate	960 $kg.m^{-3}$

used projected down at a distance of 10mm from the bottom face and extended above, suitable for the dimensions of the cylinders. The samples were then cured for 28 days under normal conditions, maintaining a temperature of 24° ± 2° Celsius and a relative humidity of approximately 95% to ensure continuous hydration of cement. The casting is explained in Fig. 4(a), (b) and (c).

Accelerated Corrosion Setup

To simulate the corrosive environment, the cast samples were subjected to the impressed current method. The samples were exposed to direct current with the help of the embedded reinforcement bar, which, in turn, accelerated the corrosion process. To simulate the corrosive environment, the samples were immersed in a 3.5% NaCl solution and subjected to an impressed current corrosion setup in accordance with ASTM G109-21 and modified ASTM C876-15 standards. A direct current was applied using a DC power supply, with the embedded reinforcement bars acting as anodes and a reference reinforcement rod as the cathode. The current density was maintained at approximately 145 $\mu A.cm^{-2}$ of exposed steel surface, as the exposed steel surface had an approximate area of 145.6 cm^2, a value chosen to balance accelerated corrosion with realistic damage progression. The corrosion process was monitored over a maximum exposure period of 10 days, during which the current was continuously applied. The exposure time required to reach predefined corrosion levels (5%, 10%, 15%, and 20%) was recorded for each test batch and is detailed in Table 2. A reference reinforcement rod

(a)

(b)

(c)

Fig. 4: a) Corrosion Inhibitor Paste, b) Coated Reinforcement Bars, c) Casted Samples.

Fig. 5: Accelerated Corrosion Setup.

(a)

(b)

Fig. 6: a) Pull out test on sample, b) Pull out test on sample, c) Failure Pattern after Pull out test.

immersed in the solution was used as the cathode, i.e., the negative terminal, and the exposed end of the reinforcement bar in the cast samples was used as the anode, i.e., the positive terminal, as explained in Fig. 5.

Pull Out Test

After the completion of the accelerated corrosion test, the samples were then tested for bond strength through the standard pull-out test. The pull-out test on the samples cast was performed in accordance with IS 2770: Part 1(1967) as shown in Fig. 6(a) and 6(b). Fig. 6(c) shows the failure pattern observed in the sample after the pullout test was completed.

RESULTS AND DISCUSSION

Accelerated Corrosion Results

The samples subjected to the accelerated corrosion setup gave insights into how the corrosion inhibitor coatings perform in a corrosive environment. The direct current was adjusted in such a way as to observe maximum corrosion that can take place in samples for a period of 10 days. The average potential values in (-mV) were obtained from the Half-Cell Potentiometer Test as per ASTM C876-15 standards. The amount of time required to reach the various levels of corrosion is explained in Table 2:

As shown in Fig. 7, the uncoated reinforcement bars corrode the fastest, showing the lowest time taken for corrosion initiation. Bars coated with henna and zinc significantly delay corrosion, with henna being more effective in preventing corrosion as the number of coatings increases. The results clearly indicate that uncoated reinforcement bars corroded the quickest, reaching 5% corrosion in just one day and 20% by the fifth day. This highlights their low corrosion resistance when exposed to harsh conditions. Applying two coats of henna (Batch 2) significantly slowed down the corrosion process compared to the uncoated bars, while four coats of henna (Batch 3) provided even greater protection, further delaying the progression of corrosion. Zinc coatings also helped reduce corrosion, but they were not as effective as henna coatings. Among the zinc-coated samples, four coats (Batch 5) performed better than two coats (Batch 4), though the

Table 2: Corrosion measurements in casted samples.

S.No	Sample	Coatings	Time taken for corrosion [min]	No. of days (approx.)	Average Measured Potential [-mV]
5 % corrosion levels					
1	Batch 1	No Coating	1482	1	364.5
2	Batch 2	2 coats of Henna	3102	2	230.3
3	Batch 3	4 coats of Henna	5124	3.5	179.4
4	Batch 4	2 coats of Zinc	2322	1.5	290.2
5	Batch 5	4 coats of Zinc	3721	2.5	234.6
10% corrosion levels					
1	Batch 1	No Coating	2954	2	472.8
2	Batch 2	2 coats of Henna	5812	4	260.7
3	Batch 3	4 coats of Henna	7933	5.5	190.3
4	Batch 4	2 coats of Zinc	3782	2.5	310.4
5	Batch 5	4 coats of Zinc	4932	3.5	276.9
15% corrosion levels					
1	Batch 1	No Coating	4951	3.5	512.3
2	Batch 2	2 coats of Henna	7521	5.5	275.4
3	Batch 3	4 coats of Henna	9451	6.5	210.8
4	Batch 4	2 coats of Zinc	5612	4	320.4
5	Batch 5	4 coats of Zinc	7869	5.5	293.5
20% corrosion levels					
1	Batch 1	No Coating	6132	5	534.8
2	Batch 2	2 coats of Henna	10430	7	291.5
3	Batch 3	4 coats of Henna	14525	10	225.4
4	Batch 4	2 coats of Zinc	9385	6.5	340.2
5	Batch 5	4 coats of Zinc	10142	7	325.5

improvement was less pronounced compared to henna-coated bars. For instance, bars with four coats of zinc reached 20% corrosion in seven days, whereas those with two coats took 6.5 days. Overall, henna coatings proved to be more effective than zinc coatings in slowing down corrosion, especially when applied in multiple layers. This could be due to the presence of organic tannins and antioxidants in henna, which form a protective barrier that slows the electrochemical reactions responsible for corrosion. On the other hand, zinc coatings work through sacrificial protection, but their efficiency may be affected by coating thickness or adhesion issues in the given experimental setup. Increasing the number of coats enhanced corrosion resistance by strengthening the protective layer, with henna showing superior performance in this study.

Pull Out Test Results

After performing the pull-out test, the following results were obtained, showing the effect on bond strength of concrete due to the corrosion levels and the presence of corrosion inhibitor coatings on the surface of the reinforcement. The results are formulated in Table 3:

Uncoated reinforcement shows the most significant decline in bond strength, reducing from 0.331 MPa (0% corrosion) to 0.193 MPa (20% corrosion). Coated reinforcement exhibits relatively better retention of bond strength. 2 and 4 coats of Henna provide significant protection, reducing bond strength loss. At 0% corrosion, the bond strength for 4 coats of Henna is 0.330 MPa, almost equal to uncoated bars. At 20% corrosion, bond strength remains at 0.295 MPa, which is 53% higher than uncoated bars. Zinc-coated samples show better performance than uncoated bars but slightly lower than henna-coated samples. Two coats of zinc at 0% corrosion result in a bond strength of 0.324 MPa, slightly lower than two coats of henna (0.329 MPa). At 20% corrosion, four coats of zinc retain 0.272 MPa, which is 40% higher than uncoated samples, as evident from Figs. 8 and 9. Four coats of henna consistently show the highest bond strength at each corrosion level, followed

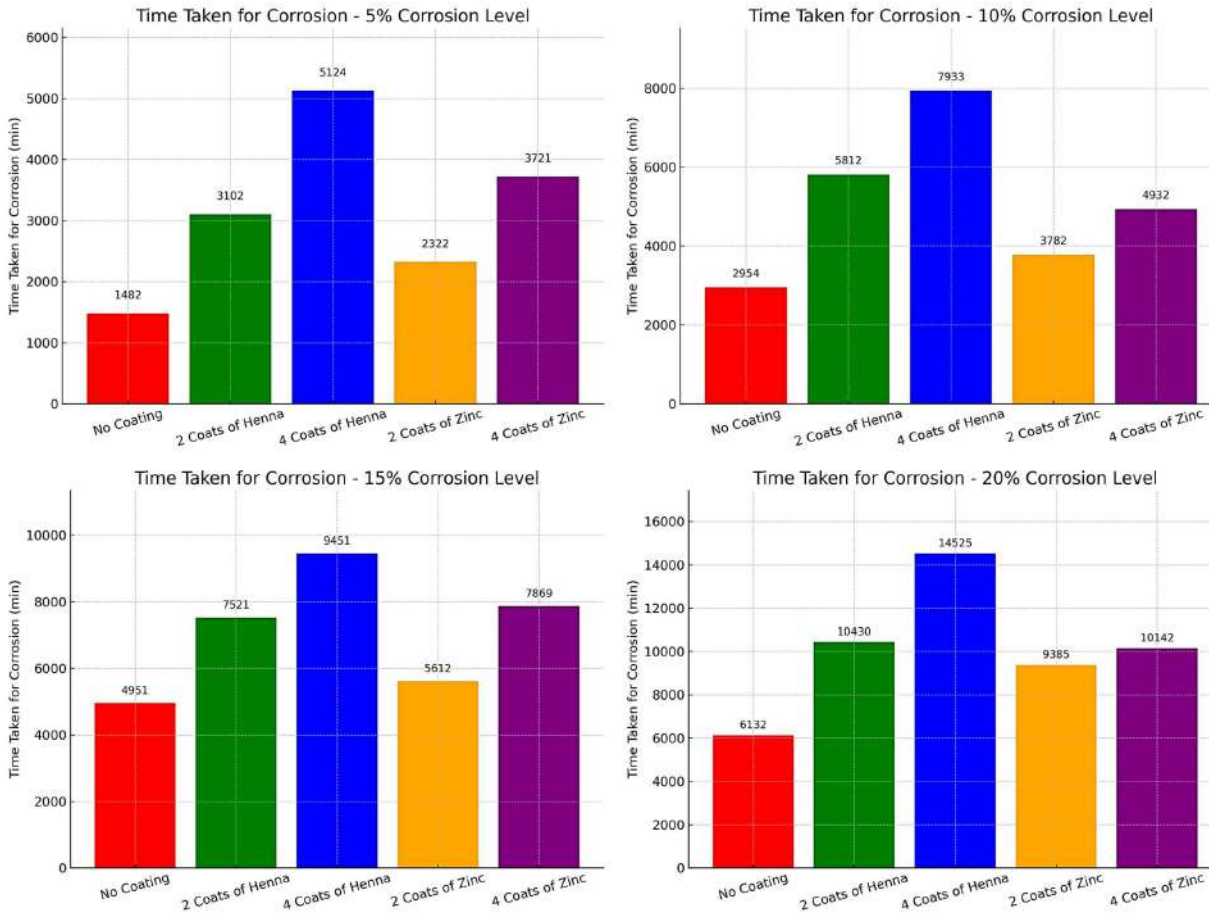

Fig. 7: Comparison of time taken to reach corrosion levels.

Table 3: Pullout Test Results.

S.No	Sample	Embedded Bar Length (mm)	Diameter of Bar (mm)	Corrosion Percentage	Coating	Average Slippage at ultimate load [mm]	Average Ultimate Load [kN]	Average Ultimate Bond Strength [MPa]
1	Batch 1	290	16	0	No Coating	6.78	48.31	0.331
2	Batch 2	290	16	0	2 coats of Henna	5.23	47.91	0.329
3	Batch 3	290	16	0	4 coats of Henna	5.04	48.12	0.330
4	Batch 4	290	16	0	2 coats of Zinc	6.12	47.21	0.324
5	Batch 5	290	16	0	4 coats of Zinc	5.89	47.11	0.323
6	Batch 1	290	16	5	No Coating	6.28	42.11	0.289
7	Batch 2	290	16	5	2 coats of Henna	5.21	46.12	0.316
8	Batch 3	290	16	5	4 coats of Henna	5.01	47.12	0.323
9	Batch 4	290	16	5	2 coats of Zinc	5.91	46.23	0.317
10	Batch 5	290	16	5	4 coats of Zinc	6.01	45.89	0.315
11	Batch 1	290	16	10	No Coating	4.13	38.12	0.262
12	Batch 2	290	16	10	2 coats of Henna	4.89	45.12	0.310
13	Batch 3	290	16	10	4 coats of Henna	4.91	47.1	0.323
14	Batch 4	290	16	10	2 coats of Zinc	4.31	43.21	0.296
15	Batch 5	290	16	10	4 coats of Zinc	4.41	44.17	0.303
16	Batch 1	290	16	15	No Coating	3.51	31.41	0.215
17	Batch 2	290	16	15	2 coats of Henna	4.65	43.12	0.296
18	Batch 3	290	16	15	4 coats of Henna	4.83	45.13	0.310
19	Batch 4	290	16	15	2 coats of Zinc	4.15	40.13	0.275
20	Batch 5	290	16	15	4 coats of Zinc	3.92	41.21	0.283
21	Batch 1	290	16	20	No Coating	3.13	28.17	0.193
22	Batch 2	290	16	20	2 coats of Henna	4.51	42.14	0.289
23	Batch 3	290	16	20	4 coats of Henna	4.78	43.05	0.295
24	Batch 4	290	16	20	2 coats of Zinc	3.81	38.12	0.262
25	Batch 5	290	16	20	4 coats of Zinc	3.51	39.61	0.272

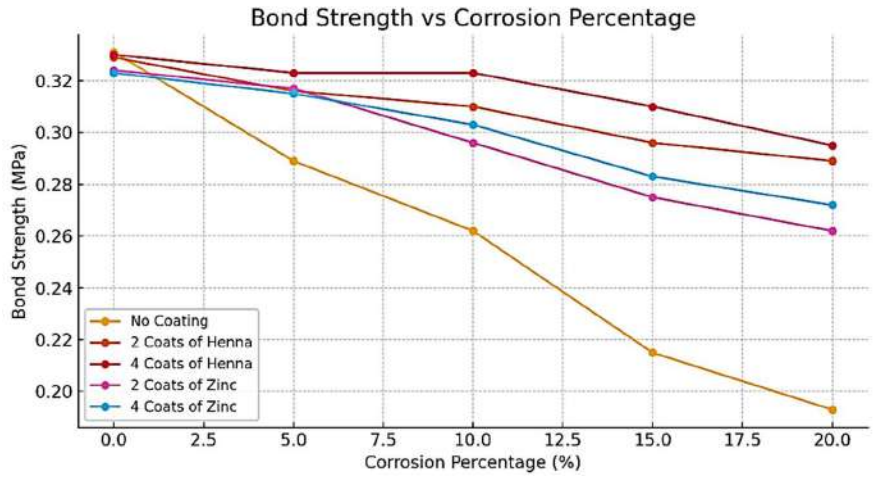

Fig. 8: Bond Strength vs. Corrosion Percentage.

by four coats of zinc. Two coats of each inhibitor perform better than uncoated bars but are less effective than four coats. Rust formation increases volume, causing internal stresses that reduce adhesion. Henna, an organic inhibitor,

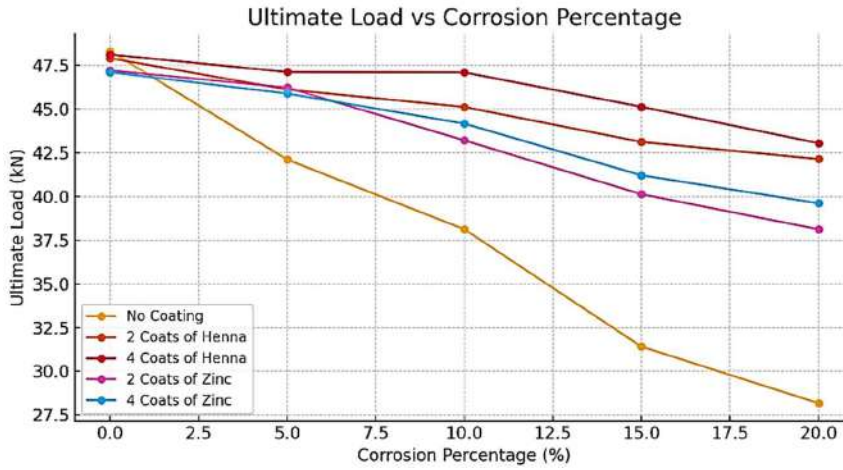

Fig. 9: Ultimate Load vs. Corrosion Percentage.

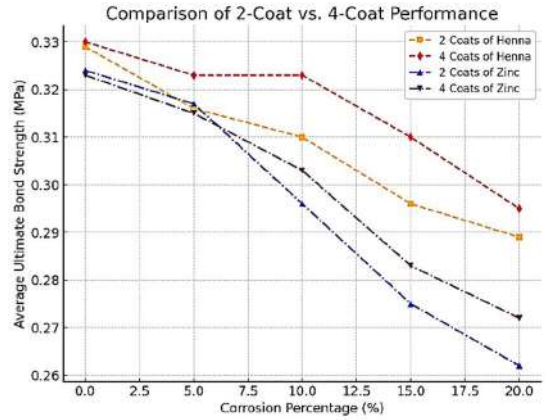

Fig. 10: 2 coat vs. 4 coat performance.

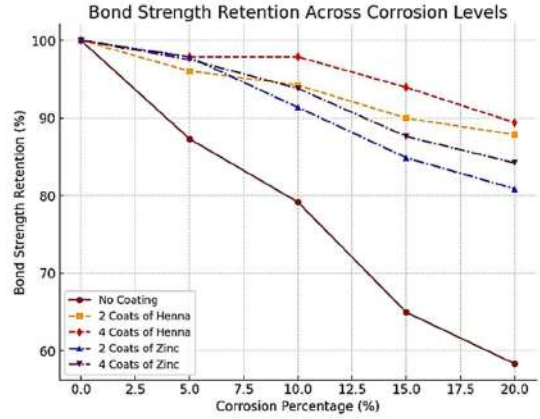

Fig. 11: Bond Strength Retention.

forms a passive layer that delays chloride and oxygen penetration. Zinc coating provides a sacrificial protective barrier, reducing oxidation of the underlying steel. Ultimate load decreases with increasing corrosion, following similar trends to bond strength. Uncoated bars exhibit the sharpest drop, from 48.31 kN (0% corrosion) to 28.17 kN (20%

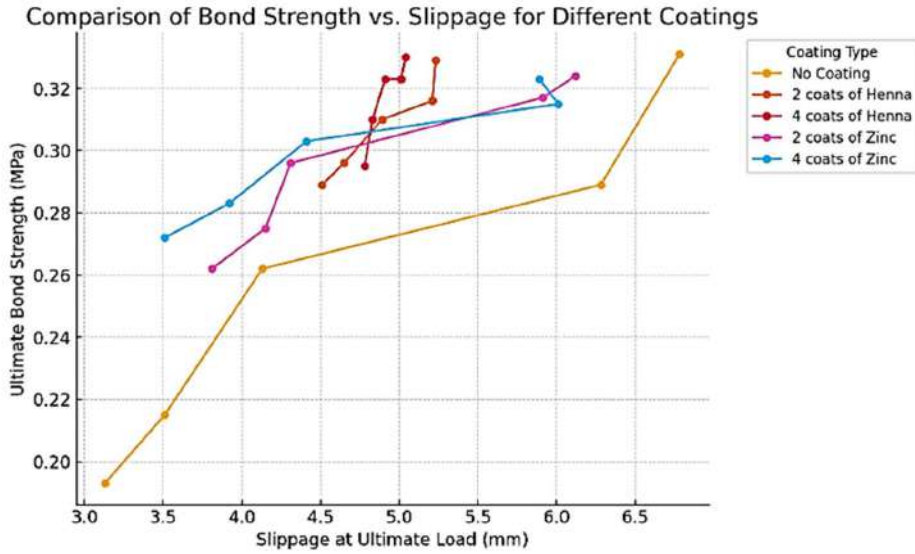

Fig. 12: Bond Strength vs. Slippage.

corrosion). Henna-coated bars maintain the highest load capacity, with four coats of henna performing best across all corrosion levels. Zinc-coated bars also show improved performance over uncoated samples, but slightly lower than henna-coated samples.

As evident from Figs. 10 and 11, henna coatings maintain higher bond strength than zinc coatings at all corrosion levels. Uncoated bars show a steep decline, retaining approximately 58% at 20% corrosion. Henna-coated bars retain the highest bond strength, with four coats of henna maintaining approximately 90% retention at 20% corrosion. Zinc coatings also improve retention, with four coats of zinc holding approximately 84% at 20% corrosion. More coating layers significantly slow degradation, reinforcing the protective benefits of both organic and inorganic inhibitors.

As evident from the above Fig. 12, bond strength decreases as slippage increases, indicating that higher slippage leads to weaker bond performance. Samples with coatings (Henna or Zinc) exhibit slightly higher bond strength than uncoated samples, likely due to better adhesion and reduced corrosion effects. As corrosion percentage increases, both bond strength and slippage decrease. This is due to corrosion-induced damage weakening the bond between concrete and reinforcement. Henna-coated bars generally show slightly higher bond strength than zinc-coated bars at similar corrosion levels, suggesting better adherence properties. The superior bond strength retention observed in henna-coated reinforcement bars is due to multiple factors. First, the surface texture of the dried henna coating-particularly when mixed with used engine oil-results in a micro-rough, slightly porous finish. This rough texture enhances mechanical interlock between the steel and surrounding concrete, unlike epoxy-based coatings, which may create a smooth barrier. Second, the polyphenolic compounds in henna, such as lawsone, are known to chemically adsorb onto steel surfaces via coordinate bonding with iron atoms. This creates a stable passive layer that not only reduces corrosion but also inhibits rust expansion, which is a major contributor to bond degradation in corroded rebars. Additionally, this passive layer limits moisture ingress and chloride diffusion, reducing micro-cracking and concrete-rebar de-bonding typically associated with corrosion. These combined effects result in enhanced long-term bond performance even at high corrosion levels. Although zinc is a well-established sacrificial coating in corrosion protection, its comparatively lower performance in this study may be due to the fact that zinc coating primarily protects through anodic dissolution (sacrificial action), but once consumed or disrupted, it may no longer prevent localized corrosion.

Cost Analysis: Henna vs Zinc Coatings

The cost analysis highlights significant differences between henna and zinc coatings in terms of material price, coverage efficiency, and environmental impact. In terms of material cost, henna powder is more economical, ranging from ₹70 to ₹250 per kg, whereas zinc coatings are comparatively expensive, costing between ₹450 to ₹1400 per kg as per various online retailers (Amazon and IndiaMart). Despite zinc's higher material cost, its coverage efficiency is superior, achieving approximately 14 m² per kg per coat, whereas henna covers around 7 m² per kg per coat. When comparing the cost per square meter for a single coat, henna proves to

Table 4: Cost Analysis: Comparative.

Parameter	Henna	Zinc
Cost	70 – 250 per kg	450 – 1400 per kg
Coverage Efficiency	$7m^2$ per kg per coat	$14m^2$ per kg per coat
Cost per m^2 (1-coat)	Rs. 10 – Rs. 36	Rs. 32 – Rs. 100
Application Process	Simple; can be applied manually	Requires specialized equipment (e.g., hot-dip galvanization)
Environmental Impact	Eco-friendly, biodegradable	Requires proper disposal procedures

be a more cost-effective option, costing approximately ₹10 to ₹36 per m², while zinc coatings range between ₹32 to ₹100 per m². From an application standpoint, henna offers a simpler, manual application process that requires minimal equipment, making it accessible for on-site applications with limited resources. In contrast, zinc coatings often demand specialized equipment, such as hot-dip galvanization setups, which adds to the overall labor and installation costs. In terms of environmental impact, henna holds a clear advantage as it is eco-friendly and biodegradable, posing minimal environmental risks. Conversely, zinc coatings require careful disposal procedures due to potential environmental concerns associated with heavy metal residues. Overall, henna offers a cost-effective, environmentally friendly, and easier-to-apply alternative, making it a promising choice for corrosion protection in certain structural applications. Combining both systems as a hybrid coating approach could provide an optimal balance between cost, performance, and environmental sustainability, as shown in Table 4.

ENVIRONMENTAL SUSTAINABILITY AND PRACTICAL IMPLICATIONS

Use of Zinc as an inhibitor coating presents significant environmental challenges, as shown in Fig. 13. The galvanization process is highly energy-intensive, operating at temperatures around 450°C and involving flux chemicals like ammonium chloride or zinc ammonium chloride, which contribute to greenhouse gas emissions and toxic waste generation (Kumar et al. 2019). Moreover, during the service life of zinc-coated steel, zinc ions can leach into the environment, especially in wet, acidic, or coastal conditions. Studies have shown that zinc runoff from galvanized surfaces can contaminate surrounding soils and aquatic systems, potentially impacting microbial activity, plant growth, and aquatic organisms (Kumar et al. 2024). Further, the accumulation of zinc in water bodies has been linked to bioaccumulation in aquatic life, with potential long-term ecological consequences. Due to these risks, many regulatory frameworks require controlled disposal or treatment of zinc waste and by-products, adding to the environmental management burden (Kumar et al. 2025). While zinc remains

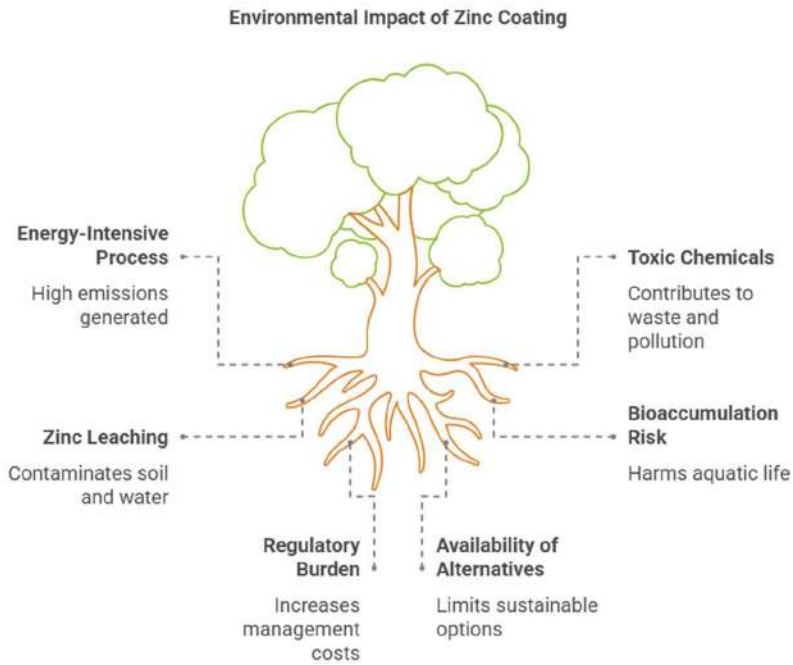

Fig. 13: Environmental Impact of Zinc Coating.

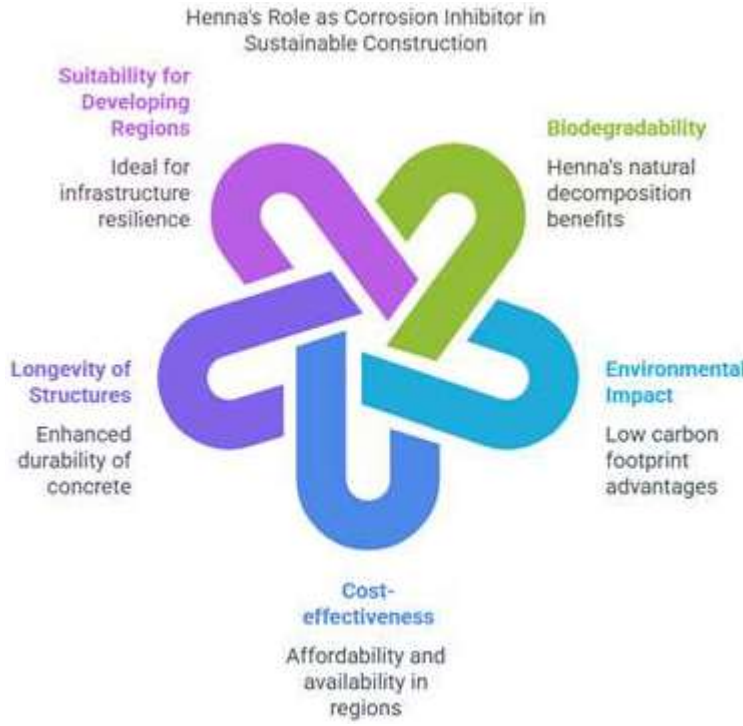

Fig. 14: Henna's role in Sustainable Construction.

an effective corrosion barrier, its environmental footprint, including energy consumption, emissions and potential ecotoxicity, limits its suitability in sustainable infrastructure development, especially when greener alternatives like biodegradable plant-based inhibitors are available.

The use of henna as a corrosion inhibitor presents a significant advancement toward sustainable construction practices, as shown in Fig. 14. Unlike synthetic inhibitors, henna is a biodegradable, plant-based material with a low environmental footprint. Its effectiveness in reducing reinforcement corrosion directly translates to a reduction in steel deterioration and replacement over the structure's lifecycle. This, in turn, minimizes the need for frequent maintenance, conserves natural resources, and reduces the energy and emissions associated with steel production and repair activities. By enhancing the longevity of reinforced concrete structures, the approach contributes to the goals of green infrastructure and sustainable urban development. Furthermore, the affordability and availability of henna make it particularly suitable for application in developing regions, where cost-effective and eco-friendly engineering solutions are essential for large-scale infrastructure resilience.

Recent studies conducted discuss the Life Cycle Assessment (LCA) methodologies on the commercially available coating methods to prevent corrosion in steel, particularly in marine environments. The two most common methods used are Alkyd-Based sol-gel Coatings and Commercial Antifouling Coatings. A study conducted in 2024 analyzed the LCA of uncoated steel versus steel coated with an alkyd-based sol-gel system in marine settings. The findings revealed a significant 46% reduction in overall environmental impact for the coated system compared to the uncoated one. However, the study also identified that components like 3-glycidyloxypropyltrimethoxysilane and n-propanol contributed notably to environmental burdens, suggesting a need for bio-based alternatives to develop greener solutions (Borgaonkar & McNamara 2025, Song & Feng 2020). Another investigation focused on two commercial antifouling coatings-one based on Copper Oxide and the other on Zinc Oxide. Through a cradle-to-grave LCA, it was determined that the production phase had the highest environmental impact, primarily due to the use of chemical compounds. Laboratory tests further indicated strong bio-toxicity and contaminant diffusion in marine environments for both coatings, highlighting the necessity to replace toxic components with more environmentally friendly alternatives (Rossini et al. 2019, Suer et al. 2022, Detty et al. 2014).

Furthermore, the use of used engine oil as a binder improved the hydrophobic characteristics and ease of application of the henna coating. It is important to recognize potential environmental and health concerns associated with its use. Used engine oil may contain residual heavy metals, combustion by-products, and other contaminants that could

pose ecological risks if not managed properly. Although the oil in this study was used solely for laboratory-scale coating and was applied in small quantities, its large-scale or open-environment application would necessitate stringent containment and disposal practices to prevent leaching or contamination of soil and water systems. Future work should consider exploring environmentally safer binder alternatives-such as natural resins or plant-based oils—that provide similar adhesion and moisture resistance while aligning better with green construction practices.

While the short-term performance of henna-based coatings in an accelerated corrosion environment has demonstrated promising results, the long-term stability of these organic coatings under real-world exposure conditions remains a critical aspect for future validation. Organic compounds like lawsone and tannins present in henna may be susceptible to degradation when subjected to prolonged moisture exposure, fluctuating pH conditions (e.g., carbonation), or elevated temperatures commonly experienced in tropical or marine climates. Such environmental variability could affect the durability and adhesion of the coating over time. Although the hydrophobicity imparted by the used engine oil binder may initially reduce moisture ingress, its performance over extended periods under cyclic wet-dry or freeze-thaw conditions is not established. Therefore, future studies should focus on evaluating the stability and protective capacity of henna coatings under varied environmental conditions.

CONCLUSIONS AND FUTURE SCOPE

Using corrosion inhibitors to delay the rate of corrosion in concrete is a well-established and efficient method. However, the importance lies in understanding the location of corrosion in concrete. Corrosion occurs at the surface of the reinforcement bars, which are embedded inside the concrete. Once these bars start to corrode, rust forms on the surface of the reinforcement bars, imparting stresses on the surrounding concrete, resulting in the formation of cracks and ultimately leading to spalling of concrete. Therefore, there is a need to use innovative methods to control corrosion on the surface of the reinforcement bars. As proven from the research conducted, the use of corrosion inhibitors coated on the surface of the reinforcement bars provides good protection against corrosion. The coatings of corrosion inhibitors delay the decay of the protective film over the surface of the reinforcement bars, which occurs due to a change in the pH of the concrete from the ingress of chlorides, sulphates, and other harmful chemicals. As evident from the above research, coatings of henna powder and zinc powder were effective in delaying the rate of corrosion by almost 100% and 40%, respectively. Four coats of henna delayed corrosion progression to the 20% level by up to 14,525 minutes (~10 days), compared to 6,132 minutes (~5 days) for uncoated samples—representing a 137% improvement in time to the critical corrosion level. This is due to the presence of organic tannins and antioxidants in henna, which form a protective barrier that slows the electrochemical reactions responsible for corrosion. However, another parameter to study was how the provision of coatings affects the bond strength of concrete. Upon investigating, it was observed that Henna coatings maintain higher bond strength than Zinc coatings at all corrosion levels, with almost 90% retention levels of the bond strength at 20% corrosion levels. Uncoated bars experienced a 42% reduction in bond strength at 20% corrosion levels, demonstrating significantly lower protection.

Further the cost analysis concluded that providing Henna coating on the steel surface was cost-effective at ₹10 – ₹36 per m^2, with a simple manual application process. This is due to the fact that the surface texture of the henna coating prepared was rough, which further aided and enhanced the bond strength of the concrete. This signifies that, rather than relying of the epoxy-based coatings method on the reinforcement surface to control the corrosion, which is a costlier method, materials like henna powder can be efficiently used as coatings on the surface of the reinforcement bars to control the corrosion along with minimal change in the bond strength of concrete, resulting in more durable and long-lasting concrete. This novel approach differentiates itself from epoxy-based and admixture-based techniques by targeting corrosion precisely at the source-on the steel surface-while maintaining bond strength. The results demonstrate that henna coatings can deliver the dual benefits of corrosion inhibition and bond preservation, offering a low-cost, environmentally sustainable alternative to conventional protective systems. An aspect that requires further investigation is the long-term durability of organic coatings. Since these coatings are organic in nature, they are susceptible to degradation over time. Exploring the sustained effectiveness of these organic inhibitor coatings in providing corrosion protection over extended periods presents a valuable area for future research. Once studied, this will further strengthen the effectiveness of organic inhibitors in mitigating corrosion, reduce dependency on industrial-based coatings for the protection of reinforcement steel in concrete, and potentially reduce the overall cost of structural repairs.

The use of natural inhibitors such as henna offers a promising pathway toward sustainable construction, combining durability enhancement with environmental responsibility. Although this study focused on the individual performance of zinc and henna coatings, combining both in

a hybrid coating system could be explored in future studies to achieve a balance between the sacrificial protection of zinc and the passive barrier action of henna. This concept is speculative at this stage and was not part of the current experimental scope.

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Cellulose-Based Materials as a Sustainable Alternative to Plastics: Mitigating Environmental Pollution Through Biodegradability and Reduced Toxicity

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ABSTRACT

This review examines the potential of using cellulose materials for overcoming environmental issues such as pollution, microplastics, and ecological toxicity as sustainable alternatives to petroleum-based plastics. Unlike plastics, which persist in the environment and break down into microplastics, cellulose materials readily degrade into non-toxic organic compounds, thus reducing pollutants in soil and water. The review outlines the relatively low environmental impact of cellulose production from renewable materials such as timber, agricultural waste, and non-timber flora. Cellulose, unlike petroleum-based polymers, is produced with lower energy inputs, greenhouse gas emissions, and greater carbon capture during plant growth. Sustainably harvested and farmed cellulose strengthens its circular economy relationship, clocking in with boastful compostability, and in some instances, recyclability. The described processes, including the manufacture of nanocellulose, chemically and mechanically treat the cellulose, improving its strength, flexibility, moisture resistance, and expanding its application in packaging, biocomposites, textiles, and medical devices. There are still some disadvantages, such as high costs, lack of industrial composting, and absence of enabling legislation. The review calls for greater advocacy of policy change, technological improvements, and public awareness campaigns aimed at promoting the use of cellulose. Most importantly, it demonstrates why these materials are needed to reduce pollution inflicted by plastics, protect biodiversity, enhance sustainability, and manage waste through responsible consumption.

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INTRODUCTION

The origin of plastics dates back to the early 20th century, when they were introduced as substitutes for natural materials like ivory, horn, and tortoiseshell (Andrady & Neal 2009, Gilbert 2017). With the invention of synthetic polymers such as Bakelite in 1907 and the development of petroleum-based plastics in the 1950s, plastics quickly became integrated into modern society due to their moldability, durability, lightweight, and cost-efficiency (Thompson et al. 2009, Geyer et al. 2017). Today, plastics are indispensable across various sectors, including packaging, electronics, textiles, automotive, and healthcare. Global plastic production has grown from 2 million tonnes in 1950 to over 390 million tonnes in recent years, underscoring its widespread acceptance and utility (PlasticsEurope 2021). Their chemical resilience and ability to meet specific functional needs at low costs have made plastics central to modern industrial and consumer applications.

However, the rapid proliferation of plastics has created far-reaching environmental, health, and socioeconomic consequences. Plastics are primarily derived from non-renewable fossil fuels and are largely non-biodegradable, persisting in ecosystems for hundreds of years (Jambeck et al. 2015). Each year, approximately 8 million metric tonnes of plastic waste enter the oceans, contributing to global environmental degradation (Lebreton et al. 2017). Approximately 79% of plastic waste is in landfills or the natural environment, while only 9% is recycled

(Geyer et al. 2017). Weathering and degradation processes fragment plastics into microplastics (<5 mm), which are now detected in marine organisms, soils, food, water, and even human tissues (Jambeck et al. 2015). Furthermore, plastics can leach toxic additives such as phthalates and bisphenol A (BPA), which are associated with endocrine disruption, developmental abnormalities, and increased risks of cancer and reproductive disorders in humans and wildlife (Godswill & Godspel 2019). In food systems, plastics compromise safety by leaching into food products, especially under high temperatures or acidic conditions (Groh et al. 2019, Obuzor & Onyedikachi 2023). Despite these well-documented risks, global responses remain inadequate. Many policy interventions are still nascent or inconsistently enforced, while sustainable alternatives have yet to achieve widespread adoption (Giacovelli 2018).

In light of these concerns, cellulose, a naturally abundant, biodegradable polymer derived from plant cell walls, has attracted renewed attention as a viable substitute for petroleum-based plastics. Sourced from wood, agricultural residues, and lignocellulosic biomass, cellulose is the most prevalent biopolymer on Earth (Klemm et al. 2005, Mujtaba et al. 2023). Unlike synthetic polymers, cellulose and its derivatives are compostable, less toxic, and derived from renewable resources (Maraveas 2020a). Technological advancements have significantly enhanced the material properties of cellulose. For instance, nanocellulose—produced via mechanical or chemical disintegration of cellulose fibres-exhibits superior strength, barrier properties, and flexibility, making it suitable for packaging, biomedical devices, and composites (Dufresne 2013, Shaghaleh et al. 2018). Additionally, specific chemical changes like acetylation have resulted in derivatives such as cellulose acetate, which improves resistance to water and durability, therefore broadening its application scope in coatings, films, and fibres (Habibi et al. 2010, Shaghaleh et al. 2018). Recent studies underscore the environmental advantages of cellulose-based alternatives, particularly in reducing microplastic generation, toxic leachates, and carbon footprints associated with plastic lifecycle emissions (Foroughi et al. 2021, Jing et al. 2024).

This review analyzes the possibilities of cellulose-containing materials as substitutes for plastics of environmental impact, characteristics of materials, technologies for processing materials, and economic aspects. It highlights current innovations, identifies the regulatory and market challenges impeding widespread adoption, and provides a comparative assessment of biodegradability, toxicity, and life cycle sustainability. The findings are relevant to stakeholders such as scientists, policymakers, business professionals, and eco-organizations targeting the shift towards sustainable materials. This review is distinctive in its holistic evaluation of cellulose-based biomaterials as potential substitutes for plastics. Beyond characterizing the materials, it assesses the recent technological developments, biodegradation, toxicity, and life cycle impacts of cellulose-based plastics. Moreover, the review examines regulatory, market, and economic obstacles that inhibit widespread adoption; these factors are often neglected by other studies. This integrative approach enhances cross-sectoral efforts to propose meaningful innovations, policies, and marketing strategies aimed at harnessing cellulose composites to alleviate plastic pollution.

ENVIRONMENTAL PERSISTENCE AND BIODEGRADABILITY

Functional Comparison of Plastics and Cellulose-Based Materials Across Industrial Applications

Plastics and cellulose-based materials serve as two distinct material classes-synthetic and fossil-derived, the other natural and bio-based-yet both have played transformative roles in industrial development. An integrated overview of their applications and functions aids in understanding the material shift currently in relation to environmental sustainability (Table 1. Due to their ease of use, cost-effectiveness and barrier properties, plastics form the bulk of the packaging industry (Wu et al. 2021). Polypropylene (PP), Polyethylene (PE), and polyethylene terephthalate (PET) are popular for food containers, films and bottles owing to their gas and moisture impermeability, which preserves the product's quality (Wu et al. 2021). However, the long-lasting durability of these materials results in waste. On the other hand, paper, nanocellulose films, and cellulose acetate are more cellulose-based materials that are increasingly being researched for use in packaging (Romão et al. 2022, Yekta et al. 2023, Ren et al. 2024). Though traditionally limited by poor moisture resistance, recent coatings and chemical modification innovations have significantly enhanced their barrier properties, making them competitive in short-lifespan applications.

Due to their strength and chemical resistance, plastic materials like PVC and polystyrene are used in piping, insulation, and fittings (Revuelta 2021). Cellulose-based boards and bio-composites are gaining traction as eco-friendly alternatives in interior cladding, insulation, and lightweight construction panels (Saini & Ledwani 2024). While not yet mainstream for heavy-duty infrastructure, modified cellulose offers advantages in biodegradability, indoor air quality, and renewable sourcing.

Plastics reduce vehicle weight, enhancing fuel efficiency. ABS, nylon, and composites are used in vehicle interiors, exterior trims, and under-hood components (Gupta & Singhal 2022). Cellulose-reinforced composites (e.g., nanocellulose-polymer blends) are emerging as substitutes in dashboards, door panels, and insulation layers (Karak 2024, Carvalho et al. 2024). Though still under development, they provide comparable mechanical strength with significantly reduced environmental footprints.

Plastics are vital in healthcare for sterile, disposable items such as syringes, IV bags, and diagnostic equipment (Greene et al. 2022). Biocompatible and biodegradable cellulose is used in wound dressings, drug delivery systems, and biosensors (Khan et al. 2024). Nanocellulose hydrogels and scaffolds are being actively researched in tissue engineering (Tamo 2024). However, challenges remain in ensuring sterility, mechanical strength, and cost-competitiveness for wide clinical deployment.

Plastics are used in greenhouses, mulch films, irrigation systems, and silage wraps (Maraveas 2020b). While effective, their disposal contributes to soil and microplastic contamination. Biodegradable cellulose-based films and coatings derived from agricultural waste are being developed to support controlled release, moisture retention, and compostability, thus offering circular economy advantages (Giordanengo 2024, Riseh 2024).

Synthetic fibres like polyester and nylon dominate textiles, offering strength and flexibility but contributing significantly to microplastic pollution. Cellulose-based alternatives, such as lyocell, modal, and viscose, are already commercialized and provide renewable, biodegradable options (Wang et al. 2021). These materials are widely used in fashion, upholstery, and hygiene products, though water and chemical use in production must be optimized.

Biodegradability of Plastics and Cellulose-Based Materials in Different Environments

Plastic and cellulose-based material degradation rates vary greatly by environment (Table 2). Traditional petroleum-derived plastics like polyethylene and polypropylene can persist in landfills for years due to their resistance to microbial activity and climatic conditions (Raddadi & Fava 2019). Due to photodegradation, UV radiation converts these plastics to microplastics without mineralizing them, slowing their breakdown in marine settings (Gewert et al. 2015). Certain plastics, including polyvinyl chloride (PVC), withstand environmental deterioration and degrade slowly in soil (Hopewell et al. 2009).

Cellulose degrades faster than plastic (Yaradoddi et al. 2020). The cellulose breaks down within weeks to months under certain conditions (Azwa et al. 2013). According to Erdal & Hakkarainen (2022), cellulose breaks down in composting in three to six months. Briassoulis et al. (2019) found that bacteria may destroy cellulose sheets in seawater after a few months. Abiotic degradation of cellulose occurs in the soil as bacteria and fungi transform cellulose into glucose, subsequently converting it into carbon dioxide and water (Datta 2024).

Table 1: Comparison of typical plastic applications and corresponding cellulose-based alternatives.

Application Sector	Typical Plastic Materials	Cellulose-Based Alternatives	Remarks
Packaging	PE, PP, PET, PS	Cellulose films, nanocellulose coatings, cellulose acetate	Improved barrier properties through functionalization/coating
Textiles	Polyester, Nylon, Acrylic	Lyocell, Modal, Viscose	Derived from regenerated cellulose, biodegradable but chemical-intensive
Healthcare	PVC, PP, PET, ABS	Cellulose hydrogels, nanocellulose scaffolds, biofilms	Suitable for wound dressings, drug delivery, sterility remains a challenge
Construction	PVC, Polystyrene, Polycarbonate	Cellulose composites, fiberboards, insulation panels	Offers a renewable alternative for interior applications
Automotive	ABS, PP, Nylon, composites	Cellulose-reinforced composites, nanocellulose-polymer hybrids	Emerging use in lightweight panels and trims
Electronics	ABS, PC, HIPS	Transparent nanocellulose films, flexible substrates	Early-stage development, potential in green electronics
Agriculture	PE films, PP containers	Biodegradable cellulose mulch films, coated cellulose packaging	Supports compostability and moisture retention
3D Printing & Bioplastics	PLA blends, ABS	Cellulose-PLA composites, cellulose acetate filaments	Enhancing printability and biodegradability in additive manufacturing
Cosmetics & Personal Care	Microbeads in gels, PE containers	Cellulose microbeads, molded pulp packaging	Replacing banned plastic microbeads, improving sustainable packaging

Enzymes and bacteria generally facilitate cellulose breakdown. Bacteria and fungi synthesize cellulases and hemicellulases that decompose cellulose polymers into smaller units such as cellobiose and glucose (Lynd et al. 2002, Datta 2024). In rich soil and compost, these bacteria break down cellulose into organic molecules to synthesize necessary organic chemicals (Datta 2024). Zeghal et al. (2021) observed that marine bacteria and fungi increase cellulose degradation. By degrading cellulose into glucose, microbes get energy. Synthetic plastics decompose slowly in microbiological breakdown processes because they resist enzymatic activity (Gewert et al. 2015).

Studies have shown that cellulose degrades differently from plastics (Puls et al. 2011, Grzybek 2024). Slezak et al. (2023) found that cellulose-based biodegradable film degraded after six months when buried in soil, whereas plastic films did not after a year. Cellulolytic marine microorganisms break down cellulose-based materials fast, whereas plastics endure for years (Briassoulis et al. 2019). Polylactic acid (PLA) takes approximately twice as long to break down in composting as cellulose acetate (Tsuji 2013).

Cellulose is more biodegradable than conventional and certain bioplastics. Polar ice and deep-sea sediments contain microplastics, according to Thompson et al. (2004). Moreover, these microplastics do not degrade; instead, they fragment, leading to enduring pollution. Consequently, plankton, fish, and humans have ingested them, potentially posing health hazards and changing food chains (Rochman 2018). Conversely, cellulose-based products decompose into organic chemicals that are environmentally benign. Cellulose particles are quickly metabolized by bacteria, reducing their long-term build-up in ecosystems. Thus, cellulose is considerably less detrimental to natural ecosystems.

Impact on Ecosystems (Terrestrial and Aquatic Organisms)

Plastics negatively impact terrestrial and aquatic ecosystems. In urban settings, terrestrial animals may consume or become ensnared in plastic waste, leading to damage or death (Gall & Thompson 2015). Plastics have been detected in the digestive systems of numerous species, including fish, birds, and mammals, within marine ecosystems (Lusher et al. 2013). The consumption of plastics might cause physical obstructions, malnutrition, and exposure to hazardous substances, resulting in population decreases (Awuchi & Awuchi 2019, Ghosh et al. 2023). Conversely,

Table 2: Biodegradability rates and environmental impact of cellulose-based vs. petroleum-based plastics.

Material Type	Environment	Degradation Time	Influencing Factors	Environmental Impact	References
Traditional Petroleum Plastics	Soil	>500 years	Low microbial activity, slow photodegradation	High emissions, significant microplastic pollution	Andrady 2011, Gewert et al. 2015
Traditional Petroleum Plastics	Marine	>500 years	UV exposure, low biodegradation	High water use, no carbon sequestration	Thompson et al. 2004, Jambeck et al. 2015
Bioplastics (PLA)	Composting Facility	3-6 months	Elevated temperature, high microbial activity	Moderate environmental impact, reduced emissions	Tsuji 2013
Cellulose-Based Materials	Soil	1-3 months	High microbial activity, moderate moisture levels	Low emissions, positive carbon sequestration during growth	Skrzypczak et al. 2023, Salimi et al. 2024
Cellulose-Based Materials	Marine	1-3 months	Microbial degradation, moderate water conditions	No microplastic pollution, moderate water use	Briassoulis et al. 2019

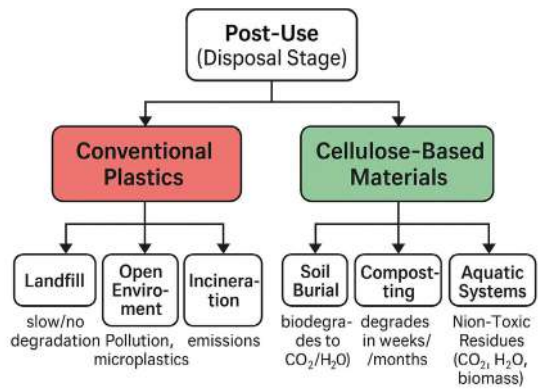

Fig. 1: Environmental fates of plastics and cellulose-based materials.

cellulose-derived compounds are biodegradable and pose negligible dangers to organisms (Erdal & Hakkarainen 2022). Upon introduction into terrestrial or marine habitats, they degrade into non-toxic organic molecules, thereby diminishing ingestion and entanglement hazards while alleviating chemical contamination. Moreover, cellulose decomposition facilitates nutrient cycling in soil ecosystems, fostering microbial proliferation and enhancing soil health (Zhan 2024).

The utilization of cellulose-based materials may substantially reduce the enduring environmental hazards associated with traditional plastics (Fig. 1). Cellulose, owing to its biodegradable characteristics, does not contribute to microplastic pollution, establishing it as a sustainable alternative for packaging, fabrics, and disposable products. Shifting from petroleum-derived plastics to cellulose-based substitutes can diminish the buildup of enduring plastic waste in the environment, mitigating its detrimental impacts on ecosystems and human health (Klemm et al. 2005).

LIFECYCLE ANALYSIS OF CELLULOSE-BASED MATERIALS

Resource Extraction and Production

Impacts of cellulose sourcing from wood, plants, and agricultural residue: Cellulose, the most abundant organic polymer, comes from wood, plants, and agricultural waste. To manufacture cellulose fiber, trees, cotton, or post-harvest agricultural byproducts are pulped to eliminate lignin and other impurities (Klemm et al. 2005). Cellulose extraction can harm the environment depending on the feedstock and methods. Sustainable Forest Management can reduce habitat loss, carbon emissions, and biodiversity loss from deforestation (Condé et al. 2022). Forest Stewardship Council (FSC) accreditation supports responsible forest management to prevent environmental deterioration from cellulose extraction from wood (Cashore et al. 2006). Instead of throwing away or burning sugarcane bagasse or straw, reusing them saves the environment. These leftovers add to greenhouse gas emissions and energy usage during transit and processing (Joshi et al. 2015). Cellulose extraction leaves less carbon footprint than petroleum-based polymers because it is ecologically benign and uses plant-based raw materials that store carbon during growth (Rajendran et al. 2025).

Compared to fossil-based plastics, cellulose extraction has a substantially lower carbon footprint (Foroughi et al. 2021). Producing 1 kg of bleached kraft pulp emits approximately 0.3–0.6 kg CO_2-eq, while polyethylene and polypropylene production emit 1.8–2.5 kg CO_2-eq per kg (Kim et al. 2022). Cellulose-based materials can also sequester 1.75–12.3 tons CO_2 per hectare per year through carbon storage during plant growth (Kim et al. 2022, Forfora et al. 2024).

Energy, water, and emissions comparison with petroleum-based plastics: Refining crude oil and natural gas produces polyethylene and polypropylene. Due to their energy usage, these activities increase greenhouse gas emissions and global climate change (Hopewell et al. 2009**).** Making plastic polymers from crude oil requires a lot of water and energy. Virgin polyethylene production consumes 80–90 MJ/kg primary energy, far exceeding the 30–50 MJ/kg required for cellulose-based products (Schirmeister & Mülhaupt 2022, Ritzen et al. 2024). However, cellulose manufacturing consumes less energy and emits fewer greenhouse gases. According to Van Schoubroeck et al. (2018), cellulose and other bio-based products have less impact on the environment in terms of non-renewable energy usage and global warming. However, processing cellulose in big pulping facilities may be energy and water-intensive. Kraft pulping can consume 30–60 m^3 of water per tonne of pulp, generating effluents that, if poorly treated, cause water pollution (Badar & Farooqi 2012, Latha et al. 2018).

Use Phase and Performance Comparison

Cellulose-Based Products: durability, performance, and usability: Cellulose is used in construction, biomedical devices, textiles, and packaging (Felgueiras et al. 2021). Cellulose products are useful and frequently endure a long time; however, petroleum-based polymers may perform better (Wang et al. 2021). Cellulose-based packaging materials lack polyethylene in water resistance and tensile strength (Wang et al. 2021). Aziz et al. (2022) suggest synthesizing cellulose acetate or adding nanocellulose to make cellulose tougher, more flexible, and long-lasting than plastics. Cellulose acetate films exhibit tensile strengths of 60–1570 MPa, comparable to around 20 MPa in low-density polyethylene (Wang et al. 2021). Thereby, making cellulose-based materials to outperform petroleum-based ones. Consumer products and food packaging benefit from these lightweight, biodegradable, phthalate- and BPA-free plastic alternatives (Piergiovanni & Limbo 2016). Cellulose fabrics like rayon and lyocell are more breathable, absorbent, and comfortable than polyester or nylon (Ahmed & Akhtar 2017, Salleh et al. 2021). The Lyocell biodegrades within 6–8 weeks in soil, whereas polyester persists for decades or longer (Wang et al. 2021).

Packaging, textiles, and industrial uses compared to traditional plastics: In recent years, cellulose-based polymers have emerged as promising plastic substitutes for single-use goods, including bags, wraps, and containers.

Plastics protect against gases and moisture better than cellulose, although enhancing its water resistance and barrier qualities is difficult (Siró & Plackett 2010). Cellulose-based fibres like lyocell and viscose are replacing synthetics in textiles. Nanocellulose coatings can achieve oxygen permeability rates as low as 0.0006 to 0.009 $cm^3 \cdot \mu m/(m^2 \cdot day \cdot kPa)$ under dry conditions, which is significantly lower than that of conventional PET films, typically ranging from 1 to 3 $cm^3 \cdot \mu m/(m^2 \cdot day \cdot kPa)$. This superior barrier performance makes nanocellulose an attractive alternative for packaging applications requiring high oxygen resistance (Nair et al. 2014). In textiles, cellulose fibres achieve 90% biodegradation under aerobic conditions, compared to <5% for polyester and nylon (Egan & Salmon 2022). These materials offer higher moisture absorption, breathability, and biodegradability (Ahmed & Akhtar 2017). Bio-composites and the building sector are using cellulose-based goods more because they can replace petroleum-based materials like plastics and fibreglass. In the construction, aerospace, and automotive industries, cellulose nanofibers can strengthen and lighten bio-composites (Dufresne 2013, Hasan et al. 2020). Adding 5-10 wt% nanocellulose can enhance the tensile modulus of composites by 30-60%, while reducing weight, making cellulose-based composites attractive for automotive, aerospace, and construction applications (Hasan et al. 2020).

End-of-life Scenarios

Compostability, recyclability, and biodegradation pathways: Cellulose-based polymers have considerable benefits compared to petroleum-based plastics in terms of end-of-life outcomes (Fig. 2). Cellulose is naturally biodegradable, breaking down into organic molecules via microbial and enzymatic activities in natural settings, leaving no hazardous leftovers (Bisaria & Ghose 1981). Conversely, plastics can endure for decades, breaking down into microplastics that exacerbate environmental pollution (Thompson et al. 2009). The compostability of cellulose-based materials is a significant advantage. Numerous cellulose products, including paper-based packaging and cellulose films, are appropriate for composting in both industrial and domestic environments, decomposing into water, carbon dioxide, and biomass within months (Song et al. 2009). Cellulose-based packaging achieves approximately 90% mass loss within 180 days under simulated home composting conditions. Under industrial composting conditions, it meets EN 13432 standards by achieving >90% biodegradation within 12 weeks (Song et al. 2009). Cellulose acetate, a chemically modified cellulose derivative, exhibits significantly reduced biodegradability, especially when the degree of substitution is ≥2.5 (Yadav & Hakkarainen 2021). To degrade, chemically modified cellulose products like cellulose acetate may need high temperatures and microbial activity (Tsuji 2013). The recycling of cellulose-based materials is achievable yet complex. Although paper and cardboard are widely recycled, other cellulose products, like textiles and composites, necessitate specialized recycling methods due to chemical additives and heterogeneous ingredients. Paper and cardboard recycling achieves approximately 70–90% fibre recovery over 4 to 6 recycling cycles, depending on the process conditions and fibre quality (Belle et al. 2024). Improving the recyclability of cellulose-based products is a vital research domain that underpins the feasibility of a circular economy.

Evaluating circular economy potential: Reuse, recovery, and recycling efficiency: The circular economy concept seeks to reduce waste by optimising material use via reuse, recycling, and recovery (MacArthur 2013). The biodegradability, recyclability, and renewability of cellulose-based materials render them very attractive in this context compared to plastic materials (Table 3). Cellulose elements in paper and cardboard can be recycled multiple times before becoming unusable, after which they can be composted or converted into energy (Thompson et al. 2009). Compared to virgin pulp production, paper and cardboard recycling reduces energy consumption by up to 60%, making it a more energy-efficient and environmentally sustainable option (Belle et al. 2024).

The extraction of nutrients from cellulose-based materials

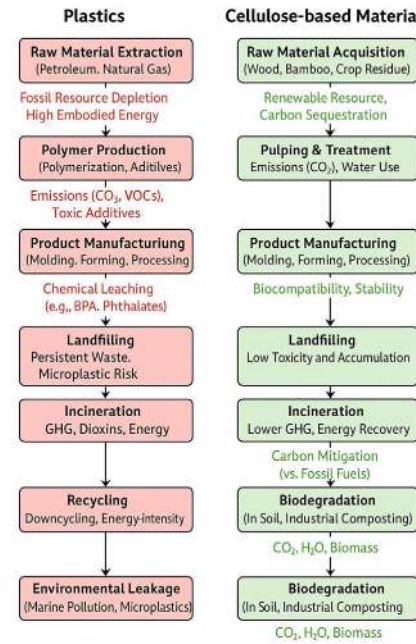

Fig. 2: Life cycle assessment of plastics and cellulose-based materials.

through industrial composting or anaerobic digestion can enhance regenerative farming methods (Tjeerdsma & Militz 2005). Furthermore, improvements in cellulose chemistry, particularly the synthesis of nanocellulose, facilitate the creation of high-performance products from recycled cellulose, thus diminishing dependence on virgin raw materials and prolonging product lifecycles (Dufresne 2017). Nanocellulose produced from recycled paper retains over 80% of the tensile strength and modulus compared to that from virgin pulp, supporting its suitability for high-value reuse in circular material applications (Kargupta et al. 2023).

TOXICITY AND MICROPLASTICS REDUCTION

Comparative Toxicology of Plastics and Cellulose-Based Materials

Chemical additives frequently utilised in plastics, particularly those sourced from petroleum, can improve attributes such as flexibility, durability, and transparency. Bisphenol A (BPA) and phthalates, commonly found in polycarbonate plastics and polyvinyl chloride (PVC), present considerable issues. BPA acts as an endocrine disruptor, imitating oestrogen and disrupting hormonal activities in humans and wildlife, resulting in reproductive, developmental, and metabolic issues (Rubin 2011). Chemicals used to make plastic flexible, phthalates, can cause fertility problems, developmental disorders, and cancer (Meeker et al. 2009). Leachate studies show that plastics can release a wide range of hazardous compounds. For example, polycarbonate plastics can leach bisphenol A (BPA) in concentrations ranging from 0.036 to 1.1 mg/L under various conditions, while flexible PVC may release phthalates at levels up to 40% by weight, particularly during use involving contact with food or biological tissues (Hahladakis et al. 2023). Plastic leachates are released from various polymers such as PVC, PE, PP, PS, and PET through weathering, UV exposure, and microbial degradation (Omidoyin & Jho 2024). In marine environments, PBDE concentrations in plastics ranged from 0.3 to 9900 ng/g, while HBCD levels in expanded polystyrene fragments have been recorded as high as 14,500 µg/g (Hirai et al. 2011, Taniguchi et al. 2016, Jang et al. 2017). In freshwater systems like Taihu Lake, leachates include BPA (28–560 ng/L), nonylphenols

Table 3: Mechanical properties, applications, and challenges of cellulose-based and petroleum-based plastics.

Material	Tensile Strength	Water Resistance	Applications	Challenges	Proposed Solution	References
Cellulose Acetate	Moderate	Moderate	Packaging, film applications	Limited water resistance	Development of hydrophobic coatings	Moon et al. 2011, Klemm et al. 2005
Nanocellulose	High	Low	Bio-composites, medical devices	High production cost, scalability issues	Advancements in enzymatic processes, nanotechnology	Dufresne 2017
PLA (Bioplastic)	Moderate	High	Food packaging, medical use	Costly production, limited recyclability	Expansion of recycling infrastructure	Tsuji 2013
Polyethylene (Petroleum)	Very High	Very High	Industrial and consumer plastics	High microplastic pollution	None (non-biodegradable)	Siró & Plackett 2010
Cellulose Films	Low to Moderate	Low	Packaging, textiles	Low consumer awareness, high perceived cost	Public awareness campaigns on biodegradability	Verma et al. 2024
Polyvinyl Alcohol (PVA)	Low	Moderate	Food packaging, medical applications	Solubility in water limits applications	Blending with other biopolymers for strength	Sirviö et al. 2020
Polyhydroxyalkanoates (PHA)	High	Moderate	Biodegradable plastics, agricultural films	High production cost	Process optimization, policy support	Gewert et al. 2015
Starch-Based Bioplastics	Low	Low to Moderate	Single-use items, compostable bags	Weak mechanical properties	Blending with nanocellulose for strength	Garavito et al. 2024
Cellophane	Moderate	Low	Packaging, textile industry	Low durability in humid conditions	Surface modification for improved performance	Moon et al. 2011
Polycaprolactone (PCL)	High	High	Biomedical, packaging, compostable items	Limited scalability, high production costs	Scaling production, blending with other polymers	Dufresne 2017

(262–1443 ng/L), and OPFRs (~1097 ng/L) (Lu et al. 2011, Xiao-Ju et al. 2012, Yan et al. 2017). Soil contamination is also significant, with approximately 3905 tons of PAEs accumulated in Chinese soils from 1958 to 2019 (Bi et al. 2021) and up to 72% of DEHP from plastic products ending up in the soil (Wang et al. 2017). These data underscore the widespread occurrence of plastic leachate and its ecological significance across multiple environmental compartments. Conversely, cellulose-based products typically do not include these hazardous chemicals, as they are derived from natural, plant-based sources. Certain chemically modified cellulose derivatives, including cellulose acetate, may include additives; nonetheless, these are generally less toxic and pose lesser risks than synthetic plastics (Moon et al. 2011). Cellulose acetate films leached bisphenol A (BPA) at around 0.5 µg/L under standard use conditions. The leaching rate was significantly lower compared to conventional synthetic polymers. This level highlights the safer environmental profile of cellulose acetate in applications involving water contact (Islam et al. 2023). Cellulose derivatives employed in food packaging, textiles, and biomedical applications frequently obtain safety certifications from regulatory agencies such as the U.S. Food and Drug Administration (FDA) and the European Food Safety Authority (EFSA) (Klemm et al. 2005). Moreover, the biodegradability of cellulose materials mitigates accumulation in ecosystems, thereby diminishing long-term toxicological effects.

Microplastics are already pervasive in the environment and present considerable threats to both human and animal health. Plankton, fish, birds, and mammals in aquatic and terrestrial ecosystems eat microplastics. Microplastics can induce physical obstructions, reduced food consumption, and malnutrition in marine animals (Wright et al. 2013). Additionally, microplastics can absorb persistent organic pollutants (POPs), including PCBs and PAHs, from the environment. Microplastics release monomers and additives such as bisphenol A (BPA) and phthalates, which are known to leach from plastic matrices and disrupt endocrine function even at very low concentrations (Prata et al. 2020). These leachates induce oxidative stress, evidenced by increased reactive oxygen species and lipid peroxidation in zebrafish and mice exposed to polystyrene particles (Lu et al. 2016, Deng et al. 2017). Exposure to 5–20 µm PS particles in mice led to significant accumulation in the liver and kidney, causing metabolic disruption and oxidative damage (Deng et al. 2017). Immunotoxic effects include suppressed dendritic cell activation and cytokine imbalances, while mussels exposed to microplastics showed altered immune gene expression and reduced immune competence (Saravia et al. 2014). Additionally, endocrine-disrupting compounds leached from plastics have been linked to hormonal imbalances and reproductive effects at low doses, highlighting the risk of chronic exposure through ingestion of up to 52,000 microplastic particles per person annually (Cox et al. 2019). Once in the food chain, these contaminants bioaccumulate and biomagnify (Rochman 2018). More individuals are afraid of eating microplastics from seafood, water, and air. Microplastics may cause oxidative stress, immune system disruption, inflammation, and other health issues, although researchers are still studying them (Prata et al. 2020). Microplastics can emit harmful chemicals like bisphenol A (BPA) and phthalates, and harm the ecosystem. Cellulose-based products do not degrade into hazardous microplastics. Since microorganisms and enzymes break down cellulose, eating glucose and other naturally occurring components is harmless. This feature considerably reduces bioaccumulation and health concerns (Klemm et al. 2005).

The Role of Cellulose-Based Materials in Mitigating Microplastics in Ecosystems

Cellulose-based polymers reduce microplastic contamination better than petroleum-based plastics. Cellulose, a natural polymer, biodegrades in soil, rivers, and seas without leaving toxic residues. However, synthetic polymer breakdown microplastics can endure generations (Andrady 2011). Switching to cellulose-based cutlery, packaging, and straws can reduce coastal microplastic pollution. Biodegradable and compostable cellulose-derived products reduce microplastic pollution in aquatic habitats. Marine microbes may easily break down these toxins into non-toxic organic molecules, as reported by Song et al. (2009).

Multiple studies have proven the prevalence of microplastics in aquatic ecosystems. Every year, 4.8–12.7 million metric tonnes of plastic debris enter the world's waters, contributing to microplastics (Jambeck et al. 2015). The Great Pacific Garbage Patch has dangerous quantities that are harming marine life (Eriksen et al. 2014). Briassoulis et al. (2019) compared cellulose-based film degradation in terrestrial and marine ecosystems to plastics. The study found that plastic films decay slowly, whereas cellulose films break down entirely within months. This suggests that employing cellulose polymers instead of petroleum plastics might dramatically shorten microplastics' ecosystem time. A second Mediterranean Sea case study examined cellulose-based marine debris reduction alternatives. Biodegradable cellulose-based fishing gear and packaging may reduce plastic pollution by 30% over ten years if waste management is successful (Bergmann et al. 2015). These findings show that cellulose-based products not only prevent microplastic generation but also reduce exposure to toxic leachates and endocrine disruptors associated with conventional plastics.

As such, cellulose-based alternatives contribute both to ecosystem health and to reduced chemical risks in the food chain.

REGULATORY AND ECONOMIC CONSIDERATIONS

Overview of International and National Regulations on Single-Use Plastics and Bioplastics

In recent years, some nations have established regulations to prevent plastic pollution, particularly single-use plastics. The EU's Single-Use Plastics Directive (2019) bans straws, cutlery, and polystyrene food containers. This law requires plastic bottles to be made from recycled material by 2025 and sets collection and recycling targets. As worries about plastic pollution in landfills and waterways have increased, Kenya, Canada, and India have banned or severely restricted single-use plastics. The 2019 Basel Convention modifications ban the transboundary movement of plastic waste; thus, governments must acquire authorisation before exporting non-recyclable plastic waste (Ahmed 2019). These restrictions aim to halt the flow of plastic waste from affluent to poor nations, where a lack of waste treatment infrastructure degrades the environment. Bioplastics and other eco-friendly materials are being promoted in numerous countries to decrease plastic waste. The EU's Circular Economy Action Plan (2020) aims to increase biodegradable material consumption and improve the bio-based product market. National governments, including the US and Japan, have provided financial incentives and research initiatives to create

Table 4: Global regulations promoting biodegradable alternatives.

Year	Country/Region	Regulation/Initiative	Policy/Action	Reference
2002	Bangladesh	Plastic Bag Ban	First country to ban thin plastic bags due to drain blockage and flooding.	(UNEP 2018)
2015	USA	Microbead-Free Waters Act	Banning plastic microbeads in rinse-off cosmetics to prevent water contamination.	(NOAA 2015)
2016	Germany	Plastic Bag Tax	Introduced a voluntary agreement leading to ~60% drop in bag usage.	(DW 2016)
2017	Kenya	Plastic Bag Ban	Strictest penalties globally, including fines and jail terms for production/use.	(Griffin and Karasik 2022)
2018	Spain	Plastic Bag Charge	Mandatory charge for lightweight plastic bags, exemptions for very lightweight bags.	(European Commission 2019)
2019	European Union	Single-Use Plastics Directive	Ban on cutlery, straws, plates, targets for recycling and collection.	(European Commission 2019)
2020	France	Ban on Plastic Cutlery & Plates	Part of a wider anti-waste law banning disposable plastic items.	(Frontier Group 2023)
2021	China	Phased Plastic Ban	Gradual restrictions on plastic bags, straws, and delivery packaging.	(Library of Congress 2021)
2022	Colombia	Single-Use Plastics Prohibition	Law banning 14 single-use plastic products from 2022 to 2030.	(ADBioplastics 2024)
2022	India	Single-Use Plastic Ban	Ban on manufacture, import, stocking, distribution, sale and use of listed plastic items.	(MoEFCC 2023)
2023	Canada	Single-Use Plastics Ban	Prohibits the sale and manufacture of plastic checkout bags, cutlery, straws, and other items.	(Government of Canada 2023)
2025	European Union	Packaging and Packaging Waste Regulation (PPWR)	Requires all packaging to be recyclable or reusable by 2030, mandates compostability criteria.	(European Commission 2025)
2023	France	Plastic Bottle Sales Reduction Target	Goal to cut single-use plastic bottle sales by 50% by 2030.	(Le Monde 2023)
2021	New Zealand	Single-Use Plastics Ban	Banned single-use plastic items such as cotton buds, drink stirrers, and fruit labels.	(Crux 2021)
2022	Nigeria	Single-Use Plastics Ban	Scheduled to ban single-use plastics, including plastic bags and straws, to combat environmental pollution.	(Sustainable Plastics 2024)
2024	United Arab Emirates	Single-Use Plastics Ban	Plans to ban single-use plastic food containers and cutlery to reduce plastic waste. The country aims to achieve zero plastic waste by 2030 as part of its larger environmental vision.	(UAE Stories 2024)

bioplastics and cellulose-based alternatives to conventional plastics (OECD 2014). Bioplastics are growing in popularity, yet cellulose-based products are still behind petroleum-based polymers. Government subsidies, tax cuts, and research grants are needed to promote cellulose-based alternatives. The European Bio-based Industries Joint Undertaking funds research and development of bio-based materials, primarily cellulose-derived polymers (Bio-based Industries Joint Undertaking 2022). Additionally, certain nations' public procurement regulations encourage biodegradable and compostable materials in public services and packaging, which expands these markets. The USDA BioPreferred Program in North America encourages bio-based goods, notably cellulose-based ones. This campaign encourages bio-based purchases and use through government procurement laws. After many public governments banned single-use plastics and required biodegradable or compostable alternatives, cellulose-based goods are becoming the standard. Table 4 outlines a chronological progression of key global regulatory actions targeting plastic pollution by promoting biodegradable alternatives.

Many developing nations face significant regulatory and implementation challenges. The global initiative to mitigate plastic pollution is affected by weak enforcement of bans on plastic materials, insufficient waste collection infrastructure, and ambiguous regulations concerning biodegradable plastics (Islam et al 2024). Capacities for enforcement and compliance monitoring do not exist in most countries that officially restrict single-use plastics (Islam et al. 2024); hence, these countries continue to consume plastic. Developing regions face an additional challenge of limited administrative capacity paired with fragmented governance structures. Inadequate infrastructure for managing waste stagnates the processes involved in properly collecting and sorting plastic waste needed for recycling. Investment-related incentives are dampened due to logistical challenges and market uncertainty facing recycling systems (OECD 2018, Gerassimidou et al. 2022). Furthermore, undefined and unenforceable rules concerning biodegradable plastics allow for the continued existence of harmful products meant to be regulated in nature without being entirely removed from ecosystems. Policy changes, infrastructure development alongside coordinated multilateral agreements, and defined international guidelines would help fill the noted gaps (Islam et al. 2024).

Economic Viability of Cellulose-Based Materials

Cellulose-based polymers are uneconomical because they cost more to make than petroleum-based plastics. Costs depend on the delicate procedures involved in collecting and processing cellulose from premium sources like wood pulp or agricultural byproducts (Shen et al. 2010). However, economies of scale, well-established production techniques, and decreased raw material costs due to natural gas and crude oil abundance favour traditional plastics. Cellulose-based products must be mass-produced to be profitable. Improved production should lower per-unit costs. Dufresne (2017) suggests that nanocellulose production might save costs by improving extraction and refining efficiency. Moreover, escalating petroleum prices and heightened regulatory restrictions on plastic use may improve the economic viability of cellulose-based alternatives, particularly as the external environmental and social costs of plastic pollution are increasingly incorporated into market pricing.

Nonetheless, augmenting cellulose manufacturing entails numerous technical and economic obstacles. Industrial-scale operations necessitate significant investments in research and infrastructure, especially for sophisticated processing methods such as enzymatic hydrolysis and nanocellulose extraction (Klemm et al. 2005). Furthermore, the shift from small-scale niche production to mass manufacture must tackle technological challenges with product uniformity, performance, and integration with current manufacturing processes. For developing nations, economic barriers are particularly acute. High capital costs, limited technical expertise, and inadequate financial incentives constrain local production of cellulose-based materials.

Market adoption is also contingent upon consumer acceptance and willingness to invest in sustainable options. There is a growing consumer preference for environmentally friendly products in select markets, which could potentially boost demand for cellulose-based materials. Nonetheless, broad adoption will require further cost reductions and improvements in product performance to ensure competitiveness with the durability and functionality of conventional plastics in applications such as packaging and consumer goods (Siró & Plackett 2010).

Barriers to Widespread Implementation

Several technological and economic challenges impede the widespread adoption of cellulose-based materials (Li et al. 2024, Wang et al. 2024). Enhancing the properties of cellulose-derived products-such as moisture resistance, flexibility, and strength-requires continuous innovation (Zhao et al. 2021). For instance, cellulose films and packaging often lack the barrier properties inherent in petroleum-based plastics, rendering them less suitable for specific food packaging applications (Wang et al. 2018). Furthermore, modifying cellulose to improve its properties, such as conversion to cellulose acetate or nanocellulose, can increase costs, undermining its competitive edge. Economic challenges include the substantial initial

investment required for new manufacturing processes and the limited availability of sustainably sourced raw materials on a large scale. While cellulose is abundant, sustainable sourcing and industrial-scale processing can incur significant expenses (Wang et al. 2024). Additionally, cellulose-based materials must compete with established petroleum-based plastics, which benefit from extensive infrastructure investments and economies of scale.

Consumer behaviour also poses a challenge, as many consumers are unaware of the environmental benefits of cellulose-based alternatives or may hesitate to switch due to concerns about performance and cost (Filho et al. 2022). Addressing these behavioural barriers necessitates educational initiatives and improvements in the affordability and functionality of cellulose-based products (Filho et al. 2022). A critical barrier to the widespread adoption of cellulose-based materials is the insufficient infrastructure for recycling and composting (Parveen et al. 2024). While cellulose is biodegradable and compostable, numerous chemically modified or composite cellulose products necessitate certain conditions for efficient composting or recycling. Cellulose acetate, frequently utilized in packaging, often fails to disintegrate in home composting settings and generally requires industrial composting facilities for thorough degradation (Song et al. 2009). The current infrastructure, which is predominantly designed for conventional plastics, limits the recycling capability for cellulose-based products (Parveen et al. 2024). Enhancing this recycling infrastructure to include biodegradable and cellulose-based items will necessitate substantial expenditures in sorting technologies, industrial composting facilities, and consumer education. In developing nations, these infrastructure deficits are even more pronounced. Most waste management systems focus on landfill and informal recycling, with minimal capacity for industrial composting or specialised cellulose recycling. Unless these infrastructural inadequacies are rectified, the prospective environmental advantages of cellulose-based materials may remain unfulfilled.

FUTURE DIRECTIONS AND INNOVATION OPPORTUNITIES

Technological Innovations

Recent advances have made cellulose-based polymers a viable alternative to plastics. Researchers have transformed cellulose's nanoscale structure to boost its mechanical strength and flexibility (Ray et al. 2021). This is shown in nanocellulose, which contains cellulose nanocrystals -CNCs and cellulose nanofibrils -CNFs (Du et al. 2019, Dufresne 2019). Its high strength-to-weight ratio makes it ideal for packaging, coatings, and composites, and its mechanical qualities rival petroleum-based polymers (Dufresne 2017). Besides mechanical qualities, research on cellulose-based material water resistance has garnered a lot of interest. Due to its hydrophilic nature and rapid moisture absorption, natural cellulose is restricted in moisture-sensitive packaging. Acetylation and hydrophobic coatings boost the performance of water resistance, packaging, and food contact (Klemm et al. 2005). Cellulose and biodegradable polymers like polylactic acid have been combined to generate composite materials with increased moisture resistance and durability.

Modern cellulose processing uses ionic liquids and deep eutectic solvents to dissolve cellulose (Taokaew & Kriangkrai 2022). These safe solvents degrade cellulose without harsh chemicals, reducing its environmental impact (Sirviö et al. 2020). Enzymatic processing optimizes cellulose materials to reduce emissions and energy use compared to kraft pulping (Isikgor & Becer 2015).

Cellulose three-dimensional printing is novel. Cellulose-based additive manufacturing can mass-produce biodegradable, custom-shaped goods for building, medicine, and other industries (Chen et al. 2020). Nanocellulose-based 3D printing inks are expanding the use of cellulose materials in eco-friendly, high-performance manufacturing (Chauhan et al. 2024, Kim et al. 2024). Fig. 3 presents a SWOT analysis of cellulose-based materials, highlighting their biodegradability and renewable origin as strengths while identifying scalability and competition from conventional plastics as key challenges.

Policy and Industry Integration

To advance cellulose-based products, policymakers and industry stakeholders must work together. Restricting single-

Fig. 3: SWOT Analysis of Cellulose-Based Materials as Alternatives to Conventional Plastics.

use plastics can accelerate the transition to biodegradable materials such as cellulose (OECD 2018). Comprehensive cellulose recycling and composting rules are needed to balance end-of-life management with environmental aims (Sikorska et al. 2021, Gadaleta et al. 2023). Collaboration between industry and research institutions is necessary for innovation and production scalability. Research and development are needed to improve the performance of cellulose-based materials and decrease their cost to compete with petroleum-based polymers. The packaging, textile, and consumer products sectors want sustainable solutions (Siró & Plackett 2010). Companies in these areas should integrate cellulose-based goods into their supply chains. Public-private partnerships (PPPs) can help innovate cellulose-based products. This type of partnership accelerates technology development and commercialization by pooling expertise, assets, and finance. The EU Bio-based Industries Joint Undertaking (BBI JU) funds cellulose product development projects. Industries, academic institutions, and governments should collaborate on this endeavour to tackle commercial and technical problems. PPPs may also align industry practices with regulations by boosting green production and waste management. Government incentives or co-investments may assist cellulose-based material producers in developing and embracing new technologies. PPPs can speed cellulose-based product development and deployment by fostering cross-sector collaboration.

Research Gaps and Prospective Investigations

Cellulose-based materials have advanced, but further study is needed to enhance their environmental advantages. Comprehensive lifecycle assessments (LCAs) are used to examine the environmental implications of cellulose-based products from extraction to disposal (Foroughi et al. 2021). Though renewable, cellulose extraction and processing have different environmental implications depending on feedstock and manufacturing procedures (Shen et al. 2010). More research is needed to improve these processes while reducing energy, water, and pollutants. The biodegradation of cellulose acetate and other chemically modified cellulose derivatives is crucial to study. Undamaged cellulose is biodegradable, although chemical changes may hinder it. Future research should aim to make modified cellulose materials biodegradable so they can break down in soil and water without leaving a deposit (Song et al. 2009). Cellulose-derived items in the bio-circular economy are a promising new research area. The bio-circular economy emphasizes waste reduction, renewable resources, and recycling (MacArthur 2013). Cellulose fits this paradigm since it's renewable, biodegradable, and recyclable. Effectively integrating cellulose materials into circular supply networks requires additional research. Research should focus on recycling composites and chemically modified cellulose, which are unsuitable for current procedures. New cellulose uses, like bio-composites and eco-friendly packaging, can boost bio-circular economy performance. According to Tjeerdsma & Militz (2005), governments and companies must collaborate to provide composting, recycling, and reuse infrastructure for cellulose-based goods to contribute to a sustainable economy.

CONCLUSIONS

Cellulose-derived materials offer a promising pathway to mitigate plastic pollution due to their biodegradability, compostability, and lower greenhouse gas emissions compared to petroleum-based plastics. They naturally decompose in terrestrial and marine environments, avoiding the long-term persistence and toxic effects associated with microplastics. Despite these advantages, technical and economic barriers remain. Unmodified cellulose lacks sufficient water resistance and mechanical strength, while chemical modifications such as cellulose acetate and nanocellulose introduce additional costs and processing challenges. Scaling production and improving material performance are essential to enhance market competitiveness. Achieving a broader transition to cellulose-based materials requires coordinated action. Industry should invest in R&D to develop cost-effective and environmentally friendly production processes. Governments can support this shift through targeted legislation, tax incentives, and investment in recycling and composting infrastructure. Public procurement policies and consumer education will further drive demand and market growth. Future efforts must focus on overcoming affordability, scalability, and end-of-life management challenges. Advancing cellulose-based solutions through innovation, public-private collaboration, and informed policy will contribute significantly to a sustainable and circular economy. In addressing the global plastic crisis, cellulose-based alternatives represent a critical step towards a cleaner and more resilient future.

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Risk Assessment of Groundwater Contamination by Heavy Metals in the Gangetic Plain: A Multivariate Statistical and Index-Based Study

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ABSTRACT

Groundwater in the Gangetic Plain of India is increasingly vulnerable to heavy metal contamination, raising serious public health concerns. This study analyzed 12 heavy metals (As, Cr, Ni, Mn, Fe, Zn, Pb, Cu, Se, Mo, Cd, and Co) in 30 groundwater samples using Inductively Coupled Plasma Mass Spectrometry (ICP-MS). Statistical evaluations included Shapiro-Wilk normalization, Pearson correlation (SPSS v25), and Principal Component Analysis (OriginLab v10.15). Heavy Metal Pollution Index (HPI), non-carcinogenic Health Risk Assessment (HI), and carcinogenic risk (CR) analyses were performed. The results showed that 70% of the samples from Ballia exceeded the HPI threshold (>100), with the highest value being 328.77. Lead (Pb) and Arsenic (As) were the dominant contributors to non-carcinogenic risk, with HI values peaked at 28,334.8. The carcinogenic risk values for As and Ni exceeded the acceptable limits in all districts, with Prayagraj and Ballia showing total CR values of 2.17 and 3.00, respectively. Strong correlations among metals (e.g., Cd–Mn, r = 0.80) suggest anthropogenic origins, particularly from industrial and agricultural sources. These findings highlight the urgent need for routine monitoring, point-source control, and localized treatment to ensure groundwater safety.

INTRODUCTION

Despite various studies on heavy metal contamination in India, an integrated assessment combining multivariate statistical analysis, pollution indexing, and comprehensive health risk evaluation remains scarce, particularly in the Gangetic Plain. This area, characterized by intense agricultural and industrial activities, faces growing pressure on groundwater resources but lacks detailed, region-specific investigations. This study addresses this gap by applying an integrated approach using Principal Component Analysis (PCA), Pearson correlation, Heavy Metal Pollution Index (HPI), and both non-carcinogenic and carcinogenic risk assessments (HQ, HI, and CR). By evaluating groundwater samples from three districts-Ballia, Lakhimpur, and Prayagraj-this study provides a comprehensive understanding of pollution levels, source identification, and associated health risks. The findings aim to support evidence-based groundwater management and inform mitigation strategies in one of India's most densely populated and ecologically sensitive regions, the Indo-Gangetic Plain (IGP).

MATERIALS AND METHODS

Study Region and Sampling

The Gangetic Plain is located in the north-central part of India, where the state of Uttar Pradesh covers the largest part. This region, lying along the Ganges River, is marked by diverse hydrogeological conditions and extensive agricultural

and industrial activities that can substantially impact groundwater quality. In this study, 30 groundwater samples were collected from three districts, Prayagraj, Lakhimpur, and Ballia, in the Central Plain Zone of this region. The geographical coordinates of the districts are listed in Table 1. All samples were collected in 500 mL high-density polyethylene (HDPE) bottles that were pre-cleaned and acid-washed with 10% nitric acid to prevent metal contamination. After collection, each sample was promptly sealed, stored at 4°C, and transported to the laboratory for analysis within 48 h, following the standard sampling protocols of the Bureau of Indian Standards (BIS) (BIS 2015). The sampling location map of the study area is shown in Fig. 1.

Heavy Metals Selection and their Multivariate Graphical Analysis

Based on the frequent detection of elements in the groundwater of this region, 12 heavy metals—viz. As Cr, Cd, Ni, Pb, Mn, Fe, Co, Cu, Zn, Se, and Mo were selected for quality monitoring analysis. The concentrations of these heavy metals were quantified using Inductively Coupled Plasma Mass Spectrometry (ICP-MS), following the standard operating procedures of the APHA (APHA 2012).

Table 1: Description of the study area.

Sl.No.	Name of the District	Geographical Coordinate	Source water	Population (Lakh) (Census 2011)
1.	Prayagraj	25.4358° N 81.8463° E	Groundwater	59.54
2.	Lakhimpur Kheri	27.9450° N 80.7821° E	Groundwater	40.21
3.	Ballia	25.8307° N, 84.1857° E	Groundwater	32.39

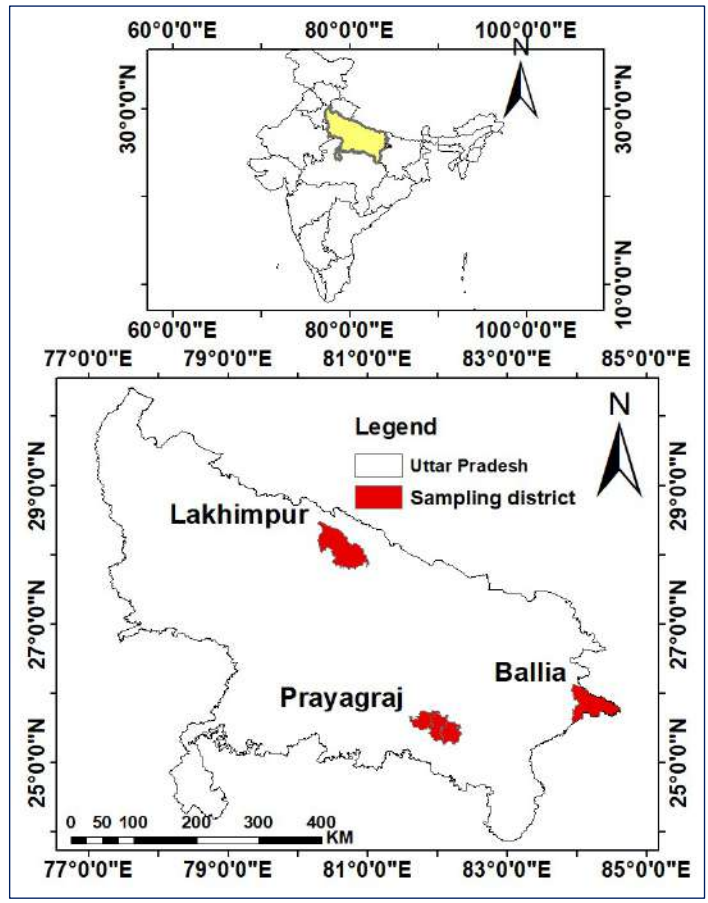

Fig. 1: Sampling Location map.

Table 2: Sub-Index calculation of the average of Heavy metals.

Heavy Metals	Mean Concentration (Ci) [μg.L^{-1}]	Max. Permissible Value (BIS 2012)	Unit weightage [Wi]	Sub Index [SI]
As	12.1262	10	0.16	0.19
Cr	1.1475	50	0.13	0.002984
Cd	2.4855	3	0.13	0.11
Ni	6.821833	20	0.09	0.030698
Pb	28.1305	10	0.16	0.45
Mn	370.3008	100	0.03	0.11
Fe	1498.373	300	0.02	0.10
Co	0.429	100	0.06	0.00
Cu	21.29517	50	0.06	0.03
Zn	574.2563	5000	0.05	0.01
Se	1.166	10	0.08	0.01
Mo	1.108167	70	0.05	0.00
HPI = $\sum SI$				104.3

To examine inter-metal correlations and identify potential sources of contamination, the Pearson correlation coefficient (PCC) was calculated using the SPSS software (version 25). Before using SPSS, all data were normalized using the Shapiro-Wilk test to ensure their suitability for parametric correlation. Multivariate statistical techniques, such as Principal Component Analysis (PCA), were employed using OriginLab software (version 9.6.5) to identify the underlying factors contributing to heavy metal distribution across the study area. Moreover, the Box plot technique was used for graphical representation and to enhance the understanding of data trends and distribution.

Heavy Metal Pollution Index (HPI)

The Heavy Metal Pollution Index (HPI) was used to assess the overall water quality. This method uses a weighted arithmetic mean, which involves two steps: (1) assigning a rating to each parameter with an appropriate weight and (2) identifying the parameters contributing to pollution for inclusion in the index. The rating scale ranged from 0 to 1. For each parameter in the HPI calculation (Table 2), the unit weight (Wi) was inversely proportional to the recommended standard (Si). HPI can be expressed mathematically as follows: (1) (Mohan et al. 1996).

$$\mathrm{HPI} = \frac{\sum_{i=1}^{n} W_i Q_i}{\sum_{i=1}^{n} W_i} \quad \ldots(1)$$

where 'Qi' represents the sub-index of the ith parameter, 'Wi' denotes the unit weight of the ith parameter, and 'n' is the total number of parameters included in the analysis. The sub-index (Qi) for each parameter is calculated using Eq. (2):

$$\mathrm{Qi} = \sum_{i=1}^{n} \frac{\{Mi - Ii\}}{(Si - Ii)} \times 100 \quad \ldots(2)$$

Where Mi, Ii, and Si represent the monitored, ideal, and standard permissible values of the ith parameter, respectively. A critical HPI value of 100 is considered the threshold for safe drinking water. In this study, a modified classification scale was adopted, categorizing HPI values into four levels: low, medium, high, and very high, as detailed in Table 3.

Health Risk Assessment Framework

The assessment of health risks from metal pollutants in groundwater includes consideration of both non-carcinogenic and carcinogenic risks. The risk was quantified according to the United States Environmental Protection Agency (USEPA) guidelines for human health risk assessment (USEPA 2005). The average daily intake (ADI) of metals via oral ingestion and dermal pathways was estimated using Equations (3) and (4), respectively.

$$\mathrm{ADI_{ingestion}} = \frac{\mathrm{C \times IR \times EF \times ED}}{\mathrm{BW \times AT}} \times \mathrm{CF} \quad \ldots(3)$$

$$\mathrm{ADI_{dermal}} = \frac{C \times SA \times SAF \times DAF \times ED \times EF}{BW \times AT \times PEF} \times \mathrm{CF} \quad \ldots(4)$$

The Average Daily Intake (ADI) represents the daily exposure to heavy metals, expressed in mg.kg^{-1}.day^{-1}, and is calculated using the parameters shown in Table 4.

Potential non-carcinogenic risks are evaluated through the Hazard Quotient (HQ) (Eq. 5), which is the ratio of the

Table 3: HPI value for drinking water used in this study.

Category	HPI Range
Low	<40
Medium	40-70
High	70-100
Very High	>100

Table 4: Exposure Parameter used in Health Risk Assessment.

Parameter	Description	Value	Unit	Reference
C	Concertation of Heavy Metal	-	mg	-
IR	Ingestion Rate	3.0	L/day	ICMR 2009
EF	exposure frequency	365	Days/year	ICMR 2009
ED	Exposure duration	30	Years	ICMR 2009
BW	Body Weight	57.5	kg	-
AT	Average time	10,950	Days	ED*365
CF	Conversion Factor	10^{-6}	kg/mg	-

exposure level of a specific element to its reference dose (RfD), as defined by the USEPA (2011) (Table 4) (Saw et al. 2023a). The Hazard Index (HI), which accounts for multiple substances under a single exposure pathway, is determined as the sum of the HQs for all analyzed metals (Eq. 6).

$$HQ = \frac{ADI}{RfD} \quad \ldots(5)$$

$$HI = \sum HQ \quad \ldots(6)$$

The carcinogenic risk (CR) was calculated by multiplying the oral slope factor (Sf) by the exposure level (ADI) (Eq. 7), which is an important measure in risk assessment. The Sf values are listed in Table 5. The acceptable range for CR or CR_{total} is defined as 1×10^{-6} to $^{4-}10\times1$ (USEPA 2009). A CR or CR_{total} less than 1×10^{-6} indicates a negligible carcinogenic risk, whereas a CR or CR_{total} greater than 1×10^{-4} signifies an unacceptable carcinogenic risk (USEPA 2001).

$$CR = ADI \times Sf \quad \ldots(7)$$

Quality Assurance and Quality Control (QA/QC)

All samples were handled carefully to avoid contamination and ensure reliability. Instrument calibration was performed before analysis, and the accuracy of the analysis was verified by analyzing the reference standard of water (NIST 1640a and NIST 1643b). The analytical precision was verified by calculating the relative standard deviations, which remained within acceptable limits (<5%) for all elements.

Table 5: Oral reference dose and oral slope factor value (Shekoohiyan et al. 2021, USEPA 2009).

Heavy metals	Rf (mg/kg/day)	Sf [$mg.kg^{-1}.day^{-1}$]
As	0.0003	1.5
Cd	0.001	6.3
Cr	0.003	0.5
Ni	0.02	0.91
Pb	0.0035	0.0085
Mn	0.14	-
Fe	0.7	-
Zn	0.3	-
Cu	0.04	-
Co	0.0003	-
Se	0.005	-
Mo	0.005	-

RESULTS AND DISCUSSION

Graphical Representation of Heavy Metals

The box plot representation of all 12 targeted heavy metals is shown in Fig. 2a-l, of these 12 elements, As, Cr, Cd, Ni, and Pb are carcinogenic, whereas the remaining seven (Mn, Fe, Co, Cu, Zn, Se, and Mo) are non-carcinogenic to human health (Mahato et al. 2017, Saw et al. 2023a). The concentration of Fe (1498.07±975.6 $\mu g.L^{-1}$) in the groundwater of the collected water sample exceeded the guideline value (300 $\mu g.L^{-1}$) set by the WHO and BIS (Fig. 2a). The concentrations of Cu and Zn (Fig. 2b-c) were well within the acceptable limits, with average values of 21.29±14.9 $\mu g.L^{-1}$ and 574.25±1106.4 $\mu g.L^{-1}$, respectively. Co and Mo (Fig. 2d-e) were detected at minimal concentrations, indicating that there was no immediate concern. Se showed a high value of 11.345 $\mu g.L^{-1}$, exceeding the BIS guideline (10 $\mu g.L^{-1}$) (Fig. 2f). Cd value ranged from 0.03 to 9.73 $\mu g.L^{-1}$, with an average of 2.48±2.7 $\mu g.L^{-1}$, approaching the 3$\mu g.L^{-1}$ specified by both BIS and WHO (Fig. 2g). Arsenic (As) levels were considerably elevated, reaching a maximum of 102.6 $\mu g.L^{-1}$, with an average of 12.12±21.2 $\mu g.L^{-1}$, surpassing the 10 $\mu g.L^{-1}$ threshold recommended by both standards, indicating a potential health risk (Fig. 2h). The elevated concentrations of heavy metals such as arsenic, lead, and chromium in the groundwater of districts such as Ballia and Lakhimpur may be attributed to both natural geogenic sources and anthropogenic influences, particularly agricultural runoff (e.g., phosphate fertilizers and pesticides) and industrial discharges from activities such as sugar processing and small-scale manufacturing (Sharma et al. 2019, Singh & Singh 2021). Cr (Fig. 2i) and Ni (Fig. 2k) were below their respective BIS limits of 50 $\mu g.L^{-1}$, with average values of 1.14±1.4 $\mu g.L^{-1}$ and 6.82±3.8 $\mu g.L^{-1}$, respectively. Pb (Fig. 2j) showed alarmingly high

Table 6: Percentage of sample exceeding BIS limits for Heavy Metals.

Heavy metal	BIS Limits [μg.L^{-1}]	% of sample
Arsenic (As)	10	47
Lead (Pb)	10	37
Chromium (Cr)	50	30
Cadmium (Cd)	3	23
Nickel (Ni)	20	27
Iron (Fe)	300	17
Manganese (Mn)	100	13.3
Zinc (Zn)	5000	0
Copper (Cu)	50	0
Selenium (Se)	10	0
Cobalt (Co)	Not specified	0
Molybdenum (Mo)	Not specified	0

values, peaking at 118.3 μg.L^{-1} with an average of 28.13± 29.2 μg.L^{-1}, far exceeding both the BIS and WHO permissible limits, underscoring severe contamination risk. Mn (Fig. 2l) levels were also markedly high, ranging from 7.7 to 1167 μg.L^{-1}, averaging 370.30±360.8 μg.L^{-1}, surpassing the BIS limit of 100 μg.L^{-1} (Table 6).

The variation in the concentration range of these targeted heavy metals in this region is attributed to the competing influences of the diagenesis, physicochemical weathering, sediment texture, geology, and geochemistry of individual metals in the Ganga River Basin (Kumar et al. 2019, Singh et al. 2006, Ukah et al. 2019). Moreover, leachable ions from the soils and the lithogenic origin of minerals in groundwater may also vary the quality of water in this region (Khan & Rai 2023).

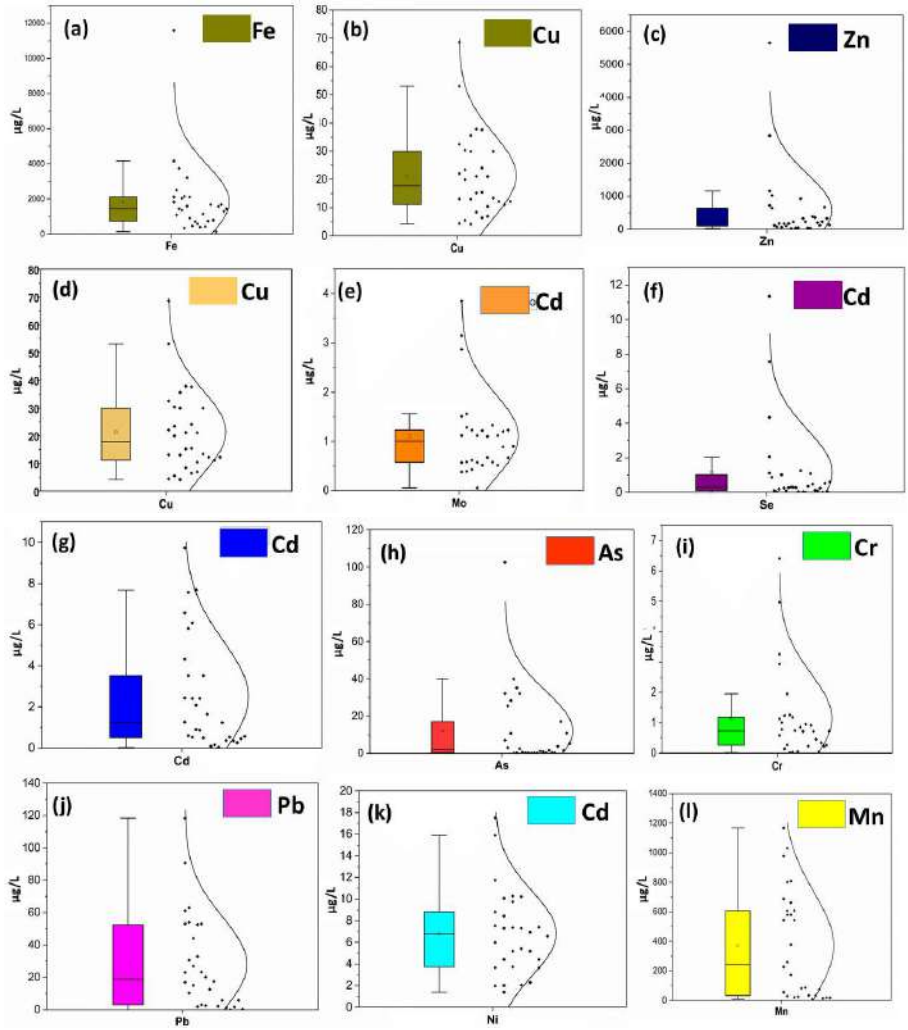

Fig. 2: Box plot diagramwith kernel density estimate of heavy metals (a) Fe, (b) Cu, (c) Zn, (d) Co, (e) Mo, (f) Se, (g) Cd, (h) As, (i) Cr, (j) Pb, (k) Ni, and (l) Mn.

Pearson Correlation Matrix

The Pearson correlation matrix of the heavy metal content in the groundwater of the study area is shown in Fig. 3. To better interpret the relationships among heavy metals, correlation coefficients were grouped by strength: strong ($r > 0.7$), moderate ($0.5 \leq r \leq 0.7$), and weak ($r < 0.5$). Strong correlations were observed between Cd and Mn (r = 0.80), Fe and Co (r = 0.79), and Pb and As (r = 0.75), suggesting common anthropogenic sources such as industrial discharge or agrochemical runoff (Mahato et al. 2023). Moderate correlations were observed between Cr and Ni and Zn–Cu, which may indicate shared transport or geogenic pathways (Singaraja et al. 2015). Weak correlations were noted for pairs such as Mo–Zn, implying dissimilar origins or independent mobility in groundwater. Conversely, cobalt (Co) exhibited negative correlations with elements such as arsenic (As) (r = -0.17) and lead (Pb) (r = -0.13), indicating potential differences in their sources or environmental interactions (Saw et al. 2023a). Overall, this correlation matrix revealed potential metal sources and interactions in water samples, aiding in the identification of contamination origins and assessment of environmental risks.

Principal Component Analysis (PCA)

The PCA plot in Fig. 4 depicts the heavy metal correlations and concentration distribution across the study area using two principal components, PC1 and PC2, which varied by 35.6% and 19.3%, respectively. Data points for each district are color-coded (Ballia-Black, Lakhimpur-Kheri- Red, and Prayagraj-Green) with 95% confidence ellipses. The blue arrows signify the contribution of each heavy metal to the principal components. The elements like Se, Co, Ni, and Cu contributed significantly to PC1; this pattern suggests a common anthropogenic origin, as these elements are often associated with industrial effluents, electroplating waste, alloy manufacturing, and phosphate-based fertilizers. While Cr, Mo, and As are more strongly associated with PC2. The deviation in the distribution was attributed to regional variations in heavy metal concentrations driven by environmental or human factors (Mishra et al. 2018). PC1, with an eigenvalue of 4.27, strongly represented the key factors influencing heavy metal distribution. Moreover, PC2 had an eigenvalue of 2.31, bringing the cumulative variance explained to 54.9% (Table 7).PC1 and PC2 captured over half of the data variability, with heavy metals (Se, Co, Ni, Cu,

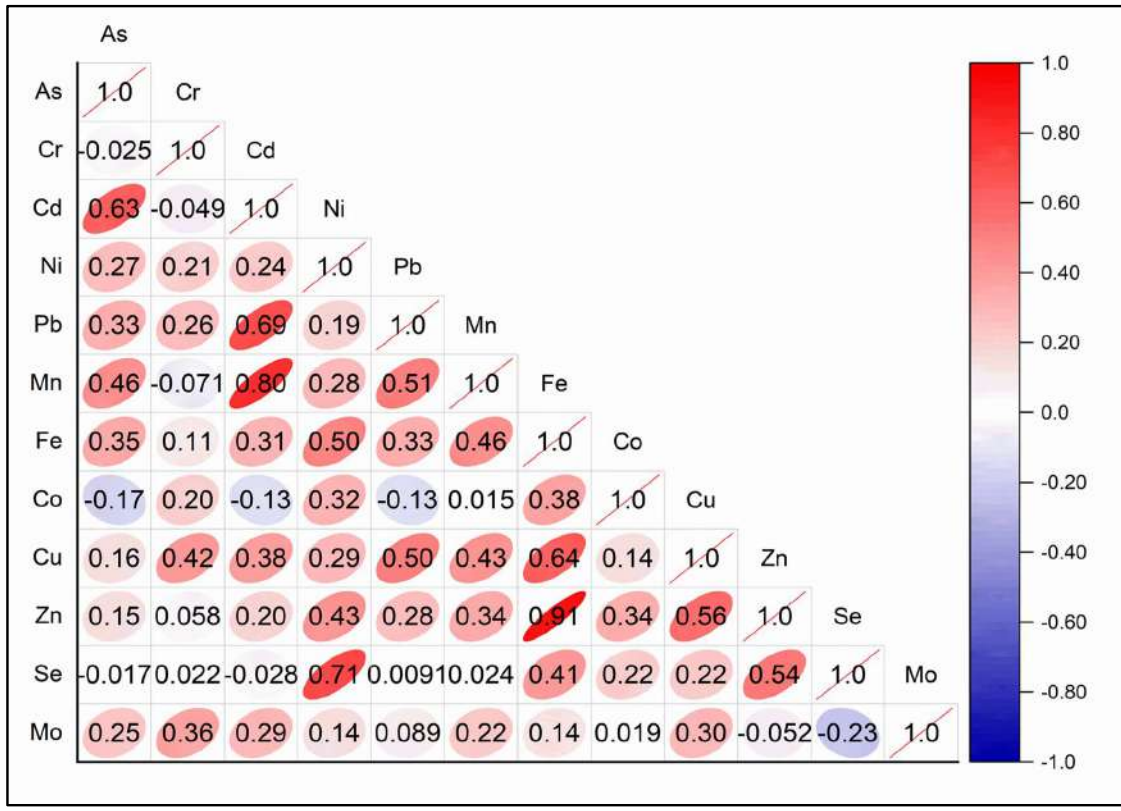

Fig. 3: Pearson correlation matrix among the heavy metal content.

Table 7: Eigenvalue and percentage of variance of PCA.

Principal Component Number	Eigenvalue	Percentage of Variance (%)	Cumulative (%)
1	4.26	35.58	35.58
2	2.31	19.28	54.86
3	1.53	12.78	67.64
4	0.99	8.30	75.95
5	0.85	7.11	83.07
6	0.61	5.13	88.21
7	0.49	4.13	92.35
8	0.29	2.43	94.79
9	0.24	2.06	96.85
10	0.16	1.39	98.25
11	0.13	1.09	99.35
12	0.07	0.64	100

Cr, Mo, and As) strongly contributing to these components, indicating regional sources or environmental impacts (Shakeri et al. 2021). The first four components explained 75.9% of the variance, defining the heavy metal pollution profiles for Ballia, Lakhimpur, and Prayagraj.

Heavy Metal Pollution Index (HPI)

Table 8 presents the HPI values and their corresponding pollution categories for the water samples collected from the three districts: Lakhimpur, Prayagraj, and Ballia. HPI values were categorized into four groups: low (HPI < 50), medium (50 ≤ HPI < 75), high (75 ≤ HPI < 100), and Very High (HPI ≥ 100).

In Lakhimpur, four samples (40%) were classified as Low with HPI values ranging from 11.07 to 27.27, two samples (20%) as Medium (48.39–59.28), two samples (20%) as High (70.61 and 70.71), and two samples (20%) as Very High (126.27 and 144.44) shown in Fig 5. While in Prayagraj, five samples (50%) fell under the Low category (14.14–43.69), two samples (20%) under the Medium category (55.22–59.42), and three samples (30%) under the Very High category (156.24–188.16). In Ballia, all 10 samples exhibited severe contamination, with three samples (30%) falling under the Medium category (55.22–62.46) and seven samples (70%) classified as Very High (130.38–328.77). The highest HPI value (328.77) was recorded in Ballia (Sample 26), while the lowest (11.07) was observed in Lakhimpur (Sample 8). The data showed that the Ballia district has the highest frequency of Very High pollution levels, emphasizing the need for targeted interventions to address the critical pollution levels in this district. Lakhimpur displayed a mix of categories, while Prayagraj presented moderate to high pollution levels, indicating the need for sustained monitoring and remedial actions.

Health Risk Assessment

Non-carcinogenic risks of heavy metals: The potentiality of non-carcinogenic risks of heavy metals via oral ingestion was evaluated using the indices HQ and HI. The calculated values of the same were tabulated in Table 9. For non-carcinogenic risk, an HQ value exceeding 1 indicates potential adverse effects, while a value less than 1 is considered safe for drinking. The results showed substantial variation in metal

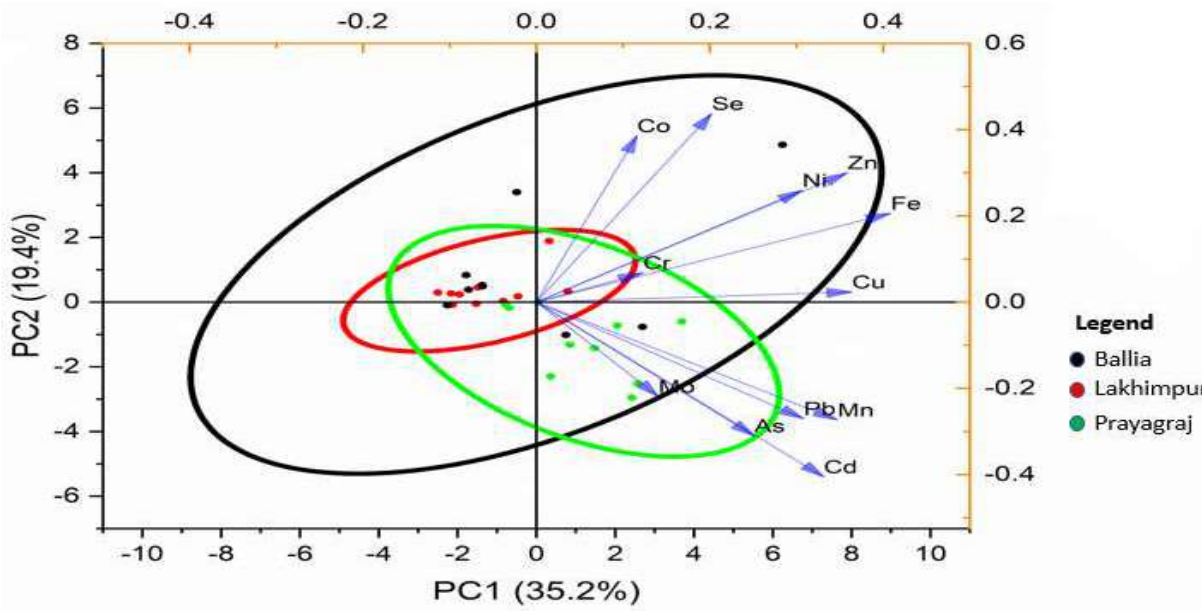

Fig. 4: PCA plot of heavy metal concentrations in Ballia, Lakhimpur, and Prayagrajdistricts of Uttar Pradesh.

Table 8: HPI Value and its category for all samples.

Locations	Sample No.	HPI value	Category L-Low, M-Medium, H-High, VH-Very High
Lakihimpur	1	144.4383	VH
	2	48.39563	M
	3	70.61014	H
	4	126.2699	VH
	5	27.27457	L
	6	59.27744	M
	7	70.71346	H
	8	11.07056	L
	9	16.49459	L
	10	21.73648	L
Prayagraj	11	34.24993	L
	12	43.69198	M
	13	21.3887	L
	14	59.424	M
	15	188.1626	VH
	16	175.9942	VH
	17	55.22373	M
	18	14.13937	L
	19	156.2387	VH
	20	20.865	L
Ballia	21	287.1517	VH
	22	152.6187	VH
	23	130.384	VH
	24	313.7766	VH
	25	172.1715	VH
	26	328.7666	VH
	27	55.22373	M
	28	58.15205	M
	29	188.0822	VH
	30	62.462	M

concentrations across samples and districts. In particular, Ballia exhibited alarmingly high concentrations of Fe, Pb, Cd, and As, with HQ values indicating potential health risks. For instance, sample 21 from Ballia reported an HI of 22,664.90, suggesting a severe pollution level. Pb, As, Fe, and Cd are the primary contributors to HQ, with their extremely high values pushing the HI to a higher value. In terms of contributions to non-carcinogenic risk, Pb and As were the dominant contributors in Ballia and Prayagraj, with HI values far exceeding safe thresholds. In Lakhimpur, Cd and Mn were the primary contributors. These patterns suggest that contamination sources and exposure risks vary geographically and require location-specific mitigation strategies.

Carcinogenic risks of heavy metals: The total carcinogenic risk (CR_{Total}) ranged from 0.714 in Lakhimpur to 3.004 in Ballia, with Prayagraj showing the highest individual risk for arsenic (2.166), as shown in Table 10. These values are significantly higher than the USEPA acceptable limit (1×10^{-4}), indicating an elevated cancer risk due to prolonged exposure. Among all elements, As and Ni were the primary contributors to the total carcinogenic risk.

CONCLUSIONS

This study highlights the serious extent of heavy metal contamination in the groundwater of the Gangetic Plain, particularly in the Ballia district, where 70% of the samples exhibited very high HPI values, with the highest recorded HPI being 328.77. Among the 12 metals analyzed, Pb, As, Fe, Cd, and Mn frequently exceeded BIS permissible limits. Health risk assessments revealed that Pb and As were the most hazardous, with Hazard Index (HI) values reaching up to 28,334.8 in Ballia, indicating severe non-carcinogenic health risks. In addition, carcinogenic risk (CR) values for Pb, Cd, As, Ni, and Cr were evaluated. The total carcinogenic risk exceeded the acceptable limit of 1×10^{-4} in all three districts, with Prayagraj showing the highest

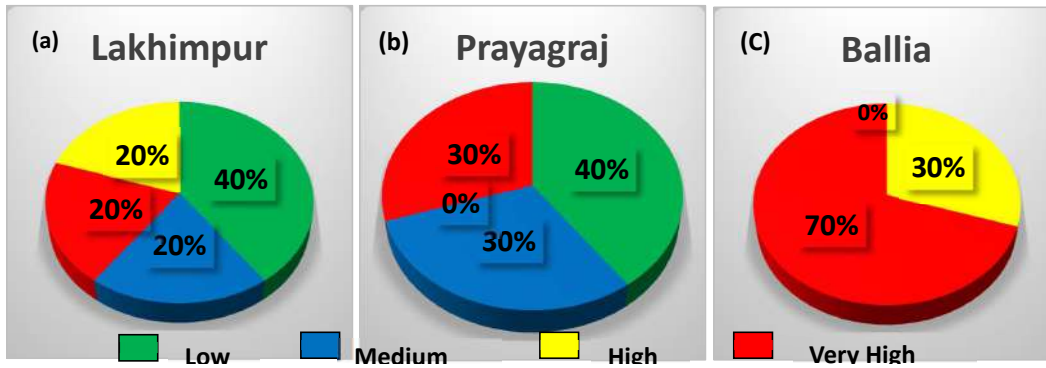

Fig. 5: Pie chart diagram of HPI of a) Lakhimpur, b) Prayagraj, and c) Ballia.

Table 9: HQ and HI values of non-carcinogenic health risk of heavy metals.

Location	Sample No.	HQ												HI
		Mn	Fe	Zn	Cu	Se	Mo	Co	Pb	Cd	As	Ni	Cr	
Lakhimpur	1	84.50	136.77	125.22	42.40	11.63	11.69	0.63	10643.48	132.52	1228.70	19.67	0.22	12437.42
	2	20.11	81.73	111.48	17.06	9.13	5.95	0.54	2946.09	63.13	524.35	9.59	0.04	3789.18
	3	10.50	107.70	20.37	26.01	1.10	6.10	0.56	4006.09	55.30	1876.52	5.26	0.02	6115.52
	4	63.84	102.30	12.30	16.97	0.16	6.47	0.57	10956.52	94.43	6.26	30.67	0.03	11290.51
	5	7.54	25.88	30.32	19.85	1.93	6.00	0.64	1781.74	90.78	27.83	9.77	0.01	2002.29
	6	96.61	118.88	11.63	39.61	10.64	13.41	2.70	2619.13	52.17	441.74	26.30	0.17	3433.00
	7	8.96	68.16	29.80	28.74	2.56	15.76	0.64	5330.43	171.65	113.04	19.03	0.04	5788.82
	8	29.98	34.81	17.75	5.72	0.99	3.97	0.66	335.65	8.87	36.52	5.37	0.01	480.30
	9	31.90	53.40	29.96	20.14	2.87	4.07	0.52	532.17	16.17	43.48	5.15	0.00	739.84
	10	140.94	44.12	4.58	13.58	3.23	5.37	0.47	436.52	3.13	9.57	3.63	0.00	665.15
Prayagraj	11	12.35	30.35	39.10	7.18	3.08	7.04	0.59	2190.43	129.39	34.78	19.20	0.03	2473.53
	12	2.87	85.64	7.21	30.55	2.87	6.05	0.58	3038.26	38.61	10.43	15.61	0.04	3238.74
	13	11.89	32.28	9.37	5.40	0.37	5.37	0.64	373.04	254.09	204.35	5.97	0.00	902.77
	14	27.63	55.45	161.30	17.45	13.15	4.49	0.55	4686.09	55.83	146.96	9.53	0.03	5178.45
	15	202.02	157.42	57.01	39.03	0.31	12.83	1.17	9222.61	368.35	5589.57	11.56	0.03	15661.89
	16	363.80	187.23	201.57	89.41	21.50	9.29	0.43	9389.57	685.57	50.43	19.04	0.03	11017.87
	17	215.52	125.66	39.63	15.94	0.00	11.69	1.10	1022.61	251.48	280.00	13.54	0.01	1977.17
	18	4.98	59.69	7.66	10.96	3.65	12.73	1.43	231.30	36.52	66.09	14.14	0.03	449.18
	19	256.47	86.38	981.74	69.27	78.99	6.99	2.68	5725.22	217.04	4415.65	41.48	0.03	11881.95
	20	6.84	10.87	5.05	8.38	118.38	0.57	0.64	0.00	27.13	33.91	45.69	0.02	257.49
Ballia	21	298.17	279.06	493.39	46.41	45.29	9.39	0.17	15754.78	789.91	4925.22	23.03	0.07	22664.90
	22	225.99	310.36	178.26	49.33	11.37	32.87	0.24	7688.70	450.78	650.43	21.99	0.10	9620.43
	23	246.63	117.84	67.06	14.49	5.01	29.95	0.92	300.87	635.48	6940.87	26.82	0.00	8385.94
	24	434.91	152.05	62.64	27.55	1.25	11.48	1.34	20573.91	1015.30	2977.39	18.13	0.01	25275.97
	25	383.85	159.80	34.66	31.37	0.94	13.88	0.66	4055.65	607.30	6113.04	26.69	0.04	11427.89
	26	226.40	239.33	20.94	27.47	2.50	16.28	0.05	9130.43	802.43	17843.48	25.46	0.04	28334.82
	27	215.52	125.66	39.63	15.94	0.00	11.69	1.10	1022.61	251.48	280.00	13.54	0.01	1977.17
	28	300.86	96.82	116.54	9.03	5.53	12.52	0.33	46.96	48.00	1893.91	19.33	0.01	2549.83
	29	202.02	157.42	57.01	39.03	0.31	12.83	1.17	9222.61	368.35	5589.57	11.56	0.03	15661.89
	30	6.40	107.33	22.94	49.04	6.26	40.17	0.99	3504.35	59.48	913.04	17.14	0.11	4727.26

Table 10: CR value of Carcinogenic risk of heavy metals.

Locations	CR(Avg)					CR_{total}
	Pb	Cd	AS	Ni	Cr	
lakhimpur	0.010	0.013	0.194	0.457	0.040	0.714
Prayagraj	0.018	0.096	2.166	0.693	0.032	1.219
Ballia	0.009	0.039	0.487	0.666	0.018	3.004

CR due to elevated Arsenic (2.166) and Ballia reaching a total CR of 3.004, highlighting significant long-term health risks from chronic exposure. Particularly, Ballia, immediate interventions such as point-source identification, regular monitoring, and installation of localized treatment systems. However, this study is limited by a small sample size and single-time sampling, which may not capture seasonal variations. Future research should include a large sample set, multi-seasonal monitoring, expanded spatial coverage, and integration of hydrogeochemical modeling and source apportionment techniques to better understand pollutant dynamics and inform targeted mitigation strategies.

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Factors That Determine the Efficiency of Public Environmental Protection Expenditures of OECD Countries: Super Efficiency DEA and Panel Data Analysis

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ABSTRACT

One of the most crucial tools used by governments in addressing environmental issues is public environmental protection expenditure. This study aims to assess the efficiency of public environmental protection expenditures and identify the factors influencing this efficiency. In this study, we use environmental data of 30 OECD countries between 2008 and 2020 employing a two-stage Data Envelopment Analysis (DEA) methodology. In the first stage, we utilize the super-efficiency DEA model. Public environmental protection expenditures are considered as inputs, while carbon dioxide emissions, renewable energy production, forest area percentage, and particulate matter concentration in the air are treated as outputs. In the second stage, we conduct a classical panel data analysis, using the efficiency scores obtained in the first stage as the dependent variable. Independent variables include population density, urbanization, industrialization, per capita national income, and primary energy intensity. The empirical findings reveal a negative relationship between efficiency scores and both population density and primary energy intensity. Conversely, urbanization and industrialization exhibit a positive relationship with efficiency scores. No significant relationship is found between per capita national income and efficiency scores. These results suggest that urbanization and industrialization may affect the efficiency of public environmental protection expenditures. The study contributes to the literature by combining Super-Efficiency DEA with panel data analysis and by addressing a notable gap in empirical research on the efficiency of public environmental protection expenditures, specifically in OECD countries, offering policy-relevant insights for sustainable fiscal planning.

INTRODUCTION

Environmental protection expenditures made by the public sector are critical in combating environmental pollution and ensuring sustainable growth and development. These expenditures clearly show the policies and strategies that states implement for environmental issue protection. Considering that environmental problems cause negative externalities and need to be solved, environmental protection and the effectiveness of environmental policies are the most important in terms of public interest. Negative externalities from environmental issues make the market mechanism's activities inadequate for addressing environmental concerns. Public environmental protection expenditures are at the forefront of these intervention tools. For this reason, the role of environmental protection expenditures in the effectiveness of environmental policies can be discussed. However, despite the growing importance of environmental expenditures in shaping green policy agendas, little is known about how efficiently these public resources are used, particularly within OECD countries. Assessing this efficiency is crucial for ensuring fiscal sustainability and maximizing environmental impact amid limited budgets and increasing ecological risks.

In this context, the study aims to calculate the effectiveness of environmental protection expenditures and to determine the factors that determine effectiveness. To achieve this, we prefer a two-stage approach: DEA is used to measure relative efficiency among countries, while panel data analysis identifies the main explanatory factors behind efficiency differences. The study employs a two-stage data envelopment analysis (DEA) method to examine the effectiveness of public environmental protection expenditures and the factors determining this effectiveness. In this study, a two-stage analysis was conducted. In the first stage, Data Envelopment Analysis (DEA), which is a non-parametric mathematical method, was applied. In the second stage, panel data analysis, an econometric method, was employed to examine the factors affecting efficiency scores. During the construction of the dataset, special attention was paid to ensuring data continuity across all countries and years, and to avoiding missing data in the selected variables. In this context, the application part of the study covers 30 OECD countries and the data set for the years 2008-2020.

The DEA model in the analysis is the super-efficiency model. Unlike traditional DEA, the super efficiency model also allows for evaluating decision-making units at 100% efficiency. While there are decision units with the same efficiency level in traditional DEA, the ranking can be done more clearly with the super-efficiency model.

For the econometric model in the research, we first apply the F test to evaluate the suitability of the classical model. Then, we perform the LM test to determine the suitability of the random effects model. Finally, we use the Hausman test to choose between the fixed and random effects models. As a result of these tests, we did estimates based on the random effects in the applied model. In addition, based on the results of the model specification tests, the presence of issues such as heteroscedasticity, autocorrelation, and cross-sectional dependence was detected. Despite these problems, the random effects model was selected for the analysis, as it was found to be the most appropriate specification for the data. Against these deviations, we follow the Driscoll-Kraay (DK) method, which is one of the effective estimation methods.

The structure of this article is organized as follows: Chapters 1 and 2 provide the introduction and a review of the relevant literature. Chapter 3 outlines the research methodology. Section 4 presents the empirical findings along with a detailed discussion. Finally, Section 5 concludes the paper with a summary of the main results and offers policy recommendations.

LITERATURE REVIEW

Governments focus on achieving sustainable development and preventing environmental pollution with environmental protection expenditures within public expenditures. Researching the factors that determine the effectiveness of these expenditures is important information for public authorities. Various studies have been conducted within this framework, and some of them can be summarized as follows. To comprehensively understand the efficiency of environmental protection expenditures, this section reviews the relevant literature by grouping studies into three thematic categories: (1) studies using DEA only, (2) studies using DEA combined with regression analysis, and (3) studies focusing on OECD versus non-OECD countries.

Taskin & Zaim (2001) calculated environmental efficiency for selected years using DEA, incorporating employment and capital as inputs and GDP and CO_2 emissions as outputs. He et al. (2018) conducted a super-efficiency DEA to evaluate China's provincial ecological efficiency between 2013-2018. Similarly, Zhang et al. (2019) employed DEA to measure environmental protection expenditure efficiency in China using pollution reduction outputs. Barrell et al. (2021) applied DEA to 30 EU countries (2005-2015), showing that higher spending does not always result in better environmental outcomes.

Zaim (2004) applied pooled least squares alongside DEA to examine structural changes in the U.S. manufacturing sector. Li & Wang (2014) used Tobit regression to explore how economic development, fossil fuel usage, and trade openness influence environmental efficiency. Lacko & Hajduová (2018) applied a two-stage DEA to EU countries, analyzing greenhouse gases and GDP per capita. Wang (2018) studied Chinese provinces using DEA and Tobit regression, identifying GDP per capita as positively associated with efficiency, while urbanization and industrialization showed negative effects.

Shuai & Fan (2020) used a super-efficiency DEA and Tobit model to evaluate regional green economic efficiency in China. Jialu et al. (2022) applied a super-efficiency SBM model with OLS to assess government spending on environmental protection in China, finding that urbanization and population size negatively affect efficiency. Iram et al. (2020) used DEA-SBM and panel regression to assess OECD countries, noting that energy efficiency is a stronger determinant than economic growth.

Le Gallo & Ndiaye (2021) applied a spatial Durbin model on 28 OECD countries, revealing that nations often adjust environmental expenditures based on regional peers. Arjomandi et al. (2023) used PMG-ARDL to explore the dynamic effect of environmental policy stringency on environmentally adjusted GDP in OECD countries, showing that environmental spending boosts short-term output but may slow long-term

green productivity growth. Yasmeen et al. (2023) analyzed energy efficiency in OECD countries using Malmquist-Luenberger and super SBM-DEA models, finding that green technology adoption and environmental taxes significantly enhance energy productivity while reducing energy intensity across countries with varying efficiency levels.

MATERIALS AND METHODS

In the two-stage DEA, firstly, the relative efficiency of each decision-making unit is calculated; in the second stage, the efficiency score is subjected to regression using potential dependent variables to determine the factors that have a statistically significant effect on efficiency. Banker & Natarajan (2008) stated that using classical panel models instead of the widely used Tobit regression model in the second stage of two-stage DEA, the econometric model estimation would yield more consistent results. McDonald (2009) obtained results that support the study of Banker & Natarajan (2008) and argued that the use of Ordinary Least Squares (OLS) would be sufficient in two-stage DEA. Simar and Wilson (2011) criticized the regression models suggested by both McDonald (2009) and Banker & Natarajan (2008) in their study. They stated that applying OLS directly without performing the necessary econometric pre-tests would render the model inconsistent. In this study, we apply both the super-efficiency Data Envelopment Analysis (DEA) model and conventional panel data techniques.

In this study, the Simar & Wilson (2011) critique regarding the use of DEA efficiency scores in second-stage regressions is acknowledged. Although bootstrapping is a commonly proposed remedy to address the serial correlation and bias concerns in DEA-based efficiency scores, this study employs a super-efficiency DEA model, which is based on a different theoretical foundation than traditional DEA models. As noted in recent literature (e.g., Shuai & Fan 2020, Jialu et al. 2022), the super-efficiency approach enables a complete ranking of fully efficient decision-making units, making it suitable for second-stage econometric analysis. Furthermore, to mitigate econometric issues such as heteroscedasticity, autocorrelation, and cross-sectional dependence, robust estimation was conducted using the Driscoll-Kraay standard errors in the panel regression. Therefore, instead of applying the Simar and Wilson bootstrapping procedure, this study follows a two-stage DEA methodology with established econometric controls to ensure reliable inference.

Super Efficiency Model

Data envelopment analysis is an approach developed by Charnes et al. (1978) to measure the relative efficiency of decision-making units and has two basic assumptions. These assumptions are constant returns to scale developed by Charnes, Cooper and Rhodes (CCR) and variable returns to scale developed by Banker, Charnes and Cooper (BCC) (Charnes et al. 1978, Banker et al. 1984). In data envelopment analysis, CCR and BCC assumptions are applied as input-oriented and output-oriented. While the aim is to obtain the current output with minimum input in input-oriented data envelopment analysis, the aim is to obtain maximum output with current input in output-oriented data envelopment analysis. While both approaches give the same results under the constant returns to scale assumption, the results may differ under the variable returns to scale assumption (Coelli, 2005).

In addition to the models mentioned, the super efficiency model developed by Andersen and Petersen (1993) ranks the effective decision-making units among themselves, thus providing a clearer result in the efficiency ranking. Since the use of super-efficiency models increases the sensitivity of the data envelopment analysis, more stable and unique results are obtained. In addition, it can be more clearly revealed how the changes to be made by the decision-making units will affect the efficiency scores in question (Zhu 2001). In this context, by using the super-efficiency model in the study, the effective decision-making units are also ranked within themselves, allowing for a more comprehensive evaluation.

The Super-Efficiency model is a method derived from classical Data Envelopment Analysis (DEA) and allows effective Decision-Making Units (DMUs) to be compared with each other. It allows ranking of effective units by allowing their efficiency scores to take values greater than 1. In the super-efficiency model, the decision-making unit being evaluated is removed from the model, providing a differentiation between the effective units. In this case:

$$\min_{\theta,\lambda} \theta \qquad \ldots(1)$$

Subject to:

$$\sum_{j\neq 0}^{n} \lambda_j x_{ij} \leq \theta x_{i0}, \quad \forall i$$

$$\sum_{j\neq 0}^{n} \lambda_j y_{rj} \geq y_{r0}, \quad \forall r$$

$$\lambda_j \geq 0, \quad \forall j \neq 0$$

For a given number of decision-making units (DMUs), each DMU j (j=1,…, n) uses the following inputs and outputs:

x_{ij}: Use of i input by j DMU

y_{rj}: Production of r output by the j-th DMU

λj: Weights of DMUs

θ: Efficiency Score

In this model:

- If θ*>1, the unit is considered super-efficient and performs better compared to other efficient units.
- If θ*=1, the unit is efficient and is at the same level as other efficient units.
- If θ*<1, the unit is not efficient.

Super efficiency is used to rank especially efficient DMUs. Because in classical DEA, all efficient units have θ*=1, while, thanks to super efficiency analysis, it is possible to distinguish between these units.

Panel Regression Analysis and Driscoll-Kraay Estimator

There are 3 models in the classical panel data method: pooled least squares, fixed effects (FE) and random effects (RE) models.

In this context, the F test is used to determine the appropriate one between the fixed effects model and the pooled least squares (PLS) model, while the Breusch and Pagan (1980) LM Test is used to compare the random effects model and the pooled least squares regression model. If the null hypothesis is not rejected in both tests, the classical least squares model is preferred. In comparing fixed and random effects models, if the Hausman test rejects the null hypothesis that individual effects are unrelated to other independent variables, the fixed effects model is preferred to the random effects model (Das 2019).

$$= \frac{\frac{RRSS - URSS}{N - 1}}{\frac{URSS}{N(T-1) - k}}$$

H0: PLS Model Valid

H1: FE Model Valid …(2)

It is formulated as F test. In the F test formula, RRSS indicates the remaining sum of squares under the null hypothesis, and URSS indicates the remaining sum of squares under the alternative hypothesis. (Pesaran 2015).

$$LM = \frac{NT}{2(T-1)}\left[1 - \frac{\hat{u}'(I_N \otimes J_T)\hat{u}}{\hat{u}'\hat{u}}\right]^2$$

H0: PLS Model Valid

H1: RE Model Valid …(3)

Breusch & Pagan 1980 LM Test is formulated as above. If the estimated statistics reject the null hypothesis, the random effects model would be an appropriate model choice since it can be concluded that the heterogeneity present in the panel data and the nature of the heterogeneity are random. (Das 2019).

$$H = \left(\widehat{\beta_{RE}} - \widehat{\beta_{FE}}\right)' \left[\mathrm{Var}\left(\widehat{\beta_{FE}}\right) - \mathrm{Var}\left(\widehat{\beta_{RE}}\right)\right]^{-1} \left(\widehat{\beta_{RE}} - \widehat{\beta_{FE}}\right)$$

H0: RE Model Valid

H1: FE Model Valid …(4)

Hausman, (1978) The test is formulated as above. The null hypothesis underlying the Hausman test is that the fixed effects and random effects estimators are not significantly different. This test statistic has an asymptotic $\chi 2$ distribution. If the null hypothesis is rejected, the random effects model will consequently be inappropriate; instead, the fixed effects model would be more appropriate. (Gujarati 2003).

When performing panel data analysis, it is also important to check the existence of heteroscedasticity, autocorrelation and cross-sectional dependence errors in addition to model determination tests because the presence of these deviations will prevent the effectiveness of the estimation.

In this context, one of the tests recommended to determine whether there is a heteroscedasticity problem in the model is the Levene, Brown and Forsyth test. In this test, the hypothesis "H0 = There is no heteroscedasticity" is tested.

In addition, the Durbin-Watson and Baltagi-Wu tests are used to test whether there is an autocorrelation problem in the established model, and the hypothesis "H0 = There is no autocorrelation" is tested, and it is decided whether H0 will be accepted or rejected according to the critical value of the statistical value obtained (<2).

The last of the deviations that disrupts the effectiveness is the existence of cross-sectional dependence. Pesaran-CD and the Fees tests can be used to test whether there is cross-sectional dependence. In these tests, the hypothesis "H0 = There is no cross-sectional dependence" is tested.

$$y_{it} = \beta_0 + \beta_1 x_{1it} + \beta_2 x_{2it} + \beta_3 x_{3it} + \beta_4 x_{4it} + \beta_5 x_{5it} + u_{it} \quad \ldots(5)$$

In a panel data model, the random effects model is expressed as follows, with the dependent variable. , the independent variables and the error term . In the context of testing for biases that distort efficiency, the Driscoll-Kraay estimator is used in fixed and random effects models in cases where there are heteroscedasticity, autocorrelation and heteroscedasticity problems in the model. This method was developed by Driscoll & Kraay (1998) to avoid biased estimators for consistent coefficient analysis.

RESULTS AND DISCUSSION

In the first stage of the study, the effectiveness of public environmental protection expenditures will be calculated using DEA. Environmental protection expenditures will be

used as an input in the DEA. Verifying the effects of public environmental protection expenditures is important in the context of the discussion on the impact of fiscal policy on sustainable development (Krajewski 2016). While assessing public environmental protection expenditures as inputs, the studies by Jialu et al. (2022), Sun et al. (2016), Wang (2018), and Zhang et al. (2019) were used. In calculating the effectiveness of public environmental protection expenditures, P: M. 2.5 particulate matter exposure (Ma et al. 2021, Wu & Guo 2021), forest areas of countries (Arltová & Kot 2023, Barrell et al. 2021), renewable energy production of countries (Barrell et al. 2021, Koçak et al. 2021) and carbon dioxide emissions (Gómez-Calvet et al. 2020, Lacko et al. 2023, Zhou et al. 2016) were determined as outputs. All variables are shown in Table 1.

The study initially aimed to calculate the effectiveness of central government environmental protection expenditures in 30 OECD countries between 2008-2020. These countries are Australia, Austria, Belgium, Chile, the Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Israel, Italy, Japan, Latvia, Lithuania, Luxembourg, the Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey, United Kingdom. In the study, the effectiveness analysis of environmental protection expenditures of 30 OECD countries for 2008-2020 was carried out using the DEA method. Canada, Costa Rica, New Zealand, Colombia, Estonia, the United States, Mexico, and Korea from OECD countries were not included in the study due to various deficiencies in data. The data to be used as an input was obtained from the IMF database, and the data to be used as outputs were obtained from the World Bank, OECD and Energy Institute databases

After the efficient frontier is determined in DEA, the inefficient DMU can improve its performance to reach the efficient frontier by increasing current output levels or decreasing current input levels. Efficiency calculations are usually based on the assumption that inputs should be minimized and outputs should be maximized. However, especially in studies on environment and energy, undesirable outputs that should be minimized are included in the production model (Scheel 2001). Efficiency analyses using undesirable outputs will provide legislators with the opportunity to determine policies on how to manage desired outputs and improve environmental standards (Zofıo & Prieto 2001). Therefore, in studies using DEA, both desired (good) and undesirable (bad) factors are present (Seiford & Zhu 2002). The factors in question in this study are P.M. 2.5 exposure and CO2 emission outputs. These outputs are defined as "undesirable output" or "bad output". This is because it is not desired to maximize these outputs. In addition, in studies on energy and the environment (estimation of efficiency with pollutants or modelling environmental performance), DEA using undesirable outputs seems to be quite popular (Zhou et al. 2008). For this reason, in the study, the undesirable output (u) of CO_2, emission and PM 2.5 exposure was converted into the desired output with the transformation f (u) = 1/u (Golany & Roll 1989, Koçak et al. 2021, Scheel 2001).

Since a significant part of the studies on environmental protection strategies focuses on inputs rather than outputs, input-oriented DEA models were preferred in this study (Barrell et al. 2021). To determine the effectiveness of OECD countries' environmental protection expenditures, the EMS 3.1 program was used in the study, considering its ease of use and access.

Table 2 calculates the effectiveness of OECD countries' environmental protection expenditures using the Super Efficiency model for each year between 2008 and 2020. The difference between the super-efficiency model and traditional DEA is that it also evaluates the effective decision-making units. Therefore, it is possible that they cannot provide full effectiveness for decision-making units with an efficiency score below 100% in the analysis. However, decision-making units with an efficiency score above 100% can determine the most effective decision-making unit according to how much they exceed the efficiency limit. While decision-making units share the same efficiency order in traditional DEA, the order can be established more clearly in DEA using the super-efficiency model.

According to the super efficiency scores obtained as a result of the analysis, Chile, Finland, Germany and Iceland (except for 2008) stand out among 30 countries in terms of the efficiency of public environmental protection

Table 1: Variables Used in the Super Efficiency Model and Their Explanations.

Variables	Indicators	Values	Source
Output 1- Renewable Energy	Renewable Energy Production	Twh	Energy Institute
Production	Forest Area	Km^2	World Bank
Output 2- Forest	Average P.M. 2.5 per capita exposure	M^3 microgram	OECD
Output 3- P.M. 2.5.	CO2 Emissions	CO2 equivalent,	
Output 4- CO_2	Environmental Protection Expenditures	thousand tonnes	OECD
Input 1- EPE		% GDP	IMF

Table 2: Super efficiency analysis on public environmental protection expenditures.

KVB		2008	2009	2010	2011	2012	2013	2014	2015	2016	2017	2018	2019	2020
1	Australia	0.9267	0.9406	0.8023	0.7433	0.6652	0.665	0.7138	0.8682	0.8798	0.8473	0.7969	0.8894	0.8989
2	Austria	0.2486	0.2233	0.1955	0.2283	0.2415	0.266	0.3036	0.2935	0.3136	0.3012	0.2845	0.2749	0.2988
3	Belgium	0.1108	0.1098	0.0876	0.0782	0.0781	0.0842	0.1032	0.0983	0.1026	0.0837	0.0853	0.0889	0.0945
4	Chile	1.9897	1.7571	2.0039	1.9164	2.0551	2.04	1.9004	1.6743	1.6833	1.7354	1.8065	1.5961	1.6469
5	Czech Republic	0.1044	0.1426	0.0789	0.0678	0.0671	0.091	0.0923	0.0788	0.1103	0.0898	0.0838	0.0917	0.096
6	Denmark	0.3511	0.401	0.3956	0.431	0.4391	0.3932	0.4307	0.435	0.4439	0.4092	0.4027	0.4374	0.4994
7	Finland	0.826	0.8017	0.8476	1.0847	1.1761	1.3429	1.3557	1.6003	1.8502	2.0584	1.902	2.1857	2.3342
8	France	0.1656	0.1811	0.1786	0.1914	0.1991	0.2001	0.2182	0.2051	0.2182	0.2032	0.209	0.2195	0.2481
9	Germany	2.6987	2.8052	2.8491	3.0434	2.6878	2.472	3.0951	2.8925	2.317	2.104	1.7478	1.9008	1.839
10	Greece	0.0878	0.1027	0.1025	0.0958	0.0811	0.06	0.0741	0.0663	0.0654	0.0626	0.0651	0.0688	0.0723
11	Hungary	0.1482	0.1667	0.1444	0.1305	0.1469	0.1122	0.0935	0.0782	0.1872	0.1281	0.1389	0.1264	0.1389
12	Iceland	1.9549	0.4841	1.2326	2.7755	2.7132	2.7687	2.3301	2.4578	2.0122	1.7953	1.9376	2.0495	1.4929
13	Ireland	0.1516	0.1706	0.1764	0.2294	0.2523	0.3122	0.3366	0.37	0.3621	0.3525	0.3872	0.3999	0.4389
14	Israel	0.1192	0.1422	0.1214	0.1351	0.1407	0.1363	0.1535	0.1296	0.1397	0.1263	0.132	0.1261	0.1368
15	Italy	0.1981	0.2327	0.2507	0.2784	0.3123	0.3203	0.3133	0.2678	0.2761	0.253	0.2489	0.2449	0.2451
16	Japan	0.2269	0.2105	0.2442	0.2115	0.2155	0.2312	0.2455	0.2598	0.259	0.2642	0.2877	0.3036	0.3161
17	Latvia	0.3606	2.5161	0.8829	0.3756	0.3855	0.3821	0.4532	0.4285	0.5288	0.5829	0.5411	0.5147	0.7012
18	Lithuania	0.2475	0.1325	0.1404	0.2732	0.239	0.3579	0.3522	0.3302	0.3788	0.4401	0.4401	0.3865	0.4685
19	Luxembourg	0.3382	0.1862	0.2935	0.2989	0.3133	0.2795	0.3577	0.3247	0.3566	0.3361	0.3269	0.307	0.3537
20	Netherlands	0.0825	0.0898	0.0843	0.0874	0.0888	0.0905	0.0994	0.0916	0.0896	0.0797	0.0845	0.0909	0.1107
21	Norway	0.3411	0.2784	0.2642	0.2524	0.2887	0.2793	0.2735	0.2301	0.2158	0.1867	0.1975	0.1846	0.1798
22	Poland	0.1183	0.1374	0.1212	0.1453	0.1886	0.1757	0.1879	0.1864	0.2623	0.2456	0.1803	0.1954	0.2112
23	Portugal	0.2443	0.3065	0.2443	0.2466	0.2891	0.2919	0.3409	0.2831	0.3064	0.2349	0.243	0.2468	0.2471
24	Slovakia	0.1258	0.1054	0.1059	0.1242	0.1202	0.1142	0.1401	0.1068	0.1308	0.12	0.1221	0.1262	0.1331
25	Slovenia	0.205	0.1198	0.2308	0.1864	0.2091	0.1961	0.1824	0.1557	0.2735	0.374	0.3245	0.3176	0.3596
26	Spain	0.341	0.4041	0.3993	0.3767	0.3984	0.4594	0.4074	0.347	0.3528	0.3055	0.3058	0.3094	0.3333
27	Sweden	0.4983	0.5699	0.5515	0.5478	0.5779	0.5724	0.6102	0.6167	0.5883	0.472	0.4187	0.45	0.5053
28	Switzerland	0.2391	0.2113	0.2149	0.2298	0.2392	0.2272	0.2874	0.2296	0.2246	0.2234	0.2434	0.2445	0.2645
29	Republic of Türkiye	0.263	0.3096	0.2915	0.2624	0.2981	0.3349	0.3787	0.347	0.3716	0.3675	0.4096	0.5668	0.7408
30	United Kingdom	0.2184	0.2427	0.2376	0.2746	0.3092	0.3818	0.3923	0.4182	0.4291	0.4327	0.5126	0.5425	0.5873
	OECD	0.46438	0.48272	0.45912	0.510733	0.513873	0.521273	0.540763	0.529037	0.52432	0.507177	0.495533	0.516217	0.533097

expenditures. Germany, in particular, has demonstrated a very successful performance in terms of the efficiency of environmental protection expenditures in all years except 2018. On the other hand, Turkey experienced a break in 2008, and its efficiency scores were largely stable between 2009 and 2017. However, Türkiye's efficiency scores have continuously increased remarkably since 2017.

It is seen that the average efficiency scores of public environmental protection expenditures of OECD countries that applied DEA between 2008 and 2020 are in the range of 50%. This means that although there are big differences in efficiency scores between countries, it is possible to say that the OECD has caught a trend for the relevant period.

The second stage of the study, the panel data analysis, will be used to determine the factors determining the efficiency of public environmental protection expenditures of 30 OECD countries. The literature review conducted to determine the variables for the econometric method to be applied in this section of the study has guided. In the econometric model to be applied to estimate the determinants of the effectiveness of public environmental protection expenditures, super efficiency scores (Jialu et al. 2022, Shuai & Fan 2020) were determined as the dependent variables and population density (Antonelli & De Bonis 2019, Iram et al. 2020, Jialu et al. 2022, Tu et al. 2017), GDP per capita (Jia & Liu 2012, Jialu et al. 2022, Yasmeen et al. 2023), urban population (Wang 2018, Tu et al. 2017), industrialization (Wang 2018) and primary energy intensity (Shuai & Fan 2020) were determined as the independent variables. The econometric model to be used in the study within the framework of the determined variables.

$$\log SES_{it} = \beta 0_{it} + \beta 1 \log PD_{it} + \beta 2 \log\_GDPPC_{it} + \beta 3 UP_{it} + \beta 4 I_{it} + \beta 5 PIE_{it} \quad \ldots(6)$$

Information about the variables used in the model is given in Table 3.

In Table 4, the results of the tests performed to decide on the estimation method of the model are given in the table. According to the results of the F test conducted to decide between the classical model and the fixed effects model, the H0 hypothesis suggests the classical model is rejected, and the fixed effects model comes to the fore.

Table 3: Variables used in the panel regression analysis and their explanations**.**

Variable	Abbreviated	Definition	Source
Super Efficiency Scores	SES	Environmental Protection Expenditure Efficiency Score	Our Own Calculation
Population Density	PD	Number of people per km2	World Bank (2024)
GDP per Capita	GDPPC	GDP per capita in dollars	World Bank (2023)
Industrialization	I	Percentage in GDP	World Bank (2024)
Urban Population	UP	Percentage of total population	World Bank (2024)
Primary Energy Intensity	PIE	Ratio of primary energy consumption to GDP	Energy Institute (2023, 2024)

Table 4: Model determination tests**.**

	F Testi	Breusch- Pagan LM Testi	Hausman Testi
Test Statistics	132.64 (0.00)*	1918.38 (0.00)*	2.31 (0.8052) *

Note: The values in parentheses are significance (prob.) values.
* Indicates statistical significance at the 1% level.

Table 5: Deviations that deteriorate effectiveness.

Deviations That Deteriorate Effectiveness	Tests	Test Statistics	p-values	Null Hypothesis	Decision
Heteroscedasticity	Levene, Brown ve Forsyth	W0 = 4.72	0.000000	H_0: No Heteroscedasticity	Rejected
		W50= 3.375	0.000000		
		W10 =4.146	0.000000		
Autocorrelation Test	Durbin-Watson	0. 96803598	0.000	H_0: No Autocorrelation	Rejected
	Baltagi–Wu LBI	1.1447572	0.000		
Cross-sectional dependency test	Pesaran -CD	2.703	0.0069	H_0: No Cross-Section Dependence	Rejected
	Frees	*2.332*	*0.000*		

*The critical value used for the Durbin-Watson and Baltagi-Wu tests is 2.

**The critical value for the Frees test at a 95% confidence level is 0.2620.

According to the Breusch-Pagan LM test results conducted to decide between the classical model and the random effects model, the H0 hypothesis suggests the classical model is rejected, and the random effects model comes to the fore. Finally, according to the results of the Hausman test conducted to decide between the fixed effects model and the random effects model, the H0 hypothesis suggests the random effects model is accepted. Thus, the fixed effects model is invalid. As a result of this evaluation, it was decided that the most appropriate model for the study was the random effects model.

The results of the tests conducted on heteroscedasticity, autocorrelation and cross-sectional dependency of the model used in the study are given in Table 5. According to the Levene, Brown, and Forsyth test results conducted to determine whether there is a heteroscedasticity problem, H0 was rejected because the variances of the units were not equal, and it was seen that there was a heteroscedasticity problem. Durbin-Watson and Baltagi-Wu tests were applied to test whether there was an autocorrelation problem in the model used. The statistical values obtained from the tests are less than 2, which is the critical value for the random effects model. In this case, it is possible to talk about the existence of first-degree autocorrelation in the random effects model used in the study. Finally, the existence of inter-unit correlation, i.e., cross-sectional dependency, in the model was tested using the Pesaran-CD and Fees tests. According to the results obtained, both the Pesaran-CD and the Fees tests gave the same result; it was concluded that there was cross-sectional dependency in the model used in the study.

Table 6: Determinants of the effectiveness of environmental protection expenditures, re and dk estimation results

Depended var: logSES, sample (N):30 countries, T: 13 years (2008-2020), N*T= 390		
Variables	RE (Random Effect)	DK (Driscoll-Kraay)
Constant	-2.234 (0.123)	-2.234 (0.152)
logPD	-0.465 **(0.000) ***	-0.465 **(0.000) ***
logGDPPC	0.0879 (0.394)	0.088 (0.499)
UP	0.0285 **(0.001) ***	0.0285 **(0.011) ***
I	0.027 **(0.000) ***	0.027 **(0.025) ***
PIE	-0.1555 **(0.000) ***	-0.1555 **(0.019) ***
R^2	0.3596	0.3453

Note: The values in parentheses are significance (prob.) values.
* Indicates statistical significance at the 1% level.

As a result of the model determination tests, all deviations (heteroscedasticity problem, autocorrelation, and cross-section dependency) that disrupted the effectiveness of the random effects model that was decided to be used in the study were detected. Therefore, in the study, an estimate was made using the Driscoll-Kraay (DK) method, which is one of the effective estimation methods, against these deviations.

The analyses conducted to estimate the determinants of the effectiveness of public environmental protection expenditures are given in Table 6. According to the results obtained from both estimation methods, population density, urbanization, industrialization, and primary energy density yielded significant results. In this context, the independent variables of population density (logPD) and primary energy density (PIE) in the model have a negative and significant relationship with the dependent variable super-efficiency scores (logSES). This result, which is in line with expectations, shows us that as countries' population density and primary energy density increase, the expected level of effectiveness from environmental protection expenditures decreases. According to the analysis results, a 1% increase in population density will cause a 0.47% decrease in environmental protection expenditures. In comparison, a 1% increase in primary energy density will cause a 0.15% decrease in the effectiveness of environmental protection expenditures.

Urbanization (UP) and industrialization (I) in the model have a positive and significant relationship with the dependent variable, super efficiency scores (logSES). This result shows us that a 1% increase in urbanization in OECD countries will provide a 0.029% increase in the effectiveness of environmental protection expenditures. At the same time, a 1% increase in industrialization will give a 0.027% increase in the effectiveness of environmental protection expenditures. It is seen that the logGDPPC variable, another variable in the model, the results suggest that the variable in question has no meaningful impact on the effectiveness of environmental protection expenditures. When the results obtained are compared with the literature, it is noteworthy that there are results consistent with the literature and that differ.

Population density, one of the independent variables used in the study, has a negative and significant relationship with super-efficiency scores. According to the analysis, a 1% increase in population density causes a 0.47% decrease in the effectiveness of environmental protection expenditures. Another independent variable used in the study is primary energy density. According to the results of the analysis, a negative and significant relationship exists between primary energy density and super-efficiency scores. The analysis

results indicate that a 1% increase in primary energy density causes a 0.15% decrease in the effectiveness of environmental protection expenditures.

It is seen that there is a significant and positive relationship between urbanization, one of the variables included in the analysis, and the effectiveness of environmental protection expenditures. According to the results of the analysis, a 1% increase in urbanization will provide a 0.029% increase in the effectiveness of environmental protection expenditures. Therefore, it is possible to say that the increase in urbanization increases the effectiveness of environmental protection expenditures.

Another variable included in the analysis is industrialization. According to the results obtained from the analysis, there is a positive and significant relationship between the effectiveness of environmental protection expenditures and industrialization. According to the results of the analysis, a 1% increase in industrialization rates provides a 0.027% increase in the effectiveness of environmental protection expenditures.

The last variable used in the analysis is GDP per capita, and it did not show a significant result on the effectiveness of environmental protection expenditures. Although per capita national income is seen as one of the factors causing environmental problems, it was found in the study that it did not affect the effectiveness of environmental protection expenditures. The most important reason for this is that the effectiveness of expenditures was measured, not environmental effectiveness. However, significant results were obtained in the studies using this variable in the literature.

DISCUSSION

Public environmental protection expenditure is the main object of protecting the environment and encouraging the sustainable use of natural resources. Also, there is a main contribution of the public environmental protection expenditure as a fiscal policy on sustainable development. As a result, the research validates which factors affect the efficiency of public environmental protection expenditure. For this purpose, we focus on what determines the effectiveness of environmental protection expenditures. Therefore, it is significant to have metrics that measure the effectiveness of environmental protection expenditures

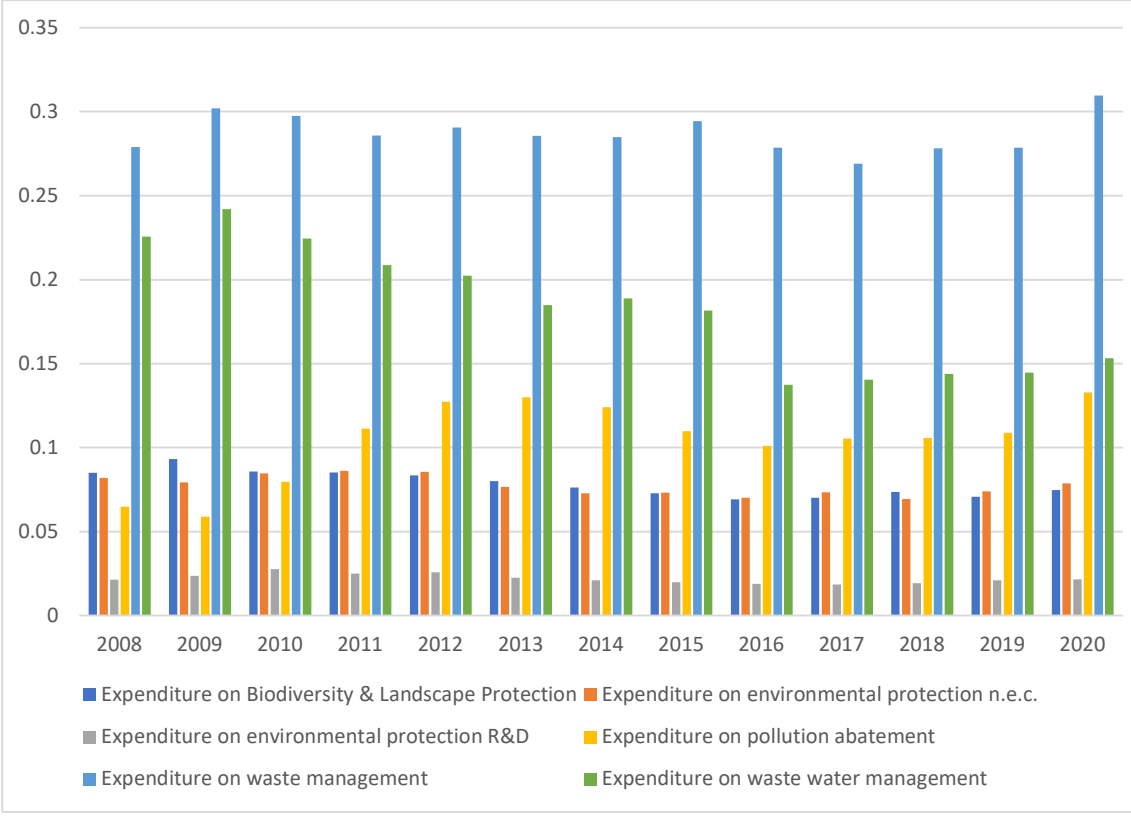

Fig. 1: OECD averages of types of environmental protection expenditures, 2008-2020.

and include information about the results. Using the data envelopment analysis (DEA) methodology, it is possible to measure the efficiency of these expenditures. DEA also allows comparing performance measures of countries with similar scores and determining which countries are performing best, given the inputs they use and the outputs they produce.

The functional classification system of public expenditures (COFOG) has divided environmental protection expenditures into six main headings. Countries classify each environmental protection expenditure they make under this heading and transparently share the purpose of these expenditures: These expenditures are waste management, wastewater management, pollution abatement, biodiversity protection, R&D expenditures related to environmental protection, and expenditure on environmental protection not elsewhere classified (n.e.c.). The subject of this study is how the effectiveness of the expenditures made with this classification affects environmental problems.

As shown in Fig. 1, the average composition of environmental protection expenditures in OECD countries reveals that waste management and wastewater management consistently represent the largest share of total spending between 2008 and 2020. This indicates a clear prioritization of basic environmental services within public expenditure structures.

Increasing population density, on the other hand, has an adverse impact on the effectiveness of environmental protection expenditures. Because of resource use, infrastructure needs and environmental degradation accelerate in regions with more people. This requires environmental protection expenditures to be more extensive and comprehensive. In addition, environmentally friendly solutions such as public transportation systems and green areas may be insufficient as the population density increases, and environmental protection expenditures may be insufficient to solve these problems. Wang (2018), Jialu et al. (2022), and Le Gallo & Ndiaye (2021) also emphasized in their studies that the priority of expenditures to be made in regions with high population density will change towards other needs of the population. Moreover, the control difficulties brought by the dense population in highly populated regions also reduce the effectiveness of environmental expenditures.

In other words, increasing primary energy density negatively affects the effectiveness of environmental protection expenditures. Primary energy density expresses the ratio of primary energy resources used in production to national income. The primary resources mentioned here are fossil fuels, primarily oil. In today's world, where the importance of green transformation, green industrial zones, and combating waste is discussed, the fact that production is carried out with primary energy resources also reduces the effectiveness of environmental protection expenditures. Shuai & Fan (2020), Li & Wang (2014), and Lacko et al. (2023) also showed in their studies that primary energy density has a negative effect on environmental efficiency. However, Donkor et al. (2022) observed that fossil fuel consumption does not significantly affect environmental quality.

With increasing urbanization, the efficient use of resources in cities, the need for green infrastructure, the fight against waste, and environmental investments in environmentally friendly technologies have increased. In addition, urbanization increases environmental awareness by providing more economic and social development; thus, society's demand for environmental protection expenditures increases, and these expenditures reach wider audiences. In line with the results obtained in Le Gallo & Ndiaye's (2021) studies, they stated that countries with high urbanization rates make more environmental expenditures, increasing the demand for environmental quality by increasing public awareness. However, while our findings reveal a statistically significant positive relationship between urbanization and industrialization and efficiency scores, it is important to interpret this cautiously. Urbanization and industrialization can also exert additional pressure on the environment through increased pollution, land use, and resource consumption. For instance, Shuai & Fan (2020) highlight that the environmental effects of urbanization vary significantly across regions. Therefore, for environmental expenditures to be effective under growing urbanization and industrialization, they must be supported by strong environmental regulations and targeted investments in green technologies and infrastructure.

There are a number of studies in the existing literature inconsistent with our results (Wang 2018, Li & Wang 2014). According to these studies, increasing industrialization will increase environmental pressures, negatively affecting expenditures' effectiveness. Nevertheless, He et al. (2018) have obtained consistent results with our study. Accordingly, the support of green organized industrial zones by countries with growing production capacities, the support of companies to zero-emission policies, and the public, encouraging companies to use renewable energy by making various public expenditures, are activities that can positively affect the effectiveness of environmental protection expenditures.

According to Wang (2018) and Li & Wang (2014), economic growth has both positive and negative effects on environmental effectiveness and the efficiency of protection expenditures. Indicated by Donkor et al. (2022) and Jialu et

al. (2022), while the increase in per capita GDP contributes to environmental improvements, excessive economic growth in some regions can lead to environmental degradation, such as CO_2 emissions. Noted by He et al. (2018), a balance should be established between economic growth and environmental policies, and environmental protection expenditures should be aligned with sustainable development goals.

CONCLUSIONS

According to our research, environmental protection expenditures made by states are used more effectively in places where urbanization and industrialization increase; however, the situation is vice versa in cases where population density and primary energy density increase. Environmental protection expenditures made to combat problems such as waste management, infrastructure investment, or air pollution, which increase with urbanization and industrialization, may produce more successful results in urbanizing and industrializing regions since they try to meet social needs. Although urbanization and industrialization are seen among the factors that positively affect the effectiveness of environmental protection expenditures in the econometric model, an important point that draws attention is the smallness of the coefficient in the effect they create (for both determinants, nearly 0.028). This situation indicates that urbanization and industrialization are determinants of the effectiveness of environmental protection expenditures, but this effect is not sufficient.

These findings offer policy-relevant implications. For instance, in regions with high population density and primary energy intensity, where expenditure efficiency is lower, governments may focus on improving public transport systems, optimizing energy use, and implementing environmental regulations tailored to local needs. Conversely, in urbanized and industrialized areas, expanding green infrastructure and supporting clean technologies can enhance the marginal impact of environmental expenditures.

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Biopolymers Production Strategies and Their Usage as Clean Material for Environmental Remediation

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ABSTRACT

The renewable sources, biodegradability, and customizable physicochemical features of biopolymers make them viable alternatives to synthetic materials. Their use in wastewater, air, and soil remediation offers promising answers to pollution problems. This comprehensive analysis encompasses the natural extraction, microbial biosynthesis, and chemical polymerization of biopolymers. Chitosan, alginate, bacterial cellulose, and polyhydroxyalkanoates (PHAs) are excellent biopolymers for wastewater treatment because they effectively adsorb heavy metals, dyes, and organic contaminants. Additionally, biopolymer-based membranes, composites, and hydrogels are garnering attention for air filtration and soil stabilization. Functional modifications have enhanced the efficiency and environmental sustainability of biopolymers through the application of synthetic biology and nanotechnology. This paper explores the potential of biopolymer-based environmental remediation technologies to replace synthetic materials in sustainable pollution management, highlighting recent advances, challenges, and prospects.

INTRODUCTION

The escalating environmental burden from non-biodegradable synthetic polymers, derived primarily from fossil fuels, has become a significant global concern because of their persistence, toxicity, and contribution to pollution in water, soil, and air. These materials not only deplete non-renewable resources but also generate long-lasting waste that threatens ecosystems and human health (Kibria et al. 2023, Islam et al. 2024). In response, biopolymers have garnered increasing attention as a sustainable alternative. Derived from renewable sources and often biodegradable, biopolymers offer promising advantages in environmental compatibility, resource efficiency, and functional versatility. This review examines recent strategies in biopolymer production and their emerging applications in environmental remediation, highlighting how these natural and engineered materials can effectively replace synthetic polymers to mitigate pollution and support a circular bioeconomy (Samir et al. 2022, Edo et al. 2025).

Biopolymers play a vital role by competing with non-biodegradable synthetic polymers, offering unique advantages such as eco-friendliness and high biodegradability. Moreover, they can be biosynthesized from various biological resources. Biopolymers possess significant market potential due to their extensive range of applications. Biopolymers are found in multiple sources, including microbial and animal origins, and most are obtained from agricultural waste. Lignocellulosic-based agricultural residues are gaining market traction from

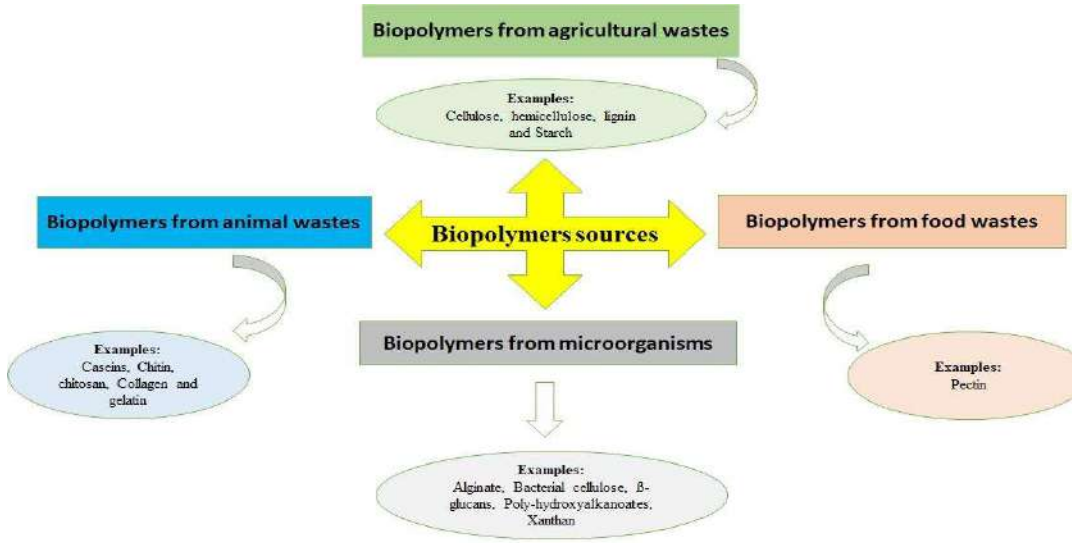

Fig 1: Biopolymers sources.

Table 1: Biopolymer origin, synthesis, and sources.

Biopolymer origin	Types	Examples	Sources
Biomass	1. Polysaccharides	Starch, cellulose, chitosan, alginate, carrageenan, pectin, and gums or their derivatives.	wild or genetically modified microorganisms
	2. Proteins	Gelatin, casein, whey, and collagen	Animal and plant origin
	3. Lipids	Waxes	Beeswax and carnauba wax
Synthesized from bioderived monomers		Polylactic acids (PLA)	Renewable agro-wastes
Bioderived monomers	-	Poly(hydroxyalkanoate)s (PHAs), poly(hydroxybutyrate)s (PHBs), bacterial cellulose, xanthan, gellan, pullulan.	Wild or genetically modified microorganisms

agricultural wastes due to their substantial global production (Rai et al. 2021, Iqbal et al. 2025). Biopolymers are defined as large molecules synthesized by microbial, plant, and animal cells, composed of highly repetitive chemical units. Fig. 1 illustrates various natural sources of biopolymers, along with examples.

The biochemical composition of biopolymers primarily comprises polysaccharides (cellulose, starch, chitosan, chitin, alginic acid, hyaluronic acid, and pectin), proteins (collagen, elastin, albumin, fibrin, gluten, and soy proteins), and nucleic acids (DNA and RNA), with primary sources derived from plant, animal, and microbial origins. Investigating the physical, chemical, biological, and mechanical properties of biopolymers enables their application in various industries, including food, pharmaceuticals, medicine, and environmental sectors (Hassan et al. 2019). Biopolymers synthesized through natural processes, including bioplastics, pullulan, dextran, xanthan, bacterial cellulose, microbial exopolysaccharides, and capsular polysaccharides, are widely utilized in medical, agricultural, agro-industrial, packaging, and environmental applications (Francis et al. 2013, Chaabouni et al. 2014, Manubolu et al. 2024, Lad et al. 2024). Based on the literature and data, biopolymer production methods include extraction from agricultural waste and animal sources, as well as synthesis via classical chemical methods (e.g., polylactic acid, PLA). Additionally, polymers are produced from indigenous and genetically modified microorganisms. Table 1 shows various biopolymer production strategies. According to the literature, technical advancements in synthesizing biopolymers from natural sources and bioderived feedstocks have been noted (Volf and Popa 2018, Chen et al. 2019, George et al. 2020).

NATURAL BIO-BASED POLYMERS CAN BE HARNESSED WITH PARTIAL MODIFICATION AS AN EFFECTIVE PRODUCTION STRATEGY

In recent years, bio-based polymers have seen a surge in demand for their versatile applications. Primarily, modification of functional groups and their properties is sought in recent technical advancements to meet our

industrial applications (Das et al. 2024). Table 1 presents three significant types of biopolymers, along with their origins and sources.

Functionality of Biopolymer

The production of biopolymers from renewable biomass has become one of the most widely adopted sustainable alternatives to fossil-fuel-based synthetic polymers. Unlike traditional plastic synthesis, which relies on nonrenewable petrochemicals and generates persistent waste, biopolymers are often biodegradable, non-toxic, and derived from abundant natural resources. This shift aligns with growing environmental regulations and rising consumer demand for eco-friendly materials (Pinaeva & Noskov 2024, Jha et al. 2024).

Among the most promising strategies in recent years is the cell factory approach, in which microorganisms are genetically engineered to convert simple carbon sources—typically glucose, glycerol, or lignocellulosic hydrolysates-into high-value polymer precursors. Glucose, in particular, is favored for its low cost, wide availability, and compatibility with many microbial systems. Through in vivo chemical synthesis and metabolic engineering, researchers have significantly advanced the microbial biosynthesis of various biopolymer building blocks (Mitra et al. 2020, de Souza & Gupta 2024).

Over the past three decades, several notable milestones have been achieved in this field:

Glucaric acid: Produced using engineered E. coli strains (Moon et al. 2009), glucaric acid is a precursor to biodegradable polyesters and has potential applications in detergents, hydrogels, and biomedical devices. Its production exemplifies how central metabolism can be rerouted to yield value-added products from glucose.

Putrescine: This diamine, synthesized by Corynebacterium glutamicum and E. coli (Qian et al. 2009), serves as a monomer for nylon-4,6, a biodegradable polyamide. Biosynthetic production of putrescine replaces the energy-intensive petrochemical routes typically required for polyamide synthesis.

3-Hydroxybutyrate (3HB): A key monomer in the synthesis of polyhydroxybutyrate (PHB), 3HB is produced by various bacteria, including Ralstonia eutropha (Jung et al. 2010). PHB exhibits thermoplastic properties similar to polypropylene, making it a potential substitute for petroleum-derived plastics in packaging and agriculture.

1,4-Butanediol (BDO): Traditionally produced via petrochemical synthesis, BDO is now biosynthesized by engineered microbes, including E. coli and Clostridium species (Oliver et al. 2013, Kumar et al. 2020). BDO is a versatile precursor to biodegradable plastics, including polybutylene succinate (PBS) and polybutylene terephthalate (PBT).

These advances underscore not only the functional versatility of biopolymers but also the potential for modular customization, enabling the design of polymers with specific mechanical, thermal, or chemical properties tailored to diverse applications, including biomedicine, agriculture, packaging, textiles, and electronics. Significantly, the functionality of biopolymers is determined not only by their monomeric composition but also by molecular weight, branching, crystallinity, and interactions with other molecules. Advances in synthetic biology and protein engineering now enable researchers to fine-tune these properties by modifying biosynthetic enzymes or incorporating non-natural building blocks into the polymer backbone (Arif et al. 2022, Khalil et al. 2025).

Despite these successes, current biosynthetic approaches face several technical limitations. Low titers and yields in industrial-scale fermentation, high recovery and purification costs, limited host tolerance to toxic intermediates, and substrate competition within central metabolism all constrain growth and productivity. To overcome these challenges, efforts are directed toward optimizing host strains, developing co-culture systems, and integrating dynamic pathway regulation to balance growth and production. Furthermore, combining metabolic engineering with process innovations such as continuous fermentation or in situ product recovery is expected to enhance overall efficiency and reduce costs. The functionality of biopolymers derived from biomass not only fulfills sustainability goals but also offers a broad spectrum of application-specific properties. Continued innovation in microbial engineering and bioprocess design will be essential for translating these materials into scalable, commercially viable solutions (de Souza & Gupta 2024, Del Hierro et al. 2024).

Synthetic Biology as a Tool to Modify Biopolymers

Synthetic biology has emerged as a transformative tool for modifying and producing biopolymers with enhanced efficiency, precision, and sustainability. Traditional one-step microbial production of polymers, while promising, often suffers from low yields, slow growth rates, and inefficient substrate conversion, particularly with wild-type or unoptimized strains. Moreover, these processes typically rely on chemical catalysts and harsh solvents for polymer extraction and purification, increasing environmental and economic burdens (Anderson et al. 2018, Kaur et al. 2024).

Commonly produced biopolymers include chitin, alginate, polylactic acid (PLA), and polyhydroxyalkanoates

(PHAs). For instance, chitin, extracted primarily from crustacean shells, is limited by its animal origin, raising sustainability and allergenicity concerns. Similarly, alginate, derived from brown algae, faces challenges due to seasonal availability and batch variability, which can affect product consistency. PHAs and PLA, although microbial in origin, often require complex feedstocks and multiple downstream purification steps because of mixed metabolic byproduct accumulation (Sharma et al. 2024, Kaur et al. 2024).

A key limitation of conventional metabolic engineering is the difficulty in controlling pathway fluxes, which can lead to unintended accumulation of intermediates or metabolic burden that compromises cell growth. Additionally, the limited range of naturally occurring monomers restricts the mechanical and functional diversity of biopolymers, limiting their potential applications. Synthetic biology addresses these challenges by enabling fine-tuned control of gene expression, modular pathway design, and the incorporation of non-natural monomers (Aravind et al. 2015, 2016, Arif et al. 2024). For example, engineered strains of E. coli have been developed to produce cellulose nanofibers with customized lengths and crystallinity. At the same time, synthetic pathways in Cupriavidus necator have been used to create novel polyhydroxyalkanoates (PHAs) with side chains that confer elasticity and biodegradability. These modifications not only enhance the functional properties of the polymers but also streamline production by eliminating unnecessary enzymatic steps (Zhang et al. 2022).

CRISPR-based genome editing and biosensor-guided pathway optimization have enabled dynamic regulation of biosynthetic pathways, allowing microbial systems to adjust in real time to fluctuations in precursor availability or metabolic stress. This results in more robust production systems that are resilient under industrial fermentation conditions (Xin et al. 2025). However, challenges remain. Many engineered strains still face scale-up issues, including instability of synthetic pathways during prolonged fermentations and sensitivity to industrial stressors, including pH and shear forces. Moreover, regulatory and safety concerns around the use of genetically modified organisms (GMOs) in open environments or consumer products may slow the commercial deployment of such technologies. Despite these limitations, the integration of synthetic biology with computational modeling, machine learning, and high-throughput screening holds promise to accelerate the development of next-generation biopolymers. These polymers can be fully bio-based, biodegradable,

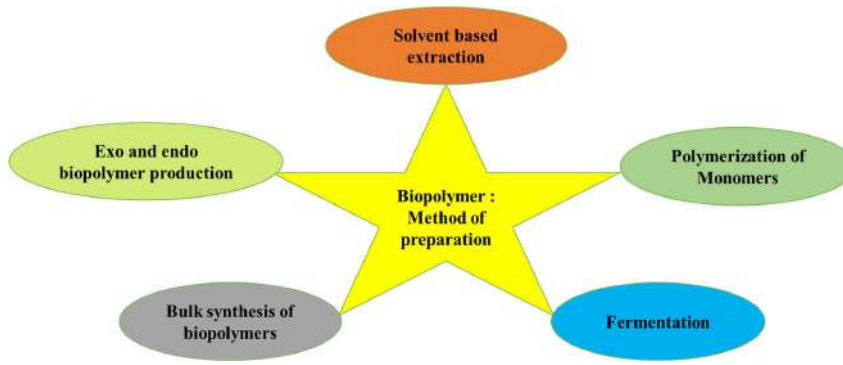

Fig. 2: Method of preparation of biopolymers.

Table 2: Overview of the biopolymer synthesis pathway.

Direction of polymer synthesis	Glucose	Glucose	Glucose	Glucose
	Glucose 6-phosphate	Glucose 6-phosphate	Acetyl coenzyme A	Glucose 6-phosphate
	Glucose 1-phosphate	Glucose 1-phosphate	Oxaloacetate	Fructose 6-phosphate
	Glucuronic acid - glucose	Glucuronic acid - glucose	Fructose 6-phosphate	Glucosamine 6-phosphate
	Cellulose	N-acetylglucosamine 1-phosphate	Mannose 6-phosphate,	N-acetylglucosamine 6-phosphate
		UDP-N-acetylglucosamine	Mannose 1-phosphate	N-acetylglucosamine 1-phosphate
		Hyaluronan	GDP-mannose	UDP-N-acetylglucosamine
			GDP-mannuronic acid	Chitin
			Alginate	Chitosan

and tailored for specific applications (Palladino et al. 2024).

PRODUCING BIO-BASED MONOMERS BY FERMENTATION AND/OR VIA CONVENTIONAL CHEMISTRY FOLLOWED BY POLYMERIZATION

Method of Preparation

Biopolymers possess excellent biological and biodegradable properties, but they lack specific mechanical properties, including low chemical resistance, limited processing capacity, and short storage duration. Various methods can be implemented to achieve maximum yield while retaining the properties of biopolymers and overcoming challenges (Pinaeva & Noskov 2024). Fig. 2 illustrates various methods for preparing biopolymers.

Fermentation: This method utilized bacteria, fungi, and algal species to produce specific types or different groups of biopolymers, which were produced using specific substrates as the sole carbon source (Chang et al. 2015). Major biopolymers (Alginate, bacterial cellulose, dextran, Hyaluronic acid, etc.) use glucose and/or sucrose as primary substrates. Very few groups of polymers (Gellan and pullulan) are produced using industrial waste as substrate. Table 3 provides detailed information on the types of polymers, substrates used for Fermentation, and polymer-producing microorganisms.

Polymerization: The monomeric form of polymers is highly prepared for the synthesis of microstructures. In this method, the polymerization of monomers occurs in a series of sequential reactions, with each step representing the functionalities of the monomers and their steric effects. For example, in the formation of alkene units, more straightforward steps are required, whereas carbonyl groups necessitate more complex steps. In the presence of strong acids, alkane units are polymerized (Doyle et al. 2010). Similarly, the production of Polycaprolactone (PCL) by two methods, which include 1. polycondensation of hydroxycarboxylic acid and 2. ring-opening polymerization of ε-caprolactone (Udayakumar et al. 2020). Table 2 provides an overview of the biopolymer synthesis pathway.

Solvent-based extraction: In solvent-based extraction, the process is governed by mechanical operations, including sifting, filtration, and centrifugation of biomass for biopolymer extraction (Faidi et al. 2019). To improve efficacy, biopolymers were extracted from pretreated biomass using various solvents (Mahmood and Moniruzzaman, 2019). Similarly, to mitigate solvent toxicity, green solvents such as ionic liquids, deep eutectic solvents, bio-derived solvents, non-halogenated solvents, and accelerated solvent systems have been used to extract polymers from biomass (Gu and Jérôme, 2013).

Endo and exo biopolymer production: Endo polymers, such as polyhydroxyoctanoate (PHO), possess unique characteristics and low melting temperatures, enabling the formation of lightweight composites (Van de Velde & Kiekens 2002, Ujang et al. 2009). These polymers are produced intracellularly by eubacteria. Similarly, Ganoderma applanatum, Collybia confluens, and Pleurotus eryngii were identified as potential sources of endopolymer. These fungi can be cultivated using Mushroom Complete Medium (MCM) (Yang et al. 2007, Jeong et al. 2008, Moradali & Rehm 2020). In exopolymer production, submerged cultures of fungal species have been widely employed, and parameters such as carbon and nitrogen sources, pH, temperature, and agitation have been standardized to optimize exopolymer production from fungal mycelia. For example, Paecilomyces japonica was used to optimize the production of maximal dry-weight biomass for extracting exopolymers (Bae et al. 2000). Similarly, Paecilomyces tenuipes C240 was studied to optimize factors using a One-Factor-at-a-Time Approach and an orthogonal matrix (Xu et al. 2003). Besides fungi, Ganoderma lucidum mushrooms and Phellinus linteus KCTC 6190 were studied to optimize mycelial growth. Similarly, Mushroom Complete Medium (MCM), Yeast Malt (YM), and Potato Malt Peptone (PMP) were studied to standardize exo-biopolymer production. PMP medium was the best medium for maximal polymer production (Kim et al. 2002). For a comparative study, Cordyceps militaris exhibited maximal mycelial growth at 7.5 days and maximal exopolysaccharide formation at 9.5 days (Park et al. 2001).

Bulk synthesis: Biopolymers are extracted and synthesized from various sources, including microbes, plants, and renewable natural sources such as food and animal waste (Kaplan 1998). Extraction methods vary by source. Biopolymers are generally produced under submerged conditions in fed-batch mode. For example, PHB was synthesized by optimizing carbon and nitrogen sources using reactor-fed bacteria of the species Ralstonia eutropha. Optimal biopolymer production depends on factors such as pH, substrate concentration, retention time, and substrate feeding rate. Similarly, genetic algorithms for fed-batch cultivation have been studied using nutrient feeding rates and dilution rates to maximize PHB production (Khanna & Srivastava 2005, Lai et al. 2013, Stanley et al. 2018).

PRODUCING BIO-BASED POLYMERS DIRECTLY VIA MICROORGANISMS

Alginate

Alginates are water-soluble, linear, anionic heteropolysaccharides. They are distributed in the cell walls of algae in the family Phaeophyceae, which include *Laminaria*

Table 3: Substrate and biopolymer-producing microorganisms.

Sl. No.	Type of Biopolymers	Producing microorganism	Substrate used	References
1.	Alginate	*Pseudomonas* and *Azotobacter* spp. (mostly *A. vinelandii*)	Sucrose	(Valentine et al. 2020, Dudun et al. 2021)
2.	Bacterial cellulose	*Gluconacetobacter, Agrobacterium, Aerobacter, Achromobacter, Azotobacter, Escherichia, Rhizobium, Sarcina,* and *Salmonella* sp.	Glucose and sucrose	(Chawla et al. 2009, Almihyawi et al. 2024, Mishra et al. 2022)
3.	Cyanophycin	*Cyanobacteria, Acinetobacter* spp., *Bordetella* spp., and *Desulfitobacterium hafniense*	Arginine and protein hydrolysate	(Solaiman et al. 2011, Aravind et al. 2016, Zou et al. 2022)
4.	Dextran	*Leuconostoc, Streptococcus*, *Lactobacillus* sp., *L. mesenteroides, Gluconobacter* sp., and *Pediococcus pentosaceus*	Sucrose and maltodextrins	(Patel et al. 2010, Wang et al. 2023, Baek et al. 2025)
5.	Gellan	*Pseudomonas elodea* and *Sphingomonas* spp., *S. paucimobilis*	Industrial waste products	(Fialho et al. 2008, Sá-Correia et al. 2002, Wu et al. 2011)
6.	Hyaluronic acid	*Streptococcus zooepidemicus, S. equi,* and *Pasteurella multocida*	Glucose, amino acids, nucleotides, salts, trace elements, and vitamins	(Kogan et al. 2007, Zakeri et al. 2017, Shikina et al. 2022)
7.	PHAs	*Cupriavidus necator* and *Phaeodactylum tricornutum*	Starch, alcohol, and industrial waste products	(Koller et al. 2010, Morlino et al. 2023)
8.	Poly-ε-lysine	*Streptomyces albulus*	Glucose	(Hamano et al. 2011)
9.	Pullulan	*Aureobasidium pullulans, Tremellales enterica, Cytaria* sp., *Cryphonectria parasitica,* and *Rhodotorula*	Industrial waste products	(Singh et al. 2008, Cruz-Santos et al. 2023, West, 2022)
10.	Xanthan gum	*Xanthomonas campestris*	Glucose and sucrose	(Palaniraj et al. 2011)

hyperborea, Macrocystis pyrifera, Laminaria digitata, and *Ascophyllum nodosum*. Besides algae, many bacterial species, such as *Pseudomonas* and *Azotobacter*, also produce alginate-like polymeric materials (Sabra & Deckwer 2005, Abka-Khajouei et al. 2022).

Dextran

Dextrans are hydrophilic polysaccharides produced by species like *Leuconostoc mesenteroides* and *Streptococcus mutans*. It has α (1-6)-linked glucan side chains attached to the 3-positions of the glucose units, forming the backbone. Class 1 - α (1 → 6)-linked d-glucopyranosyl backbone modified with side chains of d-glucose branches with α (1 → 2), α (1 → 3), and α (1 → 4)-linkage, class 2 - a backbone structure of alternating α (1 → 3) and α (1 → 6)-linked d-glucopyranosyl units with α (1 → 3)-linked branches, whereas class 3 - a backbone structure of consecutive α (1 → 3)-linked d-glucopyranosyl units with α (1 → 6)-linked branches. Dextran's physical and chemical properties generally vary depending on the source and production methodologies (Saboktakin et al. 2010, Díaz-Montes 2021).

Xanthan

Xanthan is a β-(1, 4)-linked heteropolymer composed of pentasaccharide units found in *Xanthomonas* species. This polysaccharide is widely used in food products as a thickening and gelling agent (Rehm 2010, Martínez-Burgos et al. 2024)

Gellan

Gellan is a heteropolymer widely extracted from *Sphingomonas* species and is a β-(1, 3)-linked, containing tetrasaccharide units (West 2021).

Curdlan

Curdlan, a β-(1,3)-linked homopolymer, is isolated chiefly from a few species, including *Agrobacterium, Rhizobium*, and *Cellulomonas* (Al-Rmedh et al. 2023).

Polyhydroxyalkanoates (PHA)

PHA is a unique and ideal example of intracellular biopolymers, mainly produced by many bacterial species. It contains β-hydroxy fatty acids, where the R group ranges from methyl to tridecyl. In particular, the main biopolymer is PHB (polyhydroxybutyrate), a prominent member of the PHA family. In addition, many copolymers are synthesized, including PHB family members such as poly (hydroxybutyrate-co-hydroxyvalerate) (PHBV), poly (hydroxybutyrate-co-hydroxyhexanoate) (PHBH), and poly (hydroxybutyrate-co-hydroxyoctanoate) (PHBO) (Vicente et al. 2023).

Cyanophycin

Cyanophycin is a polyamide most widely extracted from cyanobacteria. Biochemically, it consists of a repeating

heteropolymer composed of dipeptide units of aspartate and arginine. Cyanophycin is commonly used as a water softener and dispersant (Markus et al. 2023).

ε-poly-l-lysine

ε-poly-L-lysine is a polyamide, similar to cyanophycin, and is widely found in the bacterial species *Streptomyces albulus*. It is a homopolymer; lysine is one of the main amino acids present in this polymer. In the food industry, ε-poly-L-lysine is used as a food preservative and adsorbent (Pan et al. 2019).

PRODUCING BIO-BASED POLYMERS VIA ALGAE

Biopolymers are produced from algae in 3 ways: algal Fermentation, algal cell factories, and adding additives to algal biomass. In Fermentation, algal enzymes produce biopolymers from algal biomass (Khan et al. 2018). Fig. 3 shows three ways to produce biobased polymers from algae.

Algae undergo photosynthesis, producing essential nutrients that are used to synthesize biopolymers (Costa et al. 2018). Compression of algae and additives is the most common method used to prepare biocomposites (Ciapponi et al. 2019). Biopolymers such as Alginate, PHA, PHB, Carrageenan, Fucoidan, and κ-carrageenan from various algal sources were isolated using different methods, including solvent extraction, Microwave-assisted extraction, Ultrasound-assisted extraction, and Subcritical water extraction (Kartik et al. 2021). Yield (%) from these methods varies from source to source and extraction method. 4.50% of PHB was extracted from algal sources by using $CHCl_3$ with benzoic acid and MeOH with H_2SO_4 as solvent (Rueda et al. 2020), and 78.75% of κ-carrageenan was extracted from seaweed *Kappaphycus alvarezii* by using solvent 1-Butyl-3-methylimidazolium acetate by the Subcritical water extraction method (Gereniu et al. 2018).

By comparing all other biological sources, algae are one of the most promising sources for the production of biopolymers due to their scalability in production and the availability of biopolymer extraction strategies. Moreover, it can synthesize a wide range of bioproducts, including carbohydrates, lipids, pigments, polysaccharides, proteins, polymers, and other biocompounds. Due to their Low-cost production and sustainable nature, biopolymers from algae serve as the best model organism for producing various bioproducts (Khoo et al. 2019, Parsons et al. 2020, Lutzu et al. 2021). Table 4 summarises various biopolymers and biopolymers produced by algae.

Comparative Insight on the Scalability of Algal-Based Biopolymer Production Methods

Among the various approaches to producing biopolymers from algae, namely algal Fermentation, algal cell factories, and additive-assisted biomass processing, the most scalable method is continuous Fermentation using engineered algal strains in closed photobioreactors. This approach offers several key advantages: it enables precise control over growth conditions, maximizes biomass productivity, and supports

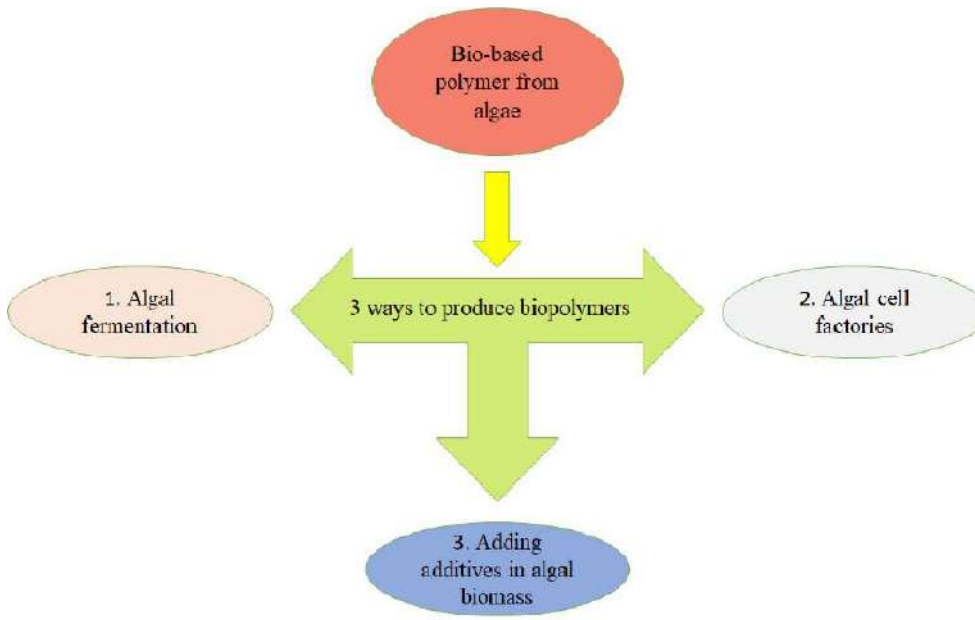

Fig. 3: Ways to produce bio-based polymers from algae.

the high-yield production of target biopolymers, such as polyhydroxyalkanoates (PHAs) and polyhydroxybutyrate (PHB). Genetic enhancements can further improve strain efficiency, substrate utilization, and tolerance to stress, making algal cell factories highly adaptable for industrial-scale applications. In contrast, direct enzyme-mediated or additive-based extraction from algal biomass is comparatively less scalable due to variability in biomass composition, dependence on seasonal availability, and batch-to-batch inconsistency (Gaur et al. 2024, Adetunji & Erasmus 2024).

Similarly, advanced extraction techniques, including microwave-assisted and solvent-based methods, offer higher purity and yield but are limited by high energy consumption, equipment costs, and environmental considerations-factors that challenge their economic viability at commercial scales. Therefore, while these techniques are valuable at laboratory and pilot levels, their transition to full industrial deployment is less straightforward. Overall, the use of genetically optimized algae in controlled bioreactor systems represents the most scalable and sustainable pathway for consistent, high-volume biopolymer production, particularly when integrated with downstream biorefinery processes (Gautam et al. 2024, Cannavacciuolo et al. 2024).

ENVIRONMENTAL REMEDIATION APPLICATIONS OF BIOPOLYMERS

Environmental pollution from industrialization, agricultural runoff, and urbanization has necessitated the search for sustainable remediation solutions. Conventional remediation strategies, such as synthetic chemical adsorbents, incineration, and physicochemical treatments, often result in secondary pollution, high costs, and energy-intensive processes. In contrast, biopolymer-based materials derived from renewable natural resources offer biodegradability, biocompatibility, non-toxicity, and efficiency in removing various contaminants (Awogbemi et al. 2023, Al-Hazmi et al. 2024).

Biopolymers, including chitosan, alginate, cellulose, starch, xanthan gum, and microbial exopolysaccharides, have significant potential for addressing water pollution, soil contamination, air purification, and hazardous waste management. These materials operate through diverse mechanisms, including adsorption, filtration, chemical binding, encapsulation, and microbial-assisted degradation. The following sections provide an in-depth exploration of their applications across various environmental remediation domains (Kaur et al. 2024, Al-Hazmi et al. 2024).

Biopolymer-Based Materials for Wastewater Treatment

Water pollution is among the most pressing global challenges, with sources including industrial effluents, agricultural runoff, and domestic wastewater. Biopolymers have attracted significant attention as effective and sustainable materials for treating contaminated water (Fakhri et al. 2023).

Adsorption of heavy metals and toxic ions: Heavy metals, including lead (Pb), cadmium (Cd), chromium (Cr), mercury (Hg), and arsenic (As), are toxic pollutants that accumulate in the environment, posing serious health risks. Biopolymer-based adsorbents offer efficient, cost-effective, and environmentally friendly alternatives for heavy-metal removal (Verma et al. 2021).

Chitosan-Based Adsorbents: Chitosan, a deacetylated derivative of chitin, is widely studied for its amino (-NH□) and hydroxyl (-OH) groups, which enable metal-ion chelation. Modified chitosan nanocomposites (e.g., chitosan-metal oxide hybrids, chitosan-carbon composites) enhance

Table 4: Biopolymer-producing algae.

Sl. No.	Biopolymer	Algal species	References
1.	Polyhydroxy alkanoates (PHA)	*Ulva* sp.	(Steinbruch et al. 2020)
2.	Polyhydroxy butyrate (PHB)	*Nostoc* sp.	(Morales-Jiménez et al. 2020)
3.	Polyhydroxy butyrate (PHB)	*Synechocystis* sp.	
4.	Polyhydroxy butyrate (PHB)	*Porphyridium purpureum*	
5.	Polyhydroxy butyrate (PHB)	*Chlorella* sp.	(Naresh Kumar et al. 2020)
6.	Polyhydroxy butyrate (PHB)	*Scenedesmus* sp.	
7.	Alginate	*Sargassum muticum*	(Flórez-Fernández et al. 2019)
8.	Fucoidan	*Nizamuddinia zanardinii*	(Alboofetileh et al. 2019)
9.	Fucoidan	*Saccharica japonica*	(Saravana et al. 2018)
10.	Carrageenan	*Mastocarpus stellatus*	(Ponthier et al. 2020)
11.	κ-carrageenan	*Kappaphycus alvarezii*	(Gereniu et al. 2018)

adsorption efficiency by increasing surface area and stability. Alginate-Based Adsorbents: Alginate, extracted from brown algae, contains carboxyl (-COO^-) groups that effectively bind heavy metals. Alginate-based hydrogels and beads have been used in continuous-flow systems for wastewater treatment (Siddiqui et al. 2025).

Cellulose and Starch Derivatives: Functionalized carboxymethyl cellulose (CMC) and starch-based bioadsorbents exhibit strong interactions with metal ions, providing an additional biodegradable option for water purification (Godiya et al. 2019). Chitosan's effectiveness largely stems from its abundant amino (–NH_2) and hydroxyl (–OH) groups, which facilitate strong chelation with metal ions. For example, recent work has demonstrated that modifying chitosan with poly(vinyl alcohol) and nano-silica can significantly enhance its Cr(VI) adsorption capacity. Additionally, studies have shown that chitosan-based adsorbents retain high efficiency across multiple adsorption-desorption cycles, highlighting their potential for cost-effective and long-term use in industrial wastewater treatment (Alkhaldi et al. 2024).

Alginate, derived from brown algae, contains carboxyl (–COO^-) groups that bind heavy metals effectively. Recent advances include Ca-alginate beads embedded with magnetic nanoparticles, which achieve high adsorption efficiency for Pb(II) ions and enable facile magnetic separation of treated water (Ayach et al. 2024). Furthermore, integrating alginate with chitosan to form interpenetrating polymer networks has improved mechanical stability and adsorption performance, making these hybrid materials promising for scalable water treatment systems (Sundararaman et al. 2024).

Cellulose derivatives, such as carboxymethyl cellulose (CMC), offer versatility due to their modifiable structures. Recent research indicates that grafting polyethylenimine onto CMC enhances its adsorption capacity for Cd(II) and Pb(II) ions by increasing the density of active binding sites (Ghanbari et al. 2024). Similarly, starch-based adsorbents functionalized with amine or thiol groups have produced nanocomposites with enhanced porous structures, resulting in improved removal efficiencies for Hg(II) and As(V) (Sahu et al. 2024).

Across these studies, kinetic analyses often show that adsorption on biopolymer-based materials follows pseudo-second-order kinetics, suggesting chemisorption as the dominant mechanism. The adsorption isotherms frequently conform to the Langmuir model, indicating monolayer adsorption on a homogeneous surface. These mechanistic insights are crucial for optimizing adsorbent performance in real-world applications (Sundararaman et al. 2024).

Modifying biopolymers, such as chitosan, alginate, cellulose, and starch derivatives, has enhanced their adsorption capacities and improved their operational stability in dynamic treatment environments. Their natural abundance, low cost, and biodegradability make them particularly attractive for sustainable wastewater treatment strategies. Integrating these advanced materials into continuous-flow systems enables effective remediation while reducing secondary pollution and overall treatment costs (Ghanbari & Zare 2024).

Removal of organic pollutants and dyes: Organic pollutants-including synthetic dyes, pharmaceuticals, and pesticides-are persistent contaminants in wastewater that pose serious environmental and health risks. Their chemical stability and resistance to degradation make them challenging to remove using conventional treatments. Biopolymers, due to their natural abundance, biodegradability, and tunable functional groups, have emerged as promising materials for the removal and degradation of these compounds (Negrete-Bolagay et al. 2021, Peramune et al. 2022, Manubolu et al. 2024).

Chitosan, a cationic biopolymer rich in amino (–NH_2) and hydroxyl (–OH) groups, exhibits a strong affinity toward anionic dyes such as methylene blue and malachite green, resulting from electrostatic attraction and hydrogen bonding. Chemical modifications or blending with other polymers can further enhance its performance to improve mechanical stability and adsorption capacity (Vijayasree & Manan 2023, Kurczewska 2022). Alginate, derived from brown algae and featuring carboxyl (–COO^-) groups, is effective for adsorbing cationic dyes such as rhodamine B. Recent studies on alginate-based hydrogels have shown that tuning the porosity and functional group density can lead to high removal efficiencies even in complex textile effluents (Wang et al. 2022, Dhanalekshmi et al. 2021).

Biopolymers can support semiconductor photocatalysts in degrading organic dyes under light irradiation. For example, TiO_2-chitosan composites combine the excellent adsorption properties of chitosan with the photocatalytic activity of TiO_2, resulting in enhanced degradation of dye molecules under visible light. Similarly, biopolymer-ZnO hybrids have been shown to stimulate the production of reactive oxygen species (ROS), which accelerate the degradation of complex organic dyes (Weon et al. 2023, Mendis et al. 2023).

Incorporating activated carbon into biopolymer matrices further improves dye removal by leveraging the high specific surface area and porosity of activated carbon. Combined with biopolymers such as chitosan, cellulose, or xanthan gum, the resulting composites exhibit enhanced dye adsorption kinetics and capacities. For instance, chitosan–activated

carbon composites have been reported to rapidly adsorb methylene blue, making them suitable for treating textile wastewater (Rehman et al. 2023, Kolya et al. 2023, Annu et al. 2024).

Biopolymer-based membranes for water filtration: Biopolymer-based membranes and hydrogels have emerged as advanced solutions for water purification, combining sustainability with high filtration efficiency. Membranes fabricated from biopolymers such as chitosan and cellulose acetate exhibit high porosity, mechanical strength, and favorable surface charge properties. These features enable effective removal of bacteria, viruses, and suspended solids from water. Chitosan-based microfiltration (MF) membranes can achieve high rejection rates for microbial contaminants, while cellulose acetate ultrafiltration (UF) membranes offer robust performance in terms of flux and fouling resistance (Gough et al. 2021, Mamba et al. 2021, Fijoł et al. 2022).

Advances in membrane technology have led to the development of nanofiltration (NF) membranes by incorporating nanoparticles into the biopolymer matrix. Modified membranes, for example, chitosan–TiO_2 or cellulose–ZnO hybrids, enhance the separation of multivalent ions and organic contaminants, providing additional functionalities such as photocatalytic degradation of pollutants. These systems achieve higher selectivity and improved permeate quality, making them attractive for selective separation processes (Li et al. 2023, Spoială et al. 2021).

Biopolymer-based hydrogels, formed by cross-linking polymers such as chitosan, alginate, or cellulose, offer an alternative strategy for pollutant removal. Their highly tunable pore structures and responsiveness to environmental stimuli (e.g., pH and temperature) enable controlled adsorption and subsequent desorption of pollutants. This controlled release is particularly valuable for designing innovative water treatment systems that

Table 5: Biopolymers in Environmental Applications.

Biopolymer	Application	Target Pollutant	Efficiency/Capacity	Reference
Chitosan	Heavy Metal Adsorption	Multi-metal	99% removal	(Ashraf et al. 2024)
Alginate	Heavy Metal Adsorption	As, Pb, Zn	67.42%, 95.31%, and 93.96%	(Spoială et al. 2021)
Cellulose	Heavy Metal Adsorption	As, Hg, Pb	177.1, 110.2 and 234.2 mg/g	(Zhan et al. 2018)
Starch+ Cellulose	Heavy Metal Adsorption	Pb, Zn, Cu	66.66, 58.82, and 47.61 mg/g	(Anghel et al. 2019)
Xanthan Gum	Heavy Metal Adsorption	Cd, Cu, Pb, and Zn	16.0 mg/g, 8.5 mg/g, 38.3 mg/L, and 7.2 mg/L	(Ko et al. 2022)
Chitosan	Wastewater Treatment	Dyes, Heavy Metals	99% and 98%	(Ayach et al. 2024)
Alginate	Wastewater Treatment	Organic Pollutants	89.3% removal	(Marques-da-Silva et al. 2022)
PHA	Wastewater Treatment	Acid Orange 7	96.44% removal	(Chang et al. 2022)
Pectin	Wastewater Treatment	Suspended Solids	-	(Jha and Mishra 2024)
Chitosan	Air Filtration	PM2.5	99.5%	(Hao et al. 2022)
Cellulose	Air Filtration & VOC Removal	Dust, Allergens, Microbes	99%	(Lippi et al. 2022)
Gelatin	Air Filtration & VOC Removal	VOCs, Formaldehyde	95%	(Kadam et al. 2021)
Chitosan	Soil Remediation	Heavy Metals	99%	(Pal et al. 2021)
Alginate-hydrogel	Wastewater	Hydrocarbons	78.8%	(Farid et al. 2024)
Pectin functionalized metal-organic frameworks	Soil Remediation	Pesticides	99%	(Liang et al. 2022)
pectin/chitosan/zinc oxide nanocomposite	Wastewater	Carbamazepine	68%	(Attallah et al. 2020)
Bacterial Cellulose	Wastewater	Microplastics	99%	(Faria et al. 2022)
Bacterial Cellulose	Bioremediation	Oil Spill Absorbents	-	(Fürtauer et al. 2021)

require regenerability and precise pollutant management (Rana et al. 2024, Ahmadi et al 2024).

Biopolymer Applications in Air Purification

Air pollution from particulate matter (PM), volatile organic compounds (VOCs), and toxic gases poses significant threats to human health and the environment. Biopolymer-based solutions have emerged for filtering airborne contaminants and catalyzing the degradation of pollutants (Gough et al. 2021, Ji et al. 2023). Table 5 lists various biopolymers and their environmental applications.

Electrospinning can produce chitosan nanofiber mats with high surface area and interconnected porous structures. These mats effectively capture delicate particulate matter (such as PM2.5 and PM10) and exhibit inherent antimicrobial properties, thereby improving indoor air quality. Functionalized cellulose membranes have been designed to enhance the removal of dust, allergens, and microbial contaminants. Their excellent mechanical and chemical stability makes them suitable for both indoor and industrial applications (Zhang et al. 2017, Lv et al. 2018, Borah et al. 2024).

Combining biopolymers with activated carbon yields composite filters that harness carbon's high adsorption capacity while retaining the biopolymer's biodegradability and processability. Such composites efficiently capture VOCs from indoor and industrial air environments (Akhtar et al. 2024). By immobilizing TiO_2 onto biopolymer supports (such as chitosan or cellulose), researchers have developed photocatalytic materials capable of degrading air pollutants such as NO_x and VOCs under light irradiation. This combination benefits from the biopolymer's adsorption properties and TiO_2's ability to generate reactive species that degrade contaminants (Balakrishnan et al. 2022, Wei et al. 2023). Biopolymers also serve as matrices for immobilizing enzymes that break down toxic pollutants. These bio-filters leverage microbial enzymatic activity to transform and remove contaminants from the air in an energy-efficient and eco-friendly manner (Abdelhamid et al. 2024).

Soil Remediation Using Biopolymers

Soil contamination from heavy metals, oil spills, pesticides, and industrial waste can reduce soil fertility and harm the environment. Biopolymers offer multiple approaches for remediating contaminated soils, including pollutant stabilization, nutrient delivery, and erosion control (Dhanapal et al. 2024). Chitosan forms complexes with heavy-metal ions through its amino and hydroxyl groups, reducing metal bioavailability in soil. This binding limits plant metal uptake and minimizes leaching into groundwater (Ahmad et al. 2017, Zheng et al. 2024).

Alginate hydrogels can encapsulate and immobilize heavy metals, reducing their mobility and bioavailability. These hydrogels help contain contaminants within the soil, thereby reducing the risk of environmental spread and plant uptake (Colin et al. 2024). Biopolymer matrices made from starch can be engineered to release nutrients gradually over time. This controlled-release mechanism minimizes nutrient runoff and soil depletion, supporting sustainable agricultural practices (Firmanda et al. 2024, Govil et al. 2024).

Coating seeds with chitosan has improved germination rates and enhanced plant resilience to environmental stresses. This treatment not only boosts early seedling growth but also protects against soil-borne pathogens (Samarah et al. 2020, Paravar et al. 2023). Hydrogels synthesized from xanthan gum and alginate enhance soil water retention and help prevent erosion. These materials support plant growth in arid environments and stabilize soils against wind and water erosion (Bajestani et al. 2025, Ali et al. 2024). Biodegradable mulch films derived from biopolymers are used in agriculture to reduce water evaporation, suppress weed growth, and maintain optimal soil temperatures. As they naturally degrade over time, they contribute to sustainable land reclamation practices (Menossi et al. 2021, Mansoor et al. 2022).

Biodegradation and Bioremediation Applications

Biopolymer-based carriers play a crucial role in supporting microbial-assisted degradation of pollutants, thereby enhancing overall bioremediation efficiency (Ayilara & Babalola 2023). Encapsulating bacteria within chitosan matrices creates a protective environment that enhances microbial survival and activity. In bioreactor applications, these encapsulated microbes degrade organic pollutants more efficiently due to sustained high-density microbial populations (Das et al. 2024).

Bioremediation beads composed of alginate or cellulose provide controlled release of biodegrading microbes into contaminated environments. These beads create a stable microenvironment that supports prolonged microbial activity, resulting in efficient pollutant degradation (Dzionek et al. 2016). Bacterial cellulose forms highly porous, lightweight sponges that absorb oil while allowing water to pass through. These properties make them practical for marine oil spill cleanup and reduce the environmental impact of oil contamination (ben Hammouda et al. 2021, Li et al. 2024). Chitosan-based materials have been developed into oil absorbents that are both biodegradable and efficient at selectively adsorbing oil from water. Their high adsorption capacity and ease of recovery provide a sustainable approach for oil spill containment and remediation in both marine and

industrial settings (Mallik et al. 2022, Basem et al. 2024, Kaczorowska & Bożejewicz 2024).

FUTURE PERSPECTIVES AND CHALLENGES

Biopolymer-based environmental remediation strategies have demonstrated promising results, but challenges remain regarding scalability, cost, and long-term stability. Future research should focus on:

- Enhancing the mechanical strength and durability of biopolymer materials for large-scale remediation applications.
- Developing multifunctional biopolymer composites that integrate adsorption, catalysis, and biodegradation into a single system.
- Optimizing production processes to reduce costs and increase biopolymer availability for environmental applications.

CONCLUSIONS

Biopolymers have emerged as a compelling alternative to synthetic polymers, offering biodegradability, renewability, and functional versatility for environmental remediation. Their successful application in wastewater treatment, air purification, and soil restoration demonstrates their potential to mitigate pollutants ranging from heavy metals and dyes to microplastics. However, real-world implementation still faces significant hurdles, including biodegradation efficiency under mixed-contaminant conditions, high production and downstream processing costs, and limited mechanical robustness in large-scale deployments. To accelerate translation from the laboratory to the field, future research should prioritize improving the structural and chemical stability of biopolymer-based materials in complex, real-world environments while optimizing biosynthetic pathways to enhance yield, purity, and economic feasibility. Additionally, developing multifunctional composites capable of addressing multiple contaminants simultaneously is crucial. Scaling up cost-effective production methods using waste-derived substrates or engineered microbial systems and assessing environmental fate and lifecycle impacts under diverse remediation scenarios.

Equally important are policy and regulatory frameworks that can facilitate the shift toward biopolymer adoption. Incentives for biopolymer-based product development, stricter regulations on persistent plastics, and public procurement programs favoring biodegradable alternatives can significantly accelerate market uptake. Furthermore, standardizing testing protocols and safety assessments for environmental applications will be crucial for regulatory approval and public trust.

With continued interdisciplinary collaboration—spanning biotechnology, materials science, environmental engineering, and policy—biopolymers can play a transformative role in enabling a circular, sustainable bioeconomy.

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Mitigating the Potential of Garlic and Turmeric in Aflatoxin-Contaminated Feeds of *Oreochromis niloticus*

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ABSTRACT

Fish feed contamination by mycotoxins presents serious challenges to farmers, as consuming aflatoxin-contaminated feed can result in toxin accumulation in fish, potentially posing risks to human health. This study assessed the detoxifying effects of garlic and turmeric powders on mycotoxin-contaminated feed and their impact on the growth and hematological parameters of juvenile *Oreochromis niloticus* (17.18±0.798g). Conducted in circular concrete tanks (0.5 m deep and 0.58 m in diameter), the experiment involved eight treatments (TDs) with three replicates each: TD1 (mold-free feed), TD2 (feed contaminated with *Aspergillus flavus* mold), TD3 (20 g garlic.kg^{-1} contaminated feed), TD4 (40 g garlic.kg^{-1} contaminated feed), TD5 (60 g garlic.kg^{-1} contaminated feed), TD6 (20 g turmeric.kg^{-1} contaminated feed), TD7 (40 g turmeric.kg^{-1} contaminated feed), and TD8 (60 g turmeric.kg^{-1} contaminated feed). Feeding trials spanned ten weeks to evaluate the effects of garlic and turmeric in mitigating aflatoxin impacts on fish growth and nutrient utilization. Some of the key findings are: Aflatoxin levels in analyzed feeds before (2.6448 µg.kg^{-1}) and after (123.168 µg.kg^{-1}) the inclusion of *Aspergillus flavus* varied. Feed processing methods, such as pelleting and drying, reduced aflatoxin concentrations. Significant reduction ($P<0.05$) in weight gain at TD2 (9.7 g) was observed compared to other treatments with the inclusion of garlic and turmeric. TD6 (turmeric at 20 g.kg^{-1}) showed the most pronounced improvement in fish growth parameters, with the highest final weight (16.07 g), weight gain (14.00 g), feed intake (10.98 g), and specific growth rate (2.95%.day^{-1}), while also achieving the lowest feed conversion ratio (0.78). Though pelleting and drying contributed to reducing aflatoxin levels, aflatoxins did not necessarily impact protein efficiency ratio (PER), nor did garlic and turmeric significantly enhance it. The inclusion of garlic and turmeric showed an improved nutrient utilization in *Oreochromis niloticus* despite the presence of aflatoxins in the feed.

INTRODUCTION

Fish are an essential and affordable source of protein in the diets of many consumers in Nigeria and other developing countries. However, mold growth in fish feed poses a significant challenge for farmers, especially in tropical regions where storage facilities are inadequate (Marijani et al. 2019). High moisture levels in fish feed promote the proliferation of molds, which can produce toxic substances known as mycotoxins. These compounds, including aflatoxins, ochratoxins, and fumonisins, are harmful to both fish and humans who consume contaminated fish. Among them, aflatoxin is the most prevalent and is frequently found in locally produced animal feeds, including fish feed (Odoemelam & Osu 2009). Under favorable conditions for mold growth, aflatoxin contamination can occur in feed ingredients and finished feeds. Ogunbanwo (2005) reported that major feed ingredients are highly susceptible to aflatoxin contamination, whether in the field or during storage. This poses a serious challenge to farmers, as fish consuming aflatoxin-contaminated feed may accumulate toxins that negatively impact human health (Pietsch 2020).

Aflatoxins are toxic secondary metabolites primarily produced by *Aspergillus* species, such as *Aspergillus flavus* and *Aspergillus parasiticus* (Abila 2003). These toxins commonly contaminate oilseed crops such as cottonseed, peanut meal, and corn, as well as wheat, sunflower, soybean, and fish meal. Four major types – aflatoxin B1 (AFB1), aflatoxin B2 (AFB2), aflatoxin G1 (AFG1), and aflatoxin G2 (AFG2) – are direct contaminants of grains and finished feeds (Pitt 2000, Marijani et al. 2019). The presence of aflatoxins in fish feed raises serious concerns due to their adverse health effects, which are further exacerbated by climate change (Nešić 2018). Rodrigues et al. (2011) emphasized that mycotoxin contamination results from poor agricultural practices, inadequate storage, and improper handling by farmers, grain processors, and feed millers. Aflatoxins, which are classified as potential carcinogens, represent a major food safety risk (Bennett & Klich 2013, Ilesanmi et al. 2023).

Aflatoxin contamination in feed and food has led to significant economic losses and health risks for both animals and humans. Various strategies have been developed to mitigate mycotoxin contamination, including pre-harvest, harvest, and post-harvest measures aimed at controlling mold growth or detoxifying contaminated products. Among these strategies, the use of plant-based detoxifiers has gained attention due to their safety and environmental friendliness compared to chemical treatments. Research suggests that plant extracts from garlic, ginger, and turmeric, along with their essential oils, can inhibit *Aspergillus* growth and reduce aflatoxin production (Agbebi et al. 2013).

This study, therefore, investigates the detoxification effects of garlic and turmeric powder on mycotoxin-contaminated feed and its impact on the growth and nutrient utilization parameters of Nile tilapia (*Oreochromis niloticus*) – a widely cultured and economically important fish species in Nigeria.

MATERIALS AND METHODS

Experimental Site

The experiment was carried out in the Department of Biology, Federal College of Education, Osiele, Abeokuta, Ogun State. The experiment was conducted in a circular concrete tank (0.5 m depth and 0.58 m diameter).

Inclusion of Aflatoxin in the Formulated Feed

The feed ingredient was purchased and pelleted at the Agro-Allied Company mill at Odo Eran, Abeokuta. The experimental feed was prepared in the laboratory and formulated as in Table 1. The compounded ingredients were sprinkled with a small amount of distilled water to make the feed moist and then mixed with a cultured strain of *Aspergillus flavus* from the Microbiology Department, University of Lagos. The mixed feed was covered with a plastic sac for 72 h to encourage mold growth in the feed.

Fresh garlic bulbs and turmeric root were purchased, dried, and ground at the Mile-1 Market, Kebbi State. They were rinsed with clean water and grated before being sun-dried. The dried form of each was then ground into powder with a locally fabricated hand grinder. The experimental feed was mixed appropriately according to treatment. There were 8 treatments (TD) in 3 replicates each adopted for the study (Table 2). These are TD1 (feed uncontaminated/mold-free), TD2 (contaminated feed of *A. flavus*), TD3 (20 g of garlic.kg^{-1} of moldy feed), TD4 (40 g of garlic.kg^{-1} of moldy feed), TD5 (60 g of garlic.kg^{-1} of moldy feed), TD6 (20 g of turmeric.kg^{-1} of moldy feed), TD7 (40 g of turmeric.kg^{-1} of moldy feed), TD8 (60 g of turmeric.kg^{-1} of moldy feed). The compounded feeds were pelletized with a pelleting machine and sun-dried immediately. Thereafter, the feed was kept in an airtight container for further study.

The proximate analysis of all diets was determined before the start of the experiment. Briefly, a known weight of the feed sample was dried at 105°C until a constant weight was achieved, and the moisture content was calculated based on weight loss. The dried sample was incinerated at 550°C, and the remaining weight was used to calculate ash content. This was followed by the Kjeldahl method, where nitrogen compounds were converted to ammonia, distilled, and titrated to calculate crude protein, on the assumption of 16% nitrogen in proteins. The crude fat (lipids) was determined using the Soxhlet method, involving solvent extraction and weighing the fat residue. Crude fiber is measured by degreasing the sample, digesting it with sulfuric acid, drying, and then incinerating it to obtain the fiber content from the weight difference.

The carbohydrate content of the feed was calculated as follows:

CHO = 100 - (moisture + ash + crude protein + fiber + lipid) (AOAC 2006)

Determination of Aflatoxin

The mycotoxin analysis of each feed treatment was done at the Nigerian Stored Product Research Institute, Ilorin Laboratory, before the commencement of the experiment with the HPLC method modified from Barbas et al. (2005).

Sample Preparation and Extraction

The sample preparation began by accurately weighing 5.0 g of the homogenized test material using a calibrated analytical

balance (±0.0001 g precision). The weighed sample was transferred into a 50 mL polypropylene centrifuge tube, and 20 mL of a methanol-water mixture (85:15, v/v) was added. The mixture was vortexed for 30 sec to ensure thorough wetting, followed by mechanical shaking at 250 rpm for 2 h at 4°C to minimize degradation. After extraction, the mixture was centrifuged at 10,000 rpm for 10 min (4°C), and the supernatant was carefully decanted into a clean glass vial.

Extract Concentration and Defatting

The collected supernatant was concentrated to near dryness under a gentle stream of nitrogen at 40°C using an evaporator. The residue was reconstituted in 5 mL of 10% NaCl (w/v) solution to precipitate interfering compounds. For defatting, 10 mL of redistilled n-hexane (HPLC-grade, ≥99.9% purity) was added, and the mixture was vigorously shaken for 1 min before phase separation. The upper hexane layer (containing lipids) was discarded, while the lower aqueous-methanol phase was retained for further cleanup.

Solid-Phase Extraction (SPE) Cleanup

A disposable silica gel SPE column (500 $mg.mL^{-1}$, 55-105 µm particle size, 60 Å pore size) was preconditioned with 10 mL of methanol, followed by equilibration with 10 mL of deionized water. The defatted extract was loaded onto the column at a flow rate of 1 $mL.min^{-1}$ under vacuum. Sequential washing steps were performed to remove matrix interferences:

- First wash: 30 mL of n-hexane (discarded).
- Second wash: 3 mL of ethyl acetate (discarded).
- Third wash: 3 mL of methylene chloride (discarded).

The target analytes (aflatoxins B_1, B_2, G_1, and G_2) were eluted with 6 mL of chloroform-acetone (90:10, v/v) at a controlled flow rate of 0.5 $mL.min^{-1}$. The eluate was collected in a salinized glass tube and evaporated to dryness under nitrogen. The residue was reconstituted in 200 µL of methanol and filtered through a 0.22 µm PTFE syringe filter before HPLC injection.

HPLC Analysis

Instrumentation and Chromatographic Conditions

- HPLC System: Agilent 1260 Infinity II (or equivalent), equipped with a fluorescence detector (FLD).
- Column: C18 reversed-phase column (150 mm × 4.6 mm, 3.5 µm particle size) maintained at 40°C.
- Mobile Phase:
- A: Water-methanol (60:40, v/v) with 0.1% formic acid.
- B: Methanol-acetonitrile (50:50, v/v).
- Gradient Program:
- 0–5 min: 20% B → 50% B (linear).
- 5–10 min: 50% B → 80% B (linear).
- 10–15 min: 80% B (isocratic).
- Post-run re-equilibration min at initial conditions.
- Flow Rate: 1.0 $mL.min^{-1}$.
- Injection Volume: 20 µL.
- Detection:
- FLD Settings:
- Excitation: 365 nm.
- Emission: 435 nm (for aflatoxins B_1 and G_1).
- Emission: 465 nm (for aflatoxins B_2 and G_2, via post-column photochemical derivatization).

Quality Control and Validation

- Calibration Standards: Aflatoxin working standards (0.5–50 $\mu g.L^{-1}$) were prepared in methanol.
- Recovery Test: Spiked samples (5, 10, 20 $\mu g.kg^{-1}$) were analyzed in triplicate, with recoveries between 85–110%.
- Limit of Detection (LOD): 0.05 $\mu g.kg^{-1}$ (S/N = 3).
- Limit of Quantification (LOQ):0.15 $\mu g.kg^{-1}$ (S/N = 10).

Data Analysis

Quantification was performed using Agilent OpenLab CDS ChemStation (v.2.4) or equivalent software. Peak identification was based on retention time (±2% tolerance) and spectral matching.

The compounded feeds were pelletized with a pelleting machine, dried immediately with an electric dryer at 45 °C for 24 hrs, and then kept in labeled airtight containers for further

Table 1: The dietary composition of formulated feed used for the experiment.

Ingredient	Feed [kg]
Maize	22.5
Groundnut cake	30.50
Fishmeal	15.50
Soya-bean meal	30.50
Mineral premix*	0.50
Methionine	0.25
Lysine	0.25
Total	100

[1] *Contains VitA 4000000IU, Vit D. 800000IU, Vit. E 40000 mg, Vit. K3 800 mg, Vit. B1 1000 mg, Vit. B2 6000 mg, Vit. B6 5000 m, Vit. B12 25 mg, Niacin 6000 mg, Patothenic acid 20000 mg, Folic acid 200 mg, Folic acid 200 mg, Biotin 8 mg, Manganese 300000 mg, Iron 80000 mg, Zinc 20000 mg, Cobalt 80 mg, Iodine 400 mg, Selenium 40 mg, Choline 800000 mg.

use. The proximate analysis of all diets was determined before the start of the experiment according to AOAC (2006).

Experimental Procedure

12-week-old healthy juvenile fish (17.18±0.798 g) were selected and arranged in a group of 10 juvenile fish per tank. The 1.2 m3 circular concrete tanks used were 24 for the experiment for 10 weeks. The juvenile *Oreochromis niloticus* were obtained from Taiwo Farm, Ndele, Ota, Ogun State, acclimated to the experimental environment for 7 days, and maintained on Top® feed (45% CP) before the treatments.

Fish in every treatment group received 3% body weight of the prepared diet. The fish were fed the experimental diets twice a day at 9:00 am and 4:00 pm. The fish fed in each treatment group were monitored daily in the morning, at noon, and in the evening for swimming movement, breathing, possible bruises, and mortality. Weekly weight changes were noted using a weighing scale, and the aquarium water was changed every two days. After 10 weeks, the potential effects of garlic and turmeric on aflatoxin-induced feed were studied based on observed experimental growth and hematological characteristics.

Assessment of Growth Performance Parameters and Survival Rate

Average fish growth performance was assessed based on weight gain, average daily growth, specific growth rate, feed conversion ratio, feed efficiency ratio, and protein efficiency ratio were evaluated to determine the effects of the various treatments.

Average Weight Gain (AWG)

The average weight gain represents the variation between the starting weight and the weight after the trial. This is calculated according to (Sepahdar et al. 2009) using the formula:

Final mean weight (g) - Initial mean weight (g) = the average weight gain (g).

Average Daily Growth (AWG)

The average weight gained per day was calculated as:

ADG (mg) = (Average Weight gain (mg))

(Duration of the experiment (days)) (Hung et al. 1989)

Percentage Weight Gain (%WG)

Percentage weight gain is weight gained over time relative to the total weight, and it is calculated as:

(%WG) = (Weight gain)

(Initial Weight) X 100 (Sepahdar et al. 2009)

Specific growth rate (SGR)

Specific growth rate (SGR) is the coefficient of the percentage increase in fish weight per day.

Specific growth rate, [SGR (%.day^{-1})] = 100 (log W2-log W1)

(T2-T1) (Arnanson et al. 2009)

Where W1 and W2 are the weights at time T1 and T2, respectively.

Feed conversion ratio (FCR)

Feed conversion ratio (FCR) represents the ratio or rate of efficiency with which the bodies of fish convert feed into the desired output, and it is calculated as follows:

Feed Conversion Ratio (FCR) = (Total dry feed fed (g))

(Total wet weight gain (g)) (Sepahdar et al. 2009)

Protein-efficiency-ratio (PER)

This is the connection between the amount of protein in feed and the moist weight increase of fish.

Protein Efficiency Ratio (PER) = (Wet weight gain (g))

(Amount of Protein fed (g)) (Sepahdar et al. 2009).

Physico-Chemical Analysis of Culture Medium

A daily assessment of temperatures, conductivity, dissolved oxygen (DO), pH, and total dissolved solids (TDS) of aquaria is done using digital methods. The digital instruments used are as follows:

- Temperature meter with the range of 0.1-80.0°C and 32.0-176.0°F.
- A digital pH meter, (JUANJUAN®) with the range of 0.00-14.00pH.
- The EC meter made in Pakistan with a range of 0-9900 µs.cm^{-1}
- The RCYAGO® Dissolved Oxygen Meter (range is 0.0-20mg.L^{-1}) from the United States
- The TDS meter made in Pakistan with a range of 0-9999 mg.L^{-1}

Statistical Analysis

The growth and nutrients utilization data, as well as water quality parameters, were subjected to One-Way Analysis of Variance using statistical analysis software (SAS 1999). Duncan's Multiple Range Test (DMRT) was used to evaluate significant averages between treatments at a probability level of 5%.

RESULTS AND DISCUSSION

During the first 3 days of the trial, the fish showed little reaction to the experimental feed, but they had assumed

full feeding by the 5th day. The experimental fish were active throughout the experimental period and showed no stressful movement or sustained any external injuries across the Treatments.

The analysis of the total aflatoxin present in the initial feed (A) bought before the inclusion of the *Aspergillus flavus* species was observed to be 2.6448 $\mu g.kg^{-1}$ (Aflatoxin G2 - 0.0005 $\mu g.kg^{-1}$, Aflatoxin G1 - 0.0054 $\mu g.kg^{-1}$, Aflatoxin B2 - 0.1066 $\mu g.kg^{-1}$ and Aflatoxin B1 - 2.5302 $\mu g.kg^{-1}$) but after the mixture with Aspergillus flavus (B), the concentration was higher at 123.168 $\mu g.kg^{-1}$ (Aflatoxin G2 - 0.072 $\mu g.kg^{-1}$, Aflatoxin G1 - 0.84 $\mu g.kg^{-1}$, Aflatoxin B2 - 22.678 $\mu g.kg^{-1}$ and Aflatoxin B1 - 99.589 $\mu g.kg^{-1}$). Meanwhile, after the inclusion of the plant extracts (garlic and turmeric) according to the treatments and feed was pelleted, the total aflatoxin for TD1, TD2, TD3, TD4, TD5, TD6, TD7, and TD8 were 2.5908 $\mu g.kg^{-1}$, 24.743 $\mu g.kg^{-1}$, 23.515 $\mu g.kg^{-1}$, 23.713 $\mu g.kg^{-1}$, 24.181 $\mu g.kg^{-1}$, 23.493 $\mu g.kg^{-1}$, 22.215 $\mu g.kg^{-1}$ and 25.431 $\mu g.kg^{-1}$, respectively, as stated in Table 2.

The proximate analysis of the experimental feed was analyzed as stated in Table 3. The results recorded for moisture, ash, protein, lipid, CHO and fiber dry matter and energy range from 40.12-39.62, 8.51-8.451, 7.03-6.88, 6.30-5.83, 94.35-93.60 and 17.81-17.58 ($kJ.g^{-1}$), respectively.

There were variations in the value of aflatoxin in the analyzed feeds. It was observed that the processing activities (pelleting and drying) had a reducing effect on the concentration of the total aflatoxin.

There was no significant difference (P>0.05) in the initial weight of the experimental fish; they were evenly distributed into the tanks. Meanwhile, after the experimental period, there was a significant difference (P>0.05) in the final weight and weight gain of the experimental fish across the treatments (Table 4 and Fig. 1). TD6 had the highest final weight and weight gain, which was significantly different (P>0.05) from the others. TD2 was significantly low (P>0.05), both in the final weight (11.62 g) and weight gain (9.74 g). In the amount of the feed intake recorded, the experimental fish fed with TD1 (10.90 g) and TD6 (10.98 g) had the best response significantly (P<0.05) to the feed, and the feed intake of the experimental fish was significantly low (P<0.05) in the TD2 (9.91 g) and TD8 (9.89 g) treatments.

The records during the experimental setup are presented in Table 5. The culture medium temperature was within 24.300C and 27.200C. The pH was within the range of 7, that is, 6.90 and 7.03. Also, electrical conductivity was within the cultured system range lowest being 366.33 $\mu.cm^{-1}$ and the highest being 416.67 $\mu.cm^{-1}$. The oxygen (DO) level was observed to be the optimum value for cultured fish, ranging between 3.64 $mg.L^{-1}$ and 4.59 $mg.L^{-1}$. In addition, total dissolved solids (TDS) were also within the cultured system range (114.67 $mg.L^{-1}$ and 205.00 $mg.L^{-1}$) (Table 5).

The protein intake TD1 (4.91) and TD6 (4.94) were the highest and significantly different (P<0.05) from others, and TD2 (4.46 g) and TD8 (4.45 g) were significantly low (P<0.05). Meanwhile, the protein efficiency ratio was significantly low in TD2 (2.23) and TD4 (2.21).

Based on the record from the period of the experiment, the experimental fish with the highest value of feed conversion ratio was TD2 (1.05) and significantly different (P<0.05)

Table 2: Total Aflatoxin analysis of the experimental feed.

Parameters	A	B	TD1	TD2	TD3	TD4	TD5	TD6	TD7	TD8
Aflatoxin G2 [$\mu g.kg^{-1}$]	0.0005	0.072	0.0005	0.008	0.005	0.006	0.008	0.004	0.004	0.008
Aflatoxin G1 [$\mu g.kg^{-1}$]	0.0054	0.840	0.0075	0.075	0.058	0.066	0.069	0.056	0.042	0.06
Aflatoxin B2 [$\mu g.kg^{-1}$]	0.1066	22.678	0.0974	0.591	0.785	0.832	0.426	0.838	0.855	1.621
Aflatoxin B1 [$\mu g.kg^{-1}$]	2.5302	99.589	2.4854	24.069	22.667	22.809	23.678	22.596	21.314	23.742
Total [$\mu g.kg^{-1}$]	2.6448	123.168	2.5908	24.743	23.515	23.713	24.181	23.493	22.215	25.431

Table 3: Proximate analysis of the experimental feed samples.

Samples	Moisture [%]	Ash [%]	Protein [%]	Lipid [%]	CHO [%]	Fiber [%]
TD1	3.15	18.02	38.81	6.1	8.36	25.56
TD2	6.17	15.67	39.30	6.1	8.92	23.84
TD3	2.67	16.80	39.75	6.5	9.80	24.48
TD4	3.82	15.27	39.13	6.3	10.27	25.21
TD5	4.47	14.11	38.34	6.2	12.20	24.68
TD6	4.87	9.68	39.25	6.0	12.29	27.91
TD7	3.07	18.88	39.13	8.2	15.28	15.44
TD8	5.61	12.01	39.25	8.4	13.36	21.37

Table 4: Growth parameters and survival rate of *O. niloticus* fed experimental diets.

Parameters	TDI	TD2	TD3	TD4	TD5	TD6	TD7	TD8	±SME
Initial wgt [g]	2.02^{a}	1.88^{a}	1.87^{a}	2.07^{a}	2.23^{a}	2.07^{a}	2.00^{a}	1.92^{a}	0.0715
Final wgt [g]	14.48^{b}	11.62^{d}	12.06^{dc}	12.20^{c}	10.83^{e}	16.07^{a}	14.23^{b}	13.66^{d}	0.3460
Wgt Gain [g]	12.46^{b}	9.74^{g}	10.19^{e}	10.13^{e}	10.60^{f}	14.00^{a}	12.23^{c}	11.74^{d}	0.3458
F. I [g]	10.90^{a}	9.91^{d}	9.89^{d}	10.19^{bc}	10.13^{c}	10.98^{a}	10.87^{a}	10.75^{ab}	0.1158
P. I [g.kg^{-1}]	4.25^{ab}	3.86^{d}	3.96^{c}	3.97^{c}	3.85^{d}	4.28^{a}	4.24^{ab}	4.19^{b}	0.0521
PER	2.03^{a}	1.70^{a}	1.66^{b}	1.70^{a}	1.70^{a}	1.58^{c}	1.58^{c}	1.58^{c}	0.0109
FCR	0.87^{d}	0.93^{cd}	0.97^{b}	1.01^{b}	1.16^{a}	0.78^{e}	0.88^{cd}	1.10^{a}	0.0254
SGR [%.day^{-1}]	2.80^{ab}	2.68^{c}	2.56^{c}	2.46^{c}	2.16^{d}	2.95^{a}	2.78^{ab}	2.47^{c}	0.0550
Survival [%]	100	100	100	100	100	100	100	100	0.0000

[1] Initial wgt – Initial weight, Final wgt – Final weight, Wgt Gain – weight gain - F. I – Feed Intake, P. I – Protein intake, PER – Protein Efficiency ratio, FCR – Feed Conversion Ratio, SGR – Specific Growth Rate.

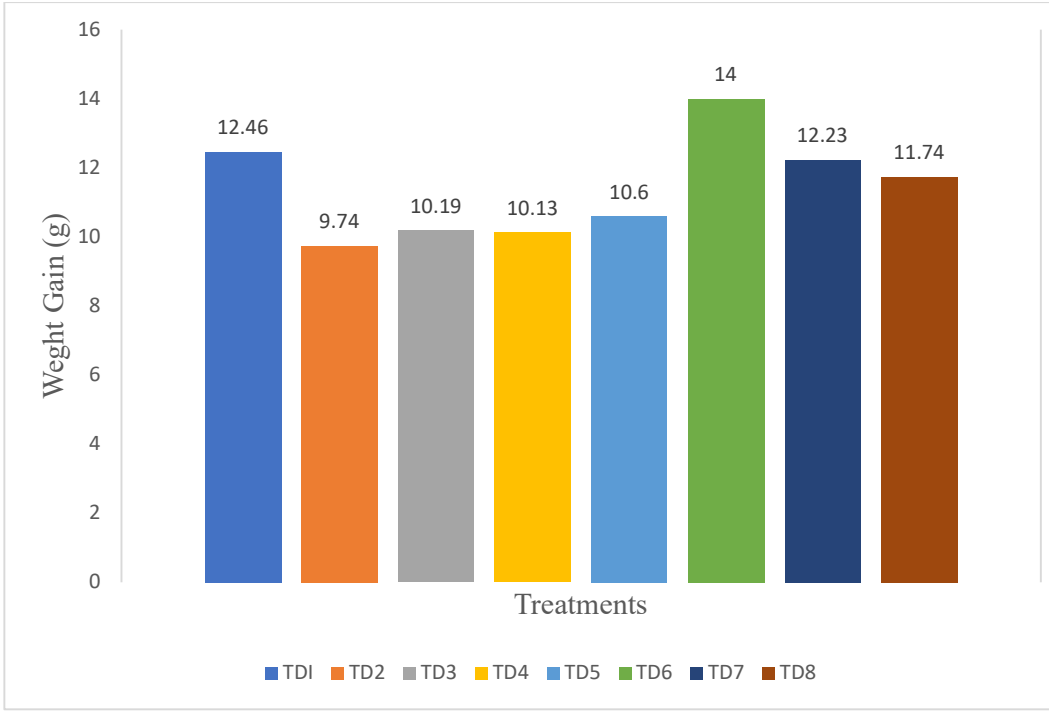

Fig. 1: Weight Gain of *O. niloticus* fed experimental diets**.**

Table 5: Average water quality parameters in the culture medium.

Parameters	TDI	TD2	TD3	TD4	TD5	TD6	TD7	TD8	±SME	Aquaculture Mean Value
EC [µ.cm^{-1}]	389.00^{b}	366.33^{c}	376.67^{bc}	384.00^{b}	410.67^{a}	416.67^{a}	381.67^{bc}	391.33^{b}	80.25	20-1500 (Boyd 2003)
TDS [mg.L^{-1}]	195.00^{b}	181.67^{d}	188.00^{c}	192.50^{bc}	205.00^{a}	203.00^{a}	193.33^{bc}	114.67^{e}	12.25	<400 (WHO 2004)
TEMP [°C]	25.43^{cd}	27.20^{a}	25.70^{bc}	26.07^{b}	25.07^{de}	24.70^{ef}	24.30^{f}	25.17^{cde}	0.11	25–30 (FAO 2006)
DO [mg.L^{-1}]	3.93^{a}	3.83^{a}	3.64^{a}	4.59^{a}	4.10^{a}	4.23^{a}	3.70^{a}	4.50^{a}	0.24	3 – 20 (Boyd 2003)
pH	6.96^{bcd}	7.12^{a}	6.98^{bcd}	7.04^{ab}	6.90^{d}	7.03^{abc}	6.95^{bcd}	6.92^{cd}	0.004	6.0–9.0 (Davis 1993)

[1] EC – Electrical Conductivity, TDS – Total Dissolved Solids, TEMP – Temperature, DO – Dissolved Oxygen.

from the others, while at TD6 it was significantly lower than the others. The specific growth rate at the TD6 was significantly higher (P<0.05) than others, but low at TD2 and TD3 significantly (P<0.05) from others. All through

the 10-week experimental period, there was no mortality; therefore, the survival rate was 100% throughout.

Discussion

The experimental feed ingredients bought directly from the feed mill contained some concentration of aflatoxin, which is an indication that the feed got contaminated by mycotoxins before it got to the farm. The report of the Biomin survey (2017) supported this, that about 74% of the ingredients used in compounding aquaculture feed are contaminated with mycotoxins, which can have a substantial negative economic impact on the aquaculture industry. Likewise, the variation in the total concentration of the aflatoxin present in the feed might be because of the differences in the handling of the feed during the pelleting and drying. The aflatoxin content of the experimental diet is higher than the standard limit level by CECC (2003) in animal feeds, which is 20 $\mu g.kg^{-1}$ and 5 $\mu g.kg^{-1}$ to 10 $\mu g.kg^{-1}$ in compounded feeds for dairy animals and lambs, respectively. The processing effect of pelleting feed can contribute to the reduction in aflatoxin concentration in the feed. This is noticed in the TD2 (mold feed) before (123.168 $\mu g.kg^{-1}$), and after (24.743 $\mu g.kg^{-1}$) the pelleting of the feed, which supported the observation made by Neme and Mohammed (2017) and Ilesanmi et al. (2024), that processing techniques can reduce the concentration of mycotoxin. Also, the extrusion technique reduces AFs by 50% to 80%, depending on the processing temperature and granule moisture content reduction (Shanakhat et al. 2018, Kabak 2006).

The growth rate parameters were recorded to track the development of *Oreochromis niloticus*. The growth observations recorded might be because of aflatoxin concentrations inside the experimental feed. The reduced weight gain observation at the TD2 of this experiment confirmed the report of low or reduced weight gain stated by Deng et. al. (2010) and Anh Tuan et. al. (2002), where fingerlings of Nile tilapia were given aflatoxin-B1 contaminated feed for 20 days, 25 days, and 56 days, respectively. Although the concentration of aflatoxin in TD2 (24.743 $\mu g.kg^{-1}$) was not the highest, TD8 had higher concentrations, but the improved weight gain at TD8 may be a result of the mitigating effect of turmeric over aflatoxin and its ability to improve weight gain (Teich et al. 2017, Shawky et al. 2022). The improved AWG recorded in TD6, TD7, TD8 and other treatments may be due to the addition of garlic and turmeric in them. Therefore, the presence of garlic and turmeric may be the reason for the improved weight gain recorded in the experiment, despite the concentration of total aflatoxin present in the feed; this was also observed by Saber et al. (2010). The inclusion of garlic extract in the feed of Juvenile *Acipenser ruthenus* demonstrated a notable improvement in feed efficiency and weight gain over a 10-week feeding period (Lee et al. 2012). Bello et.al. (2012) corroborated this importance of plant extracts when they observed a comparable rise in weight growth in *Clarias gariepinus* given meals supplemented with leftover walnut leaves and onion bulbs.

The feed intake at TD2 was significantly low ($P<0.05$) compared to others, which may be a result of the feed contamination with *Aspergillus flavus*, because there was a better feed intake in the feed without the fungi. Meanwhile, TD8 had a similar concentration level of aflatoxin (25.431 $\mu g.kg^{-1}$) with TD2 (24.743 $\mu g.kg^{-1}$), but the increase in the feed intake in TD8 and other treatments may be a result of the pleasant odour from these plant extracts (Hasegawa et al. 2015). In agreement with this, Agbon et al. (2013) stated that there is a low response to feed by juvenile *Clarias gariepinus* fed aflatoxin B1 contaminated feed for 12 weeks. Meanwhile, other treatments with the inclusion of garlic and turmeric responded well to the feed, and it may be assumed that the taste of these spices is responsible for it. This is also observed by Agbebi et al. (2013) that there is an increase in the feed intake of *Clarias gariepinus* fed a diet mixed with ginger compared to the one without ginger for 56 days. Comparing the feed intake with the treatment with the turmeric inclusion was better than that of garlic; this may be because turmeric has a more pleasant smell than garlic and a brighter color. According to this research, garlic or turmeric may have effects on the feed acceptability, which will directly influence the weight gain of the fish.

Therefore, this research showed that the reduced or no concentration of aflatoxin in the feed can improve the weight gain of *Oreochromis niloticus*. On the other hand, the acceptability of the feed with the inclusion of garlic and turmeric may be responsible for the improved weight gain experienced in this research. Meanwhile, TD3 had the lowest feed intake value but with a better weight gain compared to TD2, meaning that the plant extracts also have effects on the weight gain apart from the feed acceptability.

According to the proximate analysis of the feed and the subsequent protein intake by each treatment, TD6 had the highest protein intake compare to others but in protein efficiency ratio TD1 had the highest, this maybe as a result of the absence of plant extracts (garlic and turmeric) and low level of aflatoxin concentration in the feed that is responsible for the effective utilization of the quantity of PI available for the treatment. There was no difference in the PER of feed with turmeric inclusion (TD6, TD7 and TD8). This result was similar to what was discovered by Onyeniyoma et al. (2024) when broilers fed with turmeric inclusion feed had low PER despite being fed with high protein intake. This may probably

be as a result of the presence of anti-nutritional factors in plant extracts like flavonoids, alkaloids, tannin, phytate and saponin that are associated with nutrient impairment (Ari et al. 2012). It is noted from this research that the presence of aflatoxin in feed may not necessarily affect the PER, and plant garlic and turmeric may not improve the PER.

The feed conversion ratio, which can relate to how economical the feed is or the utilization of the feed by the fish, has its highest value in the TD2, which was significantly different from the others in the group. These results may be attributed to the deteriorating effects of aflatoxin on the food conversion of the fish, which was in confirmation of the report of Ghafarifarsani et al. (2021). TD6 after the experimental period had the best feed conversion ratio, which is significantly different from the others in the group.

The specific growth rate at TD2 and TD3 during the experimental period was significantly lower ($P<0.05$) compared to others in the group. The specific growth rate value recorded in the TD2 and TD3 might be due to the aflatoxin contamination and the low quantity of the garlic inclusion level, respectively. This was also supported by Ghafarifarsani et al. (2021) when rainbow trout were fed with aflatoxin B1 and zearalenone-contaminated feed.

The experimental fish used appeared to be in good condition during the experimental period, while fish survival was 100%. Fish respond differently to aflatoxin depending on the concentration and length of exposure (Zhang et al. 2021). Ten (10) weeks of exposing the fingerlings of *Oreochromis niloticus* to 50 ppb of aflatoxin-contaminated feed showed little or no effect on the performance of the fish (El-Banna et al. 1992). On the contrary, Effiong & Alatise (2009) in their research observed a low survival rate of catfish (*Heterobranchus longifilis*) when they were given mold-contaminated feed for 6 weeks. In fish, it has been commonly reported that aflatoxicosis comes with serious health challenges, poor growth rates, and reduced appetites (Sotolu et. al. 2014, Cagauan et. al. 2004). This research may not be able to ascertain the impact of the aflatoxin-contaminated feed on the experimental fish, but reports of Agbon et al. (2013) and Agbebi et al. (2013) established that aflatoxicosis has a negative effect on the biochemical and histological aspects of Clarias gariepinus, respectively.

The water quality parameters (temperature, pH, conductivity, dissolved oxygen and total dissolved solids) recorded weekly during the experiment were within the recommendations by the legislation (Boyd 2003, Davis 1993, FAO 2006), so the experimental feed did not have an influence on zootechnical parameters of juvenile *O. niloticus*. Fish survive and grow best in water within the recommended water quality parameters.

CONCLUSIONS

In conclusion, the addition of garlic and turmeric improved the feed intake, weight increase, and feed conversion ratios of Nile tilapia (*Oreochromis niloticus*) despite aflatoxins, indicating their potential to lessen the negative impacts of mycotoxins. The results highlight how important it is to implement natural detoxifying techniques to improve food safety and sustainability in aquaculture, especially in areas where mycotoxin contamination is a problem. However, turmeric at 20 $g.kg^{-1}$ produced the most advantageous results among the treatments, achieving the highest weight gain, specific growth rate, and feed conversion ratio, while ensuring a 100% survival rate across all treatments.

The research was just a 10-week feeding trial and did not examine the long-term physiological or histological impacts of aflatoxin exposure or the incorporation of plant extracts. Also, only two plant-based additives (garlic and turmeric) were tested, and their bioactive compound concentrations were neither standardized nor quantified. These can be for future research alongside the residual aflatoxin levels in fish tissues.

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Modeling the Effect of WWTP Bypass Events on Water Quality in Sebou River Estuary, Morocco

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ABSTRACT

Historically, urban wastewater from Kenitra was directly discharged into the Sebou estuary (68 km) through six collectors, producing environmental degradation and prompting the Kenitra wastewater treatment plant (WWTP) in 2020. Although designed to treat all urban wastewater before discharge, WWTP (19.4 km from the mouth) experiences bypass events during Eid al-Adha when organic loads surge due to widespread animal slaughter. This study aims to model the Bypass impact on Sebou estuary water quality, focusing on biochemical oxygen demand (BOD_5) as a key indicator. Given the strong dependence of water quality on hydrodynamics, a one-dimensional hydraulic model (HEC-RAS5.0.6) was used, calibrated, and validated using morphological datasets. The hydraulic simulation outputs (water levels and flow velocities) were then used in the water quality module to simulate BOD_5 dynamics. Three scenarios were examined: untreated discharge, discharge after treatment at WWTP, and Eid al-Adha bypass event. The results indicated a 90% BOD_5 reduction post-treatment, confirming WWTP efficiency. However, during the bypass event, BOD_5 surged to 4.3 $mg.L^{-1}$, significantly deteriorating Sebou estuary water quality. The pollution residence time varied from 3 days under high freshwater flow (300 $m^3.s^{-1}$) to 9 days under tidal dominance (0 $m^3.s^{-1}$). These findings highlight the urgent need for adaptive wastewater management (pre-treatment during peak periods and public awareness campaigns) during peak-load events to mitigate ecological risks and safeguard downstream communities relying on the estuary for water and livelihoods.

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INTRODUCTION

Estuaries are dynamic environments where freshwater and seawater meet, creating complex ecosystems that are highly sensitive to variations in water quality (Robins et al. 2016, Zedler 2017). Pollution peaks, often due to untreated discharges or exceptional events, can severely disrupt these fragile balances, threatening biodiversity and ecosystem services (Nizar et al. 2022a). One of the main objectives is to establish sustainable management policies and governance rules to preserve estuarine health and ensure water resource sustainability (Kettab 2014). In this context, the present study aims to address the following research question: How do pollution peaks caused by exceptional events, particularly temporary discharges of untreated wastewater with a focus on biochemical oxygen demand (BOD_5) levels, affect estuarine water quality and pollutant residence time under varying hydrodynamic conditions?

The Sebou Estuary, a vital transition zone between the freshwater of the Sebou River and the saltwater of the Atlantic Ocean, supports a rich biodiversity and is essential for maintaining the region's ecological equilibrium (Haddout et al. 2016). However, like many estuarine systems worldwide, the Sebou Estuary faces increasing pressure from anthropogenic activities, including the discharge of urban wastewater (Nizar et al. 2022a). In Kenitra, Morocco, the Kenitra Wastewater

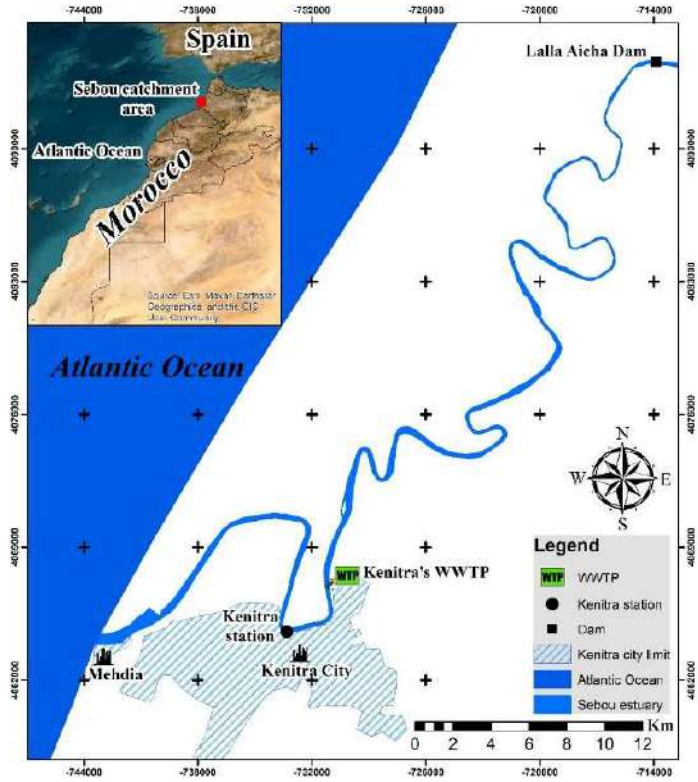

Fig. 1: Study area location.

Treatment Plant (WWTP) serves as the primary point source for treated wastewater discharge into the estuary. While the WWTP significantly improves water quality under normal operating conditions, it experiences periodic bypass events during the Eid al-Adha religious holiday due to the dramatic increase in organic loading from widespread animal slaughter. These bypass events, where untreated wastewater is directly released into the estuary, pose a significant threat to the estuarine ecosystem, raising concerns about the long-term impacts on water quality, ecosystem health, and disruption to the economic and agricultural activities in the region (Nizar et al. 2022b).

The challenge of managing wastewater during Eid al-Adha is not unique to Kenitra. As El Allaoui et al. (2017) highlight in their study of Fez city effluents, the surge in organic waste during this period can exceed the capacity of wastewater treatment facilities. Similar issues related to slaughterhouse waste management during Eid al-Adha have been highlighted by Salehin et al. (2021), emphasizing the need for effective strategies to mitigate the environmental impact of this important cultural event. The vulnerability of WWTPs to operational failures and their subsequent impact on receiving waters has also been demonstrated. For instance, Jaskulak et al. (2022) analyzed a major WWTP failure in Warsaw, Poland, where a collection system malfunction led to the release of more than 4.8 million m^3 of untreated discharge into the Vistula River. Their study revealed significant and prolonged deterioration of water quality in both the estuary and the Baltic Sea, even more than 400 km downstream from the unloading location. While the context of a system malfunction differs from the bypass events during Eid al-Adha in Kenitra, Jaskulak et al. (2022) underscore the potential for large-volume releases of untreated wastewater to have substantial and far-reaching consequences for aquatic ecosystems. These studies, including those focused on Eid al-Adha waste management, highlight the importance of understanding the consequences of such events and developing robust management plans to protect aquatic ecosystems.

Before the construction of the Kenitra WWTP in 2020, untreated wastewater from the city was discharged directly into the Sebou Estuary through six separate collectors (Nizar et al. 2022c). This situation led to significant environmental and health problems, prompting the development of a strategic plan (2015-2020) that included the construction of the WWTP to reduce pollution in the Sebou basin (Nizar et al. 2022c). The WWTP now collects and treats wastewater from Kenitra, becoming the single discharge point for treated

effluent into the estuary. However, the challenge of managing the peak organic load during Eid al-Adha remains.

The Sebou River, one of Morocco's largest waterways alongside the Moulouya and Oum Er-Rbia rivers, drains a watershed of about 40,000 km^2 (5.5 % of the country's total area) in the northwest between 4° and 7° W and 33° and 35° N. Originating in the Middle Atlas Mountains under the name Oued Guigou (Combe 1966), it flows over 600 km before emptying into the Atlantic Ocean at Mehdia, near Kenitra. The Sebou estuary, the focus of this study, covers approximately 70 km between the Lalla Aicha guard dam and the river mouth (Fig. 1). Tidal forces and freshwater releases govern its dynamics from the dam (Mergaoui et al. 2003). The Lalla Aicha guard dam plays a vital role in limiting saltwater intrusion, which historically affected upstream irrigation zones (Haddout et al. 2016).

The hydrodynamics of the estuary, including tidal influence and freshwater flow, govern the transport and fate of pollutants. The semi-diurnal sinusoidal tide, with a cycle of 12.17 hours and an amplitude of 0.97 to 3.11 meters, creates an average discharge of approximately 250 $m^3.s^{-1}$ at the mouth (Touazit et al. 2024). Tidal influence extends 45 km upstream, with patterns of filling at high tide and emptying at low tide (Mergaoui et al. 2003). The effect of wind on the flow is negligible due to the narrowness of the estuary (Haddout et al. 2016). The Sebou estuary is classified as partially mixed, reflecting the balance between river discharge and tidal mixing (Thatcher & Harleman 1981).

Previous studies on the Sebou Estuary (Combe 1966, Mergaoui et al. 2003, Palma et al. 2012, Haddout et al. 2016, Nizar et al. 2022a, 2022b, 2022c, Ech-chayeb et al. 2023, Touazit et al. 2024) have primarily focused on field measurements and the general assessment of pollution levels. While these works provide valuable baseline data, they did not investigate the combined influence of morphological and hydrodynamic conditions on pollution dynamics, nor did they address the specific impact of bypass events from the Kenitra WWTP. This gap is significant, especially considering that although the WWTP has proven effective under normal operation (Nizar et al. 2022a), it remains vulnerable to bypass events during exceptional circumstances, which can temporarily undermine its environmental benefits. The present study contributes new insights by modeling the spatial-temporal evolution of BOD_5 pollution during such events, taking into account hydrodynamic variability. This approach supports environmental managers with rapid and reliable tools for evaluating pollutant dispersion in the estuary and developing adaptive strategies under stress conditions.

One-dimensional mathematical models offer a valuable tool for this purpose due to their relative simplicity, ease of application, and suitability for management scenarios. Furthermore, a methodological approach that begins with simpler representations of the phenomena under study, such as 1D models, is often preferred. This allows researchers to evaluate the limitations of these approximations before progressing to more complex and computationally demanding models, ensuring a balanced approach between model complexity and practical applicability.

This paper aims to model Sebou water quality during WWTP bypass events at Eid al-Adha. The hydraulic regime of the Sebou River estuary was modeled using a one-dimensional model (HECRAS 5.0.6). Calibration and validation of the HEC-RAS model were performed using hydraulic and morphological datasets from 2020. The evolution of hydrodynamic variables (water levels and flow velocities) was simulated by the hydraulic model and used to model the dissolved oxygen (DO) and the biochemical oxygen demand (BOD_5) in the water-quality HEC-RAS module. Three scenarios were analyzed: discharge without treatment, reflecting pre-WWTP conditions (mean BOD_5 of 414.13 $mg.L^{-1}$), discharge after treatment, representing the standard WWTP operation (mean BOD_5 of 21 $mg.L^{-1}$), and the exceptional Eid al-Adha period, during which the WWTP is bypassed (mean BOD_5 of 990 $mg.L^{-1}$). The results highlighted the role of tidal dynamics and upstream freshwater flows in shaping pollutant dispersion. They confirmed that the WWTP significantly reduced BOD_5 levels in the estuary and offered insights into pollutant dispersion patterns and residence times.

This study is the first to assess the impact of WWTP bypass events during Eid al-Adha on water quality in the Sebou Estuary, addressing concerns highlighted by earlier works (Haddout et al. 2016, Nizar et al. 2022a) regarding periods of exceptional organic load that exceed treatment capacity. It offers new perspectives on the performance of the Kenitra WWTP, the risks linked to peak pollution episodes, and the need to incorporate such events into coastal management planning.

MATERIALS AND METHODS

Kenitra's WWTP

The Kenitra WWTP (Fig. 2) employs an activated sludge process with medium load to treat urban wastewater before discharge into the Sebou Estuary. This modern facility, covering 6 hectares, has a current treatment capacity of 700,000 equivalent inhabitants (EH) and is projected to reach 1,050,000 EH by 2030. The treatment process is divided into three modules: water treatment, sludge treatment, and biogas production. The plant also includes a control room

and an analytical laboratory for process monitoring and optimization. A key feature of the WWTP is its focus on energy sustainability. Biogas cogeneration covers over 45% of the plant's electricity needs, and photovoltaic panels provide 100% of the lighting requirements. Treated wastewater is classified as "urban wastewater" (Nizar et al. 2022b).

Normally, treated effluent is discharged into the Sebou Estuary. However, during Eid al-Adha, the Kenitra WWTP often experiences a significant surge in organic load due to widespread animal slaughter. This sudden increase exceeds the facility's treatment capacity (0.7 $m^3.s^{-1}$), resulting in a hydraulic overflow. Consequently, unplanned bypass events occur, during which untreated wastewater is directly released into the estuary (Nizar et al. 2022b).

In 2021, the WWTP conducted a trial to assess its ability to handle the Eid al-Adha wastewater flow. During this year only, the WWTP successfully treated the Eid al-Adha wastewater flow, which reached 0.6 $m^3.s^{-1}$, a level within the plant's capacity. However, during the Eid al-Adha periods of 2020, 2022, 2023, and 2024, the organic load exceeded the WWTP's capacity, resulting in bypass events. The 2021 dataset is thus used as a baseline reference to characterize treated discharges and assess their impact on estuarine water quality. Three simulation scenarios were developed to explore the influence of WWTP operations. This comparative approach provides a clearer understanding of the potential consequences of WWTP performance or failure during high-load periods and helps quantify the environmental risk associated with bypass events.

Biochemical Oxygen Demand (BOD_5) Measurement

Biochemical Oxygen Demand (BOD_5) represents the amount of dissolved oxygen consumed by bacteria to partially degrade or fully oxidize biodegradable organic matter in water (Das et al. 2024). In this study, BOD_5 analyses at the WWTP were conducted daily at the RAK laboratory (Régie Autonome de Kénitra) using water samples collected with the Endress+Hauser XE4302.2, an instrument designed for continuous water monitoring and automatic sampling. BOD_5 was determined using the OxiTop® system, a manometric respirometric technique based on pressure changes in a closed system. As microorganisms consume oxygen and produce CO_2, which is absorbed by sodium hydroxide (NaOH) pellets, allowing indirect measurement of oxygen uptake via the resulting pressure drop (Rahmati et al. 2021). Two measurement modes were employed: for raw wastewater, 164 mL of sample was analyzed and the result multiplied by 10, for treated wastewater, 43.5 mL was used and the result multiplied by 50. Since BOD_5 levels are inherently dependent on the amount of available organic matter, the measurement system was calibrated based on the volumes of the analyzed samples. Temperature stabilization is crucial for accurate BOD_5 measurements due to its influence on biological activity (Maddah 2022). All BOD_5 measurements were performed at a controlled temperature of 20°C within a thermostatically controlled cabinet.

Mathematical Hydraulic Model

The one-dimensional approach was applied in this study, as it is suitable for long-distance river courses. The HEC-RAS (5.0.7) mathematical model, developed by the Hydrologic Engineering Center of the U.S. Army Corps of Engineers (Brunner 2016), was used to simulate open-surface flows. This integrated hydraulic analysis software can simulate steady and unsteady flows, sediment transport, and other functions that aid in the design and analysis of hydraulic

Fig. 2: Overview of the location of the Kenitra's WWTP.

structures (Nizar et al. 2022a). It is important to note that while the 1D HEC-RAS model is well-suited for simulating longitudinal flow and pollutant transport in elongated estuarine systems, it does not account for lateral mixing or vertical stratification. These limitations may affect the spatial accuracy of water quality predictions in areas where multidimensional flow dynamics are significant. The Sebou estuary's water quality is strongly influenced by its hydraulic regime. The one-dimensional Saint-Venant equations, which govern the conservation of mass and momentum, form the basis of the HEC-RAS model and are expressed as follows (Brunner 2016):

$$\frac{\partial Q}{\partial x} + \frac{\partial A}{\partial t} - q_1 = 0 \quad ...(1)$$

$$\frac{\partial (VA)}{\partial t} + \frac{\partial (QV)}{\partial x} + gA\left(\frac{\partial Z}{\partial x} + S_f\right) = 0 \quad ...(2)$$

Where V is the velocity ($m.s^{-1}$), Q is river flow ($m^3.s^{-1}$), A denotes the cross-section area (m^2), q_l represents lateral inflow ($m^2.s^{-1}$), x and t are the distance of the estuary (m) and time (s), respectively, g indicates acceleration of gravity (9.81 $m.s^{-2}$), $\partial Z/\partial x$ refers to the flow surface slope (m), and S_f (Dimensionless) is the frictional slope expressed as (Nizar et al. 2022a):

$$S_f = \frac{Q|Q|n^2}{2.208\, A^2 R^{\frac{4}{3}}} \quad ...(3)$$

Where R (m) is the hydrodynamic radius, and n represents Manning's roughness coefficient ($1.m^{-1/3}.s^{-1}$). The initial estimation of Manning's roughness coefficient integrated into equation (3) is obtained using the empirical equation discovered by Chow (1973), expressed as:

$$n = (n_0 + n_1 + n_2 + n_3 + n_4).m_5 \quad ...(4)$$

Where n_0 represents the correction factor for river bed granularity, n_1 refers to the correction factor of the influence of irregularity in river, n_2 refers to the correction factor for cross-section type and shape, n_3 refers to the correction factor for the effects of obstacles, n_4 means the correction for the effect of the presence of vegetation on the banks, and m_5 denotes the correction factor for the degree of meandering in the river. The n_0 factor was assessed based on the granulometric data collected along the estuary. The other factors were also assessed during the field visits, cross-section, available photos, and aerial photos. Equations (1) and (2) were solved using the four-point implicit box finite difference scheme. The commonly used forms of the derived equations for a function *f* are presented below. The time and spatial derivatives are given in equations (5) and (6):

$$\frac{\partial f}{\partial t} \approx \frac{\Delta f}{\Delta t} = \frac{0.5\left(\Delta f_{j+1} + \Delta f_j\right)}{\Delta t} \quad ...(5)$$

$$\frac{\partial f}{\partial x} \approx \frac{\Delta f}{\Delta x} = \frac{\theta\left(\Delta f_{j+1} - \Delta f_j\right) + \left(f_{j+1} - f_j\right)}{\Delta x} \quad ...(6)$$

The value of the function f is given in equation (7):

$$f \approx \bar{f} = 0.5.\theta.\left(\Delta f_j + \Delta f_{j+1}\right) + 0.5\left(f_{j+1} + f_j\right) \quad ...(7)$$

Where θ represents the weighting factor, its default value in HEC-RAS is 1 (Brunner 2016). Solving the system of equations needs spatial discretization of the river estuary into characteristic grids and determining the geometry of the river, upstream and downstream boundaries, and initial river flow. The Sebou estuary was divided into 1310 grids, each ranging from 46 to 49 meters in length, with an average of 48 meters. For each grid, the length, cross-sectional area, and Manning friction factor were specified.

Transport Model

In the HEC-RAS model, the water quality transport module uses the advection-dispersion equation, based on the mass balance equation, with supplementary terms to incorporate lateral inflows (Brunner 2016). For BOD_5 simulation, the transport equation is expressed as follows (Brunner 2016):

$$\frac{\partial (AC_{BOD})}{\partial t} = -\frac{\partial (QC_{BOD})}{\partial x} + \frac{\partial}{\partial x}\left(AD_x \frac{\partial C_{BOD}}{\partial x}\right) - AK_1 C_{BOD} + AR_{BOD} \quad ...(8)$$

Where C_{BOD} represents the organic matter concentration ($kg.m^{-3}$), R_{BOD} denotes the organic matter release ($mg.L^{-1}$), K_1 indicates the coefficient of oxidation ($1.days^{-1}$), which was empirically calibrated using field observations, and D_x is the dispersion coefficient ($m^2.s^{-1}$), which is the key parameter that must receive appropriate evaluation. The HEC-RAS water quality module utilizes the ULTIMATE QUICKEST explicit numerical scheme developed by Leonard to solve the advection-dispersion equation. The resulting finite difference solution for equation (8) is given as follows (Brunner 2016):

$$V^{n+1}C^{n+1} = V^n C^n + \Delta t \times \left(Q_{up}C^*_{up} - Q_{dn}C^*_{dn} + D_{dn}A_{dn}\frac{\partial C^*}{\partial x}\Big|_{\mathrm{dn}} - D_{up}A_{up}\frac{\partial C^*}{\partial x}\frac{\partial C^*}{\partial x}\Big|_{up}\right) \quad ...(9)$$

Where V^{n+1} and V^n (m^3) are the water quality cell volumes at the next and current time steps, respectively, C^{n+1} and C^n ($kg.m^{-3}$) represent the constituent concentration at the current and preceding time step, respectively, C^*_{up} denotes the upstream face QUICKEST concentration ($kg.m^{-3}$), Q_{up} refers to the flow of the upstream face ($m^3.s^{-1}$), $(\partial C^*/\partial x)_{up}$ indicates the QUICKEST derivative at the upstream face ($kg.m^{-4}$), D_{up} refers to the upstream face dispersion coefficient ($m^2.s^{-1}$), and A_{up} refers to the upstream face area (cross-sectional) (m^2). The water quality module inputs are the initial concentration, boundary conditions concentrations,

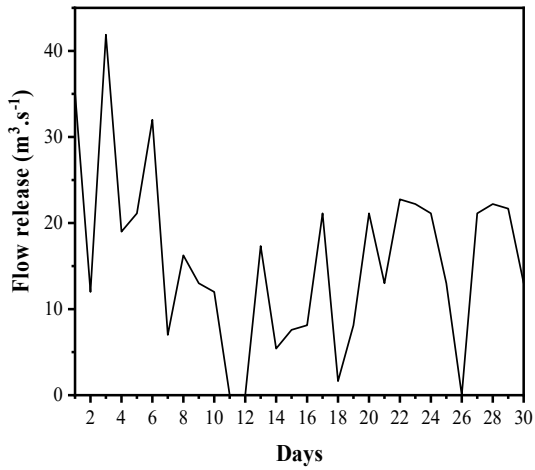

Fig. 3: Flow release by Lalla Aicha dam (upstream boundary condition), June 2021.

and dispersion coefficient. This parameter was computed using the Fischer equation (Fischer et al. 1979):

$$D_x = 10.62\left(\frac{U^2w^2}{U^*h}\right) \quad ...(10)$$

Where w is the mean channel width (m), U denotes the flow velocity ($m.s^{-1}$), U* corresponds to the shear velocity ($m.s^{-1}$), and h represents the mean channel depth (m).

RESULTS AND DISCUSSION

Calibration and Validation of Hydraulic Model

The boundary conditions of the hydrodynamic model are constituted by the flows released by the Lalla Aicha guard dam (upstream condition) obtained from the Hydraulics Directorate of Kenitra (Fig. 3) and the temporal evolution of the water level at the estuary mouth (Port of Mehdia) (downstream condition) obtained from SHOM (Maritime Hydrographic and Oceanographic Service) (Fig. 4).

In the hydraulic simulation, the hydrodynamic model was subjected to calibration and validation. The Manning roughness coefficient in the estuary is the parameter used for calibration. Calibrating a model consists of simulating a given period, knowing the initial and boundary conditions, and comparing the model outputs (water level, flow, etc.) with field measurements by adjusting the Manning coefficient, which is initially estimated by the Cowan and Chow formula. This coefficient was uniformly adjusted along the entire study reach, assuming that the sources of error influencing its variation were consistent across all grid cells.

Water level data from the Kenitra station (Fig. 1) were used to calibrate and validate the model, as these measurements can be obtained there by the National Ports Agency (ANP). The calibration process utilized data from June 1st, 2021, to June 15th, 2021. A strong agreement was obtained between the water level simulated by the model and that measured at Kenitra station (Fig. 5). To confirm the calibration results, a model validation test is usually performed. This test consists of simulating the flow regime over a period other than the one used in the calibration while keeping the same Manning coefficient values. In this study, the validation test was performed for the period ranging from June 15th, 2021, to June 30th, 2021. The output variables are well reproduced, which attests to the performance of the model.

The performance of the hydraulic model was evaluated using statistical indicators, including the root-mean-squared error (RMSE), normalized objective function (NOF), and Nash-Sutcliffe coefficient (NSC) (Abbi et al. 2025). The RMSE gives a measure of the model error, with a value of 0 indicating perfect agreement between measured and

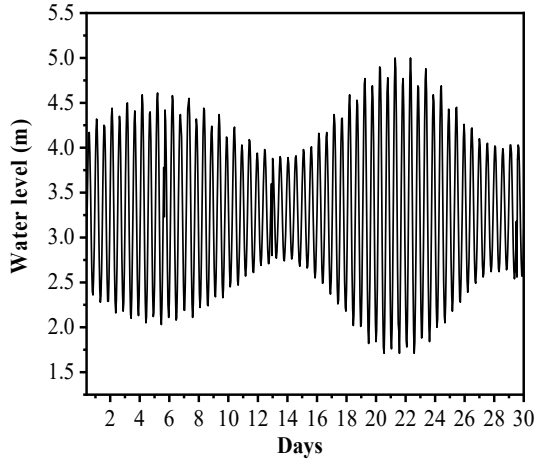

Fig. 4: Semi-diurnal tide at estuary mouth (Downstream boundary condition), June 2021.

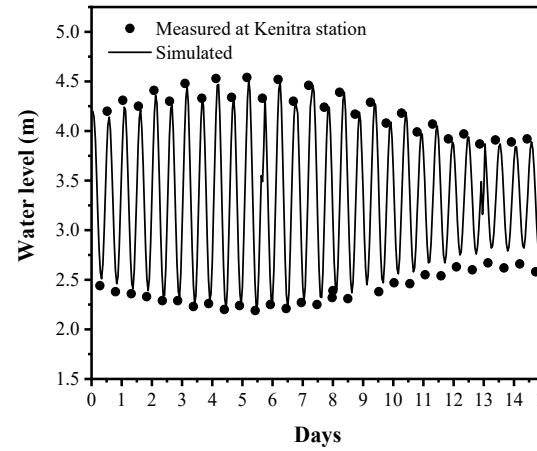

Fig. 5: Hydraulic calibration using water level from Kenitra station (from 1st to 15th June 2021).

Table 1: Statistical indicators used to assess the performance of the model.

Statistical indicators	RMSE	NOF	NSC
Calibration phase	0.18	0.05	0.98
Validation phase	0.11	0.04	0.99

simulated values. For NSC, it ranges between -∞ and 1, with values tending towards 1 indicating the strong performance of the model, and the NOF, with a value less than 1 indicating negligible model error (Abbi et al. 2025). The statistical analysis revealed an NOF value below 1, a low RMSE, and an NSC value near 1, indicating the model's efficiency and confirming the success of its calibration and validation (Table 1).

The outputs of the HEC-RAS model make it possible to estimate the spatiotemporal evolution of numerous hydraulic parameters, such as river velocity, water level, and river flow. Fig. 6 shows the velocity evolution from downstream to upstream in the section subject to study for both tide states (high tide and low tide) during spring and neap tides. A strong fluctuation is observed in the flow velocity, which is due to the influence of the estuary's morphology and bottom. The spring tide period's velocities are higher than those of the neap tide, especially downstream. At high tide, the velocities are also significant but in the opposite direction. Also, the results show sites with high velocity values (from 3 to 8 km, 15 km, 20 km).

Water Quality Simulation Results

Water quality simulations for the Sebou Estuary were conducted between June and August 2021. The dispersion coefficient, calculated using the Fischer equation, was estimated at 150.27 $m^2.s^{-1}$. Three scenarios were analyzed: untreated urban wastewater discharge, treated effluent discharge from the WWTP, and a bypass event scenario during peak pollution periods such as Eid al-Adha, where treatment is temporarily suspended.

In the primary simulation, the inflow of urban wastewater into the Sebou estuary is shown in Fig. 7, while the corresponding BOD_5 concentrations are illustrated in Fig. 8. On the day of Eid al-Adha, the discharge flow reached to 0.55 $m^3.s^{-1}$, reflecting a 14% increase compared to the average flow observed during the monitoring period (Fig. 7). Simultaneously, the BOD_5 concentration rose to 990 $mg.L^{-1}$, representing a 139% increase relative to the average concentration recorded during the same period (Fig. 8).

Fig. 9 shows the longitudinal variation of BOD_5 concentrations from the river mouth up to the Lalla Aicha guard dam under four tidal phases: high tide, falling tide, low tide, and rising tide. BOD_5 levels increase both upstream and downstream, but are significantly higher in the downstream

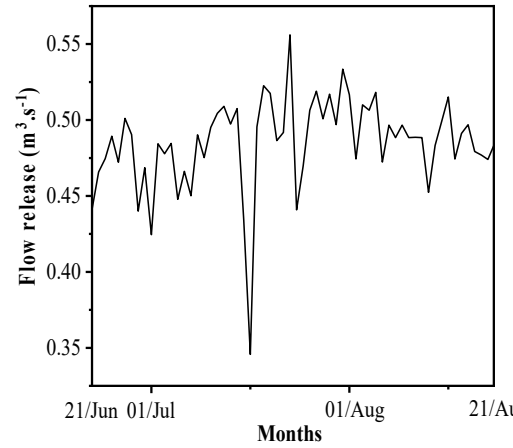

Fig. 7: Evolution of raw water flow discharged from Kenitra's WWTP (June 21st to August 21st, 2021).

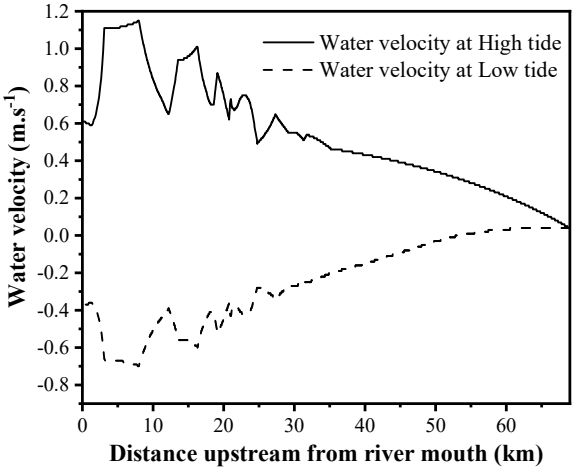

Fig. 6: Velocity profile estimated at high and low tides along the Sebou estuary.

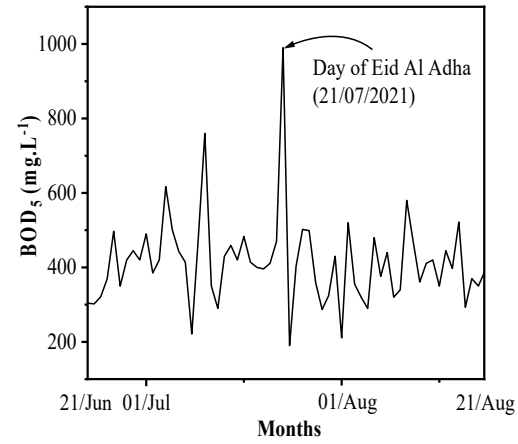

Fig. 8: BOD_5 concentration in raw water flow (lateral condition) (June 21st to August 21st, 2021).

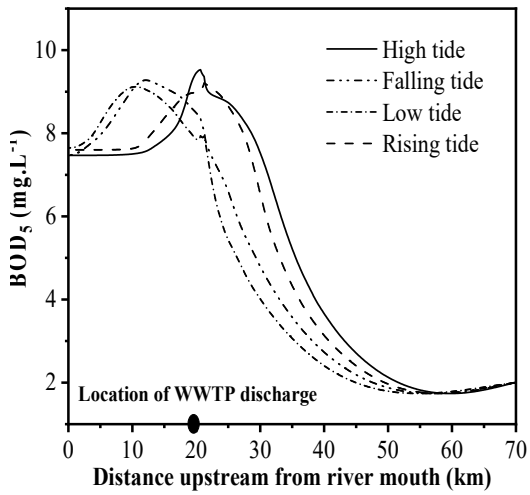

Fig. 9: Longitudinal evolution of BOD_5 along the Sebou estuary for untreated urban waters on July 21st, 2021.

sections. The BOD_5 concentrations at downstream from the WWTP during high tide exceed those observed during low tide, with a mean increase of 0.64 $mg.L^{-1}$ attributed to the dilution effect of cleaner continental waters. The movement of pollutants discharged into the estuary follows the tidal rhythm, transported downstream at low tide and driven upstream at high tide, resulting in pollutant accumulation downstream. However, this accumulation gradually decreases as a result of biochemical reactions and dispersion. Importantly, the increase in BOD_5 does not affect areas beyond 35 km from the river mouth.

The second simulation focuses on urban wastewater after treatment at the WWTP. The concentration of BOD_5 for treated urban wastewater at WWTP and discharged into the estuary is shown in Fig. 10. This reveals a considerable reduction of the BOD_5 concentration in the discharge. Previously, the untreated discharge ranged from 200 $mg.L^{-1}$ to 1000 $mg.L^{-1}$ (Fig. 8), which fails to comply with WHO guidelines for discharges into aquatic ecosystems. The installation of the WWTP significantly reduces pollution, so the BOD_5 concentrations typically do not exceed 20 $mg.L^{-1}$, except on Eid al-Adha, when they peaked at 50 $mg.L^{-1}$, which corresponds to 5% of the BOD_5 levels found in untreated discharge. This indicates that the WWTP reduced the BOD_5 levels during Eid al-Adha by 95%.

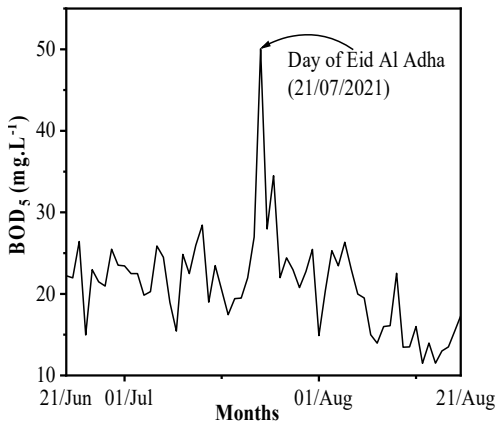

Fig. 10: BOD_5 concentration in water treated flow (lateral condition) (June 21st to August 21st, 2021).

Fig. 11 highlights the progressive reduction of BOD_5 concentrations from upstream to downstream, primarily due to biochemical degradation and dispersion processes. No significant deterioration in water quality was observed, including the Eid al-Adha period. These results confirm the effective performance of the Kenitra WWTP in handling increased organic loads under controlled operational conditions, as recorded in 2021. The reduction of BOD_5 in the Sebou estuary is given as follows:

$$\mathrm{BOD_5 reduction} = \left(\frac{BOD_5 \text{before treatment} - BOD_5 \text{after treatment}}{BOD_5 \text{before treatment}}\right) \times 100$$

The BOD_5 concentration in the Sebou River decreased from 9.8 $mg.L^{-1}$ before treatment to 0.9 $mg.L^{-1}$ after treatment, corresponding to an approximate reduction of 90%.

Simulation of WWTP Bypass During Eid al-Adha

Final simulations considered a discharge treated except that of the Eid al-Adha day (bypass in the WWTP), as shown in Fig. 12. The concentration of BOD_5 during Eid al-Adha is 990 $mg.L^{-1}$, which represents a 4591.9% increase from the average concentration observed during the monitoring period (50 $mg.L^{-1}$).

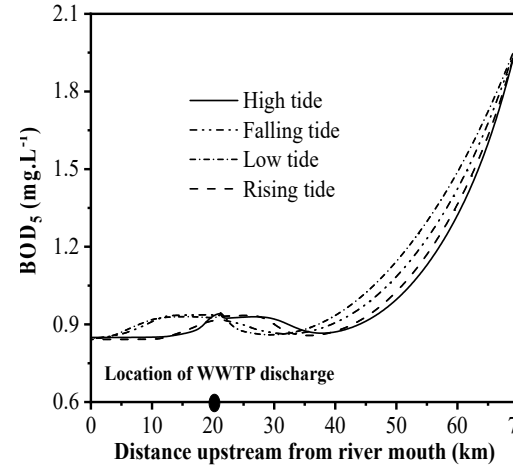

Fig. 11: Longitudinal evolution of BOD_5 along the Sebou estuary for treated urban waters on July 21st, 2021.

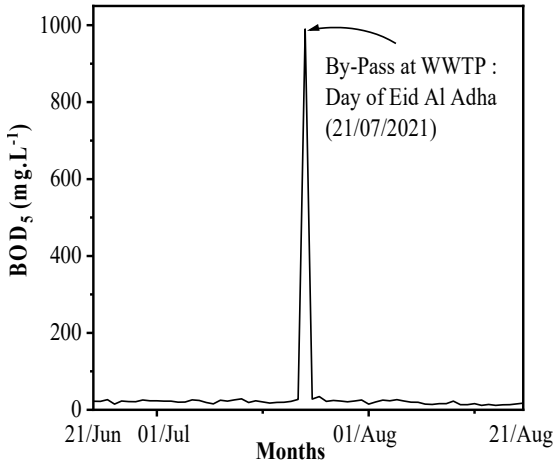

Fig. 12: BOD_5 concentration in the Bypass event at the WWTP during Eid al-Adha.

Fig. 13 shows the longitudinal evolution of BOD_5 concentrations from downstream to upstream under different residence times, following the end of the WWTP bypass event during Eid al-Adha, with a flow release of 43 $m^3.s^{-1}$. A marked deterioration in water quality was observed, particularly within the 0–35 km stretches from the estuary mouth, an area encompassing sensitive ecological zones adversely affected by decreased oxygen availability. BOD_5 concentrations rose to 4.3 $mg.L^{-1}$, reflecting a significant increase in organic pollution. Moreover, the residence time of this pollution within the estuary was approximately 7 days, reflecting the system's limited dilution and flushing capacity at this flow rate. The BOD_5 was reduced by approximately 56% during the WWTP bypass event on Eid al-Adha, compared to a 90% reduction under normal operating conditions.

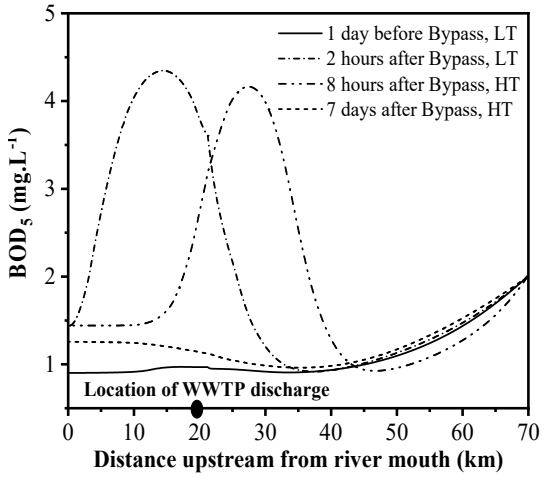

Fig. 13: BOD_5 along the estuary at different residence time after bypass event, for Q=43 $m^3.s^{-1}$, LT: low tide and HT: high tide.

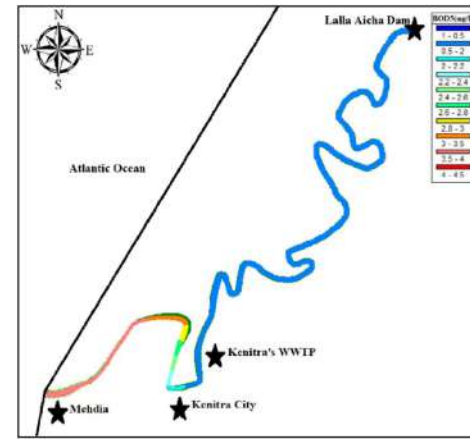

Fig. 14: BOD_5 distribution in the estuary at 2h after bypass event, for Q=300 $m^3.s^{-1}$, at low tide.

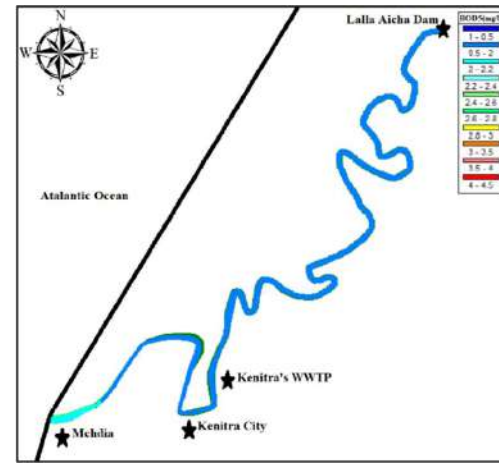

Fig. 15: BOD_5 distribution in the estuary 3 days after bypass event, for Q=300 $m^3.s^{-1}$, at low tide.

In this study, three simulations were carried out to assess the impact of freshwater flow rates released from the Lalla Aicha guard dam on the residence time of pollutants resulting from the WWTP bypass event in the Sebou estuary. These simulations considered two characteristic flow scenarios: a first with a free-flow condition at 300 $m^3.s^{-1}$ and a second simulating a closed dam at 0 $m^3.s^{-1}$. The analysis of BOD_5 discharges from the Kenitra WWTP under these different flow conditions offers valuable insights into the behavior and persistence of pollutants within the estuarine system.

For a free-flow condition at 300 $m^3.s^{-1}$, Figs. 14 and 15 show that after the discharge ended, BOD_5 released from WWTP was rapidly evacuated towards the ocean. The residence time was reduced to approximately 3 days, and a notable decrease in BOD_5 concentrations was observed. This is attributed to the enhanced flushing action, combined

with natural attenuation processes such as dispersion and biodegradation.

Conversely, under conditions where no river flow is released (0 $m^3.s^{-1}$) from the Lalla Aïcha guard dam (upstream end), Figs. 16 and 17 show that the pollutant remains within the estuary even after 9 days, maintaining a BOD_5 concentration level of approximately 2 $mg.L^{-1}$. The limited dilution and circulation due to tidal dominance result in a residence time extending up to 10 days. These findings emphasize the critical role of freshwater input in controlling pollutant dispersion and improving estuarine water quality during pollution peaks.

In this low-flow context, marine tides emerge as influential regulators of BOD_5 discharges. The dispersion and movement of pollutants within the estuary are controlled by the tidal cycles. This observation highlights the significance of not only considering flow rates but also recognizing the intricate interdependencies between tidal dynamics and the fate of pollutants in the Sebou estuary water quality during peak pollution events such as Eid al-Adha.

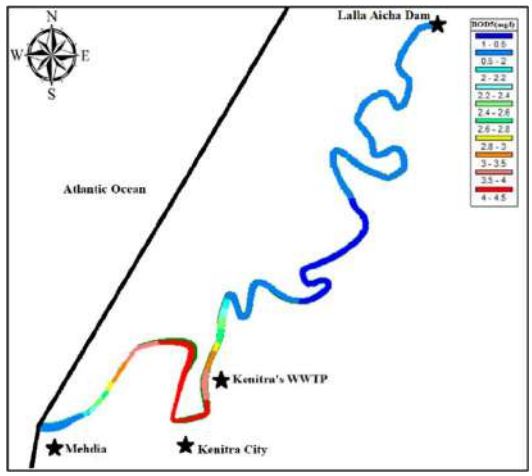

Fig. 16: BOD_5 distribution in the estuary 2h after bypass event, for Q=0 $m^3.s^{-1}$, at low tide.

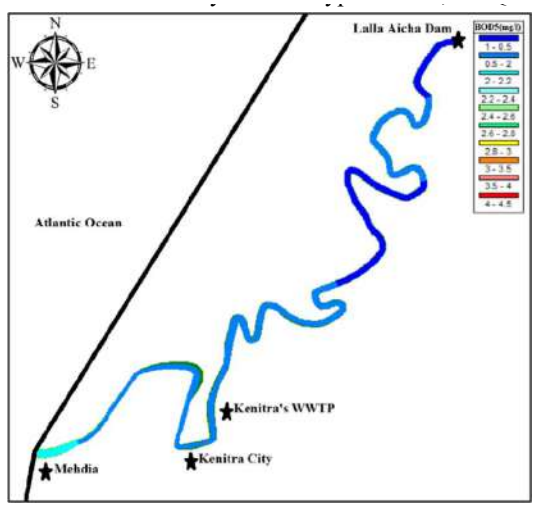

Fig. 17: BOD_5 distribution in the estuary 9 days after bypass event, for Q=0 $m^3.s^{-1}$, at low tide.

CONCLUSIONS

This study assessed the effect of bypass discharges from the Kenitra WWTP on Sebou estuary water quality during the pollution peak period associated with Eid al-Adha, using a calibrated and validated one-dimensional hydraulic model (HEC-RAS). The water quality module simulated BOD_5 variations along the estuary, based on hydraulic conditions. The findings revealed that, under normal operation, the WWTP achieves approximately 90% BOD_5 removal efficiency. However, during bypass events such as Eid al-Adha, this efficiency drops to around 56%, leading to BOD_5 concentrations of up to 4.3 $mg.L^{-1}$ in the estuary. These elevated levels can negatively affect aquatic ecosystems, particularly by reducing dissolved oxygen availability-critical for fish and invertebrates. This, in turn, poses risks to local fisheries and agricultural irrigation that rely on clean water sources. The simulation highlighted the influence of tides and river flows on the fate and dispersion of pollutants. At a freshwater flow of 300 $m^3.s^{-1}$ (freshwater dominance), complete decontamination of the WWTP bypass discharge occurs after an estimated residence time of 3 days, enabling faster evacuation of the discharge to the ocean. Under tidal dominance (0 $m^3.s^{-1}$ freshwater flow), pollutant retention increases, requiring about 10 days for full decontamination. These results highlight the necessity of addressing peak pollution events, such as those occurring during Eid al-Adha, in wastewater management strategies and WWTP design. To mitigate the impact of such events, several practical and quantitative measures can be considered. These include increasing treatment capacity, implementing pre-treatment during peak periods, launching public awareness campaigns to reduce organic waste, and increasing the flow release during Eid al-Adha to approximately 300 $m^3.s^{-1}$. This specific flow rate would help limit the residence time to 3 days, thereby significantly reducing the accumulation of organic pollutants. This study provides valuable insights and modeling tools to support informed decision-making for improved wastewater management and the protection of estuarine ecosystems.

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Effects of Climate Change on Drought: A Systematic Review of Drought Indices and Climate Change

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ABSTRACT

Global weather patterns are greatly impacted by climate change, making droughts more frequent and severe, especially in regions with limited adaptation capacity. This review evaluates the strengths and limitations of widely used drought indices in the context of climate change. Our analysis identifies the Standardized Precipitation Evapotranspiration Index (SPEI) and the Normalized Difference Vegetation Index (NDVI) as the most robust tools for monitoring drought under current and projected climate scenarios, with CMIP6 models indicating increased drought risk for vulnerable regions such as South Asia. The integration of remote sensing and artificial intelligence enhances the accuracy and adaptability of drought monitoring. The findings highlight the need for region-specific frameworks and actionable recommendations for researchers, policymakers, and technologists to improve drought resilience and management strategies.

INTRODUCTION

Environmental and climate change have been profound over the past century. Severe natural calamities, such as droughts and floods, have been triggered by global heating, leading to changes in water distribution throughout the hydrological cycle (Leng et al. 2015). Accelerated population growth and climate change have emerged as the most significant obstacles to sustainable human resource development and the conservation of natural systems. Humans have modified drought characteristics during the Anthropocene, so they may no longer be regarded as "natural hazards" in their entirety (Van Loon et al. 2016, Haile et al. 2020). Drought is generally characterised as an abnormal lack of moisture compared to a standard reference point, but it is more specifically categorised depending on the particular phase of the water cycle in which these deviations in moisture emerge (Wilhite & Glantz 1985).

The concept of drought lacks a broadly agreed-upon definition. According to McMahon and Diaz Arenas (1982), drought is a prolonged period of arid weather that affects the water supply, producing a moisture scarcity for human use. The drought phenomenon has long been a focal point of interest among ecologists. Research publications titled "Drought" have been published since at least the 1920s (Gorham & Kelly 2018), and the ecological effects of drought have long been studied. Due to climate model forecasts of increasingly frequent, severe, and pervasive water shortages, curiosity has grown in this subject in recent decades (Stocker et al. 2013). Globally, the impact of drought on terrestrial ecosystems has increased over the last century, as confirmed by many investigations (Schwalm et al. 2017, Du et al. 2018). Generally, drought severity can be measured by drought indices using drought indicators.

Drought is a complex phenomenon, often described as a prolonged period of water scarcity that results from significant moisture deficits compared to historical norms. It directly impacts agriculture, water supply, ecosystems, and economies. Historically, droughts have triggered severe consequences, including famines and ecosystem degradation. Unlike other natural hazards, drought's onset and termination are often slow and challenging to predict, making it particularly devastating. Understanding drought in the context of climate change is increasingly critical, as climate models project more frequent, severe, and widespread water shortages in the coming decades (Rahman 2017, Wilhite 2000).

Various drought indicators and indices have been developed to quantify and monitor drought severity, each tailored to specific aspects of the hydrological cycle and regional characteristics. For instance, indices such as the Standardized Precipitation Index (SPI), Standardized Precipitation Evapotranspiration Index (SPEI), and the Palmer Drought Severity Index (PDSI) are widely utilized but have varying degrees of effectiveness in capturing drought dynamics under changing climatic conditions (Dixit et al. 2022). These indices help contextualize drought severity, offering critical insights for policymakers and researchers. However, traditional methods often struggle with the challenges posed by climate change, such as non-stationarity and the increasing influence of human activities on hydrological patterns.

The main aim of this review paper is to provide a comprehensive synthesis of current research and understanding regarding the correlation between climate change and drought. The review systematically incorporates recent studies to analyze drought indices and indicators, emphasizing their applicability in evolving climatic conditions. A detailed evaluation of the 25 most widely recognized drought indices is presented, focusing on their methodological strengths, limitations, and relevance to contemporary research challenges. These indices are assessed within the context of changing climate scenarios to analyze the research undertaken on the connection between drought and climate variability. Most past reviews have focused either on specific drought indices or particular geographic regions, whereas this paper provides a broader comparative evaluation of 25 indices in the context of climate change, addressing a critical gap in current literature.

The study also explores the potential for integrating advanced technologies, such as remote sensing and artificial intelligence, to develop hybrid drought indices that address the limitations of traditional approaches. By highlighting these advancements, the review underscores the need for innovative, region-specific frameworks to enhance drought resilience.

This review synthesizes recent advancements in drought monitoring to address critical gaps in understanding how traditional and emerging indices perform under evolving climate conditions. By evaluating the robustness of drought indices across historical, CMIP5, and CMIP6 scenarios, we identify those most resilient to temperature-driven hydrological shifts and non-stationary climatic patterns. The analysis systematically compares their efficacy in capturing drought impacts across meteorological, agricultural, hydrological, and ecological domains, emphasizing regional applicability and scalability. Furthermore, we explore the potential of integrating remote sensing and artificial intelligence to overcome limitations in data resolution, socio-economic integration, and real-time adaptability. Through this synthesis, the review provides a foundation for developing adaptive frameworks that enhance drought resilience, offering actionable insights for researchers and policymakers to bridge the gap between theoretical advancements and practical implementation in water resource management.

MATERIALS AND METHODS

To achieve the aim and objectives of this research, a systematic and comprehensive approach was employed to analyse the correlation between climate change and drought, with a specific focus on drought indices and their applicability under evolving climatic conditions. The methodology began with a thorough review of existing scientific literature and research papers on climate change, drought characteristics, and drought indices, including peer-reviewed journals, IPCC reports, and datasets from CMIP5 and CMIP6 models. Relevant studies from the past two decades were prioritized to ensure the inclusion of recent advancements and findings. Publicly available datasets, including those from climate models and remote sensing technologies, were gathered to provide a robust basis for analysing drought patterns and their connection to climate variability.

The study then categorized droughts into distinct types-meteorological, agricultural, hydrological, socio-economic, ecological, groundwater, and flash droughts-to ensure a comprehensive understanding of the phenomenon. Drought indicators and indices, such as the Standardized Precipitation Index (SPI), Standardized Precipitation Evapotranspiration Index (SPEI), Palmer Drought Severity Index (PDSI), and other composite indices, were identified and evaluated, focusing on their methodological strengths, limitations, and applicability in the context of climate change. The CMIP6 Scenario Model Intercomparison Project was utilized to

analyse future drought projections under different emission and socio-economic pathways. A comparative analysis of CMIP5 and CMIP6 models was performed to understand the advancements in sensitivity and accuracy in predicting drought conditions.

The study also explored the potential of remote sensing and artificial intelligence for drought monitoring and management, examining existing hybrid drought indices and proposing frameworks for integrating advanced technologies to improve the accuracy and applicability of drought monitoring tools. Gaps in existing methodologies were identified, particularly in terms of data availability, spatial resolution, and the inclusion of socio-economic factors in drought assessment. Challenges related to non-stationarity in climate models and the increasing influence of anthropogenic activities on hydrological cycles were also analysed.

Based on the findings, region-specific and innovative frameworks were proposed to enhance drought resilience and improve monitoring and mitigation strategies. Recommendations for improving water resource governance and addressing the economic impacts of drought were included to assist policymakers in developing effective strategies. This systematic approach ensured that the research covered all aspects of the complex relationship between climate change and drought, providing a detailed evaluation of drought indices and proposing advanced methodologies for drought monitoring and mitigation. Methodology adopted for the Systematic Review is shown in Fig. 1.

A rigorous search and screening process ensured extensive coverage and reduced bias in this review. We searched Scopus, Web of Science, and Google Scholar for recent peer-reviewed drought indices and climate change literature. We also incorporated pertinent IPCC reports and carefully chosen grey literature on CMIP5, CMIP6 forecasts, and AI-based drought monitoring frameworks to capture growing trends and state-of-the-art methodologies.

About 500 publications were found using keywords such as "drought indices," "climate change," "CMIP5," "CMIP6," "remote sensing," and "AI-based drought monitoring." About 150 full-text articles were eligible after removing duplicates and assessing titles and abstracts for relevance. Approximately 50 core papers were selected for further examination based on methodological rigour, relevance to climate-based drought assessment, and contribution to indices comparative understanding. Fig. 2 shows a PRISMA-style flowchart of research inclusion and exclusion.

This narrative review synthesises information from multiple sources; however, we included only peer-reviewed and high-impact research in the final comparative matrix. Since the review was a comprehensive, comparative overview rather than a quantitative meta-analysis, GRADE or risk-of-bias scoring was not performed. The Results and

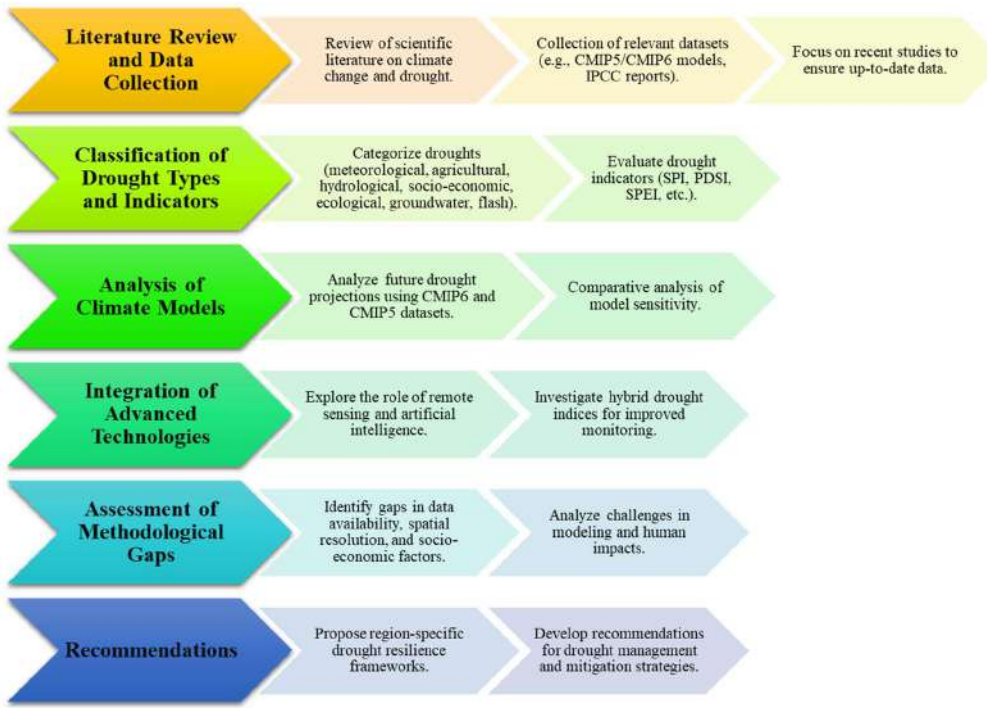

Fig. 1: Methodology Adopted for the Systematic Review.

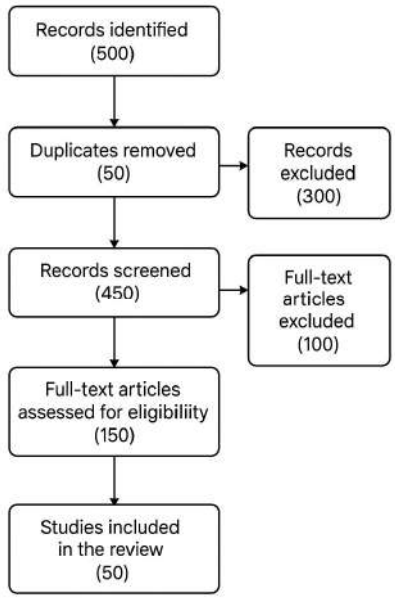

Fig. 2: PRISMA-style flow diagram of the literature screening and selection process.

Discussion sections provide theme evaluation and index ranking based on the chosen studies. The manuscript cites 123 references for contextual and conceptual support, but only 50 core studies were included in the systematic review matrix.

While this review does not directly apply artificial intelligence (AI) methods, it systematically examines how AI has been integrated into drought monitoring and index development in the existing literature. Our review process included identifying studies that utilize machine learning and other AI techniques to process remote sensing data, develop hybrid indices, and improve drought prediction accuracy. These studies were analyzed to understand the current state, advantages, and challenges of AI in drought research. Our methodology thus provides a comprehensive synthesis of AI's role in advancing drought monitoring, as reflected in recent scientific work.

Ranking System for Drought Indices

To objectively assess and compare the robustness of drought indices for monitoring under changing climatic conditions, we developed a transparent ranking system based on measurable criteria. The choice of criteria was informed by the need to evaluate indices according to their scientific rigor, practicality, and suitability for diverse climate scenarios, as recommended in the literature on drought indicators and indices (Svoboda & Fuchs 2016).

Criteria for Ranking

We selected the following six criteria, each relevant to the effective application of drought indices in operational and research contexts:

i. Climate Sensitivity: Measures the extent to which the index accounts for temperature, precipitation, and evapotranspiration, reflecting its responsiveness to climate change.

ii. Data Requirement: Evaluates the ease of obtaining the input data required for the index, including data availability and accessibility.

iii. Spatial Resolution: Assesses the suitability of the index for application at local, regional, or global scales.

iv. Temporal Resolution: Considers the frequency and flexibility of monitoring (e.g., daily, monthly, seasonal, annual).

v. Performance under Projected Climate Scenarios (CMIP5/CMIP6): Rates the index's reliability and adaptability under current and future climate model projections.

vi. Operational Usability: Reflects the ease of implementation, interpretability, and integration into existing drought early warning systems or policy frameworks.

Scoring Method

Each index was scored on a scale of 1 (low) to 5 (high) for each criterion, based on a review of published literature, expert consensus, and operational case studies. The total score for each index was calculated by summing the scores across all criteria, providing a quantitative basis for comparison. This approach ensures that the ranking is transparent, reproducible, and grounded in both scientific and practical considerations (Dikici 2020, Patil et al. 2023, Jain et al. 2015).

CLASSIFICATION OF DROUGHTS

The droughts are being recognised as a known natural calamity among water scientists, agricultural specialists, ecological researchers, geographers, and forecasters (Ashok et al. 2020). According to Wilhite and Glantz, there are four distinct types of droughts. Meteorological factors include insufficient precipitation, agricultural factors include insufficient soil moisture to sustain crop growth, hydrological factors include deficiency in streamflow and groundwater resources, and social factors include the inability to fulfil water requirements (Wilhite & Glantz 1985).

The conceptual definition of drought provides an overview of its fundamental ideas and a general explanation of the physical processes involved. This involves the lack

Table 1: Different types of drought classification and their description.

Type of Drought	Description
Meteorological Drought	Insufficient precipitation compared to normal levels over specific time scales (e.g., decadal, annual, monthly). Regional and climate-specific.
Agricultural Drought	Inadequate soil moisture to meet the needs of crops and pastures during critical growth stages, leading to reduced agricultural productivity.
Hydrological Drought	Deficiency in surface and groundwater resources, affecting streamflow, reservoir levels and the overall hydrological cycle.
Socio-Economic Drought	Water scarcity impacts society and industry, disrupting trade and reducing access to essential water-dependent commodities and services.
Ecological Drought	Prolonged water shortages that disrupt ecosystem functions, services and biodiversity, often exceeding ecosystems' adaptive capacities.
Groundwater Drought	Long-term decline in underground water supplies caused by reduced recharge rates or over-extraction.
Flash Drought	Rapid onset and intensification of drought conditions often affect multiple regions in a short timeframe.

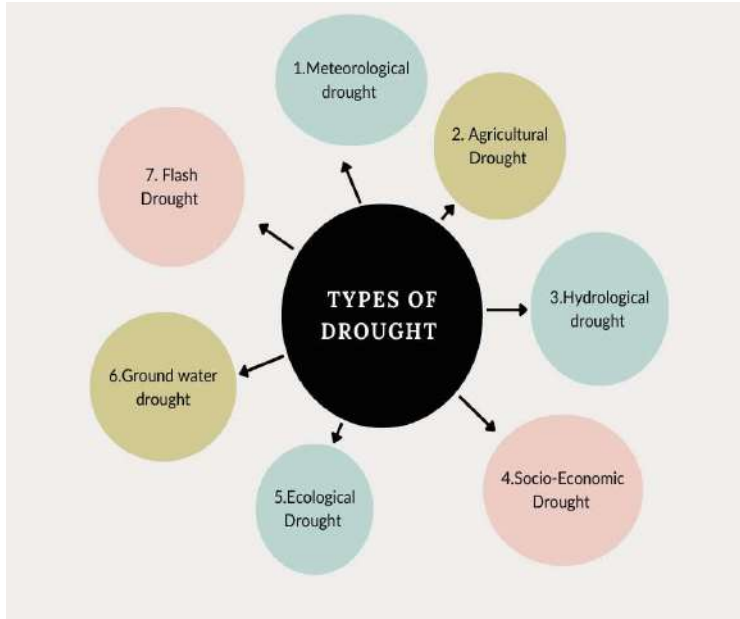

Fig. 3: Pictorial representation of the existing drought classes.

of rainfall in a meteorological drought, soil moisture in an agricultural drought, water in lakes and streams in a hydrological drought, and water availability for water management (Mishra & Singh 2010, Wilhite 2000, Mukherjee et al. 2018, Ezzahra et al. 2023). A summary visual representation of these drought classes is presented in Fig. 3 (Wilhite & Glantz 1985).

Drought problems and threats were thoroughly investigated and studied. The goal was to study its effects on agriculture, water, and ecosystems. This knowledge was used to create adaptable and sustainable strategies and solutions (Iqbal et al. 2020, Seleiman et al. 2021, Wahab et al. 2022). Further classification details are given in Table 1 (Wilhite & Glantz 1985, Mishra & Singh 2010, Mukherjee et al. 2018).

DROUGHT INDICATORS

Drought indicators and indices play a critical role in understanding, monitoring, and managing drought conditions. They provide quantitative and qualitative measures to assess the severity, duration, and spatial extent of droughts. Indicators generally rely on meteorological, hydrological, agricultural, and socio-economic data to reflect the specific components of drought, whereas indices combine multiple datasets into a single value for decision-making purposes (Salehnia et al. 2020, Kulkarni et al. 2020).

These tools are essential for researchers, policymakers, and water resource managers to identify drought conditions, predict potential impacts, and implement mitigation strategies. Effective drought indices enable a comprehensive

analysis by capturing changes in climatic factors such as precipitation, temperature, evapotranspiration, and water availability.

The evaluation of drought indices is especially relevant in the context of climate change, as shifts in temperature and precipitation patterns intensify the frequency and severity of droughts. Modern advancements, such as the integration of remote sensing data and artificial intelligence techniques, have further enhanced the accuracy of drought indicators, enabling real-time monitoring and improved predictions (Dakhil et al. 2024).

Bachmair et al. (2016) referred to drought indicators and indices collectively as tools to characterize and quantify droughts, highlighting their widespread application in global drought research.

DROUGHT INDICES

An index is typically calculated using statistical methods such as normalization or by combining multiple processes to produce a single value. Using available data, an index measures drought duration and severity, offering a comprehensive overview for decision-making. It provides a single numerical value that combines hydrological and meteorological variables, including temperature, evapotranspiration, precipitation, runoff, and other indicators of water availability. Decision support tools used to assess drought intensity, duration, and severity rely on specific indices and indicators (Botterill & Hayes 2012). In the drought-monitoring community, hydrological cycles, drought indicators, and indices are often used interchangeably (Hayes et al. 2021). Common drought indicators, such as precipitation, temperature, groundwater levels, streamflow, and soil moisture, are widely used across different regions. In contrast, drought indices are determined and analysed based on hydro-climatic factors that influence drought. These indices represent singular quantities (Hayes et al. 2021).

The key characteristics of a drought are its intensity, duration, and geographic extent. Among these, the primary factor for drought analysis is the severity of the drought (Tigkas et al. 2015). Given the multiple factors contributing to drought, several composite drought indices—RAI, PDSI, SPI, RDI, SPEI, CMI, SPDI, SRI, and others—have been developed to monitor drought conditions by integrating individual remote sensing drought indices. Here is a list of the drought indices:

Rainfall Anomaly Index (RAI): The proposal was first put forward by Van Rooy (1965). The operation of this method relies on the comparison of computed precipitation with random values that span from −3 to +3. There are ten categories assigned to the variations in precipitation. Furthermore, it is executed on both a yearly and monthly basis (Smakhtin & Hughes 2007). The aforementioned research has assessed the effectiveness of RAI in specific, uniform regions classified by moist to moderate climates. It has identified the precipitation patterns and variations, as well as the intensity and frequency of rainfall (Costa & Rodrigues 2017, Siddharam et al. 2020, Goswami 2018). While a study by Loukas et al. (2003) looked at RAI's performance in temperate climate stations in Greece, where summers often have negative precipitation values, no data on RAI's performance in dry and extremely dry climates are available. In these climates, extended warm dry periods have severely skewed precipitation and produced many zero precipitation data points (Raziei et al. 2015).

Palmer Drought Severity Index (PDSI): Palmer (1965) bases its definition of drought on soil moisture, precipitation, and temperature. Four main factors-precipitation, temperature, soil moisture, and evapotranspiration—need to be calculated through sophisticated formulations in order to calculate the PDSI, which is employed on a monthly basis. PDSI is a soil moisture algorithm (Ntale & Gan 2003, Van der Schrier et al. 2011) that is calculated for comparatively uniform areas. This drought warning system is among the most advanced and precise available. Though it is a useful tool for identifying long-term drought every month, the PDSI is not appropriate for characterizing short-term drought every week (Hong & Wilhite 2004). The categorization of precipitation patterns based on index values provides a comprehensive scale to assess the severity of deviations from average rainfall (Table 2).

Standardised Precipitation Index (SPI)*:* McKee et al. (1993) developed the SPI index in 1993. This index is calculated by dividing by the standard deviation after subtracting the mean precipitation from the actual precipitation. Calculations are based on precipitation data for 3, 6, 12, 24, and 48 months. The use of SPI varies according to the chance of precipitation at different time scales. Meteorological droughts can be assessed through the use of the SPI (Diani 2019, Bhunia et al. 2020, Li et al. 2020). Furthermore, it has the ability to forecast droughts

Table 2: PDSI Classification and its Range.

PDSI Range	Classification
4 or above	Extremely wet
0.5 to 0.99	Very wet
-0.49 to 0.49	Normal
-0.5 to -0.99	Moderate drought
-4 or less	Extreme drought

Table 3: SPI classification and range.

SPI Range	Classification
2 or above	Extremely wet
1.5 to 1.99	Very wet
-0.99 to 0.99	Normal
-1.49 to -1	Moderate drought
-1.99 to -1.5	Severe drought
-2 or below	Extreme drought

before they occur and assists in determining drought intensity (Funk & Shukla 2020). SPI classification and range are listed in Table 3. This index is less complex in terms of processing requirements compared to the Palmer index (Yihdego et al. 2019, Liu et al. 2021).

Presently, individuals responsible for organising and making decisions on drought are aware that the SPI serves multiple purposes and understand its specific significance. In addition, they acknowledge that the input data values in SPI have the potential to be altered, and they consider this to be a limitation of the index (Ji & Peters 2003). These are the values into which precipitation variations are classified by SPI, where each range signifies the extent of departure from average precipitation, offering a structured assessment of drought severity within specific regions based on SPI values.

Reconnaissance Drought Index (RDI): The MEDROPLAN coordinating meeting introduced an innovative drought detection and assessment index (Tsakiris 2004) and further elaborated upon it throughout subsequent works (Tsakiris et al. 2007). The PET methodologies were implemented in the RDI index value computation (Halwatura et al. 2015). It provides a distinct framework for classifying and comprehending various degrees of drought intensity. This criterion evaluates the severity of drought by utilising numerical values. RDI classification and range are listed in Table 4.

Soil Moisture Drought Index (SMDI): Hollinyer et al. (1993) established the SMDI in 1993. It is calculated for one year by summing the daily soil moisture readings. This measure only considers soil moisture as a single meteorological variable (Karimi et al. 2001). Recent studies have examined the SMDI's effectiveness in monitoring drought (Cao et al. 2022, Sen Roy et al. 2023). Utilising

Table 4: RDI classification and range.

RDI Range	Classification
-0.5 to -1.0	Mild drought
-1.0 to -1.5	Moderate drought
-1.5 to -2.0	Severe drought
-2 or below	Extreme drought

Table 5: SPEI classification and range.

SPEI Range	Classification
2 or above	Highly wet
1.5 to 2	Very wet
1 to 1.5	Moderately wet
-1 to 1	Normal
-2 to -1	Severely dry
-2 or below	Extremely dry

historical data as a benchmark, it is conventionally computed as the discrepancy between present-day soil moisture levels and the long-term mean. Typically, it is denoted by a standardised value between −4 (extremely dry) and +4 (extremely moist). SMDI values of 0 and negative values, respectively, denote different levels of dryness.

Standard Precipitation Evaporation Index (SPEI): The SPEI, similar to PDSI, considers the influence of reference evapotranspiration on drought severity. However, its ability to analyse several time scales allows for the identification of various types of drought and their effects on different systems (Vicente Serrano, S.M. et al. 2012a, Vicente Serrano, S.M. et al. 2012b, Vicente Serrano, S.M. et al. 2013a, Vicente Serrano, S.M. et al. 2013b). SPEI values vary from +2 to −2. SPEI classification and range are listed in Table 5

Therefore, the SPEI possesses the same level of sensitivity as the PDSI when it comes to measuring the demand for evapotranspiration, which is influenced by changes and patterns in climatic factors other than precipitation. Additionally, it is straightforward to compute and can be applied at various scales, similar to the SPI (Beguería et al. 2013).

Crop Moisture Index (CMI): The purpose of the CMI is to offer information that addresses broad-scale general inquiries rather than localized ones. The sensitivity study conducted on the Crop Moisture Index revealed that the index shows increased levels of moisture in response to rising temperatures under certain instances (Juhasz & Kornfield 1978). It is dependent on the available meteorological data, in particular, the data comprises the total precipitation and mean temperature for each week. It evaluates climate change's impact on water resources and equitable growth (Miryaghoubzadeh et al. 2019, Ampitiyawatta & Wimalasiri 2023). The evapotranspiration anomaly index and Wetness Index are added to generate the final CMI (Heim et al. 2002, Hogg et al. 2013). During the growth season, the value is near zero, stays near zero if crop moisture supply and weather conditions are normal, and returns to nearly zero at the conclusion (Palmer 1968).

Standardised Runoff Index (SRI): The SRI, according to McKee et al. (1993), requires calculating the unit standard

normal deviation of the percentile of hydrologic runoff data over a period of time. Various timeframes (such as 1-month or 9-month) and varying levels of spatial grouping for the index can be computed based on the resolution of the source data and the intended use. SPIs, such as those computed by NOAA, are determined at a climatic division level and by state agencies at a county level (Shukla & Wood 2008). It is used to identify drought patterns in a region (Nalbantis & Tsakiris 2008, Wang et al. 2013), and different methods have been used to calculate the SRI (Sheffield et al. 2012).

Munger's Index: The Munger Index, created by Robert Munger in the 1920s, is a measure of drought. This method is straightforward and commonly employed; by considering precipitation, the severity of drought is evaluated. Short-term droughts can be assessed most effectively using this index (Yihdego et al. 2019). It aids in assessing the sufficiency of rainfall for crop development, where readings below a specific threshold indicate drought conditions and higher values imply ample moisture availability. Nevertheless, it is crucial to acknowledge that the Munger Index predominantly emphasises precipitation, and variables that may impact drought, such as soil moisture and temperature, are not considered. A timeframe without a 24-hour rainfall of 1.27 mm is noted. Munger made the interesting observation that the drying-out effect of drought on plant life in forests is independent of the duration that it lasts. The approach uses a right triangle whose height and base are proportional to drought length. The mathematical expression for the severity of drought is given by the formula $0.5\ L^2$, where L is the length of the drought in days (Hogg et al. 2013).

Kincer's Index: Kincer produced a set of essential maps and charts depicting the seasonal patterns of rainfall and climatological data on the average yearly frequency of rainy days. Kincer's definition of drought is a period of 30 or more consecutive days with precipitation of less than 6.35 mm (0.25 in.) within 24 hours (Hogg et al. 2013). Furthermore, it highlights the allocation of rainfall across several seasons, taking into account the average yearly precipitation (Qiao et al. 2014). The Kincer's Index evaluates the vulnerability of a watershed to drought and identifies regions most prone to drought (Mishra & Nagarajan 2010, Wu et al. 2016). Studies indicate that the Kincer's Index shows higher accuracy compared to the PDSI and SPI (Gouveia et al. 2019).

Marcovitch's Index: To calculate a drought index, Marcovitch created an equation that combines precipitation and temperature: Drought index = $0.5 \times (N/R)^2$, where N is the cumulative count of consecutive days, lasting for at least two days, with temperatures over 32.2°C (90°F), and R denotes the overall amount of rainfall during the summer for that particular month (Hogg et al. 2013).

Blumenstock's Index: In his climatic study, Blumenstock utilised probability theory to calculate the frequencies of droughts. The drought time in days was used to calculate the index. For a drought to end, 2.54 mm (0.10 in.) of precipitation was needed within 48 hours. Using the Munger and Blumenstock indices, short-term droughts were measured (Hogg et al. 2013). When compared to other indices, it becomes apparent that various indices provide unique expressions of drought (Yihdego et al. 2019). The findings highlighted the need for using evapotranspiration and precipitation data to assess drought severity, improve drought understanding, and help develop effective drought monitoring and control technologies (Silva et al. 2021, Johnson et al. 2021, Santos et al. 2022).

Antecedent Precipitation Index (API): Antecedent precipitation refers to the precipitation that occurs before a certain storm event and impacts the relationship between runoff and that storm event. The yield from the same rainfall event on a watershed that has already been wetted by earlier rainfall is less than the yield from a rainfall event on a dry watershed (Heggen 2001). The system includes precipitation, which is a reverse drought index used for flood prediction (Hogg et al. 2013), and the API decay constant k affects API value accuracy (Heggen 2001). Soil moisture is crucial in the connection between land and atmosphere. Estimating soil moisture levels can be done by several methods, such as in situ measurements, hydrological modeling, and satellite remote sensing. The utilisation of indicators to perform an index of soil moisture conditions is another effective strategy. This study examines one index known as the API. To match the physical process, two parameters were added to the standard API. The recession coefficient is initially allowed to change with air temperature to account for evapotranspiration. The maximum API value considers the soil's maximum water-holding capacity. The adjusted API was subsequently calibrated and validated through a comparison with soil moisture measured in situ (Zhao et al. 2019). Some recent studies utilise this index to assess drought intensity, track rainfall patterns, and forecast hydrological responses (Goswami 2018, Nguyen-Huy et al. 2022).

Moisture Adequacy Index (MAI): The MAI (McGuire & Palmer 1957) index, generated from prospective evapotranspiration, compares a region's moisture need to its actual moisture supply, which includes rainfall and soil moisture. It is computed by dividing the actual moisture supply by the moisture needed and expressing it as a percentage. Indicators such as precipitation and soil moisture are used, and 100% indicates that what is available has been enough to fulfil the need (Yihdego et al. 2019). Research has shown that the use of the MAI is an extremely

effective technique to determine drought severity, optimise crop cultivation, and make accurate field assessments for agriculture (Rawat & Joshi 2010, Sarkar & Biswas 2017, Das et al. 2019).

Keetch and Byram Drought Index (KBDI): Its purpose is to evaluate the state of drought with a special focus on fire control management. It measures soil moisture depletion in hundredths of an inch, with 0 indicating no shortage and 800 indicating severe drought. The calculation relies on a soil moisture storage capacity of 203 mm (8 in.). The DI is calculated using a daily water budgeting approach that balances drought factor, precipitation, and soil moisture. For monitoring and predicting wildfires, this index is extensively used (Hogg et al. 2013). In situ soil moisture measurements might enhance wildfire threat estimates, which frequently use the KBDI (Krueger et al. 2017). Studies examine this technology's usefulness in assessing droughts and wildfires (Keetch & Byram 1968, Gouveia et al. 2019).

Surface Water Supply Index (SWSI): The SWSI is a hydrologic drought statistic developed in 1981, particularly for Colorado, based on empirical data. It incorporates snowfall, reservoir storage, streamflow, and high-elevation precipitation to improve the PDSI. A useful indicator of surface water resources is the SWSI (Wilhite & Glantz 1985, Shafer & Dezman 1982). Colorado's Drought Assessment and Response Plan incorporates SWSI and PDSI-like measurements. The SWSI is largely computed for river basins and has been adopted by other western states (Hogg et al. 2013). It includes precipitation, snowpack, reservoir storage, and runoff (Yihdego et al. 2019). The study of an area, its real-world applications, and its effectiveness in evaluating drought seriousness and predicting streamflow have been documented (Wilhite and Glantz 1985, Wu et al. 2016).

Vegetation Condition Index (VCI): The data utilised for drought identification and tracking is derived from satellite Advanced Very High Resolution Radiometer (AVHRR) radiance, namely in the visible and near-infrared spectrum. This data is modified for land climate, ecology, and weather. Kogan's 1995 research showed this approach's potential. The VCI capitalises on the strong correlation between vegetation and climate, drawing inspiration from ideas established by German biologist W. Köppen nearly a century ago in his climate classification system based on vegetation. VCI enables drought identification and serves as a potential worldwide benchmark for assessing the timing, severity, duration, and impact of drought on vegetation. Nevertheless, due to its reliance on vegetation, the VCI is predominantly valuable during the summer period of plant growth. Its us Dixit Dixit efulness is restricted during the winter months when plant growth is mostly inactive (Hogg et al. 2013). Recent studies have examined drought severity and evaluated drought trends (Wu et al. 2016, Khatri & Sharma 2019). Before 2000, the Anomaly Vegetation Condition Index (AVCI) predominantly had negative values, indicating a lack of soil moisture. Analysis of exceedance probability on an annual time scale revealed a 20% likelihood of severe drought (VCI $\leq$ 35%) and a 35% likelihood of regular drought (35% $\leq$ VCI $\leq$ 50%) occurring in Nepal (Baniya et al. 2019).

Drought Monitor Index (DM): The authors of the DM index rely on studies of various crucial indices and supplementary indicators from multiple organisations to construct the ultimate map. The main factors consist of the PDI (Palmer Drought Index), CMI, percentiles of soil moisture models, percentiles of daily streamflow, percent of normal precipitation, topsoil moisture (percent of short and very short levels) provided by the USDA, and a satellite-based Vegetation Health Index. The U.S. Drought Monitor employs a categorical framework to categorise drought severity: D0 (Abnormally dry) denotes conditions before drought, D1 (Moderate drought) indicates initial effects on crops and water supply, D2 (Severe drought) signifies significant harm to agriculture and water scarcity, D3 (Extreme drought) suggests critical losses and widespread water scarcity, and D4 (Exceptional drought) represents catastrophic impacts on agriculture and economy, coupled with severe water shortages. These categorisations facilitate communication of drought seriousness and consequences, guiding responses and allocation of resources in affected areas (Hogg et al. 2013). From 2000 to 2016, this research used the Integrated Drought Monitoring Index (IDMI) to measure agricultural drought in Tamil Nadu, the southern Indian peninsula, during the northeast monsoon season. PCI (Precipitation Condition Index), SMCI (Soil Moisture Condition Index), TCI (Temperature Condition Index), and VCI constitute the four indices of the IDMI. The indices are calculated based on time-series satellite observations of climate risks, namely infrared precipitation data from CHIRPS, the European Space Agency Climate Change Initiative (ESA-CCI), and Moderate Resolution Image Spectroradiometer (MODIS) (Kuma et al. 2021). The significance and effectiveness of measuring drought severity and vulnerability were assessed (Zargar et al. 2022, Liu et al. 2022).

Joint Deficit Index (JDI): Kao and Govindaraju (2010) developed the JDI, a measure using copula functions that integrates joint distributions of several SPIs. It accounts for precipitation and streamflow distribution while considering seasonal variations. This indicator detects incipient and protracted droughts quickly and allows for monthly

evaluation of drought conditions. It predicts how much rainfall is needed in the coming months to restore normal conditions (Mirabbasi et al. 2013). The copulas used in JDI are multivariate distribution functions that provide a link between one-dimensional marginal distributions and joint probability distributions (Nelsen 2006). Geostatistics and spatial statistics explain multi-timescale covariance using a two-parameter function. Analysis of long-term precipitation data proves covariance model. Bootstrap tests demonstrate that the Gaussian copula model assesses drought severity better than the empirical copula. It measures droughts outside the empirical copula. Second, drought is well-quantified. Finally, it clarifies the estimate uncertainty (Van de Vyver & Van den Bergh 2018). This index's versatility, applicability, and capability to monitor droughts were confirmed (Wu et al. 2016, Liu et al. 2022).

Multivariate Standardised Drought Index (MSDI): The MSDI utilises a probabilistic approach for integrating the Standardised Soil Moisture Index (SSI) and the SPI to accurately represent drought conditions. MSDI assesses drought using meteorological and agricultural data. The suggested MSDI is used in California's climate divisions and in North Carolina to assess drought conditions. Studies employing the MSDI are compared to the SSI and SPI. The findings indicate that MSDI accurately recognises the beginning and end of drought situations by taking into account both SPI and SSI. SPI largely determines drought onset, whereas SSI more closely determines drought persistence. In short, the MSDI model demonstrates that it is possible to quickly merge multiple indices using stochastic approaches (Hao & AghaKouchak 2013). The joint cumulative probability is transformed using a standard normal distribution's inverse cumulative distribution function to obtain the MSDI. Standardisation is not achieved using this method. Since the joint cumulative probability is not evenly distributed on [0,1], negative index values are more likely. The Standardised Drought Analysis Toolkit (SDAT) (Farahmand & AghaKouchak 2015) computes non-parametric standardised univariate and non-parametric bivariate MSDI drought indices (Erhardt & Czado 2018). Methods of drought characterisation and risk assessment were evaluated (Zhang et al. 2022, Trigo et al. 2021, Albajes et al. 2022).

Reconnaissance Trivariate Drought Index (RTDI): The RTDI is a composite of soil moisture, evapotranspiration, and precipitation. Meteorological and agricultural droughts are effectively represented by the RTDI and MSDI, linking climatic status. For bivariate and trivariate analysis, the most appropriate copulas are the Student and Frank's t copulas, respectively. To analyse drought onset and withdrawal characteristics, the two drought indices are formulated and evaluated. Cross-wavelet analysis (CWA) can reveal how large-scale climatic anomalies affect drought indices. This research considers Indian summer monsoon rainfall (ISMR), Multivariate ENSO Index (MEI), Southern Oscillation Index (SOI), and Indian Ocean Dipole (Dixit & Jayakumar 2021).

Effective Drought Index (EDI): Byun and Wilhite (1999) created the EDI in 1999 to address index limitations. By assessing daily water collection over time, the EDI provides a full evaluation. Its specialised design for daily drought severity estimation and more accurate water resource evaluations makes the EDI advantageous (Kim 2009). EDI values range from −2.5 to 2.5. Index readings between −1.0 and 1.0 imply near-normal conditions, while −2.0 or below indicates severe drought (Salehnia et al. 2017, Raja Azman et al. 2022).

Relative Drought Indices: The relative Standardised Precipitation Index (rSPI) and Relative Palmer Drought Severity Index (rPDSI) improve drought evaluation in shifting climates. They provide an innovative method to compare drought conditions across time and geography. In order to apply drought indices to future climates, they must first be calibrated using aggregated observational data from all stations during a reference period. This process is known as achieving the former. This approach can be utilised to evaluate the spatial displacement of drought caused by climate change. However, the latter method uses station measurements to accurately track temporal drought changes in relation to the present climate. The second approach may provide indicators that are not comparable across climate zones (Mukherjee et al. 2018, Dubrovsky et al. 2009).

Standardised Streamflow Index (SSFI): Power spectrum and detrended fluctuation analysis are the employed techniques. The presence of the yearly oscillation indicates all the streamflows. This oscillation also acts as a transition point between two regions. For frequencies below the yearly cycle (or timescales longer than 1 year), the dynamics are roughly random. However, for frequencies above the yearly frequency (or timescales shorter than 1 year), the dynamics are consistently correlated (Telesca et al. 2012). The SSFI is useful in drought evaluation and monitoring, particularly in regions with enormous river systems and complex hydrology. The tool's ability to quantify drought's effect on streamflow makes it valuable for water resource management and environmental planning (Wu 2016).

Data Fusion-Based Drought Index (DFDI): The data fusion drought index is the process of combining several sources or types of data to form a comprehensive index that evaluates drought conditions. The index thoroughly encompasses all categories of drought by utilising a range of indices and

Table 6: Comparative Matrix of Drought Indices.

Index	Input Data	Type	Scale	Applicability under CC
RAI	Precipitation	Precipitation based	Local, regional, monthly	Limited (only precipitation)
PDSI	Precipitation, Temp., Soil moisture	Evapotranspiration	Regional, monthly	Moderate (needs calibration)
SPI	Precipitation	Precipitation based	Local, global, monthly	Limited (excludes temperature)
RDI	Precipitation, PET	Evapotranspiration	Regional, monthly	High (accounts for PET)
SMDI	Soil moisture	Agricultural	Local, regional, daily	High (soil moisture focus)
SPEI	Precipitation, Temp., PET	Evapotranspiration	Local, global, monthly	High (integrates temperature)
CMI	Precipitation, Temp., Soil moisture	Agricultural	Regional, weekly	Moderate (short-term focus)
SRI	Streamflow	Hydrological	Regional, monthly	Moderate (streamflow-dependent)
Munger's Index	Precipitation	Precipitation based	Local, daily	Limited (short-term focus)
Kincer's Index	Precipitation, Temp.	Composite	Regional, monthly	Moderate (empirical)
Marcovitch's Index	Precipitation, Temp.	Composite	Local, daily	Limited (experimental)
Blumenstock's Index	Precipitation	Precipitation based	Local, daily	Limited (historical focus)
Antecedent Precipitation Index	Precipitation, Soil moisture	Hydrological	Local, regional, daily	Moderate (soil moisture integration)
MAI	Precipitation, Soil moisture	Agricultural	Regional, monthly	Moderate (agro-climatic focus)
Keetch-Byram Index	Temp., Soil moisture	Ecological	Local, regional, daily	High (fire risk under warming)
SWSI	Streamflow, Snow pack, Reservoir storage	Hydrological	Regional, monthly	Moderate (water resource focus)
VCI	Satellite imagery (NDVI)	Vegetation-based	Regional global, monthly	High (real-time monitoring)
Drought Monitor Index	Multi-source (PDSI, CMI, VCI, etc.)	Composite	Regional global, monthly	High (integrates multiple proxies)
JDI	Precipitation, Streamflow (copula-based)	Composite	Regional, monthly	High (multi-variable)
MSDI	Precipitation, Soil moisture	Composite	Regional, monthly	High (non-stationary climates)
RTDI	Precipitation, Soil moisture, ET	Composite	Regional, monthly	High (multi-variable)
EDI	Daily Precipitation, ET	Evapotranspiration	Local, regional, daily	Moderate (short-term focus)
Relative Drought Indices	Precipitation, Temp. (calibrated)	Composite	Regional, monthly	High (non-stationary climates)
SSFI	Streamflow	Hydrological	Regional, monthly	Moderate (streamflow-dependent)
DFDI	Multi-source (remote sensing, climate)	Remote-sensing	Regional global, monthly	High (AI/RS integration)

proxies linked to each specific form of drought. The primary objective of data fusion, defined as the integration and consolidation of data from various sources and sensors, is to generate a solution that is either more precise or enables specialists to access a greater quantity of information than would be possible by using individual data sources alone.

To aid comparative evaluation, a matrix-style summary is presented in Table 6 below, highlighting key characteristics of major drought indices, including their input data, applicable drought type, strengths, limitations, scale of application, and suitability under changing climate scenarios.

This paper offers a method to objectively correlate water availability and plant characteristics to assess terrestrial ecosystem water stress. A set of drought indices (DIs) was considered. The combination approach determines each time step's water stress circumstances using multivariate statistical methods, such as independent component analysis, and eco-meteorological parameters, including land use, land cover, and climate. Three case study regions with varying land use, climate regimes, and surface and atmospheric variables are provided to assess the new approach's potential to generalize DIs (Azmi et al. 2010).

Table 7: Summary of commonly used drought indices.

Index Name	Description	Strengths	Limitations
SPI	Measures drought based on precipitation deviation from normal.	Simple, widely used, adaptable to different timescales.	Does not account for temperature and evapotranspiration.
SPEI	Combines precipitation and evapotranspiration to assess drought severity.	Considers temperature impacts, robust under climate change.	Requires detailed climate data.
PDSI	Incorporates soil moisture balance to measure long-term drought.	Effective for agricultural drought monitoring.	Complex calculations, not suited for short-term drought.
CMI	Focuses on short-term agricultural droughts using soil moisture.	Relevant for agricultural applications.	Not useful for long-term drought analysis.
RDI	Considers precipitation and potential evapotranspiration.	Versatile and adaptable.	Requires accurate climatic data.
SRI	Quantifies drought using runoff data.	Hydrologically relevant.	Requires extensive hydrological data.
VCI	Monitors drought impact on vegetation using remote sensing data.	Useful for agricultural and ecological droughts.	Relies on satellite data availability.
RAI	Compares rainfall anomalies to historical averages.	Simple and easy to calculate.	Limited by the exclusion of temperature and soil factors.
EDI	Considers precipitation deficits over time.	Dynamic and responsive to recent changes.	Requires detailed precipitation records.
EDI	Quantifies drought by analyzing evapotranspiration anomalies.	Relevant under warming climate scenarios.	Requires advanced climate models.

The following Table 7 provides an overview of 25 widely used drought indices, categorised based on their focus areas, such as meteorology, hydrology, and agriculture. Each index is evaluated for its strengths, limitations, and suitability under evolving climate conditions.

This section provides a comprehensive understanding of the tools used to measure and monitor droughts. Indices and indicators continue to evolve, with recent advancements integrating modern technologies and climate models, ensuring their relevance for addressing the challenges of climate change. Recent studies demonstrate the integration of diverse drought indicators or proxies from multiple data sources to offer a thorough evaluation of drought severity. The assessment of drought monitoring, early warning, and drought assessment can be conducted effectively (Azmi et al. 2016, Mishra et al. 2018, Wu et al. 2017).

Fig. 4: Pictorial representation of the different classifications of drought indices.

Other drought indices are employed to thoroughly evaluate and describe drought conditions. These indices provide additional viewpoints on different aspects of drought, going beyond conventional measurements to offer a more detailed understanding. Drought indices, which quantify departures from typical local conditions based on historical distributions, are used to track drought (Dai 2011). Drought indices can be classified as shown in Fig. 4.

RESULTS AND DISCUSSION

This section provides a detailed explanation of the results from the literature analysis, key findings and their implications. It also discusses the challenges encountered during the research and outlines the future scope for advancing drought monitoring and management in the context of climate change.

Results

The results of this study demonstrate the complex and multifaceted relationship between climate change and drought, evaluated through a detailed analysis of drought indices, climate models and emerging trends. The findings are summarized as follows:

Analysis of Drought Indices

- Among the 25 evaluated indices, the Standardized Precipitation-Evapotranspiration Index (SPEI) and Normalized Difference Vegetation Index (NDVI) were identified as the most robust tools for monitoring drought under changing climatic conditions.
- SPEI's inclusion of evapotranspiration made it particularly sensitive to temperature increases caused by global warming, whereas NDVI proved useful for real-time monitoring of agricultural and ecological drought impacts.

Climate Model Comparisons

- Comparative analysis of CMIP5 and CMIP6 models highlighted advancements in the latter, with improved sensitivity to regional climate variations and better predictions of extreme drought events.
- The CMIP6 models projected a significant increase in the frequency and intensity of droughts in South Asia, especially under high-emission scenarios (SSP5-8.5).
- Results also showed non-linear trends in precipitation variability, indicating a potential for more severe meteorological and hydrological droughts in semi-arid regions.

Regional Projections for South Asia

The comparative analysis of CMIP5 and CMIP6 models reveals significant regional differences in projected drought patterns. For South Asia, CMIP6 models indicate a marked increase in the frequency and severity of droughts, especially under high-emission scenarios (SSP5-8.5) (Dixit et al. 2022). Projections suggest that agricultural and meteorological droughts will become more frequent, with soil moisture deficits and temperature-driven evapotranspiration playing a key role (Mukherjee et al. 2018). For example, in certain regions of South Asia, the frequency of severe agricultural droughts is projected to increase by 20–30% by the end of the century. These changes are closely linked to projected increases in temperature and shifts in precipitation patterns, which are more robustly captured by CMIP6 models compared to CMIP5.

The SPEI and NDVI indices emerge as particularly robust for monitoring these projected changes, due to their sensitivity to temperature and vegetation stress, respectively (Vicente-Serrano et al. 2012, Dixit et al. 2022). The integration of remote sensing and AI-driven models further enhances the ability to detect and predict drought severity in real time, supporting more adaptive management strategies for the region (Dakhil et al. 2024).

Integration of Advanced Technologies

a. Remote sensing and artificial intelligence showed significant potential in improving drought monitoring capabilities. For example, hybrid indices combining NDVI and surface water data offered higher spatial accuracy for drought predictions.

b. AI-based models reduced prediction errors and allowed for more adaptive monitoring frameworks tailored to regional conditions.

Role of Artificial Intelligence in Drought Monitoring

Several reviewed studies highlight the growing use of artificial intelligence (AI) techniques, such as machine learning, for processing remote sensing data and developing hybrid drought indices. AI-based models have been shown to reduce prediction errors and enable more adaptive monitoring frameworks. However, these approaches are still largely experimental or limited to well-resourced regions, and their broader application depends on data availability and computational infrastructure.

Best Performing Indices by Drought Type and Climate Scenario

To provide clear guidance for practitioners and researchers, we synthesized our findings to identify the most suitable drought indices for each drought type under current and projected climate scenarios (CMIP5 and CMIP6). This summary is based on the comparative analysis and ranking system described in Table 8.

Table 8: Recommended drought indices by drought type and climate scenario.

Drought Type	Current Climate	CMIP5 Scenario	CMIP6 Scenario
Meteorological	SPI, SPEI	SPEI	SPEI, MSDI
Agricultural	NDVI, VCI, SMDI	NDVI, SPEI	NDVI, SMDI, Hybrid/AI
Hydrological	PDSI, SWSI	PDSI	MSDI, Hybrid
Ecological	NDVI, VCI	NDVI, VCI	NDVI, Hybrid/AI

- **Meteorological Drought:** Under historical and current climates, both SPI and SPEI are widely used, but SPEI is preferable under CMIP5 and especially CMIP6 projections due to its sensitivity to temperature-driven evapotranspiration, which is increasingly relevant in warming scenarios. MSDI also shows promise for capturing multi-variable influences in future conditions.
- **Agricultural Drought:** NDVI and VCI are effective for real-time monitoring of vegetation stress, making them ideal for current and projected climates. SMDI is valuable where soil moisture data is available, and hybrid or AI-driven indices are recommended under CMIP6 for their ability to integrate diverse datasets and improve prediction accuracy.
- **Hydrological Drought:** PDSI and SWSI remain standard for long-term water resource assessment under current and CMIP5 conditions. However, under CMIP6, MSDI and hybrid indices are better suited to capture the complexity of hydrological droughts influenced by multiple climate variables.
- **Ecological Drought:** NDVI and VCI are robust for monitoring ecological impacts, and their effectiveness is enhanced under future scenarios when combined with AI and hybrid approaches for greater spatial and temporal resolution.

These recommendations provide a practical framework for selecting drought indices tailored to specific drought types and evolving climate scenarios. The following Discussion section expands on these findings and their implications for research and policy.

Ranking and Comparative Performance of Drought Indices

To objectively identify the most robust drought indices for monitoring under changing climatic conditions, we applied the ranking system described in the Methodology section. Each index was evaluated according to six criteria: climate sensitivity, data requirement, spatial resolution, temporal resolution, performance under projected climate scenarios (CMIP5/CMIP6), and operational usability. Scores for each criterion were assigned on a scale of 1 (low) to 5 (high), based on a literature review and expert consensus. Table 9 presents the scores for each index according to the six ranking criteria. The total scores indicate the overall robustness of each index for drought monitoring under changing climatic conditions. The results show that SPEI and NDVI are the most robust indices, with the highest total scores. This ranking supports their use for drought monitoring under both current and projected climate scenarios.

The results indicate that SPEI and NDVI achieved the highest total scores, reflecting their robustness for drought monitoring under current and projected climate scenarios. This ranking supports the selection of SPEI and NDVI as the most suitable indices for operational and research applications, particularly in the context of climate change.

Discussion

The findings underscore the growing complexity of drought management in the era of climate change. Several key insights and implications arise from the results:

Relevance of Drought Indices in Climate Change Context

The effectiveness of drought indices is highly dependent

Table 9: summarizes the scores and total ranking for each index.

Index	Climate Sensitivity	Data Requirement	Spatial Resolution	Temporal Resolution	CMIP6 Suitability	Operational Usability	Total Score
SPI	2	5	4	5	2	5	23
SPEI	5	4	4	5	5	5	28
NDVI	4	4	5	5	4	4	26
PDSI	3	3	3	3	3	4	19
MSDI	5	2	3	3	5	3	21

on their ability to incorporate climate variables such as temperature, evapotranspiration, and soil moisture. Indices like SPEI are better suited for capturing the multi-dimensional impacts of climate change compared to traditional indices like SPI, which focus solely on precipitation. However, challenges remain in adapting these indices to account for local socio-economic and ecological conditions.

Comparative Usability of Drought Indices Under Climate Change Scenarios

The increasing complexity of droughts under climate change necessitates a nuanced understanding of the strengths and limitations of various drought indices. The usability of each index depends not only on its methodological foundation but also on its sensitivity to evolving climate variables, data requirements, and suitability for different drought types and regional contexts. Here, we synthesize the comparative performance of widely used indices under historical and projected climate scenarios (CMIP5 and CMIP6), providing practical guidance for their application. Table 10 represents the comparative analysis of major drought indices.

Usability Under Projected Climate Scenarios

- CMIP5 vs. CMIP6: The transition from CMIP5 to CMIP6 models has improved the simulation of regional climate extremes, particularly in South Asia and other drought-prone areas. Indices that incorporate temperature and evapotranspiration (e.g., SPEI, MSDI) demonstrate greater sensitivity and reliability under high-emission scenarios (SSP5-8.5), where temperature-driven droughts are projected to increase in both frequency and severity.
- SPI remains a useful baseline tool but underestimates drought risk in warming climates due to its exclusion of temperature effects. Its simplicity and broad adoption make it suitable for initial screening but less so for future-focused risk assessments.
- SPEI is better suited for climate change contexts, as it

Table 10: Comparative analysis of major drought indices.

Index	Input Data	Drought Type	Strengths	Weaknesses	Performance under CMIP5/CMIP6	Best Use Cases
SPI	Precipitation	Meteorological	Simple, widely used, multi-scale	Ignores temperature, less sensitive to warming	Adequate for historical climates, less robust under projected warming (CMIP6)	Short-term meteorological drought, baseline monitoring
SPEI	Precipitation, Temperature/ ET	Meteorological, Agricultural	Captures temperature effects, multi-scale, climate-adaptive	Sensitive to ET estimation, data intensive	Highly robust under CMIP5/CMIP6, especially for warming scenarios	Climate change impact studies, semi-arid regions
PDSI	Precipitation, Temperature, Soil Moisture	Agricultural, Hydrological	Integrates soil moisture, long-term trends	Complex, less suitable for short-term drought, region-specific calibration needed	Useful for long-term drought under both scenarios, but calibration is critical	Water resource management, policy planning
NDVI/ VCI	Satellite Vegetation	Agricultural, Ecological	Real-time, spatially explicit, sensitive to vegetation stress	Seasonal limitations, indirect for meteorological drought	Effective for rapid drought detection under both scenarios, especially with AI integration	Agricultural monitoring, early warning systems
SMDI	Soil Moisture	Agricultural	Direct soil moisture assessment	Requires dense soil data, spatial heterogeneity	Valuable where soil data is available, especially for flash droughts under CMIP6	Crop yield forecasting, precision agriculture
JDI/MSDI	Multiple (Precipitation, Soil Moisture, Streamflow)	Meteorological, Agricultural, Hydrological	Integrates multiple variables, captures compound events	Computationally intensive, requires multi-source data	Strong performance under non-stationary, extreme events in CMIP6	Complex drought risk assessment, research applications
Hybrid/ AI-Based Indices	Remote Sensing, Climate, Socio-economic	All	Adative,customizable, high spatial/temporal resolution	Data and expertise intensive, validation needed	Promising for future scenarios, especially in data-rich regions	Integrated drought risk management, policy frameworks

integrates both precipitation and temperature, capturing the intensifying evapotranspiration and moisture deficits projected in CMIP6. SPEI is recommended for regions experiencing significant warming or variable precipitation patterns.

- NDVI/VCI and other remote sensing indices excel at real-time monitoring of agricultural and ecological droughts, especially when combined with AI for rapid data analysis. Their effectiveness is particularly notable in regions with high spatial variability and for early warning applications.
- Composite indices (e.g., JDI, MSDI, hybrid AI-based) are increasingly valuable for capturing compound and cascading drought events, which are expected to become more frequent under future climate scenarios. These indices are recommended for advanced research and operational frameworks in regions with sufficient data infrastructure.

Practical Guidance for Index Selection

- Meteorological droughts in semi-arid and warming regions: Prefer SPEI or MSDI, as these indices account for temperature-driven evapotranspiration and are responsive to projected climate variability.
- Agricultural and ecological droughts: NDVI, VCI, and SMDI are highly effective, especially when integrated with AI for real-time monitoring and prediction.
- Hydrological droughts and water resource management: PDSI and SWSI remain relevant for long-term planning, but require careful regional calibration and may benefit from integration with remote sensing data.
- Early warning and rapid response: Remote sensing indices (NDVI/VCI) and hybrid indices combining climate, vegetation, and soil data provide the most timely and spatially explicit information.
- Data-limited regions: SPI and RAI can serve as initial tools, but efforts should be made to build data infrastructure for adopting more advanced, climate-adaptive indices.

The usability of drought indices is context-dependent and should be aligned with both the type of drought and the prevailing or projected climatic scenario. SPEI and NDVI emerge as the most robust and versatile indices for monitoring drought under changing climate conditions, particularly when enhanced with AI and remote sensing technologies. However, no single index is universally optimal, a combination of indices, tailored to regional data availability and climate risks, is recommended for comprehensive drought assessment and management.

Regional Implications of Climate Model Projections

The CMIP6 projections for South Asia highlight the urgent need for region-specific drought mitigation strategies (Dixit et al. 2022). The increased frequency of flash droughts and long-term hydrological droughts underscores the importance of enhancing water resource management and adopting adaptive agricultural practices (Mukherjee et al. 2018). The findings also emphasize the value of advanced monitoring tools, such as remote sensing and AI, in providing actionable insights for policymakers and practitioners (Dakhil et al. 2024). However, it is important to note that model uncertainties and regional biases remain a challenge, and efforts should be made to validate projections with local observational data where possible (WMO & GWP 2016).

Advancements in Drought Monitoring Technologies

Integrating remote sensing and AI into drought management frameworks could revolutionize the field. Technologies such as satellite-based vegetation monitoring and AI-driven predictive models enable near real-time assessment of drought conditions, allowing for faster response times and more targeted mitigation efforts. The integration of artificial intelligence (AI) into drought monitoring represents a significant advancement, as evidenced by the reviewed literature. AI enables the rapid analysis of large datasets, improves the accuracy of drought predictions, and facilitates the development of adaptive, region-specific indices. However, challenges such as data scarcity, model interpretability, and the need for specialized expertise limit the widespread adoption of AI-driven approaches. Future research should focus on overcoming these barriers to fully realize the potential of AI in enhancing drought resilience.

Challenges in Drought Assessment and Mitigation

Despite advancements, significant challenges remain. Data availability and quality continue to hinder the application of sophisticated drought indices and climate models in many developing regions. Additionally, the increasing influence of anthropogenic activities on hydrological cycles complicates the prediction of drought impacts.

Policy Implications and Recommendations

The results emphasize the need for policymakers to prioritize investments in climate-resilient infrastructure and technologies. For instance, improving the accessibility of high-resolution climate data and implementing AI-driven drought monitoring systems could significantly enhance preparedness and response capabilities. The integration of climate, remote sensing, and socio-economic data into a hybrid drought monitoring system is illustrated in Fig. 5. This diagram illustrates the integration of climate data, remote sensing, and socio-economic information within a hybrid drought monitoring system. Climate models

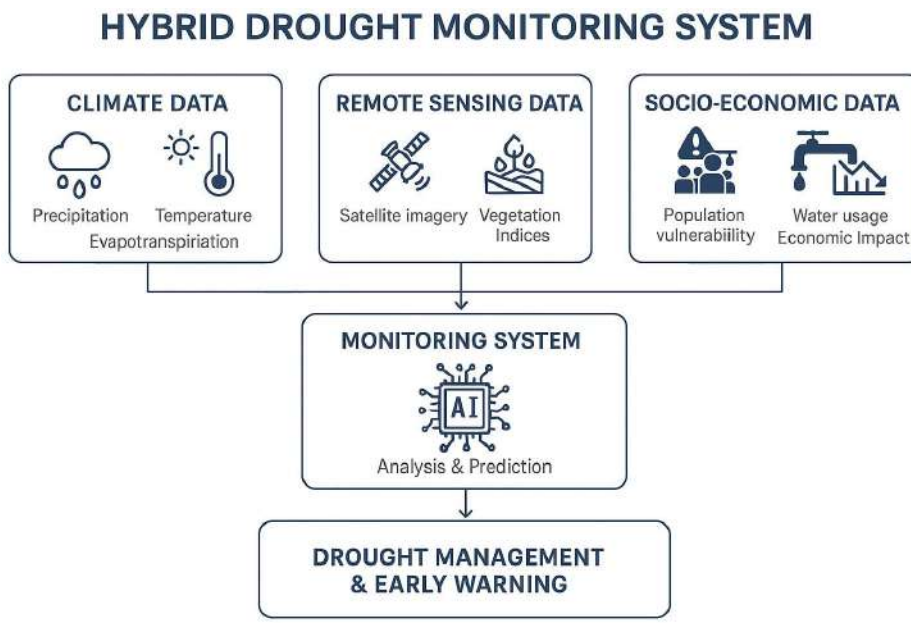

Fig. 5: Conceptual diagram of a hybrid drought monitoring system.

provide projections of temperature and precipitation, while remote sensing delivers real-time data on vegetation health, soil moisture, and surface water. Socio-economic data are incorporated to assess community vulnerability and adaptive capacity. Artificial intelligence (AI) processes these diverse data streams, enabling rapid, accurate drought prediction and early warning. The system supports adaptive management and policy decisions by providing comprehensive, actionable insights for drought resilience and water resource management.

Case Studies and Real-World Applications

In India, the Normalized Difference Vegetation Index (NDVI) has been widely used for early warning of agricultural drought. For instance, in the Marathwada region of Maharashtra, researchers have utilised NDVI data from MODIS and Landsat satellites to monitor vegetation health and detect drought onset up to several weeks before traditional ground-based indicators (Kulkarni et al. 2020). This approach has enabled local authorities to issue timely advisories and implement water-saving measures, reducing crop losses and supporting adaptive agricultural planning. Similar applications have been reported in other drought-prone regions of India, demonstrating the value of remote sensing indices for early drought detection and response.

Future Scope

While this study provides valuable insights, several areas for further research remain:

- Developing hybrid indices that integrate socio-economic factors for a more holistic assessment of drought impacts.
- Improving climate models to address non-stationarity and better capture the effects of anthropogenic activities.
- Exploring the role of groundwater and ecological droughts in shaping long-term resilience to climate change.

CHALLENGES AND GAPS IN EXISTING RESEARCH

Despite significant advancements in drought research, several challenges and gaps remain in both the methodology and technological applications, which hinder the accurate assessment and effective management of droughts under changing climate conditions. Challenges in Understanding and Managing Drought are shown in Fig. 5. The key challenges identified in this study are as follows:

Methodological Challenges

One of the primary challenges in drought assessment is the lack of uniformity in the application of drought indices. Many indices, such as the Standardized Precipitation Index (SPI) and the Standardized Precipitation–Evapotranspiration Index (SPEI), are predominantly based on precipitation data and do not adequately account for other crucial climate variables such as temperature, evapotranspiration, and soil moisture. While some indices have been adapted to include temperature and evapotranspiration, their application under non-stationary climate conditions remains a significant limitation.

Another challenge is the complexity of integrating multiple drought indices for comprehensive drought monitoring. While hybrid indices that combine remote sensing and ground-based data show promise, their development and validation are still in nascent stages, which makes their widespread application challenging.

Technological Challenges

Although advancements in satellite-based remote sensing and artificial intelligence have the potential to improve drought monitoring, challenges remain in terms of data accessibility, quality, and the integration of these technologies into operational frameworks. High-resolution satellite data can be expensive and difficult to access, especially for developing countries, which limits its use for real-time drought monitoring.

Furthermore, AI models require vast amounts of reliable and diverse data to accurately predict drought conditions. Inadequate datasets, coupled with the challenges of transferring these models to real-world applications, pose significant barriers to the deployment of AI-driven drought management solutions.

Limitations in the Application of Current Indices under Non-Stationary Climate Models

The increasing unpredictability of climate patterns due to human activities and natural variations complicates the application of traditional drought indices. Current models often fail to account for the non-stationarity of climate systems, which means that indices developed under stationary climate conditions may not perform well in future scenarios characterised by abrupt climatic shifts. The adaptation of these indices to more dynamic, non-stationary models is a crucial gap in the current research.

Additionally, the ability of existing indices to predict extreme droughts with sufficient accuracy under new climate conditions is still a matter of concern. Many indices are not sensitive enough to capture the nuances of severe drought events, especially in regions with complex climates or rapid climatic changes.

Integrating Socio-Economic Factors into Drought Assessments

Another significant gap in existing drought research is the difficulty of incorporating socio-economic factors into drought assessments. While climate data and drought indices can provide insights into environmental conditions, they often overlook the socio-economic vulnerabilities of the affected populations. Factors such as local economic dependencies, social resilience, and governance structures are critical in assessing the full impact of droughts but are challenging to quantify and integrate into existing models.

The lack of socio-economic data, coupled with difficulties in assessing the combined effects of climate and socio-economic variables, makes it challenging to develop comprehensive drought assessments that inform policy and adaptive strategies effectively. Research efforts that focus on integrating socio-economic aspects with environmental data are still limited, but are essential for improving the resilience of communities to droughts.

How Future Tools Could Resolve These Challenges

Recent advances in artificial intelligence (AI) and remote sensing offer promising solutions to many of these challenges. AI-driven models can integrate diverse datasets, including socio-economic and ecological variables, to better capture the complexity of drought impacts and vulnerabilities. By leveraging machine learning and big data analytics, these tools enable more accurate, real-time monitoring and prediction

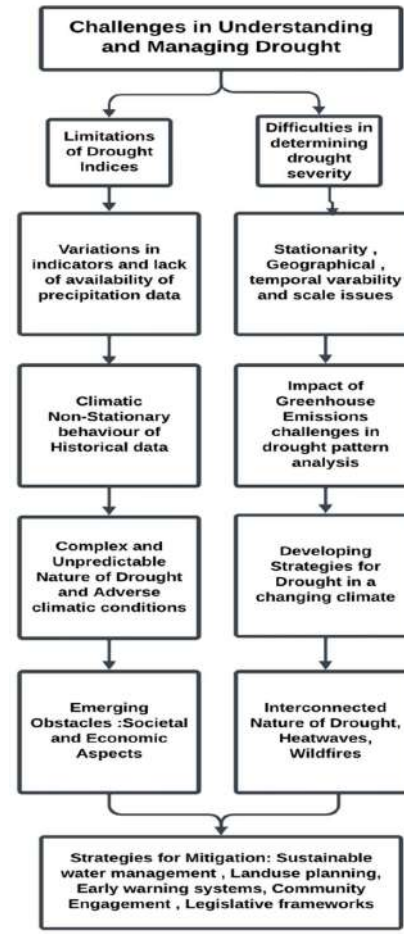

Fig. 6: Challenges in Understanding and Managing Drought.

of drought conditions, supporting adaptive management and targeted interventions. Furthermore, the integration of AI with remote sensing facilitates the development of hybrid indices that address data gaps and improve the spatial and temporal resolution of drought assessments, ultimately enhancing the resilience of vulnerable communities and ecosystems. A flow chart of challenges in understanding and managing drought is shown in Fig. 6.

RECOMMENDATIONS AND FUTURE FRAMEWORKS

To address the challenges identified in this study and improve drought management and mitigation strategies, several key recommendations are proposed. First, region-specific solutions should be prioritised, considering the unique climatic, geographical, and socio-economic conditions of each area. Implementing localized drought monitoring systems that integrate advanced technologies, such as satellite-based remote sensing, AI, and machine learning, can provide real-time data, enabling more accurate and timely responses. Additionally, drought indices should be enhanced by incorporating cutting-edge tools, such as AI-driven predictive models and hybrid indices that integrate multiple data sources, to improve their sensitivity and applicability under non-stationary climate conditions.

To support an actionable application, we recommend that SPEI and NDVI be prioritised for drought monitoring in semi-arid and warming regions due to their sensitivity to temperature-driven evapotranspiration and real-time vegetation stress. Policymakers should integrate SPEI and NDVI into early warning systems by leveraging remote sensing and AI-driven predictive models, such as Support Vector Machines (SVM), Random Forest (RF), and Deep Learning, which have proven effective in recent studies.

Policy directions should focus on improving water management systems, with an emphasis on sustainable practices, water conservation, and efficient resource allocation. Policymakers must also address the socio-economic impacts of drought by supporting vulnerable communities through adaptive agricultural strategies, investment in climate-resilient infrastructure, and the development of social safety nets. By fostering greater integration between scientific research, technological innovation, and policy development, the resilience of communities to droughts can be significantly improved, ensuring a more sustainable future in the face of climate change.

CONCLUSIONS

The findings of this study underscore the intricate relationship between climate change and drought, highlighting the significant role of advanced drought indices, climate models, and emerging technologies in enhancing drought monitoring and management. The comprehensive evaluation of 25 drought indices and climate model comparisons offers valuable insights into the growing complexity of droughts under climate change scenarios. The integration of remote sensing and AI-based tools provides a promising avenue for more accurate, real-time assessments of drought conditions, which is crucial for adaptive strategies in the face of increasingly erratic weather patterns. Moreover, the study emphasizes the need for innovative, region-specific frameworks that consider local socio-economic, ecological, and climatic conditions to enhance drought resilience. These frameworks can serve as a foundation for more effective drought management and mitigation efforts, providing tangible solutions for addressing the challenges posed by climate-driven droughts.

To translate these insights into practice, we recommend that researchers prioritise the development of hybrid indices integrating socio-economic factors and advanced technologies, policymakers invest in robust data infrastructure and region-specific monitoring systems, and technologists advance the integration of AI and remote sensing for real-time, adaptive drought monitoring. Ultimately, this research calls for continued collaboration between researchers, policymakers, and stakeholders to develop actionable strategies and ensure sustainable water resource management, particularly in regions most vulnerable to droughts.

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The Influence of Urban Green Spaces on Airborne Particulate Pollution: A Case Study of Phutthamonthon Park, Bangkok Suburb, Thailand

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ABSTRACT

Urban green spaces play a vital role in promoting public health and enhancing the quality of urban environments, particularly through their potential to mitigate air pollution. This study investigated the association between green space and airborne particulate matter (PM) of various sizes, including PM1, PM2.5, PM10, and total dust, within Phutthamonthon Park. Located in Nakhon Pathom Province, adjacent to Bangkok, Phutthamonthon Park is one of the largest suburban green spaces in the region. The park serves as a significant recreational and cultural hub that supports diverse activities, including walking, cycling, and community gatherings, while simultaneously contributing to urban air quality management through its extensive vegetation cover and open spaces. The study also assessed the influence of meteorological factors such as temperature, relative humidity, and time of day on PM concentrations. A cross-sectional survey was conducted in January 2025 using DustTrak DRX Aerosol Monitors across four zones of the park during morning, midday, and evening periods, yielding a total of 144 samples. The results demonstrated a significant reduction in total dust levels within green spaces, with peripheral zones exhibiting 50-100% higher concentrations compared to interior areas. However, no statistically significant differences were observed for PM1, PM2.5, and PM10, which remained elevated during morning hours, likely due to unfavorable atmospheric conditions. These findings underscore the complex role of urban greenery in air quality management, revealing both benefits and limitations. The study offers practical insights for urban planners and policymakers, emphasizing the importance of strategic green space design in reducing air pollution and promoting healthier urban living conditions.

INTRODUCTION

Urban expansion in recent decades has led to increasing environmental challenges. In particular, air pollution from fine particulate matter (PM) significantly affects the quality of life in urban areas (Liu et al. 2018). In this context, "urban green spaces" have been recognized as spatial components that can alleviate air pollution problems and promote a long-term, healthy urban environment (Tzoulas et al. 2007). Urban green spaces include parks, gardens, street tree areas, and natural vegetation. They play an important role in absorbing and trapping atmospheric PM through biological mechanisms, such as filtering dust via leaves (Speak et al. 2012) and stems, and absorbing pollutants on leaf surfaces (Barwise & Kumar 2020, Nowak et al. 2006). However, the efficiency of dust reduction depends on many factors, including the structure and type of vegetation, the physical characteristics of the area, greenness, vegetation density, and meteorological conditions (Vos et al. 2013).

Previous research suggested that green spaces can significantly reduce levels of large and total PM in some contexts, but their impact on fine PM, such as PM1 and PM2.5, remains uncertain, particularly in the context of tropical or rapidly expanding cities (Barwise & Kumar 2020). Fine PM is considered a primary risk

factor for long-term health problems, such as respiratory disorders and cardiovascular disease (Pope III & Dockery 2006). This underscores the importance of local pollution control (World Health Organization 2024). Additionally, factors such as temperature, relative humidity, and time of day significantly affect atmospheric PM levels and influence their buoyancy, dispersion, and deposition in open spaces (Zhang 2019). Therefore, studies that consider only green spaces while excluding these environmental factors may not sufficiently explain the phenomenon. Despite growing recognition of the role of green infrastructure in urban air quality, there remains a lack of spatially detailed, real-time assessments within large public parks in Southeast Asia. This study contributes novel evidence by applying a zone-based monitoring design to explore intra-park variation in PM concentrations. By capturing fine-scale spatial dynamics in a tropical peri-urban setting, the findings aim to inform a more context-sensitive understanding of how urban green spaces are associated with airborne PM levels. Given this knowledge gap, this study aims to analyze the relationship between urban green spaces and airborne PM levels, using Phutthamonthon Park, a large green space in the suburb of Bangkok, as a case study. The analysis encompasses four types of PM: PM1, PM2.5, PM10, and total dust, while also considering the influence of temperature, relative humidity, and time of day on PM changes. It is expected that the results of this study will enhance the understanding of the role of urban green spaces in environmental science and support urban design approaches that contribute to the promotion of public health at a fundamental level.

MATERIALS AND METHODS

This study is a cross-sectional survey conducted in January 2025 in Phutthamonthon Park, a suburb of Bangkok, Thailand. Phutthamonthon Park was selected as the study area due to its status as a large public park located in the suburb of Bangkok. This expansive green area has the potential to curb air pollution and is an important component of the urban ecosystem (Jim & Chen 2008). It has an area of 988 acres, with a width and length of approximately 2 kilometres on each side. The park boasts both natural diversity and the capacity to support various activities. Phutthamonthon Park reflects the impact of large urban green areas on air PM. In this study, the area was divided into four zones based on distance from the outer area to facilitate a clearer comparison of PM levels in each zone. The three inner zones (Zones A-C) were delineated by equal radial increments of roughly 333 metres, while Zone D comprised the remaining fringe abutting the park boundary, as shown in Fig. 1. This zoning approach enhances the interpretation of PM distribution by incorporating variation in openness, land use, and surrounding influences, with each zone exhibiting distinct physical, ecological, and anthropogenic features as follows:

Zone A (Central Zone): Located at the park's core, this area features the main Buddha statue and large open lawns interspersed with low- to medium-height trees with sparse canopies. It is the most open zone and is heavily used for physical activities and public gatherings.

Zone B (Intermediate Zone): This zone includes a large pond in the east with minimal tree cover, while the west side contains taller, more densely spaced trees forming shaded jogging paths. Built structures are located in the northeast. The area offers a mixed canopy environment with moderate recreational use.

Zone C (Inner Border Zone): Relatively enclosed, this zone has dense tree coverage in the west and north with taller trees and overlapping canopies, contrasted by open grass space in the east. The southeast includes some built structures. Human activity is limited, and small ponds are scattered throughout.

Zone D (Outer Zone): Located along the park's outer boundary, this zone borders a major road to the east and low-density communities to the north, south, and west. It comprises partially open areas and continuous strips of trees and shrubs, forming a transitional buffer between urban infrastructure and park greenery.

This research utilized a field-based data collection approach, employing the DustTrak DRX Aerosol Monitors. The DustTrak device is recognized for its precision in measuring fine PM, particularly PM2.5, which is considered a critical indicator of air pollution and a key factor affecting human health in urban settings (Sousan et al. 2016). This tool employs light-scattering technology to measure the concentration of dust particles in the air. The scattering of light from dust particles is detected and processed into data reflecting dust values of various sizes, including PM1, PM2.5, PM10, and total dust, allowing for real-time data collection and measurement of dust values appropriate for air quality research. The study also collected temperature and relative humidity data. Data was collected over three days, divided into three time periods: morning (05:00–07:00), midday (11:00–13:00), and evening (17:00–19:00). In each round, measurements were conducted across the four designated zones, with four samples taken per zone corresponding to the north, south, east, and west directions. This approach yielded 16 samples per round, resulting in 48 samples per day and a total of 144 samples throughout the study period. Temperature and relative humidity were

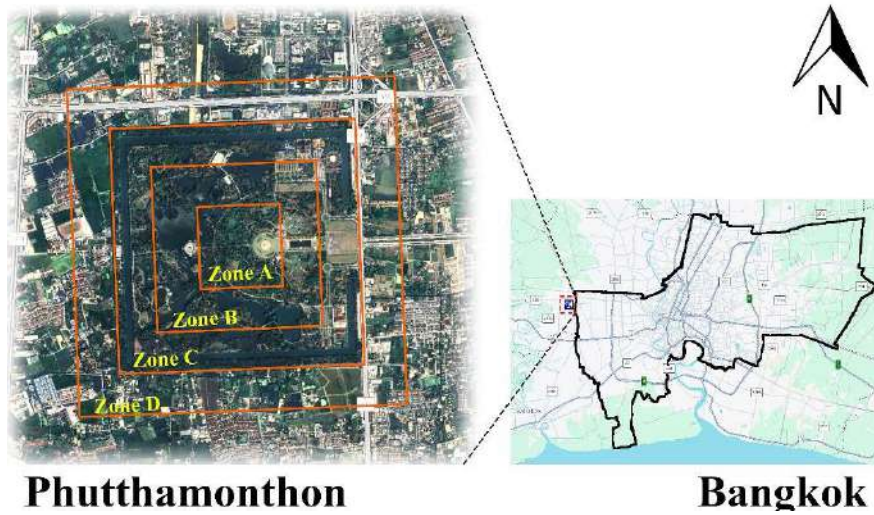

Fig. 1: Study area: Phutthamonthon Park.

Table 1: Average of PM concentrations, temperature, and relative humidity across the time period.

Parameter	Morning Mean ± SD [95% CI]	Midday Mean ± SD [95% CI]	Evening Mean ± SD [95% CI]	Significance
PM_1 [mg.m^{-3}]	0.09 ± 0.02 (0.08–0.09)	0.05 ± 0.01 (0.05–0.06)	0.04 ± 0.01 (0.04–0.05)	< 0.001
$PM_{2.5}$ [mg.m^{-3}]	0.09 ± 0.02 (0.08–0.09)	0.05 ± 0.01 (0.05–0.06)	0.04 ± 0.01 (0.04–0.05)	< 0.001
PM_{10} [mg.m^{-3}]	0.09 ± 0.02 (0.09–0.10)	0.06 ± 0.02 (0.06–0.07)	0.05 ± 0.01 (0.05–0.06)	< 0.001
Temperature [°C]	21.81 ± 1.85 (21.28–22.35)	33.36 ± 1.95 (32.79–33.93)	30.49 ± 1.69 (30.00–30.98)	< 0.001
Relative humidity [%]	91.79 ± 3.51 (90.77–92.81)	47.63 ± 11.57 (44.27–50.99)	53.60 ± 8.80 (51.05–56.16)	< 0.001

also recorded alongside PM measurements to support data interpretation.

Descriptive data were analyzed using the mean and standard deviation. The relationships between various factors, including location within the garden area, temperature, relative humidity, and the effects of different times of day, on PM concentrations were analyzed using regression analysis. In addition, the study compared the average PM levels across different locations using ANOVA at a statistical significance level of 0.05.

RESULTS AND DISCUSSION

PM, Temperature, and Relative Humidity Across Time Periods

PM1, PM2.5, and PM10 concentrations were highest during the morning, with significant reductions by midday and evening ($p < 0.001$). Morning hours also recorded the highest relative humidity (91.79%), while midday showed the highest temperature (33.36°C). All temporal variations in PM levels, temperature, and relative humidity were statistically significant (Table 1).

Spatial Distribution of Total Dust

Total dust concentrations were significantly higher in Zone D (outside the park) compared to Zones A–C (within the park) ($p = 0.003$). Regression analysis supported this finding, showing a positive association between dust levels and exterior areas (Fig. 2).

One-way ANOVA revealed significant differences in total dust concentrations among zones ($F = 2.971$, $p = 0.034$) (Table 2). Duncan's Multiple Range Test was subsequently applied to identify specific pairwise differences. Detailed results are presented in Table 3.

Post-hoc pairwise comparisons using Duncan's Multiple Range Test confirmed that Zone D (exterior) had significantly

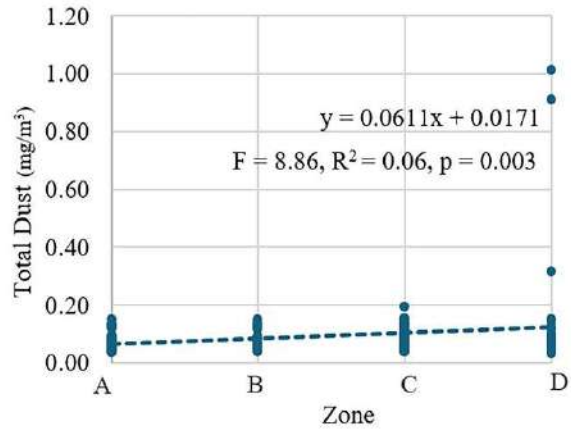

Fig. 2: Distribution of dust particles of different sizes.

Table 2: Total dust concentration by zone.

Parameter	Zone				F	Significance
	A	B	C	D		
	Mean ± SD [95% C I]	Mean ± SD [95% C I]	Mean ± SD [95% C I]	Mean ± SD [95% C I]		
Total Dust [mg.m^{-3}]	0.08 ± 0.03 (0.07–0.08)	0.07 ± 0.03 (0.07–0.08)	0.08 ± 0.04 (0.07–0.10)	0.14 ± 0.21 (0.07–0.21)	2.971	0.034

higher total dust concentrations than the interior zones (A, B, and C), which showed no significant differences among themselves (Table 3).

Effects of Temperature and Relative Humidity

Temperature was inversely related to PM1, PM2.5, and PM10 concentrations up to approximately 30 °C. Beyond this point, levels of PM increased slightly, while total dust continued to decline (Fig. 3). Relative humidity exhibited a non-linear association with PM concentrations: levels decreased until approximately 55% relative humidity, then began to rise. Total dust showed a positive linear relationship with increasing relative humidity (Fig. 4).

Influence of Time of Day

Time of day significantly affected all measured variables. PM levels peaked in the morning and declined throughout the day (Fig. 5). Correspondingly, temperature rose from morning to midday and fell by evening, while relative humidity followed an inverse pattern (Fig. 6 and Fig. 7). A strong inverse relationship was found between temperature and relative humidity.

Table 3: Comparison of mean total dust concentrations (mg/m³) across zones using Duncan's Multiple Range Test ($\alpha = 0.05$).

Zone	Mean ± SD [mg.m^{-3}]	Duncan's Grouping
A	0.08 ± 0.03	B
B	0.07 ± 0.03	B
C	0.08 ± 0.04	B
D	0.14 ± 0.21	A

Note: Means followed by different letters are significantly different at $p < 0.05$ according to Duncan's Multiple Range Test.

Discussion

The findings from this study highlight significant temporal and spatial variations in PM concentrations, temperature, and relative humidity in and around Phutthamonthon Park. These variations have direct implications for human health, especially concerning respiratory and cardiovascular outcomes (World Health Organization 2021).

The significantly higher total dust levels observed in Zone D (outside the park) compared to Zones A–C (inside the park) align with previous evidence that urban vegetation can reduce airborne particulates through interception and deposition mechanisms (Nowak et al. 2006). It is also noteworthy that Zone D exhibited a markedly higher standard deviation compared to other zones, indicating substantial variability in total dust concentrations. Upon inspection of the raw data, two exceptionally high values were identified, likely reflecting transient environmental events or measurement anomalies. These values were retained in the analysis, as they fall within plausible environmental limits and represent

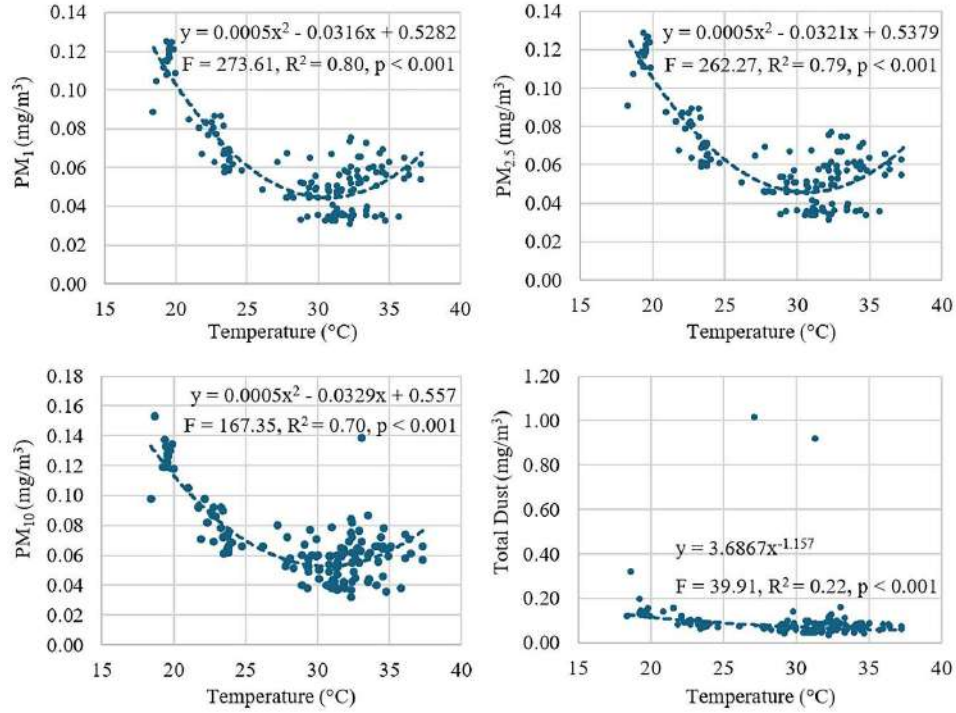

Fig. 3: Influence of temperature on the amount of dust particles of different sizes.

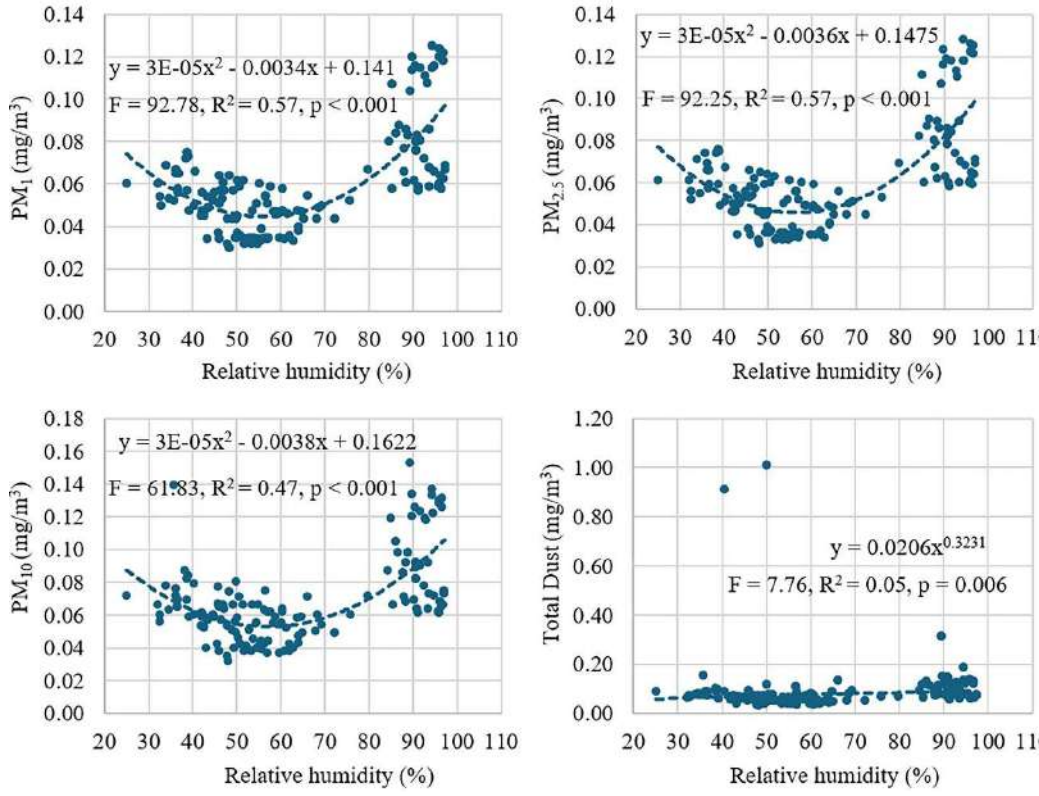

Fig. 4: Influence of relative humidity on the amount of dust particles of different sizes.

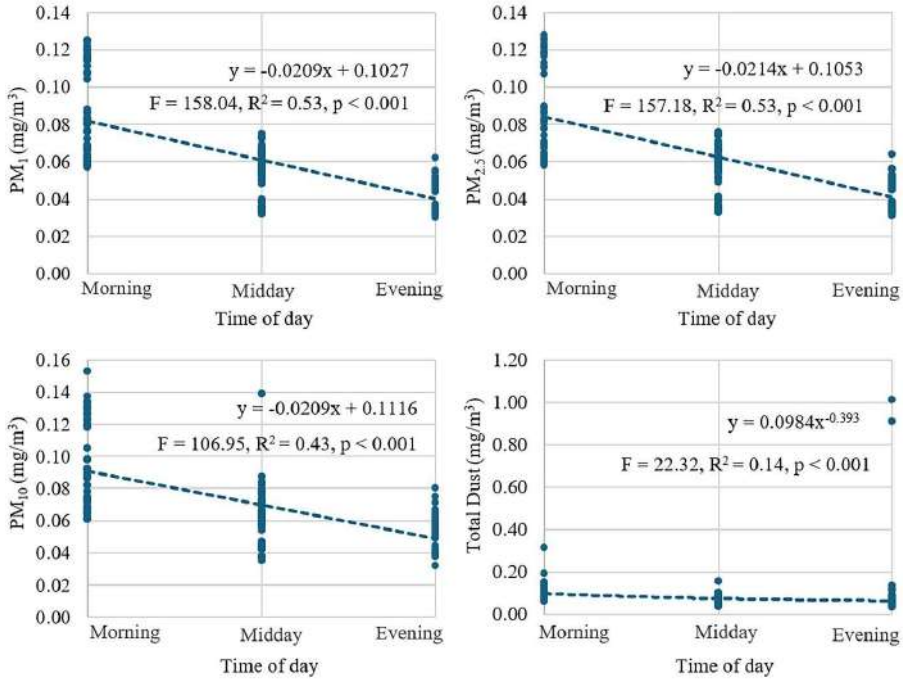

Fig. 5: Influence of time of day on the amount of dust particles of different sizes.

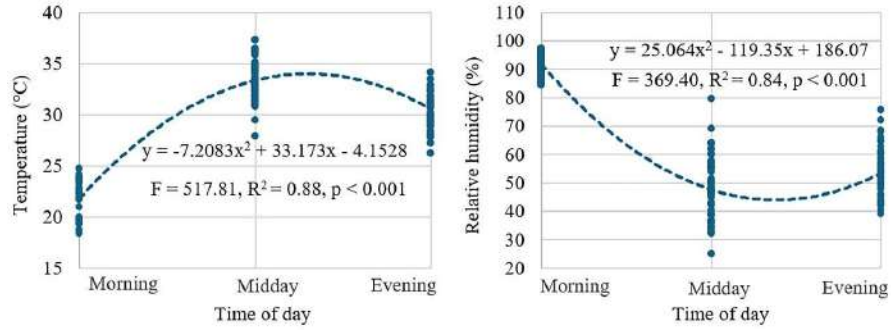

Fig. 6: Influence of time of day on temperature and relative humidity.

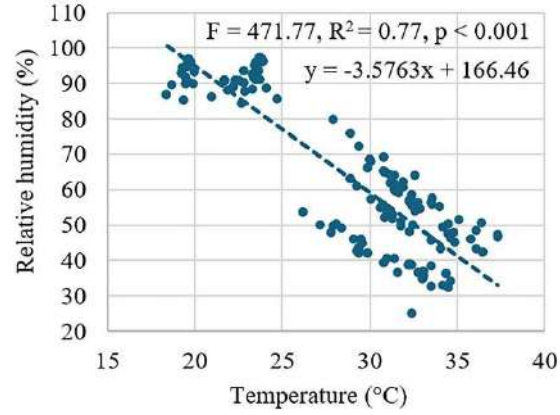

Fig. 7: Influence of temperature on relative humidity.

real-world variability. However, PM1, PM2.5, and PM10 concentrations did not differ significantly between zones. This pattern is likely explained by the buoyancy and prolonged atmospheric suspension of fine particles, which reduces their likelihood of deposition onto surfaces (Janhäll 2015, Nowak et al. 2013). Due to their small aerodynamic diameter and low settling velocity, these particles are less effectively intercepted by plant surfaces. Additionally, although the DustTrak DRX is widely used in air quality monitoring, its detection sensitivity varies by particle size and ambient humidity (Sousan et al. 2016). These factors collectively affect fine PM capture and may have contributed to the observed spatial uniformity.

Interestingly, high total dust concentrations were observed during early morning hours (05:00–07:00), when traffic volumes in the vicinity were likely still relatively low. While traffic emissions may partly account for elevated PM levels near roadside zones, these temporal patterns suggest that other environmental or atmospheric factors may also be at play. Due to the absence of time-resolved traffic data, the influence of morning vehicular activity cannot be confirmed and warrants further study.

Temporal analyses showed that PM levels peaked during morning hours and decreased throughout the day. This pattern corresponds to diurnal changes in temperature and relative humidity, with lower morning temperatures and higher relative humidity favoring PM accumulation through hygroscopic growth, where water vapor condenses onto particle surfaces, increasing their mass and reducing their dispersion in the atmosphere. These observations are consistent with Piyavadee et al. (2024), who found strong correlations between ambient PM concentrations and climatic conditions in Chiang Mai, Thailand. Specifically, early morning temperature inversions and high relative humidity were associated with higher pollutant levels. Further support comes from Bhosale et al. (2023), who reported that PM10 concentrations varied by location and time of day in Kolhapur City, India. Their findings underscore the role of urban structure and local meteorology in shaping PM distribution, similar to the elevated PM levels found in the outer areas of Phutthamonthon Park.

The non-linear relationships observed in this study between PM levels and meteorological variables (temperature and relative humidity) suggest complex atmospheric interactions. Moderate temperature and relative humidity were associated with reduced PM concentrations, while extremes led to increases-likely due to enhanced suspension or secondary particle formation (Zhang 2019, Zhang et al. 2024). This phenomenon may also be linked to the formation of volatile organic compounds and their subsequent transformation into PM under certain climatic conditions (Han et al. 2014, Jacob & Winner 2009, Seinfeld & Pandis 2016).

The results of this study confirm that green spaces in cities can reduce total dust but demonstrate limited efficacy in mitigating health-hazardous PM1, PM2.5, and PM10. These findings align with previous research by Yli-Pelkonen et al. (2017), which found that urban vegetation primarily reduces larger PM rather than fine particles. The elevated morning concentrations of PM1, PM2.5, and PM10, particularly in areas outside parks, represent a significant public health concern, as fine PM is associated with serious health risks (Brook et al. 2010), including respiratory and cardiovascular diseases. As emphasized by the World Health Organization (2024), long-term exposure to PM2.5, even at low concentrations, may increase mortality risk. While this study demonstrates that green spaces contribute significantly to reducing overall dust levels (particularly total dust), their limited impact on PM1, PM2.5, and PM10, which pose the most direct health threats, highlights the need for complementary mitigation strategies. Effective reduction of fine dust requires an integrated approach combining source control measures (Setälä et al. 2013) with urban meteorological management. These findings provide valuable evidence to inform sustainable urban planning and park design, while supporting the development of comprehensive environmental and health policies at the city level.

This study measured multiple air quality parameters (PM1, PM2.5, PM10, total dust, temperature, and relative humidity) across different zones and times, providing a detailed analysis of pollution distribution. The comparison between areas inside and outside Phutthamonthon Park offers practical insights into how green spaces influence dust levels in an actual urban environment. However, several limitations must be noted.

First, results from Phutthamonthon Park may not fully represent other urban green spaces with different vegetation types, sizes, or surrounding pollution sources. Second, the study was conducted over only three consecutive days in January, a dry, cool-season period in Thailand. Notably, this season is typically associated with heightened PM levels in Bangkok due to atmospheric inversion and stagnant wind conditions. As such, the findings may reflect peak pollution conditions rather than average year-round patterns. Seasonal factors such as rainfall, wind speed, and temperature inversions can significantly affect PM levels. Likewise, differences in human activity between weekdays and weekends (e.g., traffic volume, park usage) could influence pollution dynamics. Finally, the analysis does not detail

park characteristics (e.g., tree density, species, and canopy structure) that might influence dust capture efficiency. Future studies should extend the temporal duration and spatial scope of research to enhance data accuracy. This expansion should include longer monitoring periods across different seasons and day types, multiple study sites to improve generalizability, and assessment of climate change impacts on PM dynamics. Additionally, researchers should examine how green space design characteristics, particularly vegetation selection in public parks, influence the reduction of health-relevant PM (PM1, PM2.5, and PM10).

CONCLUSIONS

This study aimed to analyze the influence of urban green space on airborne PM levels, using Phutthamonthon Park as a case study to examine the patterns of change in different types of PM, including PM1, PM2.5, PM10, and total dust, as well as the roles of temperature, relative humidity, and time within the context of urban green space. The findings demonstrate that green spaces can significantly reduce total dust concentrations, particularly when comparing interior zones of the park to surrounding urban areas. This highlights the value of vegetation density, distance from pollution sources, and spatial configuration in mitigating airborne particulate matter.

Conversely, PM1, PM2.5, and PM10 showed no statistically significant reduction within green zones. These smaller particles, due to their aerodynamic properties and long atmospheric residence times, remain largely unaffected by vegetation alone. Furthermore, PM1, PM2.5, and PM10 concentrations were highest during morning hours and declined throughout the day, a pattern influenced by meteorological conditions, particularly temperature and relative humidity. The study also confirmed the non-linear relationship between these environmental factors and PM levels: moderate temperature and relative humidity tended to reduce PM, while extreme values led to increased concentrations. Although the study offers valuable insights, it is based on data collected during only three days in January, a period known for heightened particulate matter levels in Bangkok due to winter meteorological conditions. As such, the findings may reflect peak pollution scenarios rather than year-round averages. Future studies should expand sampling to cover multiple seasons to improve representativeness and capture broader temporal variability.

These findings underscore the complexity of PM dynamics in urban green spaces and point to the need for integrated strategies that combine urban greenery with broader environmental management measures.

POLICY RECOMMENDATIONS

Based on the study's findings, the following policy recommendations are proposed to enhance the role of urban green spaces in air quality improvement and dust pollution control:

1. Promote the development of green infrastructure in high-traffic urban zones, prioritizing areas with elevated pollution levels. While green spaces may have limited impact on fine PM, they contribute to overall dust reduction and support healthier urban environments.
2. Introduce dust control technologies in public spaces, such as misting systems to maintain optimal relative humidity levels and air purification units in high-use zones. These can complement the natural filtration offered by vegetation.
3. Design green spaces to maximize air circulation and pollutant capture by incorporating diverse, pollution-tolerant plant species with high particulate retention capacity. Tree type, canopy structure, and vegetation density should be aligned with air quality goals.
4. Encourage continued research into the interactions between vegetation, climate, and particulate matter. Future studies should explore seasonal variations, vegetation characteristics, and the effects of climate change on PM dynamics across various urban settings.

By integrating these strategies, cities can enhance the effectiveness of green spaces in improving air quality and protecting public health.

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Sustainable Reinforcement of Rubber Compound Using Recycled PET (Polyethylene Terephthalate): A Review

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ABSTRACT

Increasing environmental concerns over plastic waste and the depletion of non-renewable resources have intensified the search for sustainable alternatives in rubber reinforcement. This review comprehensively investigates the use of recycled polyethylene terephthalate (r-PET) as a green reinforcing agent in rubber compounds, replacing traditional fillers such as carbon black and silica. Experimental studies reveal that incorporating r-PET can improve tensile strength by up to 45%, enhance thermal stability by 20–30°C, and lead to energy savings of approximately 50 $MJ.kg^{-1}$ compared to virgin PET. Notably, this review is the first to integrate mechanical performance analysis with life cycle assessment (LCA), providing a dual perspective on technical feasibility and environmental impacts. Processing methods, such as mechanical blending, chemical grafting, and surface modification, have been discussed to enhance r-PET compatibility with rubber matrices. Challenges such as poor dispersion, interfacial adhesion, and thermal degradation are critically analyzed, along with mitigation strategies. The findings demonstrate that r-PET not only offers performance and cost advantages but also supports circular economy initiatives by repurposing post-consumer PET waste into high-performance rubber composite. Future research directions are proposed, focusing on hybrid reinforcement systems, compatibility enhancement, and carbon-footprint reduction.

INTRODUCTION

The growing concerns over environmental pollution and resource depletion have driven the search for sustainable alternatives in materials science. One such approach is the incorporation of recycled PET (rPET) into rubber composites. PET, which is widely used in water bottle manufacturing, contributes significantly to plastic waste. Recycling PET into rubber matrices can mitigate environmental impacts and improve the performance of rubber products. This paper reviews the current state of research on r-PET as a reinforcement for rubber compounds. It covers processing techniques, mechanical properties, environmental benefits, etc., with a focus on overcoming the limitations of industrial applications.

The rapid increase in global plastic consumption, particularly in single-use applications, has created an urgent need for recycling and repurposing plastic waste. PET (IUPAC: Poly (ethyl benzene-1,4-dicarboxylate)), being one of the most widely produced polymers, presents a significant waste management challenge (Shen et al. 2025). Traditional disposal methods, such as landfilling and incineration, contribute to pollution and greenhouse gas emissions (Elamri et al. 2015). Consequently, researchers have explored innovative ways to integrate PET waste into various materials, including rubber compounds, to promote sustainability and reduce environmental degradation.

The integration of r-PET into rubber compounds is not only a means of recycling waste but also an approach to enhancing the material properties. Studies

have shown that PET-based reinforcements can improve tensile strength, abrasion resistance, and overall durability of rubber products (Cazan et al. 2014). However, issues such as phase separation, interfacial adhesion, and compatibility between r-PET and rubber matrices need to be addressed through various modifications and processing techniques (Zhang 2024).

Moreover, research has highlighted the potential of converting PET waste into value-added products rather than discarding it. Given that PET is a thermoplastic material with excellent mechanical properties, it has the potential for reuse in various engineering applications. The recycling of PET (Rung et al. 2023) into rubber composites is not only environmentally beneficial but also cost-effective, as it reduces dependency on expensive virgin raw materials as well as Carbon Black. Additionally, the growing regulations on plastic waste management worldwide are encouraging industries to adopt more sustainable practices, making the use of r-PET in rubber a viable solution in the future.

Chemical Structure of PET

Polyethylene terephthalate (PET) is a polyester derived (Vitkauskienė & Makuška 2008) from the polymerization of terephthalic acid ($C_6H_4(CO_2H)_2$) and ethylene glycol ($HOCH_2CH_2OH$). The repeating unit of PET is shown in Fig. 1.

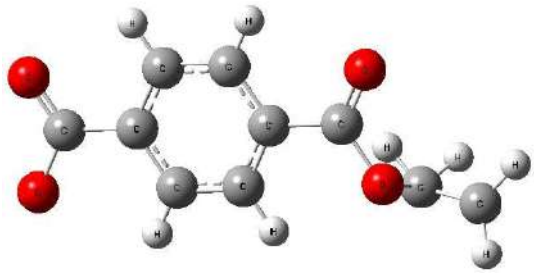

Fig. 1: PET Chemical Structure.

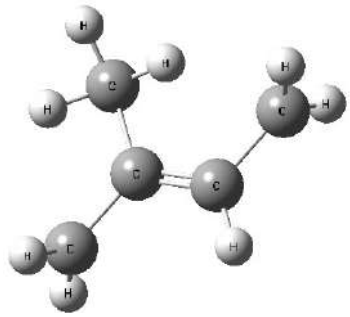

Fig. 2: Natural Rubber Chemical Structure.

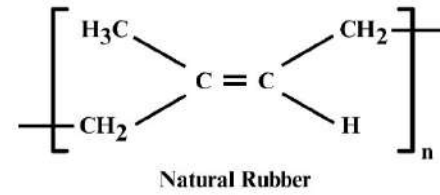

Fig. 3: Monomer of Natural Rubber.

The ester functional groups (-COO-) present in PET provide rigidity and resistance to degradation, making it a durable reinforcement material (Bagadiya et al. 2016, Quintero et al. 2019).

Natural Rubber Structure

Natural rubber (NR) is primarily composed of cis-1,4-polyisoprene, which imparts high elasticity and flexibility. The molecular formula of natural rubber is shown in Figs. 2 and 3. The presence of double bonds in the polymer chain makes NR reactive to chemical modifications, which can be exploited to enhance compatibility with r-PET (Hashim & Kohjiya 1994).

The chemical reaction between polyethylene terephthalate (PET) and natural rubber (NR) chains typically involves the interaction of their respective polymer chains through a process known as cross-linking or grafting.

In such reactions, PET, a polyester, and NR, a polyisoprene-based elastomer, can undergo various chemical modifications when subjected to specific conditions, such as heat, radiation, or the presence of catalysts. This results in the formation of covalent bonds between the polymer chains, leading to crosslinking of the NR chains or grafting of PET onto the NR structure. The reaction often involves the use of initiators or curing agents to facilitate the chemical bonding, which improves the mechanical properties and durability of the material (Nabil et al. 2011). The reaction between PET and NR can be used to enhance the strength, elasticity, and

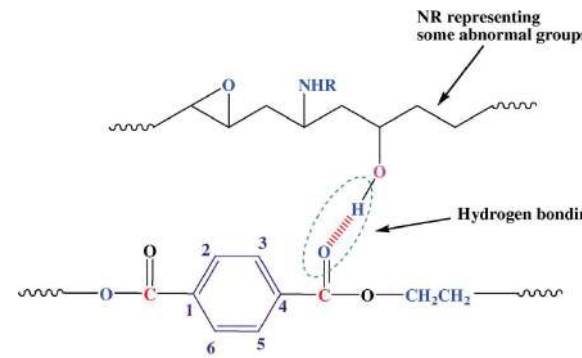

Fig 4: Proposed interaction via hydrogen bonding between the carbonyl group of PET with abnormal groups, such as the hydroxyl functional group in NR (Reddy et al. 2015).

chemical resistance of the resulting composite material, making it suitable for various industrial applications, such as the production of advanced elastomers or hybrid materials.

RECYCLED PET AS A REINFORCEMENT IN RUBBER

Recycled polyethylene terephthalate (r-PET) is increasingly used as a reinforcing material in rubber composites owing to its superior mechanical properties, environmental benefits, and cost-effectiveness. The reinforcement of rubber (Bagadiya et al. 2016) with r-PET has shown promise in improving tensile strength, durability, and thermal stability while reducing dependency on traditional fillers such as carbon black and silica (Nabil et al. 2011). This section explores the various types of rubber in which r-PET can be incorporated, its effects on different properties, and its real-world applications.

PET Reinforcement in Different Rubber Types

Recycled PET can be incorporated into various types of rubber in different forms, such as fiber, powder, and flake. It has been successfully integrated into natural rubber (NR), styrene-butadiene rubber (SBR), ethylene-propylene-diene monomer (EPDM), and nitrile-butadiene rubber (NBR). In natural rubber, PET reinforcement enhances the tensile strength and wear resistance but may slightly reduce the elongation at break owing to its rigid structure. Similarly, in SBR, which is widely used in tire and industrial applications, PET reinforcement significantly improves the resistance to wear and oxidation, contributing to a longer service life. When added to EPDM, which is commonly used in automotive seals and gaskets, PET enhances thermal stability and resistance to ultraviolet (UV) radiation, increasing its longevity in outdoor applications. In oil-resistant applications such as NBR, PET-reinforced composites exhibit superior mechanical strength and enhanced chemical resistance, making them more durable in harsh environments (Cazan et al. 2014, Hashim & Kohjiy 1994, Padhan & Gupta 2015, Popa 2011, Reddy et al. 2015, Sanchez et al. 2021, Vallejos et al. 2024).

The effectiveness of PET reinforcement in different rubber matrices depends on factors such as dispersion quality, interfacial bonding, and processing techniques used. Proper modification and compatibilization strategies can improve the overall mechanical properties of these composites, enabling their use in a wide range of industries, including automotive, construction, and industrial applications.

Effects of PET Reinforcement on Rubber Properties

The inclusion of recycled PET (r-PET) in rubber significantly alters its mechanical, thermal, and dynamic properties of rubber. From a mechanical perspective, PET reinforcement increases tensile and tear strength due to its rigid crystalline structure, making the rubber more resistant to deformation under stress (Cosnita et al. 2017). Additionally, it enhances abrasion resistance, making it particularly suitable for high-wear applications, such as tires and industrial belts. However, incorporating PET can reduce the elongation at break, potentially affecting the rubber's flexibility. This drawback can be mitigated by incorporating plasticizers or modifying the rubber matrix to maintain an optimal balance between strength and flexibility.

In terms of thermal properties, PET reinforcement contributes to improved thermal stability, enabling rubber products to withstand higher operating temperatures without significant degradation. The presence of PET also enhances flame retardancy by reducing the flammability of the composite, making it suitable for applications that require enhanced fire resistance. These thermal improvements extend the lifespan of rubber products, particularly in environments exposed to high heat and harsh conditions (Nabil et al. 2011a).

The dynamic and aging properties of PET-reinforced rubber also exhibited notable enhancements. The inclusion of PET reduces oxidative degradation and aging effects by creating a more stable composite structure that is less susceptible to environmental factors, such as moisture, heat, and UV radiation. This results in improved durability and a longer service life, particularly for outdoor applications, where rubber is exposed to weathering. Increased resistance to UV radiation further contributes to the longevity of rubber components used in construction, automotive, and industrial applications. By incorporating PET reinforcement, manufacturers can enhance the performance and sustainability of rubber products, ensuring they meet stringent

Table 1: Effect of PET Content on Mechanical Properties Across Rubber Types.

Rubber Type	PET Form	PET Content [% by wt]	Tensile Strength Change	Elongation at Break	Hardness
NR	Powder	10%	↑ 25–30%	↓ 10–15%	↑ (Nabil et al. 2011)
SBR	Fiber	15%	↑ 35–40%	↓ Slight	↑ (Cazan et al. 2014)
EPDM	Flake	20%	↑ 20–25%	No significant change	↑ (Vallejos et al. 2024)
NBR	Powder	12%	↑ 30%	↓ 10%	↑ (Reddy et al. 2015)

industry standards and environmental regulations (Zhang 2024).

Processing and Dispersion Challenges

Although r-PET offers significant benefits, several challenges must be addressed to optimize its use in rubber matrices. One of the primary concerns is poor compatibility due to PET's high crystallinity and polarity (Zekriardehani et al. 2017), which makes it inherently incompatible with most rubber matrices. This incompatibility can lead to phase separation, affecting the uniformity of the composite (Cosnita et al. 2017). To counter this, various compatibilization techniques, such as chemical modifications and the use of compatibilizing agents such as maleic anhydride or ethylene

Table 2: Comparative Life Cycle Metrics of Virgin vs. Recycled PET (Duan et al. 2024, Gracida-Alvarez et al. 2023, Zheng & Suh 2019).

Parameter	Virgin PET	Recycled PET	Benefit of r-PET
Carbon Emission [kg CO_2.kg-^{1}]	4.5	1.8	~60% reduction
Energy Consumption [MJ.kg^{-1}]	80	30	~63% energy saving
Waste Diversion	0%	~100%	Prevents landfill waste
Water Usage	High	Low	Reduced environmental load
Cost (Industrial Estimate)	High	Moderate–Low	More economical

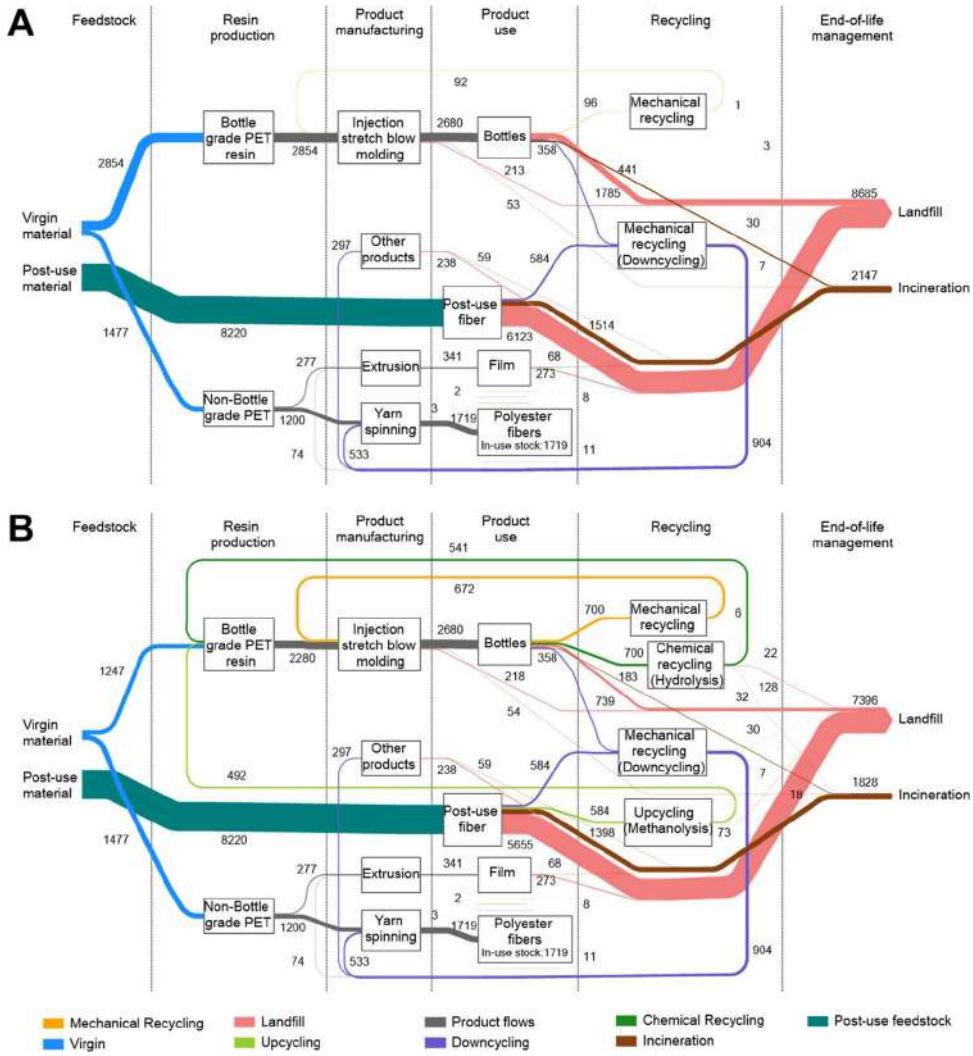

Fig. 5: Sankey diagrams of the U.S. PET bottle supply chain in 2017 for (A) the current state scenario and (B) mechanical, chemical, and upcycling scenarios. The numbers indicate thousand tonnes per year. (Gracida-Alvarez et al. 2023).

vinyl acetate (EVA) copolymers, have been employed to enhance the interfacial adhesion between PET and rubber.

Another significant challenge is the agglomeration of PET particles, which can lead to uneven dispersion within the rubber matrix. When PET particles cluster, the mechanical properties of the final composite become inconsistent, reducing overall performance (Sanchez et al. . n.d.). Effective dispersion techniques, such as high-shear mixing, ultrasonic dispersion, and mechanical milling, can help achieve a more uniform distribution of PET within the rubber.

Processing difficulties also arise because of PET's high melting point of PET (~260°C), which can degrade the rubber properties during blending. The high processing temperature required for PET can lead to the thermal degradation of the rubber matrix, negatively impacting its elasticity and flexibility. To overcome this, processing aids such as plasticizers, stabilizers, and processing oils are used to lower the processing temperature while maintaining the integrity of both the PET and rubber components.

Optimizing the formulation of r-PET-reinforced rubber composites is crucial for achieving the desired balance between their strength, flexibility, and durability. Factors such as PET content, particle size, and surface treatment methods must be carefully adjusted to enhance the compatibility and mechanical performance. By addressing these processing and dispersion challenges, the application of PET-reinforced rubber composites can be expanded across various industries, including automotive, construction, and industrial manufacturing.

Real-World Applications of PET-Reinforced Rubber

Recycled PET-reinforced rubber is gaining traction across multiple industries because of its ability to enhance mechanical performance, reduce material costs, and promote sustainability. One of the most significant applications (Gracida-Alvarez et al. 2023) is in the tire manufacturing industry, where the addition of r-PET improves rolling resistance, tread wear performance, and fuel efficiency. By integrating PET-reinforced rubber into tire treads and sidewalls, manufacturers can produce longer-lasting tires that contribute to lower carbon emissions and improved fuel economy.

In the automotive sector, r-PET-reinforced rubber is used in various components, such as door seals, gaskets, and vibration-damping materials. These parts benefit from enhanced durability, chemical resistance, and improved wear performance. The addition of PET provides better thermal stability, making the material suitable for under-the-hood applications, where exposure to heat and oil is common.

Another prominent application is in industrial belts and hoses, where PET reinforcement improves the mechanical strength and abrasion resistance. Conveyor belts, transmission belts, and hydraulic hoses require materials that can withstand high levels of stress and wear. PET-infused rubber composites offer better load-bearing capacity and extend the service life of these components, thereby reducing maintenance costs and downtime.

In the footwear and sporting goods industries, PET-reinforced rubber is used to manufacture shoe soles and sports equipment. The improved impact resistance and tensile strength make it an ideal material for high-performance athletic footwear and protective gear. Additionally, its ability to enhance energy return and flexibility makes it suitable for products that require high durability and elasticity.

The construction and roofing materials sector has adopted r-PET-infused rubber for applications such as flexible roofing sheets and waterproofing membranes. The incorporation of PET enhances UV resistance, water impermeability, and overall longevity, making it an excellent choice for building materials that are exposed to harsh environmental conditions. Furthermore, PET-based rubber composites are used in vibration isolation pads and shock-absorbing materials in buildings and bridges, where enhanced damping properties are required.

These applications demonstrate the versatility of PET-reinforced rubber and its potential to replace conventional materials while offering superior mechanical properties, cost savings, and environmental benefits (Reddy et al. 2015). As industries continue to seek sustainable alternatives, the adoption of PET-reinforced rubber is expected to expand, thereby driving innovation and efficiency in multiple sectors.

Future Prospects

The use of r-PET in rubber reinforcement is expected to increase with advancements in processing technology and material modifications. Researchers are focusing on improving PET dispersion, developing new compatibilizers, and optimizing processing conditions to maximize the benefits of rPET in rubber applications. As industries move towards sustainability, PET-reinforced rubber will play a crucial role in reducing environmental impact while maintaining high-performance standards.

MECHANISMS OF MIXING PET SCRAP WITH RUBBER

Mixing recycled PET (r-PET) scrap with rubber involves complex physical and chemical interactions that determine the final properties of the composite material. The key challenges in mixing PET with rubber include compatibility,

dispersion, interfacial adhesion, and processing difficulties of the resulting composites. Several mechanisms have been employed to effectively integrate PET into rubber matrices.

Compatibility Factors

One of the primary challenges in mixing PET with rubber is their inherent incompatibility owing to differences in polarity, crystallinity, and thermal properties (Vallejos et al. 2024). PET is a polar, high-melting thermoplastic, whereas rubber is generally non-polar and elastomeric. This mismatch leads to phase separation, negatively affecting the mechanical properties and reducing the efficiency of PET as a reinforcing material. To address this issue, several techniques have been explored to improve the compatibility of PET and the rubber matrices.

Surface modification is an effective approach to enhance compatibility. PET surfaces can be treated with silane coupling agents, maleic anhydride, or other reactive chemicals to improve their adhesion to rubber. Functionalized rubber, where specific functional groups such as carboxyl, hydroxyl, or amine groups are incorporated, can also enhance interfacial bonding with PET (Coniglio et al. 2019). Additionally, blending with compatibilizers, such as styrene-butadiene-styrene (SBS) and ethylene-vinyl acetate (EVA), has been shown to improve interfacial interactions, reduce phase separation, and increase overall mechanical stability.

Despite these efforts, achieving a uniform dispersion of PET within the rubber matrix remains challenging. The agglomeration of PET particles can occur, leading to inconsistencies in the mechanical properties. Effective dispersion techniques, such as high-shear mixing, ultrasonic dispersion, and mechanical milling, help achieve a more uniform distribution of PET within the rubber matrix. Furthermore, optimizing the processing parameters, such as temperature, shear rate, and mixing time, can further enhance the interaction between PET and rubber, thereby improving the composite performance.

By implementing these strategies, the incorporation of PET into rubber matrices can be optimized, resulting in improved mechanical properties, durability, and sustainability of the PET-reinforced rubber composites. As research in this area continues, advanced modification techniques and novel compatibilizers are expected to further enhance the compatibility between PET and rubber, thereby expanding their applications in various industrial sectors.

One of the primary challenges in mixing PET with rubber is their inherent incompatibility owing to differences in polarity, crystallinity, and thermal properties. PET is a polar, high-melting thermoplastic (Bhatt et al. 2015), while rubber is generally nonpolar and elastomeric. This mismatch leads to phase separation, which negatively affects the mechanical properties.

Challenges in Mixing

Several challenges must be addressed to achieve a uniform dispersion of PET in rubber matrices. This leads to inconsistencies in the mechanical properties, which negatively affect protection. This leads to inconsistencies in mechanical properties, negatively affecting strength, elasticity, and durability (Dimitrov et al. 2013). Effective dispersion techniques, such as high-shear mixing, ultrasonic dispersion, and mechanical milling, can help achieve better distribution and uniformity in the composite material.

Another challenge is the high melting point of PET, which requires elevated temperatures for processing. The high processing temperatures (~260°C) required to melt PET can degrade the rubber matrix, leading to a loss of elasticity and mechanical integrity. This problem can be mitigated by using processing aids, plasticizers, or compatibilizers to lower the processing temperature while maintaining the desired mechanical properties of PET and rubber.

Phase separation is another major issue in the mixing of PET with rubber. Owing to differences in polarity and solubility parameters, PET and rubber have limited miscibility, causing phase separation, which weakens interfacial adhesion and reduces overall mechanical performance. Various strategies, such as chemical grafting, surface modifications, and the use of compatibilizers such as maleic anhydride or silane coupling agents, can improve interfacial bonding and enhance the mechanical strength of the composite.

Additionally, PET's crystalline nature presents difficult in achieving proper adhesion and bonding with rubber matrices. Modifying PET through controlled degradation, glycolysis (Vitkauskienė & Makuška 2008), or reactive blending can enhance its compatibility with rubber, resulting in better dispersion and improved mechanical properties (Luo & Van Ooij 2002).

Addressing these challenges through innovative processing techniques, compatibilization strategies, and optimized formulation methods can significantly enhance the performance and viability of PET-reinforced rubber composites in industrial applications.

Modification Techniques

The modification of recycled polyethylene terephthalate (R-PET) composites is essential for enhancing their mechanical, thermal, and structural properties. Various

techniques have been explored to optimize their performance for industrial use. Chemical modification involves surface functionalization using chemical agents, such as silane coupling agents, which enhance the adhesion between PET and fillers. The incorporation of compatibilizers, such as maleic anhydride (MAH) or glycidyl methacrylate (GMA), improves interfacial bonding, whereas chemical treatments, such as alkaline hydrolysis, modify the polymer's surface energy, increasing its interaction with reinforcements.

Physical modification includes melt blending with reinforcing materials such as silica, carbon fibers, or nanoclays to enhance mechanical properties. Plasma treatment alters the surface characteristics, improving the wettability and adhesion properties of PET, and heat treatment techniques, such as annealing, increase the crystallinity and thermal stability of PET composites. Mechanical modification focuses on grinding and reprocessing to improve the particle size distribution and uniformity in composite blends. Mixing PET with impact modifiers, such as elastomers or rubber, enhances its toughness and ductility, whereas fiber reinforcement techniques using natural or synthetic fibers increase the tensile and compressive strengths of PET composites (Li et al. 1998).

To better evaluate the effectiveness of different PET modification strategies, Table 3 summarizes their functional benefits, drawbacks, and industrial applicability. Although silane treatments and chemical grafting agents such as MAH and GMA exhibit excellent bonding performance, they can be prohibitively expensive for large-scale use. Conversely, physical methods, such as alkaline hydrolysis and plasma treatment, are more environmentally favorable but may compromise molecular integrity or require complex infrastructure. Thus, the selection of an appropriate technique depends on the intended application, cost constraints, and environmental compliance.

Hybrid modifications combine chemical and mechanical techniques, such as coupling agents with fiber reinforcements, to yield composites with superior mechanical and environmental resistance. Nano-enhancements using graphene or carbon nanotubes further improve the electrical conductivity and structural integrity.

Case Studies and Applications

Several case studies have highlighted the successful implementation of R-PET composites in various industries. These applications demonstrate the potential of modified PET composites for sustainable construction and engineering solutions. In the construction industry, modified R-PET composites have been tested for use in pavement blocks, demonstrating improved durability and resistance to environmental degradation. Blended PET with sand and other reinforcements has been used to manufacture lightweight and eco-friendly architectural panels.

In the automotive sector, R-PET mixed with carbon or glass fibers is employed in vehicle interiors for lightweight and durable panels. Studies have explored PET-based composites for under-the-hood applications, where thermal stability and impact resistance are critical properties. In the packaging industry, chemically modified PET is used in food containers to improve barrier properties and recyclability. Nanocomposite enhancements have led to PET films with improved mechanical strength and resistance to permeation.

In the textile and fashion industries, PET is extensively used to produce sustainable textiles for clothing and upholstery. PET-based composites are also used in technical textiles for geotextiles and industrial applications. In electronics and electrical applications, PET composites with enhanced thermal conductivity and insulation properties are used in electronic device casings, and the integration of conductive fillers with PET matrices has led

Table 3: Comparison of Key PET Modification Techniques.

Technique	Improvement Achieved	Limitation	Application Area
Silane Coupling Agent	Enhances interfacial adhesion, improves tensile strength	Costly, sensitive to moisture, limited shelf life	Automotive parts, construction
Maleic Anhydride (MAH)	Improves compatibility via grafting	May cause discoloration, not biodegradable	Footwear, hoses, seals
Glycidyl Methacrylate (GMA)	Strong chemical bonding, improves elasticity	Expensive, processing requires peroxide initiator	Tire treads, dynamic components
Alkaline Hydrolysis	Increases surface roughness, low cost	Degrades molecular weight, harsh chemicals	Eco-building materials
Plasma Treatment	Eco-friendly, increases surface energy	Requires specialized equipment, low scalability	Medical packaging, electronics
Compatibilizer (EVA/SBS)	Improves blend uniformity, widely available	Moderate effectiveness, may affect thermal stability	General rubber goods

to the development of lightweight and flexible electronic components.

These case studies demonstrate that effectively modified R-PET composites can significantly contribute to sustainable material development and circular economy initiatives across various industries.

PROCESSING TECHNIQUES AND COMPOSITE FORMULATIONS

The processing of recycled PET (r-PET) in rubber matrices involves several techniques that influence the properties of the final composite. The selection of a suitable processing technique depends on factors such as PET content, rubber type, processing temperature, and required end-use properties. This section elaborates on the major processing techniques, key parameters, material modifications, and their effects on the mechanical, thermal, and morphological properties.

Processing Techniques

The processing of recycled polyethylene terephthalate (R-PET) composites involves several techniques that optimize their structural and mechanical properties. Melt blending is one of the most common techniques, in which PET is heated and mixed with reinforcing fillers such as silica, carbon fibers, or nanoclays to improve its mechanical strength. Extrusion is widely used to process PET composites into sheets, fibers, or molded components, providing a uniform distribution of fillers. Injection molding allows the creation of intricate composite structures with high precision and high reproducibility. Compression molding is another method in which heat and pressure are applied to form durable and high-strength composite panels (Luo & Van Ooij 2002). Advanced processing techniques, such as electrospinning and 3D printing, have also been explored for fabricating PET-based composites with enhanced functionalities (Jamshidi et al. 2005).

Composite Formulations and Key Parameters

The formulation of R-PET composites depends on several key parameters, including the type and proportion of fillers, matrix modification, and processing conditions. The selection of fillers, such as glass, carbon, or natural fibers, influences the mechanical, thermal, and barrier properties of the composite. The incorporation of coupling agents like silane or compatibilizers such as maleic anhydride improves the interfacial adhesion between PET and fillers, leading to enhanced composite performance (Razavizadeh & Jamshidi 2016). Processing parameters, such as temperature, shear rate, and cooling rate, affect the crystallinity and phase dispersion of the composites. The addition of impact modifiers can enhance toughness, whereas stabilizers improve thermal resistance, ensuring longevity in various applications.

Impact on Final Properties

The final properties of R-PET composites are significantly influenced by the processing techniques and formulations used to produce them. The mechanical properties, such as tensile strength and impact resistance, depend on the type of reinforcement and processing conditions (Nabil et al. 2011). The improved dispersion of the fillers enhanced the thermal stability, making the PET composites suitable for high-temperature applications. The addition of nanofillers or compatibilizers increases the resistance of the composite to moisture and chemical degradation, thereby improving its durability. Furthermore, proper processing techniques minimize void formation and enhance density, leading to better load-bearing capabilities in structural applications (Phetphaisit et al. 2015). The ability to fine-tune these properties renders R-PET composites versatile across various industries.

Case Studies and Industrial Applications

R-PET composites have found applications in various industries owing to their sustainable and high-performance characteristics. In the construction industry, they are used in pavement blocks, roofing tiles, and structural panels, offering a lightweight and durable alternative to conventional materials. The automotive industry employs PET composites in interior panels, bumpers, and under-the-hood components because of their excellent strength-to-weight ratio and thermal stability. In packaging, PET-based composites improve barrier properties, ensuring better food preservation and sustainability of the packaging. The textile sector benefits from R-PET fibers, which are used in clothing, upholstery, and non-woven fabrics (Popa 2011). Electronics and electrical applications include PET composites in circuit boards, insulating materials and casings for electronic devices. These case studies illustrate the adaptability of R-PET composites in various high-performance and eco-friendly applications.

MECHANICAL PROPERTIES OF PET-REINFORCED RUBBER

Experimental studies on PET-reinforced rubber composites have demonstrated significant improvements in their mechanical properties, making them suitable for various engineering applications. Research indicates that incorporating PET fibers into rubber matrices enhances the tensile strength, elongation at break, and impact resistance.

Studies have also shown that the dispersion of PET in rubber affects stress distribution, leading to more durable and resilient composites (Cosnita et al. 2014). Additionally, crosslinking techniques, such as sulfur or peroxide curing systems, play a crucial role in defining the mechanical performance of these composites.

Several factors contribute to the property enhancement of PET-reinforced rubber. The selection of reinforcement materials, particle size, and fiber orientation significantly influences the mechanical strength. The addition of coupling agents, such as silane treatments, improves the interfacial bonding between PET and rubber, reducing stress concentration points and enhancing toughness. Furthermore, the degree of crystallinity of PET fibers affects the stiffness and energy absorption capabilities of the final composite (Vallejos et al. 2024).

Material testing techniques are essential in evaluating the mechanical properties of PET-reinforced rubber. Tensile tests determine the strength and elasticity, while dynamic mechanical analysis (DMA) assesses viscoelastic behavior under different temperature conditions. Scanning electron microscopy (SEM) is used to examine the dispersion and adhesion of PET fibers within the rubber matrix (Vallejos et al. 2024). Additionally, thermogravimetric analysis (TGA) provides insights into the thermal stability of the composite, crucial for high-temperature applications.

The performance of PET-reinforced rubber under real-world conditions has been investigated across multiple industries. In automotive applications, these composites exhibit superior resistance to wear and tear, making them ideal for tire treads and suspension components (Cosnita et al. 2017). In construction, PET-reinforced rubber is used in shock-absorbing materials and flexible pavement structures (Cosnita et al. 2014). Additionally, in sports and safety equipment, these composites provide enhanced impact resistance, ensuring durability and protection of the equipment. These real-world applications highlight the versatility and efficiency of PET-reinforced rubber, further establishing its potential for sustainable material development.

THERMAL AND MORPHOLOGICAL ANALYSIS

Advanced thermal studies of PET-reinforced composites are crucial for understanding their behavior under varying temperature conditions. Thermogravimetric analysis (TGA) revealed that R-PET composites exhibited enhanced thermal stability compared to virgin PET, primarily owing to the presence of reinforcing fillers and improved interfacial bonding. Differential scanning calorimetry (DSC) was employed to evaluate the melting and crystallization behaviors of these composites, revealing that the incorporation of nanofillers and compatibilizers can significantly modify the thermal transitions and enhance the heat resistance.

The material decomposition behavior is an essential factor in assessing the longevity and performance of PET-based composites. Studies have indicated that PET decomposition occurs in multiple stages, beginning with the breakdown of weaker polymer chains and followed by more complex degradation reactions (Padhan & Gupta 2015). The presence of stabilizers and flame retardants in PET composites can delay thermal degradation, ensuring enhanced durability for high-temperature applications (Pacheco-Torgal et al. 2012). Additionally, pyrolysis studies have demonstrated that PET composites can be effectively recycled through controlled thermal decomposition processes, thereby minimizing their environmental impact.

Surface morphology analysis plays a pivotal role in determining the structural integrity of PET composite materials. Scanning electron microscopy (SEM) is widely used to examine the dispersion of fillers, detect microstructural defects, and assess fiber-matrix adhesion. Atomic force microscopy (AFM) provides insights into the surface roughness and interfacial interactions, aiding in the optimization of composite formulations (Phinyocheep et al. 2007). Well-dispersed reinforcements within the polymer matrix lead to enhanced mechanical properties and thermal stability, ensuring superior performance in practical applications.

The impact of PET content on thermal stability has been extensively studied to identify optimal compositions for various applications. Increasing the PET content generally improves the heat resistance of the composites; however, excessive PET concentrations may lead to brittleness and reduced toughness. Balancing PET with other polymers or elastomers ensures synergy between mechanical strength and thermal endurance. The experimental results highlighted that composites with controlled PET content and appropriate fillers demonstrated superior resistance to thermal degradation, making them suitable for demanding industrial applications.

ENVIRONMENTAL AND SUSTAINABILITY ASPECTS

The incorporation of R-PET in composite formulations presents significant environmental and sustainability benefits (Vallejos et al. 2024). The use of recycled materials reduces plastic waste, minimizes landfill accumulation, and decreases dependency on virgin petroleum-based resources (Miranda Vidales et al. 2014). Regulatory frameworks, such as extended producer responsibility (EPR) programs

and environmental directives, encourage industries to adopt sustainable materials and ensure compliance with eco-friendly manufacturing practices. Industry adaptation has been steadily increasing, with sectors like automotive, construction, and packaging integrating PET-based composites to meet sustainability goals (Zhang 2024). Compared with conventional rubber reinforcement methods, PET composites offer superior recyclability, a reduced carbon footprint, and enhanced durability. The long-term benefits of PET-reinforced composites include improved lifecycle performance, lower production costs, and alignment with circular economy principles, making them viable alternatives to traditional reinforcement materials.

ENVIRONMENTAL IMPACT CALCULATION

A detailed life cycle assessment (LCA) of PET-reinforced (Duan et al. 2024) composites highlights their significant environmental advantages over conventional materials. LCA studies have shown that incorporating ET in composite formulations results in lower greenhouse gas emissions, reduced energy consumption, and minimized resource depletion (Phetphaisit et al. 2015). Emissions data indicate that PET composites emit fewer carbon dioxide equivalents per kilogram compared to virgin plastics and conventional rubber reinforcements. The use of recycled PET also contributes to waste reduction by diverting plastic waste from landfills and reducing reliance on virgin polymer production.

Carbon Footprint Reduction:

- Virgin PET production emits approximately 4.5 kg CO_2 per kg of PET produced (Zheng & Suh 2019).
- Recycling PET reduces emissions by up to 60%, leading to a net reduction of approximately 2.7 kg CO_2 per kg of recycled PET used in rubber composites.

Energy Savings:

- Manufacturing virgin PET requires 80 $MJ.kg^{-1}$ of energy.
- Recycled PET requires only 30 $MJ.kg^{-1}$, leading to a significant reduction in energy consumption.

Waste Diversion:

- Incorporating r-PET into rubber reduces landfill waste and extends the lifecycle of plastic materials.

Waste reduction benefits of PET composites are evident in their recyclability and reusability, promoting a circular economy. By using post-consumer PET waste, industries can significantly lower their environmental footprint while maintaining high material performance. Economic impact assessments demonstrate that adopting PET-based composites can lead to cost savings in material procurement, processing, and waste management. (Zhang 2024). Additionally, regulatory incentives and sustainability goals encourage industries to transition towards PET composites, further solidifying their role in eco-friendly material innovation.

Comparison with Carbon Black-Reinforced Rubber

To evaluate the environmental advantage of PET-reinforced rubber, it is essential to compare it with the most commonly used conventional filler, carbon black (CB). Carbon black is a fossil fuel–derived reinforcing agent widely used in rubber compounding, particularly for tires and industrial goods. However, its production is highly energy-intensive and generates significant greenhouse gas (GHG) emissions.

A direct life cycle comparison between CB- and r-PET–reinforced rubber composites reveals considerable environmental benefits in favor of r-PET, as shown in Table 4.

In addition to lower GHG emissions and energy use, r-PET provides circular economy benefits by diverting plastic waste from landfills and reducing dependency on virgin fossil resources. While carbon black remains technically superior in some reinforcement metrics, the environmental trade-offs strongly favor r-PET, particularly for non-critical or semi-dynamic applications where sustainability is prioritized.

CHALLENGES AND FUTURE PROSPECTS

Despite the numerous benefits of PET-reinforced composites, several challenges limit their widespread adoption. One

Table 4: Life Cycle Comparison – Carbon Black vs. r-PET as Reinforcement in Rubber.

Metric	Carbon Black (CB) Reinforcement	Recycled PET (r-PET) Reinforcement	Relative Benefit
GHG Emissions [kg $CO_2.kg^{-1}$ filler]	~2.4	~1.8	~25% reduction
Energy Use [$MJ.kg^{-1}$ filler]	~100	~30	~70% reduction
Waste Diversion	None	High (from post-consumer PET)	Strong benefit
Carbon Source	Fossil	Recycled polymer (plastic waste)	Renewable-based
Circular Economy Value	Low	High	√

major limitation is the difficulty in achieving consistent mechanical properties due to variability in recycled PET feedstocks. Addressing this issue requires advanced sorting and purification techniques to enhance the quality of recycled materials. Another challenge is the relatively high processing cost associated with modifying PET composites for specific applications. Innovations in processing technologies, such as improved compatibilization methods and additive manufacturing, can help mitigate these costs.

Future innovations in PET-reinforced composites will likely focus on developing bio-based (Bhatt et al. 2016) and hybrid reinforcements to enhance sustainability and performance. Research into self-healing materials and smart composites with adaptive properties can further expand the applications of PET composites. Large-scale adoption of PET composites can be facilitated through industry collaborations, policy incentives, and advancements in recycling infrastructure. Establishing standardized testing and certification protocols will also play a crucial role in ensuring the reliability and acceptance of these materials across various industries. With continued advancements, PET-reinforced composites are poised to become a key component in next-generation sustainable materials.

CONCLUSIONS

Recycled PET presents a sustainable and effective reinforcement for rubber compounds, offering improved mechanical and thermal properties while reducing environmental impact. Through various mixing mechanisms, PET scrap can be effectively incorporated into rubber, enhancing its durability and reducing reliance on virgin raw materials. Chemical modifications, such as glycolysis and compatibilization, further enhance the feasibility of PET-rubber composites.

However, challenges such as optimizing processing conditions, ensuring homogeneous dispersion, and improving interfacial adhesion remain critical areas of research. Addressing these challenges will facilitate broader industrial adoption of PET-reinforced rubber composites.

Future advancements in chemical recycling techniques, including depolymerization and reactive extrusion, hold great promise for enhancing the performance of r-PET in rubber matrices. Additionally, more in-depth environmental assessments can provide clearer insights into the long-term sustainability benefits of such materials.

Overall, continued research and innovation in this field will pave the way for greener, more sustainable rubber products, contributing to a circular economy and reducing the environmental burden of plastic waste.

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Performance Analysis of Membrane Distillation in Desalinating and Concentrating Brine on a Pilot Scale

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ABSTRACT

Investigating desalination and brine concentration using advanced membrane and thermal processes is crucial for reducing energy consumption and costs in the desalination industry. Emerging technologies such as forward osmosis (FO), osmotically assisted forward osmosis (OAFO), pressure-assisted forward osmosis (PAFO), electrodialysis (ED), membrane distillation (MD), and solvent extraction desalination (SED) have shown promise at the lab and pilot scales but are not yet commercially viable due to operational and economic challenges. In our study, we focused on MD to evaluate desalination performance using various saline feeds, including fresh, brackish, seawater, and desalination brine from Kuwait, applying both electrical and solar heating methods. Results revealed higher water flux for brackish water compared to seawater and brine, with salt rejection unaffected by increased salinity. Energy consumption was more influenced by feed quantity than by salinity. The water flux ranged from 1.5 to 2 L per square metre per hour ($L.m^2h^{-1}$), with a water recovery of 3.3 to 4% in electrical heating mode of operation. Solar mode operation of the MD system showed a water flux of 0.95 to 1 $L.m^2h^{-1}$, with an average recovery of 2.75%. Our findings highlight the practical potential of MD for solar desalination and brine concentration in remote areas and small-scale industrial waste treatment.

INTRODUCTION

Membrane distillation (MD) has been utilised in various applications over the past decade and is acknowledged as an effective method for wastewater treatment, desalination, and brine concentration (Reddy et al. 2021, Hegde & Ribeiro 2022, Ali et al. 2011, Gourai et al. 2015, AlMallahi et al. 2024). MD technology has a history of more than three decades. However, MD technology is not available on a commercial scale due to unsolved challenges and its lower efficiency. Several laboratory and pilot-scale studies have been conducted to evaluate the viability of the MD process for different applications in the literature (Gourai et al. 2015, Tiwari et al. 2022, Adewole et al. 2022, Yan et al. 2021, Conidi et al. 2020, Bhattacharjee et al. 2017, Yadav et al. 2021, Zhong et al. 2021, Choi et al. 2019, Shalaby et al. 2022, Drioli et al. 2014, Essalhi 2014, Camacho et al. 2013). All the literature indicates that MD technology is more suitable for small-scale applications due to challenges and limitations in the process. The challenges in MD technology include the unavailability of highly efficient hydrophobic membranes, low thermal energy loss during treatment, low fouling for high-salinity water, and module design. Despite these challenges, MD technology has gained more interest among the research community for treating desalination brine (Bhattacharjee et al. 2017, Yadav et al. 2021, Zhong et al. 2021, Choi et al. 2019). In recent years, applied research on MD technology for desalination and brine concentration applications has gained more interest due to the possibility of using renewable energy and waste heat for the process. The MD process requires low-grade heat to increase the temperature of the feed solution to a range of 50–80 ºC. Therefore, the energy needed for the

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MD process can easily be provided by renewable energy sources (Hegde & Ribeiro 2022, Ali et al. 2011, Tiwari et al. 2022). Additionally, MD membranes are hydrophobic and low fouling, requiring minimal pre-treatment, which will reduce chemical costs and minimise the discharge of harmful chemicals into the environment.

On the other hand, literature reviews show that solar MD technology offers a simple and promising solution for effectively producing distilled water on a small scale (Elbar & Hassan 2020, Moossa et al. 2022, Hamwi et al. 2020). This is especially beneficial for remote areas, emergencies, and situations with limited resources. Additionally, the application of MD technology to treat small-scale rejects from industries and desalination brine from farms leads to reduced environmental impact from these rejects. However, MD technology is in the developmental stage; therefore, it requires thorough investigation of its performance in specific geographical locations. Kuwait is blessed with abundant solar energy due to its geographical location, receiving an estimated annual solar irradiation of 2,100-2,200 kilowatt-hours per square metre ($kWh.m^{-2}$) per year, with an average of 7 to 12 hours of sunshine per day (Hadi et al. 2013, Briney 2020, Geography of Kuwait 2020). Consequently, there is great potential for implementing a solar MD system in Kuwait for decentralised freshwater production and brine concentration applications. This would also help reduce greenhouse gas emissions, lessen the carbon footprint in the environment, and reduce the brine discharge problem in remote areas. There are several similar studies conducted worldwide at the laboratory and pilot-scale level (Shatat et al. 2013, Jawed et al. 2024, Kumar & Martin 2017, Al-Sairfi et al. 2023). However, conducting a study in the State of Kuwait is very important due to the higher salinity and turbidity levels in Kuwait's saline water. Additionally, the high solar radiation intensity, along with dust and wind effects, significantly impacts the quantity and quality of distilled water production. As a result, a study was carried out to evaluate the feasibility of implementing an integrated solar-powered MD technology for desalinating seawater and brine concentration by considering the current environmental conditions in Kuwait.

MATERIALS AND METHODS

Materials

The Air-Gap Membrane Distillation (AGMD) membrane module was purchased from Aquastill BV, the Netherlands. The AGMD module, with a membrane area of 5.6 m^2, features a spiral-wound design, and the active material is a super-hydrophobic polypropylene (PP) polymer membrane. The solar thermal unit was purchased from Sanvi Solar Pvt. Ltd., India. The solar thermal unit has both electrical coil heating connections and solar heating. Automated temperature (A GSP-6) and humidity (Elitech RC-5+TE) sensors were purchased through Amazon, and a solar power meter was purchased from Munro Instruments MRC Group Company, United Kingdom. Feed water samples were collected from different locations in Kuwait and analysed at the International Organization for Standardization (ISO)-certified laboratories of the Water Research Center (WRC) at the Kuwait Institute for Scientific Research (KISR), Kuwait. The instruments were calibrated and examined using international standards and techniques.

Methods

The solar MD system was fabricated in the WRC workshop by combining a solar thermal unit with an AGMD module. The required electrical heater, sensors, flow meters, and a heat exchanger were incorporated into the fabricated solar MD system to measure the required parameters. The schematic diagram is shown in Fig. 1, and the actual image of the fabricated system is presented in Fig. 2. The electrical heater in the solar thermal unit was used as a heating source to optimise the feed flow rate and temperature using second-stage reverse osmosis (RO) permeate as feed. The optimisation process was conducted inside the laboratory to avoid the solar thermal heating effect from the evacuation tubes. Optimisation experiments were conducted over a period of 2 hours, and each result was recorded at 30 min intervals. The heat exchanger was used to exchange heat from the thermal storage tank to the feed. The required feed temperature was set by adjusting the heating rate of the coil in the thermal storage tank. After the optimisation of the process was complete, optimised parameters were set to assess the desalination performance of different saline waters. Once the initial trials were completed, the solar MD system was fixed on the rooftop of the building and connected to the RO brine line as feed. Experiments were conducted over a period of six hours during the daytime, and each result was recorded at 30 min intervals for five days. During the experiments, the feed solution was supplied to the bottom of the AGMD module using a feed pump. This initial feed solution passed through the membrane module and heat exchanger and returned to the AGMD module from the top side. During this process, the AGMD module acted as a heat exchanger and increased the temperature of the incoming feed solution. Any additional temperature required for the feed solution was supplied through an electrical heater, as shown in Fig. 1. Due to the temperature difference between the feed side and the hot side of the membrane, water vapour passed through the membrane and emerged in the permeate spacer channel. By measuring the quantity and quality of the distillate, the water flux and

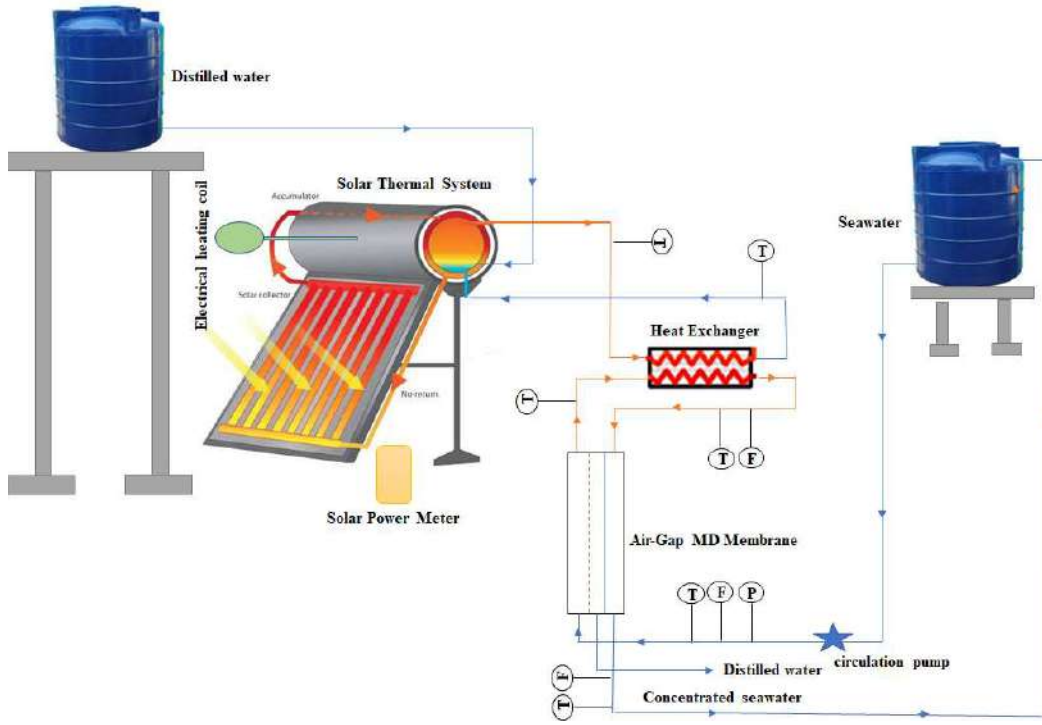


Fig. 1: Schematic process flow diagram of the membrane distillation system.

salt rejection were calculated using Equations 1 and 2. In the brine concentration experiments, MD brine was recirculated back to the feed tank. After completing the electrical heating experiments, the heater was disconnected, and solar energy was used as the heat source to heat the incoming feed. The solar MD brine concentration experiments were conducted for five days, using RO brine feed operating six hours per day. During the solar MD test, solar radiation intensity, weather temperature, humidity levels, feed-in temperature, and brine-out temperature were measured at each 30 min interval using a pre-programmed measuring instrument to record mean values. The changes in the measurement values were correlated with water production values in the Results and Discussion. The experiments conducted using the electrical heating mode of operation were triplicated, and the mean value of the results was reported.

$$J_v = \frac{\Delta V}{A_m \cdot \Delta t} \quad ...(1)$$

Where Jv is the water flux ($kg.m^2h^{-1}$), ΔV is the quantity of distillate (kg), Am is the effective distillation area (m^2), and Δt is the sampling time (h).

The rejection coefficient R will be calculated according to the following equation:

$$J_v = \frac{\Delta V}{A_m \cdot \Delta t}$$

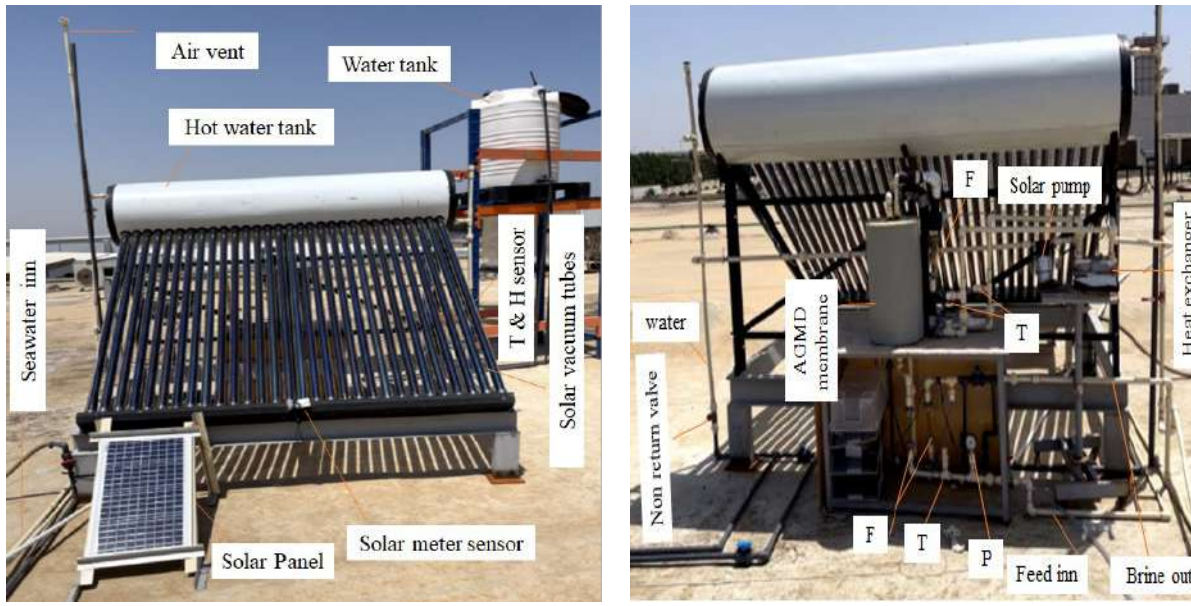
$$R = \frac{C_f - C_p}{C_f} \quad ...(2)$$

Where, C_f and C_p are the concentrations of the feed and distillate, respectively.

RESULTS AND DISCUSSION

Desalination and Brine Concentration Performance Study

The MD system was constructed as shown in Fig. 1. A leakage test and calibration of the sensors were conducted. After the system underwent preliminary testing, the operating parameters of the MD system were optimised using second-stage RO permeate as feed by varying the feed flow rate (120-240 $L.h^{-1}$) and temperature (50-80°C). The highest water flux (1.64 $L.m^2h^{-1}$) was observed at a feed temperature of 80°C and a feed flow rate of 240 $L.h^{-1}$. Therefore, an optimised feed flow rate of 120 $L.h^{-1}$ and a temperature of 80°C were considered for desalination and brine concentration applications using different saline waters.



a: Front side view b: Back side view

T: Temperature, H: Humidity

Fig. 2. a) Front side view of integrated solar membrane distillation system, and b) back side view of integrated solar membrane distillation system.

Table 1: Physicochemical Analysis of Investigated Feed Solution.

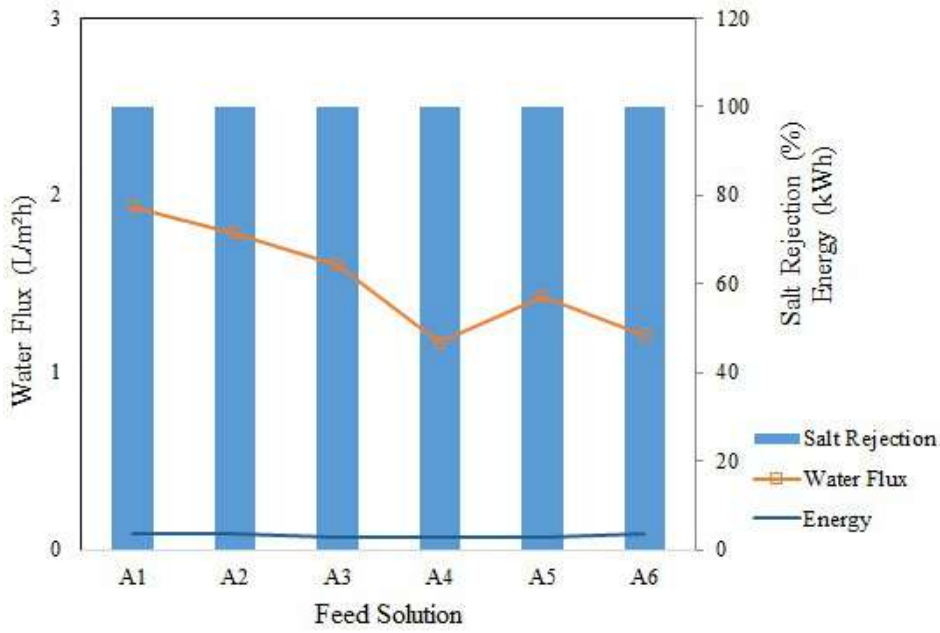
Sample Name	Parameters/Unit								
	TDS [ppm]	pH	Conductivity [ms.cm^{-1}]	Mg [mg.L^{-1}]	Ca [mg.L^{-1}]	SO_4 [mg.L^{-1}]	Na [mg.L^{-1}]	Cl [mg.L^{-1}]	K [mg.L^{-1}]
A1	4,692	7.94	7.45	123.4	590	1,400.3	910	1,957.7	35
A2	8,358	8.14	13.21	299	916	2,454.7	1,146	4,081	113
A3	43,504	7.93	61.7	1,288	825	4,901.7	11,688	22,938	393
A4	66,784	7.86	94.84	1,663.2	1,240	4,241	18,240	38,908.7	611
A5	43,560	7.96	61.7	1,350	1,288	6,190.7	12,288	25,452.4	422
A6	56,336	8.03	80	1,536	1,306	7,824.7	15,360	31,831.6	520

TDS: Total dissolved solids, ppm: parts per million, ms.cm^{-1}: milli siemens/centimeter, Mg: magnesium, Ca: calcium, SO_4: Sulfate, Na: Sodium, Cl: Chloride, K: Potassium, mg.L^{-1}: mg.L^{-1}

A1: Wafra groundwater, A2: Wafra groundwater RO brine, A3: Doha beach well seawater, A4: Doha beach well seawater RO brine, A5: Shuwaikh surface seawater, A6: Shuwaikh seawater MSF brine.

The desalination and brine concentration study was conducted using different saline feed solutions collected from Kuwait. The collected samples included different salinities ranging from 4,692 parts per million (ppm) to 66,784 ppm, including brackish groundwater and its RO brine, beach well seawater, surface seawater, RO brine, and multistage flash distillation (MSF) brine. The names of the locations and the physicochemical analysis results of the feed solutions are presented in Table 1.

The desalination study conducted using electrical heating mode showed that the water flux was higher for the low-salinity groundwater feed source than for the brine sample, as shown in Fig. 3 and Table 2. The water flux values clearly indicate that an increase in feed concentration results in a decrease in water flux (Schwantes et al. 2018, Duong et al. 2021). All the tested feed solutions showed more than 99% rejection efficiency, and the energy requirement ranged from 3 to 3.5 kilowatt-hours (kWh). Freshwater recovery ranged between 3.33% and 4%, indicating that water recovery is not significantly affected by increased feed salinity from groundwater (4,692 ppm) to RO brine feed (66,784 ppm). This indicates that the MD system is able to concentrate



A1: Wafra groundwater, A2: Wafra groundwater RO brine, A3: Doha beach well seawater, A4: Doha beach well seawater RO brine, A5: Shuwaikh surface seawater, A6: Shuwaikh seawater MSF brine.

Fig. 3: Desalination performance in electrical heating mode.

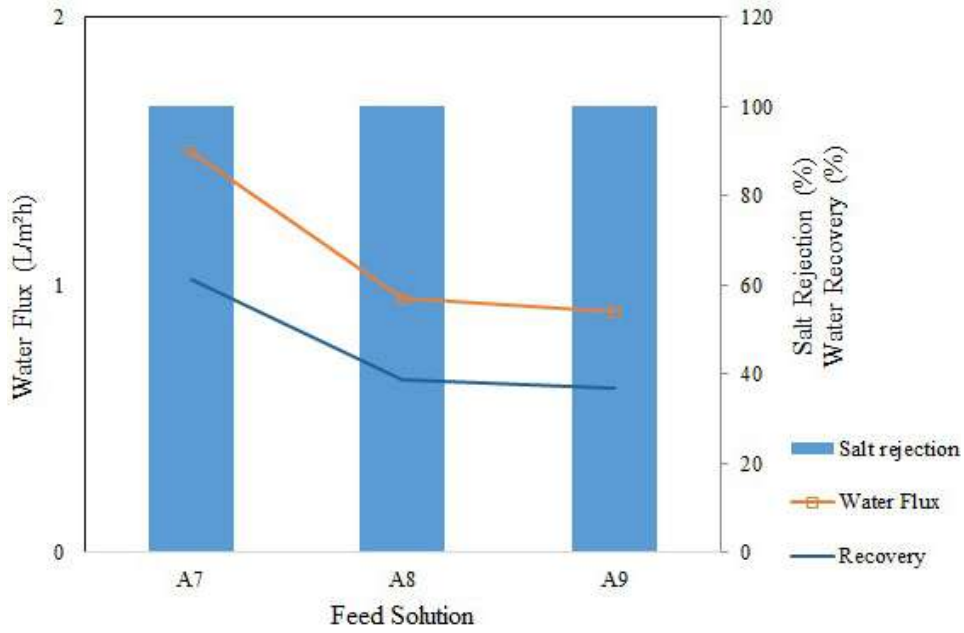
Table 2: Desalination Performance of the Investigated Feed Solution.

Sample Name	Water Flux [L.m²h^{-1}]	Energy [kWh]	Salt Rejection [%]
Wafra groundwater	1.93±1	3.5±1	99.97±1
Wafra groundwater RO brine	1.78±2	3.5±1	99.96±1
Doha beach well seawater	1.60±2	3±1	99.98±1
Doha beach well seawater RO brine	1.16±4	3±	99.99±1
Shuwaikh surface seawater	1.42±2	3±1	99.98±1
Shuwaikh seawater MSF brine	1.21±1	3.5±	99.98±1

RO brine feed to a much higher concentration level without significantly compromising salt rejection and water recovery (Alobaidani et al. 2008, Schwantes et al. 2018, Duong et al. 2021, Ugarte et al. 2024, Hamwi et al. 2022).

During the experiment, the temperature loss on the brine-side solution was calculated by measuring the temperature of the feed-in, feed-out from the membrane, and brine-out. For all the tested experiments, the temperature loss was almost 5–6°C on the brine side, and temperature recovery was 93%. The higher recovery of temperature from the brine side is due to the module design and its configuration. The tested Aquastill MD membrane has a three-channel membrane configuration and acts as a heat exchanger during the desalination process. Therefore, the Aquastill MD membrane requires less energy for the desalination process compared with other module designs, such as hollow-fiber modules and simple plate-and-frame modules (Duong et al. 2021).

After completing the initial desalination studies, the MD system was applied for brine concentration using groundwater RO brine, seawater RO brine, and seawater MSF brine as feed solutions. Tests were conducted over 6 hours, data were recorded every 30 min, and the brine solution was recirculated to the feed tank. For each test, 75 L of feed solution was taken in a closed, insulated container, and temperature variation in the feed tank was measured every 30 min. During the experiments, the temperature of the feed increased over time, but the temperature difference between the membrane feed side and the brine side adjusted automatically due to the MD module configuration. The temperature on the permeate side increased due to the higher temperature between the feed side and the permeate-side solution. A slight reduction in water flux was observed compared to experiments without brine recirculation, but salt rejection exceeded 99%. Fig. 4 shows



A7: Wafra groundwater RO brine, A8: Doha beach well seawater RO brine, A6: Shuwaikh seawater MSF brine.

Fig. 4: Brine concentration performance in electrical heating mode.

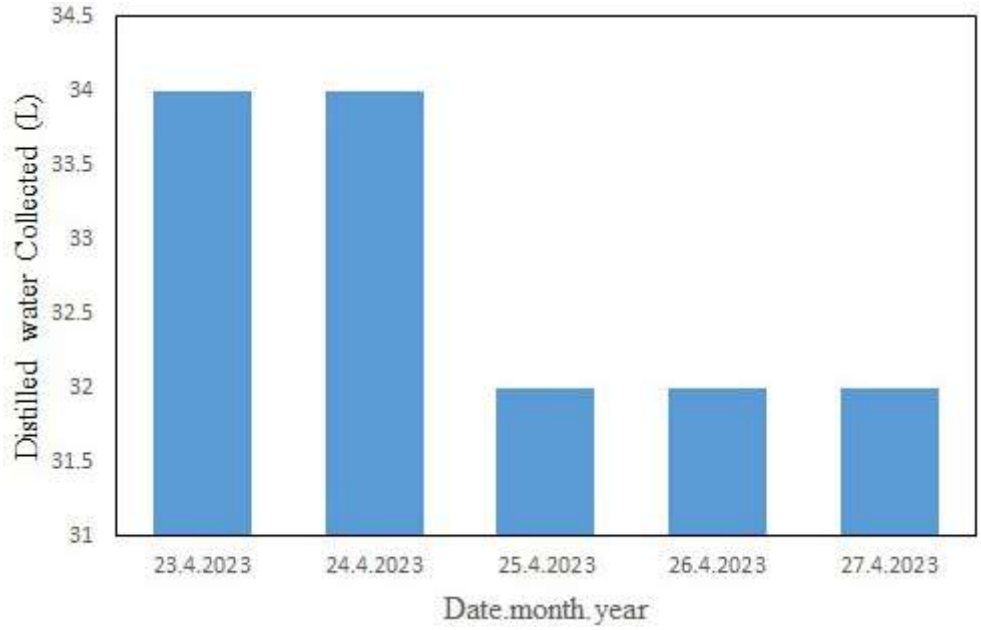
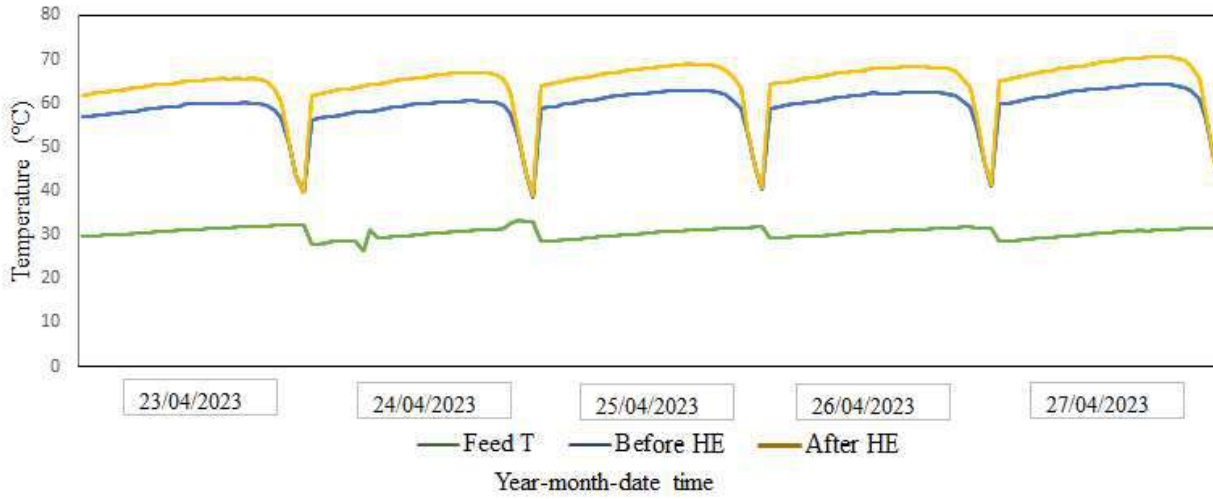


Fig. 5: Daily average distilled water production in solar mode.

the graphical representation of water flux, salt rejection, and overall recovery.

After completing the electrical-mode brine concentration study, the MD system was fixed on the rooftop of the DRP building, and its brine concentration performance was assessed using RO brine as feed. The MD system was operated for five days, running the system each day for six hours. The total feed volume was 500 L, and the brine from the system was recirculated back to the feed tank. The solar MD system operated from April 23 to April 27, 2023. During this period, the daytime temperature (T) ranged from 24.1 to 42.5°C (with a mean T of 30.83°C), humidity (H) ranged



T: temperature, HE: heat exchanger.

Fig. 6. Operating parameter during the solar MD operation.

from 23.1 to 69.7% (with a mean H of 43.32%), and the average radiation over all five days was 478.76 W.m^{2-}. The experimental results show that daily permeate production ranged from 32 to 34 L.day^{-1}, and each day produced nearly the same quantity of permeate/distilled water (Hamwi et al. 2022). This indicates that the increase in feed salinity did not significantly affect permeate quantity during the five-day operation period. This may be due to additional heat storage in the thermal tank after six hours of operation, which was utilised for the next day. There was no change in the water flux and salt rejection values throughout the study. This indicates that the membranes were not fouled or scaled due to the increased salinity and drastic changes in temperature during the day and night (changes in feed temperature). Further research is needed for year-long testing of the solar MD system and to study changes in membrane properties to observe fouling or scaling issues. The daily permeate production, variation of temperature, humidity, and system inlet and outlet temperatures are presented in Figs. 5 and 6.

Overall, the experimental results showed that the MD system is a promising technology for brine concentration applications. The integration of solar energy into the MD system for brine concentration will also help reduce the environmental impact. The research team recommends long-term (more than one year) operation of the solar MD system to accurately evaluate its performance and fouling behaviour for brine concentration applications.

CONCLUSIONS

The MD system was fabricated to operate in both electrical and solar heating modes. The desalination performance of the fabricated MD system was evaluated using different saline waters. The experimental results from the electrical heating mode showed that the fabricated MD system could desalinate saline water ranging from 4,692 ppm to 66,784 ppm without compromising salt rejection. Brine concentration studies using the electrical heating mode showed highly promising results, concentrating the feed brine solution up to 20% (near saturation level). The five-day operation of the solar MD system for RO brine concentration applications showed uniform water flux and salt rejection. This clearly indicates that solar MD technology is a promising and viable process for brine concentration.

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Sunlight-Induced Photocatalytic Degradation of Methyl Red Using Lignocellulosic Biomass of *Ricinus communis* Stem with Isotherm and Kinetic Modeling

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ABSTRACT

Nowadays, Water pollution is a major global issue brought on by the mixing of effluents from various industries, such as leather, paper, printing, cosmetics, Textile etc., containing metal ions and dyes. This impact causes severe damage to the health of humans, aquatic plants and animals. In view of removing the dyes from the wastewater, several techniques available pose certain drawbacks. To combat such issues, a rapid, cost-effective, eco-friendly, and sludge-less method for removing dye from the effluent using activated *Ricinus communis* stem as a catalyst in the solar irradiation method is discussed and reported with results. The adsorption studies carried out, followed by degradation of methyl red onto Si-RCS, shows 86% in the presence of natural sunlight, and the optimum dye concentration was 20 ppm at 0.25 g photocatalyst dosage. It depicts that the prepared adsorbent material, Si-RCS, can be used as an effective adsorbent as well as a photo-catalyst to treat textile effluents. The best fit model was found to be pseudo-second order based on the R^2 value. Among the isotherm models studied, it was found that Langmuir is the best fit model.

INTRODUCTION

Water is essential to all life for survival. Water pollution is the tainting of bodies of water, including rivers, lakes, ponds, and groundwater, as a result of the dumping of sewage, industrial waste, acid rain, oil pollution, thermal power plants, and eutrophication. One of the most significant industrial sectors involved in environmental degradation is the textile sector. There are 72 hazardous compounds released into water supplies by the dyeing industry, of which 30 hazardous compounds are difficult to remove. Each year, more than 10,000 dyes are used in the textile, leather, paper, rubber, cosmetic, pharmaceutical, plastic, and food industries (Monda 2018). The dye-containing wastewater breaks down into cancer-causing aromatic amines in the absence of dissolved oxygen, which adversely affects the animals, plants, and humans (Jameel et al. 2024). Sludge, industrial waste, and radioactive waste disposal contributes pollutes around 70% of the water resources. Since dyes are typically mutagenic, carcinogenic, and poisonous by nature, industrial effluents should be treated before being released into the environment (Hameed & El-Khaiary 2008).

When aquatic species ingest these textile effluents through the food chain, it can cause several physiological abnormalities, including kidney damage, hypertension, cramps, and occasional fever (Karthikeyan et al. 2006). A risk to survival arises from the buildup of dyes in biotic and abiotic components. Water pollution is a contributing factor to several health issues, including blood disorders, heart conditions, nervous system disorders, skin lesions, vomiting, and diarrhea, including chromosomal changes (Galenda et al. 2014).

Methyl red is a monoazo dye (2-(4-[dimethyl amino] phenyl azo) benzoic acid) used in textile dyeing, paper printing and as an indicator in acid-base titrations. The discharge of Methyl red from various industries causes harmful effects to human beings, plants, animals and the environment (Vinoda et al. 2015). The direct inhalation of methyl red damages the central nervous system, causes digestive system irritation, kidney failure and severe depression, which signifies the removal of methyl red dye before discharging into the environment. Numerous adsorbents were investigated for the removal of Methyl red by various researchers were given in Table 1.

Activated carbon is a widely used adsorbent for the removal of dyes and heavy metals, because it has a large surface area, a high microporous structure, high adsorption capacity, and special surface reactivity (Zhi et al. 2015). Various types of adsorbents are used to increase the degradation capacity in an economic way. Many researchers have attempted to develop adsorbents from various resources such as food waste, agricultural wastes, industrial wastes, etc. (Anjali et al. 2022). Natural materials from agricultural and industrial processes are abundant and could be used as low-cost adsorbents (Mahmoud et al. 2020, Karunakaran & Thamilarasu 2010). Because of their low cost, these materials can be disposed of without requiring costly regeneration after they have been used. Most of the low-cost adsorbents have the limitation of low adsorption capacity, and they generate more solid waste, which poses disposal problems.

The present work investigates the potential of *Ricinus communis* stem activated by silica gel for the degradation of the synthetic textile dye methyl red from wastewater. *Ricinus communis* is a species of flowering plant in the spurge family, Euphorbiaceae, and belongs to a monotypic genus, *Ricinus*, and subtribe, Ricininae (Manickavasagam et al. 2013).

Ricinus communis grows throughout the drier parts and near drainages of India. The annual production of *Ricinus communis* is estimated to be more than 1.0 million tons globally, of which India accounts for 60% of the production (Makeswari & Santhi 2013). The removal of heavy metals and dyes using various parts of *Ricinus communis* is listed in Table 2.

Table 1: Degradation of Methyl red using Nano particles and agricultural waste.

Pollutant	Adsorbent used
Methyl red dye	Silica nanoparticles from rice husk
	Nano Fe_3O_4
	Ag-N-Co doppedZnOnanoparticles
	Tungsten trioxide thin films
	Boron and nitrogen-doped TiO_2.
	Seaweed-mediated zinc oxide Nanoparticles
	Spent oil shale

MATERIALS AND METHODS

Adsorbent Material

The stem of *Ricinus communis* (RCS) was collected near Saravanampatti, Coimbatore District (Tamil Nadu). The adsorbent was thoroughly washed in running tap water to remove dirt and other particulate matter. The washed adsorbent was dried, powdered, and stored in an airtight container for further studies.

Preparation of Adsorbent

Preparation of silica-activated *Ricinus communis* stem (Si-RCS): *Ricinus communis* stem powder is dissolved in water and agitated in a magnetic stirrer for 2 h. Silica is added to the slurry, and agitation is continued for another 1h to attain homogeneity. The whole content is kept in a stable place for 8 h to obtain bubble free mixture. Ethanol and NaOH are added to the mixture and kept aside for 24 h to form the final product. The final product is filtered and dried in a hot air oven at 100° C for 5 h. The dried product is packed in an airtight container for further studies (Madhusudhan et al. 2012).

Preparation of the adsorbate: A stock solution of methyl red (1000 ppm) was prepared separately and diluted to the various required initial concentrations.

Instrumentation

The instruments used for the analysis of adsorbent and photocatalytic behavior are a Digital pH meter, Conductivity

Table 2: Removal of heavy metals and dyes using various parts of *Ricinus communis*.

Pollutant	Precursors	Activating agent
Dyes	Castor bean seed	$ZnCl_2$
	Ricinus communis pericarp	H_2SO_4
	Ricinus communis epicarp	$ZnCl_2$
	Ricinus communis	Citric acid
	Ricinus communis pericarp	H_2SO_4
	Ricinus communis castor leaf powder (CLP)	Dried in a hot air oven for 5 h at 105ºC
Heavy metals	*Ricinus communis* pericarp	H_2SO_4
	Ricinus communis pericarp	H_2SO_4
	Ricinus communis leaves	Tannin gel
	Castor leaf *Ricinus communis* powder	Dried and made to 63-μm particles
	Ricinus communis seed shell	Polypyrrole

bridge cell, Mechanical Bench Shaker, UV, SEM, EDX, XRD, FTIR and HPLC.

Characterization of the Adsorbents

Physico-Chemical Characterization of the Adsorbents

Yield: The yield of the adsorbent in percentage was calculated by using the formula given below

$$Yield(Y) = (M/M_0) \times 100 \quad \ldots(1)$$

Where, M =Mass of the activated adsorbent, M_0=Mass of the adsorbent

Moisture content: About 5 g of the adsorbent was placed in a china dish, weighed, and heated for 6 h at 110±20C. After heating, the dish was cooled in a desiccator and weighed. Heating and cooling were performed every 30 min until there was less than a 5 mg difference between the two subsequent weights.

The moisture content is determined by loss in weight.

$$Moistue\ content\ (\%) = [(M - X)/M] \times 100 \quad \ldots(2)$$

Where M = Mass of the material taken for the analysis(g)

X = Mass of the materials taken after drying(g)

Adsorbent pH: About 0.2 g of the adsorbent was weighed and taken in a 50 mL beaker. Thirty mL of boiled and cooled water, whose pH was adjusted to 7.0, was added and heated to boiling. The first 10 mL of the filtrate was rejected. The remaining filtrate was cooled, and the pH was determined using a digital pH meter.

Zero point charge (pHzpc): About 0.2 g of adsorbent was added to a solution of Sodium nitrate of concentration 0.01M, whose pH was adjusted with 0.1M NaOH and 0.1M HNO_3, and the final pH was measured. The results were plotted with initial pH vs. ΔpH (ΔpH =final pH-initial pH). The pH equals zero yields the pHzpc of the adsorbent.

Iodine Number

Determination of iodine value of the adsorbents: Iodine solution was titrated against sodium thiosulphate (A) and with the sample solution (B).

$$\text{Iodine value} = C \times \text{Conversion factor} \quad \ldots(3)$$

Where C = (B-A)

Surface Acidity and Basicity of the Adsorbent

Acidity: To 0.2 g of adsorbent, 25 mL of 0.5 M NaOH solution is taken in a conical flask, and it is agitated for 10 h in a closed flask. Filter it, and the filtrate is titrated against with 0.05 M HCl. Acidity and Basicity are expressed in 'm'moL.g^{-1}

Basicity: To 0.2 g of adsorbent, 25mL of 0.5 M HCl solution is taken in a conical flask, agitated for 10 h in a closed flask. Filter it, and the filtrate is titrated against with 0.05 M NaOH. Acidity and basicity are expressed in 'm' moles.

Determination of surface group (Boehm titration)

The adsorbent was well mixed with 20 mL of a different base in volumetric standards. About 20 mL of the base was added to a 50 mL conical flask containing the adsorbent. The flask was then sealed and agitated in a shaker for 3 days. The solution was filtered, and 5 mL of each filtrate was titrated with 0.1 M HCl using a water–ethanol solution of methyl red as the indicator.

The number of basic sites was calculated from the amount of HCl reacted with the adsorbent material. Then, the various free acidic groups present were derived using the assumption that NaOH neutralizes carboxyl lactones, Na_2CO_3 neutralizes carboxyl and lactones, and $NaHCO_3$ neutralizes only carboxyl groups, respectively. The results obtained can be expressed in milliequivalents per gram (Balakrishnan & Thiagarajan 2021).

Photocatalytic Studies

Batch-mode adsorption studies for individual samples were carried out to determine the effect of different parameters, such as pH, adsorbent dosage, dye concentration, and contact time. Batch adsorption studies were conducted using 250 mL flasks. The adsorbent and adsorbate solution were separated using Whatman 48 filter paper. The concentration of the dye solution was measured using a UV–VIS spectrophotometer 119. All experiments were duplicated, and only the mean values were reported. The maximum deviation observed was less than ±5% (Alzaydien 2009).

A stock solution of methyl red (1000 ppm) was prepared and diluted to the required initial dye concentrations. Degradation studies were carried out in direct sunlight from March 2017 to May 2018. Batch-mode studies were performed using the required amount of adsorbent for each bottle, to which 50 mL of solution of the required concentration and pH was added, and kept in a shaker for 10 min to obtain a homogeneous mixture. After obtaining the homogeneous mixture, it was exposed to direct sunlight. At regular time intervals, the resulting mixture was filtered using Whatman 48 filter paper, and the final concentration of methyl red dye in the filtrate was estimated by a UV–VIS spectrophotometer 119 at a maximum absorbance of 524 nm.

From the initial and final concentration, the percentage of degradation was calculated by using the following equation.

$$\%\ \text{of Degradation efficiency} = \frac{A_0 - A_t}{A_0} \quad \ldots(4)$$

Where η was the degradation ratio, $A_0$was the initial absorbance of the dye, and A_t was the absorbance of the dye after degradation.

Effect of pH

To analyze the effect of pH on the degradation of dye solutions, 50 mL of a 20 ppm dye solution was adjusted from pH 2 to 9 using 0.1 M HCl and 0.1 M NaOH. The optimum dosage of adsorbent was added and kept in direct sunlight irradiation until equilibrium was reached. After exposure to sunlight, the solution was filtered, and the absorbance was measured using a UV–VIS spectrophotometer 119.

Effect of contact time

For the determination of the rate of dye degradation by the studied adsorbent, 50 mL of a 20 ppm dye solution was analyzed for residual dye concentration at different time intervals. The pH, adsorbent dosage, and adsorbate concentration were kept constant.

Effect of Adsorbate Concentration

The effect of adsorbate concentration was studied by varying the concentration of 50 mL of the dye solution, ranging from 20, 40, 60, 80, 100, 120, and 140 ppm. The equilibrium time and pH were kept constant depending on the dye solution under consideration.

Effect of Adsorbent Dosage

The effect of adsorbent dosage, i.e., the amount of adsorbent, on the degradation of dye was studied at various dosages ranging from 0.05 to 0.35 $g.mL^{-1}$ with 20 ppm dye solution. The equilibrium time and pH were kept constant depending on the dye solution under consideration.

UV-Spectral Studies

The degradation ability of Si-RCS was analyzed for the degradation of methyl red dye, which was confirmed by measuring the absorbance at regular time intervals using a UV–Visible spectrophotometer at a wavelength of 524 nm.

RESULTS AND DISCUSSION

Characterization of the Adsorbents

Physico-chemical Characterization of the Adsorbent

The physicochemical properties of Si-RCS were evaluated and shown in Table 3.

The total phenolic and carboxyl groups present in Si-RCS were 1.32 $m.eq.g^{-1}$. The yield and iodine number provide a measure of surface area or the capacity to adsorb small molecules and are associated with the capacity to adsorb substances with low molecular weight. The pollutant uptake was high when the yield and iodine number were high. The high adsorbent moisture content demands a higher adsorbent load and also reduces the effectiveness of Si-RCS.

Table 3: Physicochemical characteristics of the Si -RCS.

Parameters	Values
Yield %	79.51
Moisture content %	6.22
pH	5.50
Surface acidity [$mmoL.gm^{-1}$]	4.63
Surface basicity [$mmoL.gm^{-1}$]	3.01
pH_{zp}	4.00
Boehm titration [$m.eq.gm^{-1}$] Basic sites	2.47
Phenolic and Carboxylic groups	1.32
Carboxyl groups	0.89
Iodine Number $mg.g^{-1}$	2156.11

The negative charge density on the surface of Si-RCS increases when the zero point charge for the investigated adsorbent is lower than the pH of the solution. Surface acidity and surface basicity values confirmed the presence of negative charge density. The presence of basic and oxygenated acidic surface groups on the adsorbent was determined by Boehm titration.

Surface Characteristics

Scanning electron microscopic spectroscopy

The adsorbent's surface features and properties were examined using scanning electron microscopy (SEM). SEM studies revealed the surface texture and porosity of the adsorbent materials. SEM images of Si-RCS are shown in Fig. 1. The SEM images of Si-RCS show the porous and rough surface of the adsorbent. The rough surface and presence of pores facilitate the adsorption of dye molecules onto the adsorbent.

Energy-Dispersive X-ray Spectroscopy (EDS or EDX)

The fundamental principle is that each element has a unique atomic structure that permits a unique collection of peaks on its X-ray spectrum, which accounts for a major portion of the characteristics of energy-dispersive X-ray spectroscopy.

The confirmation of the existence of elements for the studied adsorbent (Si-RCS) was done by EDX analysis. The elements, percentage mass of elements presented in Si-RCS (before and after degradation), were represented in the Fig. 2, and the values were also summarized in Table 4.

Energy-dispersive X-ray spectra showed the presence of O, Na, C, Ca, K, and Si in the adsorbent. These are known as the principal elements of the adsorbent Si-RCS. The main elements were carbon and oxygen, with carbon content being higher than oxygen in Si-RCS. The presence

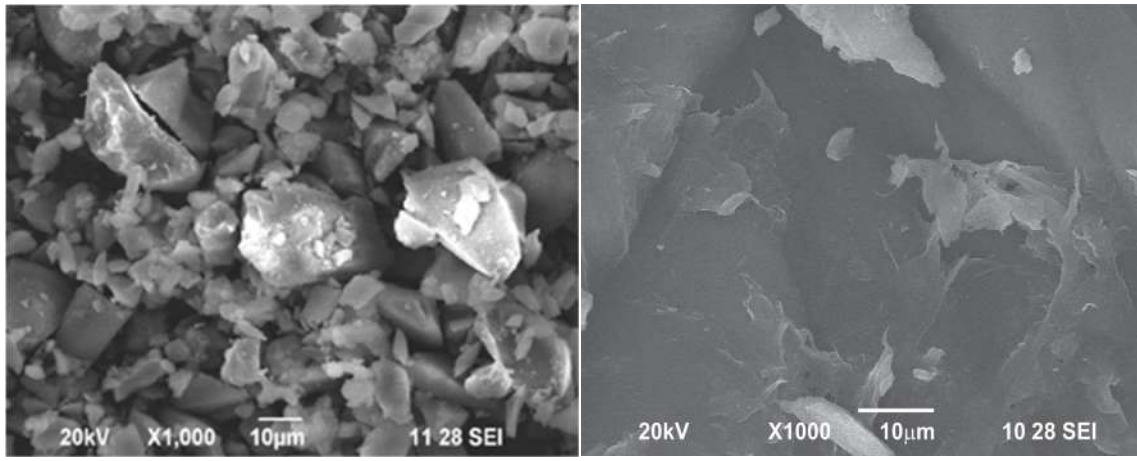

Fig. 1: SEM images of a) Si-RCS and b) Si- RCS after degradation of MR.

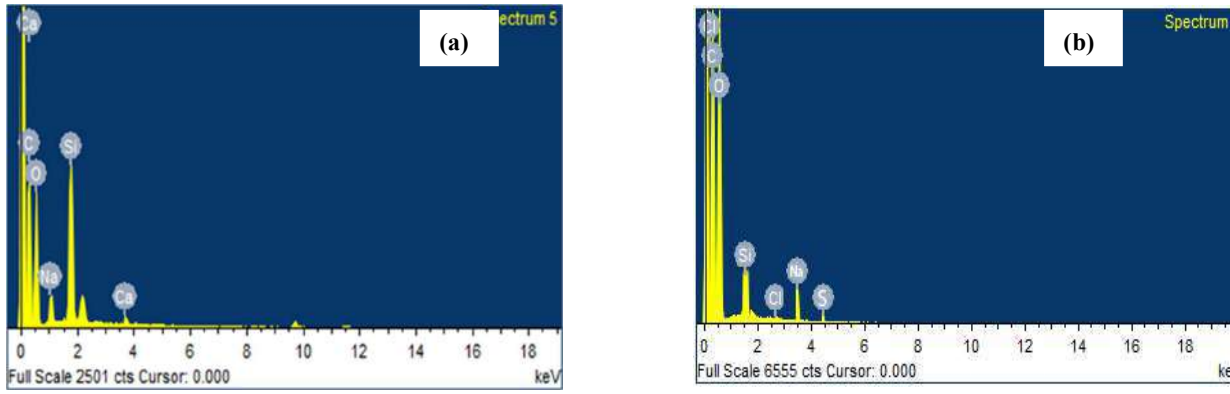

Fig. 2: EDX spectra of (a) Si-RCS and (b) Si-RCS after degradation of MR.

of Si in Si-RCS is attributed to the activation of the RC stem with silica gel.

X-ray Refractive Diffraction (XRD)

The structure of the adsorbent RCS is complex and includes both crystalline and amorphous regions. RCS displays an XRD pattern (Fig. 3a) that is comparable to that of other cellulosic materials. The smallest peak (at around 16° 2θ) denotes a less ordered polysaccharide structure, while the largest peak (at about 22° 2θ) shows the existence of highly organized cellulose.

Fig. 3b shows the XRD pattern of silica-activated RCS. Following silica activation, the crystalline character of RCS was enhanced, as evidenced by the increase in peak intensity in the Si-RCS XRD pattern. However, there are no significant changes in the XRD pattern of Si-RCS after methyl red (MR) degradation.

Table 4: Composition of Si-RCS before and after degradation of MR.

Compound	Si-RCS [Mass %]	Si-RCS-MR [Mass %]
C	25.03	32.75
O	24.62	28.02
Na	3.85	4.77
Si	42.38	10.91
Cl	-	23.47
Ca	4.12	-
S	-	0.08

Fourier Transform and Infrared Spectroscopy (FTIR)

The purpose of employing FTIR analysis was to determine the presence of functional groups with the help of characteristic peaks. Fig. 4a and 4b show the FTIR spectra of RCS and Si-RCS, respectively. The FTIR spectra of Si-RCS exhibit changes in peak intensity and modest peak-position shifts, confirming the silica activation. The FTIR spectra of Si-RCS contain a band at 3171 cm^{-1}, showing the presence of NH groups. A peak at 2878 cm^{-1} indicates

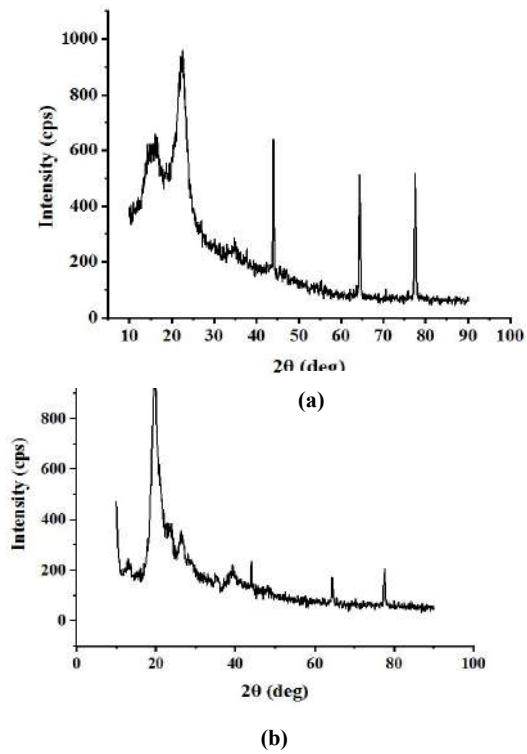

Fig. 3: XRD image of a) RCS and b) Si-RCS.

the presence of symmetric and asymmetric vibrations of methyl and methylene groups. The peak around 2338.82–2353.59 cm^{-1} indicates the presence of C–C stretching due to silica activation. The region at 1505 cm^{-1} is assigned to stretching vibrations of aromatic C=C in lignin, and the peak at 1054.06 cm^{-1} indicates the presence of lignin.

Photo-Catalytic Studies

Effect of Initial Dye Concentration

The effect of the initial dye concentration of methyl red (MR) on Si-RCS was studied using initial dye concentrations ranging from 20 ppm to 140 ppm. The experimental studies indicate a decrease in dye degradation with an increase in dye concentration, as shown in Fig. 5. The degradation efficiencies were found to decline with an increase in the initial concentration of the MR dye solution on Si-RCS. The degradation efficiency of MR onto Si-RCS decreased from 85.85% to 28.98%. The decline in degradation was due to the lack of active sites on the adsorbent surface at higher initial concentrations of MR; the ratio of the initial number of moles of dye molecules to the available adsorption surface area was high. This may also be responsible for an increase in the driving force of the concentration gradient with the rise in initial dye concentration.

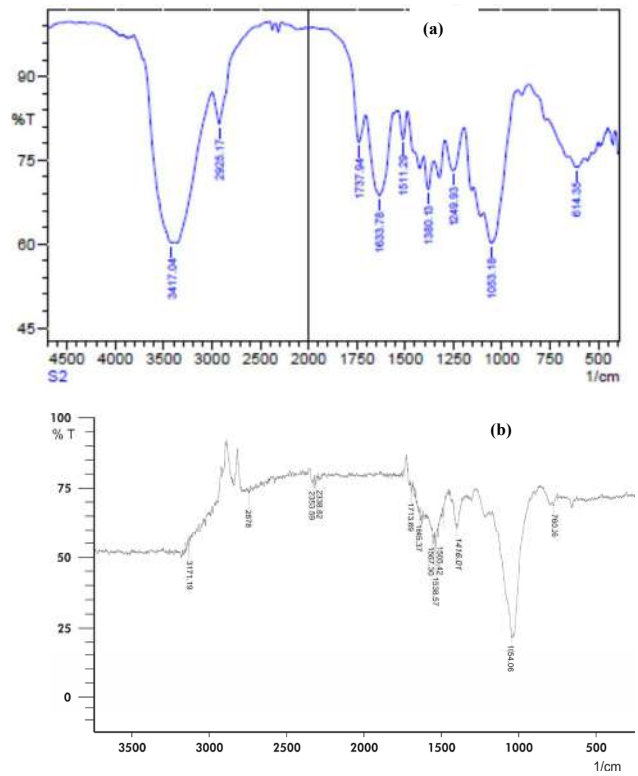

Fig. 4: FTIR image of a) RCS and b) Si-RCS.

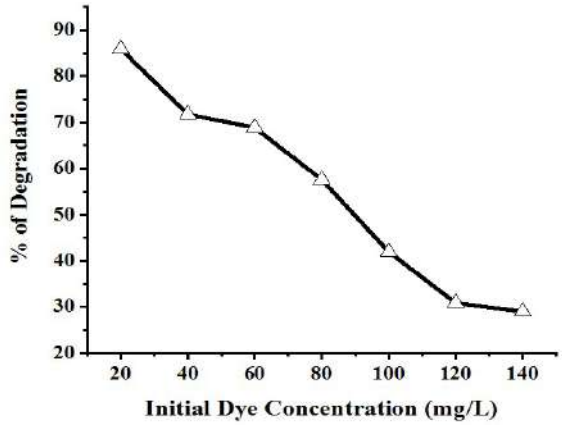

Fig. 5: Effect of Initial Dye concentration on adsorption of MR on Si-RCS.

Effect of Solution pH

The pH of the aqueous dye solution influenced the adsorption process. pH was an important factor that determined the ionizing capability of the adsorbing molecules at the given pH. The effect of the initial pH of the MR dye solution on the amount of adsorption was studied by varying the initial pH of the adsorbent Si-RCS under optimum conditions. The optimum dose of adsorbent was fixed at 0.2 g, 0.25 g, and 0.2 g, and the optimum initial dye concentration was 20 ppm for Si-RCS, respectively. The optimum contact time of Si-RCS was 100 min for the degradation of MR.

The maximum adsorption capacity of the adsorbents with respect to the initial pH of the adsorbate is shown in Fig. 6. The adsorption capacity decreased with increasing solution pH, and the optimum pH value for the adsorption of MR on Si-RCS was 4. The maximum degradation efficiencies of MR on Si-RCS were 80.58% at pH 6 and 85.85% at pH 3, respectively.

Effect of Contact Time

The percentage degradation was found to increase with

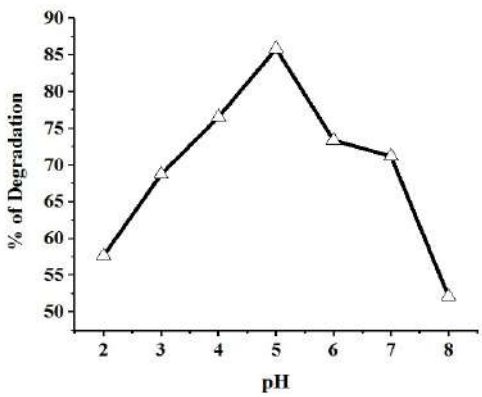

Fig. 6: Effect of pH on the adsorption of MR on Si-RCS.

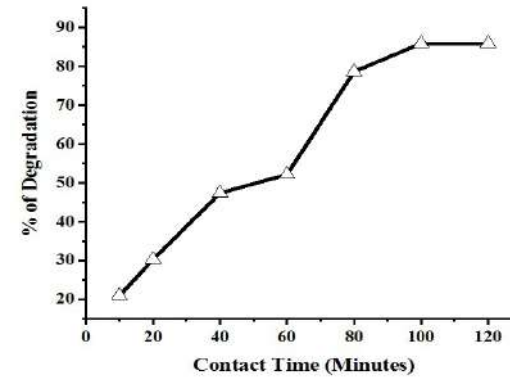

Fig. 7: Effect of Contact time on the adsorption of MR on Si-RCS.

increasing contact time, and then it became stable after attainment of equilibrium time, so the maximum degradation was achieved at 100 min for Si-RCS. This may be because initially the surface of adsorbent sites was highly vacant and the solute concentration was high, and after the equilibrium time, there was no change in the degradation of MR onto the studied adsorbents because the active sites were largely occupied at this stage. The maximum degradation efficiency of MR onto Si-RCS was 85.85% at 100 min. The effect of contact time on the degradation of MR onto Si-RCS is shown in Fig. 7.

Isotherm Modelling

An adsorption isotherm generally explains the dynamic behaviour of any adsorbate from solution to the solid adsorbent phase. The efficiency of an adsorbent for a particular adsorbate removal is represented by adsorption isotherms.

The equilibrium data analysis is important to develop the equation that accurately represents the adsorption process and can be used for design purposes (Balakrishnan & Thiagarajan 2015, 2016). Equilibrium studies determine the adsorption capacities of the adsorbent. To correlate the quantity of adsorption with the liquid-phase concentration, Langmuir, Freundlich, Temkin, and Dubinin–Radushkevich (D–R) adsorption isotherm equations were used. The adsorption isotherm models are evaluated from the values of the correlation regression coefficient (R^2).

Langmuir Isotherm

Irving Langmuir developed the Langmuir isotherm in 1916 to explain the nature of the surface coverage of an adsorbed gas on the surface at constant temperature. The monolayer adsorption on the homogeneous surface of the adsorbent by dye molecules is the main assumption of the Langmuir model (Jeyagowri & Yamuna 2016).

The mathematical expression of the Langmuir adsorption isotherm is:

$$\frac{C_e}{q_e} = \frac{1}{q_{max}} b + \frac{C_e}{q_{max}} \quad \ldots(5)$$

Hence, Ce is the concentration of MR dye solution ($mg.L^{-1}$), qe is the concentration of MR in adsorbent ($mg.g^{-1}$), qmax is the maximum adsorption capacity ($mg.g^{-1}$), and b is the Langmuir equilibrium constant. The linear plot of C_e/q_e vs C_e gives a straight line with a slope of $1/q_{max,}$ and the intercept Ce/qmax is shown in Fig. 8.

The isotherm parameters obtained from the Langmuir model for the removal of MR onto Si-RCS are shown in Table 3. The calculated and experimental values of this model are very close to each other, which indicates a good fit of the parameters to the Langmuir adsorption isotherm model. The correlation regression coefficient is the best evidence that the adsorption of MR onto Si-RCS follows the Langmuir isotherm. The R^2 values for the adsorption of MR onto Si-RCS were 0.991.

Freundlich Isotherm

The Freundlich adsorption isotherm model is a well-known equation used to explain the adsorption process. A Freundlich empirical equation can describe multilayer adsorption and the heterogeneous surface of the adsorbent for non-ideal adsorption. The linear form of the Freundlich isotherm is given as follows:

$$\text{Log } q_e = 1/n \log(C_e) + \log K_f \quad \ldots(6)$$

Where K is the Freundlich constant ($mg.g^{-1}$), n is the Freundlich exponent, q_e is the amount of adsorbed per unit mass of the adsorbent, and C_e is the adsorbate concentration at equilibrium in solution ($mg.L^{-1}$). The Freundlich constant K_f and 1/n were calculated from the slope and intercept from the plot of $\log C_e$ vs $\log q_e$ for the adsorption of MR onto Si-RCS. K_f was approximately an indicator of the adsorption capacity

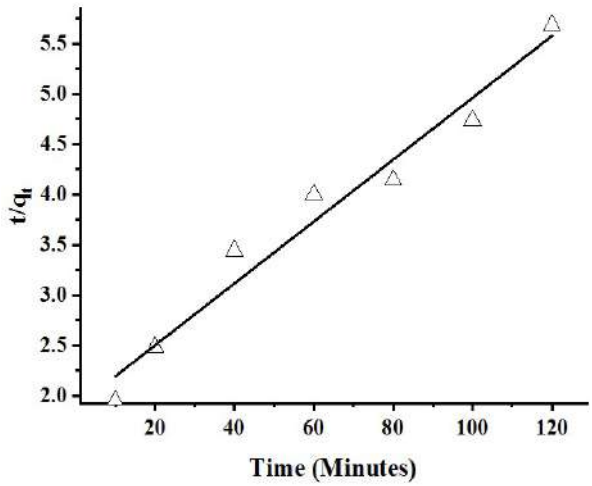

Fig. 8: Langmuir isotherm for adsorption of MR on Si-RCS.

Table 5: Isotherm parameters for the adsorption of MR onto Si-RCS.

Dye		Methyl Red
Langmuir Isotherm	Q_m [$mg.g^{-1}$]	9.58
	b [$L.mg^{-1}$]	3.86
	R^2	0.991
Freundlich Isotherm	K_F[$L.g^{-1}$]	2.912
	N	3.13
	R^2	0.924
Temkin Isotherm	α	0.637
	β	1.94
	R^2	0.905
D-R Isotherm	E [$kJ.moL^{-1}$]	1.000
	R^2	0.9195

related to the bond energy, and 1/n was the intensity of dye adsorption onto the adsorbent surface (Arunachalam et al. 2012). The extent of the exponent, 1/n value, suggested the favourability of the adsorption. The value of 1/n <1 represents a favourable adsorption condition, while 1/n >1 represents cooperative adsorption. The calculated data obtained from the Freundlich adsorption isotherm of the MR onto Si-RCS were summarized in Table 5. The Langmuir and the Freundlich isotherm models were correlated by the regression coefficients (R^2). The correlation coefficient values of MR onto Si-RCS for the Langmuir and the Freundlich isotherms are 0.991 and 0.924. From the results Langmuir adsorption isotherm was well fitted when compared to the Freundlich isotherm model, and confirms the monolayer adsorption of the dye onto the adsorbent (Pradeeba & Sampath 2017).

Temkin adsorption isotherm

Temkin isotherm describes the effect of some indirect adsorbent/adsorbate interactions on the adsorption isotherm indicates that the heat of adsorption decreases linearly with coverage. The simplified form of the Temkin isotherm is as follows:

$$q_e = \beta \ln\alpha + \beta \ln C_e \quad \ldots(7)$$

Where β = (RT)/b, where b is the constant related to heat of adsorption ($L.mg^{-1}$), α is the binding equilibrium constant, T is the absolute temperature (K), R is the universal gas constant (8.314 J), q_e is the amount of adsorbed per unit mass of the adsorbent and C_e is the concentration of the adsorbate in solution at equilibrium condition. The values of α, β and R^2 obtained from the plot of lnCe vs q_e of adsorption of MR onto Si-RCS were presented in Table 5.

Dubinin-Radushkevich Isotherm (D-R isotherm)

The adsorption capacity of the adsorbent is determined by using the Dubinin-Radushkevich Isotherm model. The mathematical expression of the D-R model is given as follows.

$$lnq_e = \boldsymbol{ln\, q_{max} - \beta\varepsilon^2} \quad \ldots(8)$$

Where q_{max} is the theoretical saturation capacity and ε is the Polanyi potential. The constants β and q_{max} can be calculated from the graph plotted between ε^2 vs In q_e for the adsorption of MR onto Si-RCS and are presented in Table 5. The constant β provides the mean free energy *E* of adsorption per molecule of dye when it is transferred to the solid surface of the adsorbent from infinity in the solution. The value of *E* provides information about the nature of adsorption-whether it is physisorption or chemisorption. The adsorption process is physisorption when *E* lies between 1 and 8 kJ.moL^{-1}, while it is chemisorption when is higher than 8 kJ.moL^{-1}. From Table 3, it was observed that the value of *E* is 1.000 kJ.moL^{-1}, confirming that weak physical forces of interaction are the driving force for the adsorption of dye onto Si-RCS (Pradeeba & Sampath 2022a).

From Table 3, it is revealed that the correlation coefficient R^2 of the Freundlich, Temkin, and D–R isotherms were lower than that of the Langmuir adsorption isotherm for the dye, indicating monolayer adsorption of the dye onto the surface of the adsorbent.

Kinetic Modelling

Pseudo-second Order kinetics

The adsorption kinetics of MR onto Si-RCS was explained by using pseudo-second order kinetics. The pseudo-second order equation is:

$$\frac{\mathrm{t}}{q_t} = \frac{1}{K_2} - \frac{t}{q_e} \quad \ldots(9)$$

Where K_2 is the equilibrium rate constant for the pseudo-second-order adsorption (g.mg^{-1}.min^{-1}), it can be calculated from the slope of the graph shown in Fig. 9, and the experimental data are summarized in Table 6. The initial adsorption rate and the rate constant k for the pseudo-second-order can be calculated from $h = kq_e^2$ (mg.g^{-1}.min^{-1}) (Vidhya et al. 2023). From the intercept and slope, the values of q_e, k, and h can be determined. The experimental data and the plot were well fitted to the pseudo-second-order kinetic model. The correlation regression coefficient R^2 and the maximum adsorption capacity q_e for the adsorption of MR were 0.9655 and 19.68 mg.g^{-1}, respectively. The calculated q_e values are in good agreement with the experimental data. From the experimental results, it is evident that the adsorption system follows the pseudo-second-order kinetic model and provides a good correlation for the adsorption of MR onto Si-RCS (Mall et al. 2005).

Elovich Equation

The Elovich equation is another rate equation that is typically used to illustrate the adsorption capacity. The equation is exhibited as:

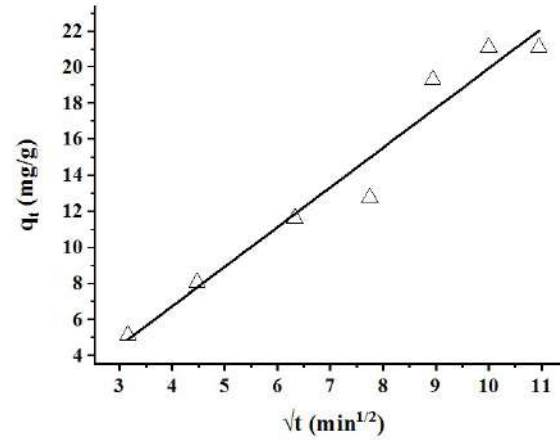

Fig. 9: Pseudo-second order model for the adsorption of MR on Si-RCS.

$$\frac{dq_t}{\mathrm{dt}} = B_E \exp\,(-A_E q_t) \quad \ldots(10)$$

Where A_E is the elution constant (g.mg^{-1}) and B_E is the initial adsorption rate [mg(g.min^{-1})]. A graph is plotted between lnt vs q, and the experimental data were summarized in Table 6. The elution constant A_E and initial adsorption rate B_E for the adsorption of MR were 0.126 and 2.41 for Si-RCS, respectively. The elution constant A_E is lower than the initial adsorption rate, which contributes to the higher adsorption capacity of MR onto Si-RCS.

Intra-particle Diffusion Model

The intra-particle diffusion model is an important tool to find out the mechanism of the adsorption process. The intra-particle diffusion model is expressed as:

$$q_{t=} K_d t^{1/2} + C \quad \ldots(11)$$

Where C is the intercept, K_d intra particle diffusion rate constant (mg.g^{-1}.min$^{-1/2}$) and q_t is the amount of MR dye adsorbed at time, t(mg,g^{-1}). A plot of qt vs √t gives a linearized form of intra particle diffusion model for the adsorption of MR by Si-RCS, which were studied, and the values were summarized in Table 4. From the results, the intra-particle diffusion rate, K_d, intercept C, and regression correlation coefficient R^2 of MR onto Si-RCS were 2.22 to 2.05 and 0.9593, respectively. The value C (intercept) gives information about the thickness of the boundary layer; if the resistance of the external mass transfer increases, the thickness of the boundary layer also increases (Pradeeba & Sampath 2022b). In the case of intra particle diffusion model, the qt vs √t plots must pass through the origin if the intraparticle diffusion is the rate-limiting step. The plot of qt vs √t for the adsorption of MR by Si-RCS does not pass through the origin, which indicates that the intraparticle diffusion model is not rate limiting step, as surface

Table 6: Kinetic parameters for the adsorption of MR onto Si-RCS.

Dye	Kinetic models	Parameters	Si-RCS
Methyl Red	Pseudo- first order equation	$k_{1[}min^{-1}]$	0.076
		$q_e[mg.g^{-1}]$	14.12
		R^2	0.8233
	Pseudo-Second order equation	$k_2[Lmole^{-1}\ sec^{-1}]$	0.0013
		h	0.503
		q_e,cal$[mg.g^{-1}]$	19.68
		R^2	0.9655
	Elovich Model	A_E $[mg.g^{-1}]$	0.126
		B_E $[mg.g^{-1}]$	2.41
		R^2	0.911
	Intra-particle diffusion	k_d $[mg.g^{-1}]$	2.22
		R^2	0.9593
		C	-2.05

adsorption and intraparticle diffusion were concurrently operating during the dye and adsorbent interaction. The comparison of correlation regression coefficient values for Si-RCS for the adsorption of MR was summarized in Table 6.

The pseudo-second order kinetic model gives a good correlation for all of the adsorption processes of MR onto Si-RCS.

Mechanism of Dye Degradation

The prime reason for using activated *Ricinus communis* Stem as a photocatalyst for the degradation of MR dyes is that when it is exposed to direct solar light irradiation, it gives electron-hole pairs. The dye degradation process is induced by the formed electron hole pairs. Even though it is photocatalytic degradation, initially, adsorption of the dye on the surface of the adsorbent occurs, followed by the photo-degradation process. In the early stages, when the dye concentration is low, there are more vacant sites available on the adsorbent's surface for adsorbing dye molecules; however, as the dye concentration increases, there are fewer vacant sites available on the adsorbent's surface for adsorbing dye molecules. Because the vacant sites are filled by the dye molecules, the rate of dye degradation decreases (Pradeeba et al. 2022). Mechanism of photo degradation occurs in two steps, i.e., Dye adsorption by adsorbent, followed by degradation under direct sunlight takes place by the following equation

$$\text{Adsorbent} + h\nu \rightarrow \text{Adsorbent}\ (e^- + h^+) \quad \ldots(12)$$

$$O_2 + e^- \rightarrow O_2^{\cdot -} \quad \ldots(13)$$

$$O_2^{\cdot -} + e^- + 2H^+ \rightarrow 2OH^{\cdot} \quad \ldots(14)$$

Oxygen plays a vital role in the degradation of dyes.

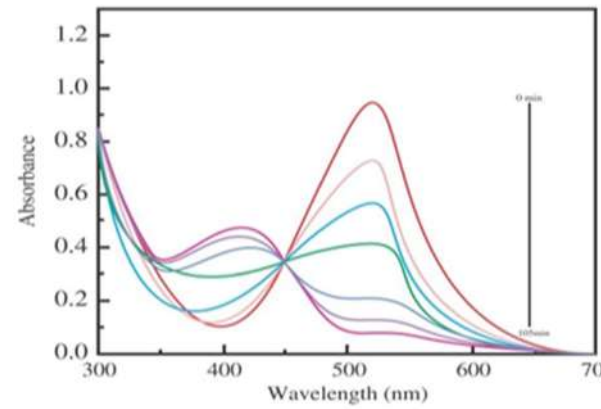

Fig. 10: Photocatalytic degradation of MR on Si-RCS.

The sunlight, oxygen, and hydroxyl ion shows considerable effects on the degradation. The degradation capacity of the dye is reduced if any of the above parameters is reduced. The formed hydroxyl radical species react with any species to form hydrogen peroxide, which is a considerably active species in the photo degradation process. Fig.10 shows the photocatalytic degradation of the dye MR by the adsorbent SiRCS.

$$2OH^{\cdot} \rightarrow H_2O_2 \quad \ldots(15)$$

The formed hydrogen peroxide cleaved to form $OH^{\cdot}$

$$H_2O_2 + h\nu \rightarrow 2OH^{\cdot} \quad \ldots(16)$$

Degradation of dye by $OH^{\cdot}$ radical

$$OH^{\cdot}{}_{ads} + \text{dye} \rightarrow \text{oxidation of the dye} \quad \ldots(17)$$

CONCLUSIONS

In the present research work, silica gel-activated adsorbent was prepared from *Ricinus communis* stem (Si-RCS). The photocatalytic removal of methyl red dye using (Si-RCS) in the presence of natural sunlight was examined. The effectiveness of methyl red dye degradation depends on initial dye concentration, contact time, and pH. In the dye concentration variation, the maximum degradation efficiency was 86% in the presence of natural sunlight, and the optimum dye concentration was 20 ppm at 0.25 g photocatalyst dosage. The adsorbent Si-RCS was utilized as a catalyst in the presence of natural sunlight to investigate the kinetic isotherm, pseudo-first-order, and pseudo-second-order for dye variation. The best fit model was found to be pseudo-second order based on the R^2 value. Adsorption isotherms like the Langmuir and the Freundlich. Temkin and DR isotherm has been analyzed for the dye variation parameter in the removal of Methyl Red. Among the isotherm models studied, it was found that Langmuir is the best fit model.

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Divergent Energy Paths - A Trend Analysis of Global Energy Growth Across Economic Classifications

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ABSTRACT

This study examines global energy growth trends from 1990 to 2022, utilizing secondary data from sources like the IEA, the World Bank, and the United Nations. The analysis focuses on indicators such as total energy consumption, fossil fuel and renewable energy consumption, energy intensity, and carbon emissions. The study employs various econometric techniques, including stationarity testing using the Augmented Dickey-Fuller (ADF) test to examine the time series data for unit roots. A semi-logarithmic trend model is used to estimate the long-run trends of energy indicators, and a Kinked Exponential Growth Model is applied to capture variations in growth across different sub-periods, accounting for potential structural breaks. The data was tested for stationarity using the Augmented Dickey-Fuller (ADF) test, showing a non-stationary process at the level but stationary at the first difference (I(1) process). The semi-logarithmic trend model revealed significant differences in growth rates across economic classifications. For developed countries, Japan (5.05%) and the United States (5.5%) had high growth rates, while New Zealand and the UK showed negative growth. In developing countries, China (6.7%), India (5.8%), and South Africa (4.7%) showed strong growth, whereas Nigeria (2.3%) and Pakistan (3.5%) had lower rates. The discontinuous growth analysis revealed steady positive growth for most countries, while Denmark and Finland experienced minimal or negative growth in certain periods.

INTRODUCTION

Energy plays a central role in shaping the trajectory of human civilization (Smil 2017). From the earliest days of harnessing fire and water to today's complex networks of fossil fuels, nuclear power, and renewable energy systems, societies have depended on energy not only for survival but also for development, innovation, and economic growth. In the contemporary world, energy serves as the lifeblood of economies, powering industries, enabling transportation, lighting up cities, and supporting digital infrastructure. However, the manner and pace at which different countries consume and produce energy vary significantly, depending largely on their level of economic development (Vlachogianni & Valavanidis 2013). Globally, energy demand continues to rise, driven by population growth, urbanization, and industrial expansion (Avtar et al. 2019). However, the path toward meeting this demand is neither uniform nor equitable. While developed countries have progressed toward energy diversification and decarbonization, developing and underdeveloped nations continue to grapple with energy poverty, reliance on traditional fuels, and limited access to advanced energy technologies. These asymmetries have created divergent energy trajectories, where each group of countries, classified by their economic standing, exhibits distinctive patterns in energy growth, transition, and sustainability (Khan et al. 2021).

International energy markets, trade dependencies, geopolitical alliances, and transnational investments significantly influence national energy strategies, particularly in developing and underdeveloped countries (UDCs). For instance,

while developed nations often lead in innovation and technology exports, many resource-rich developing countries continue to function primarily as raw material suppliers in the global energy value chain. This asymmetry creates structural dependencies that limit the autonomy of low-income economies in crafting independent and sustainable energy strategies to achieve energy security. Furthermore, access to global finance and climate funds is often mediated through complex eligibility criteria and institutional prerequisites that many underdeveloped countries struggle to meet (Chaudhury 2020). These external constraints, when combined with internal challenges such as poor governance, weak infrastructure, and limited human capital, further exacerbate disparities in energy development. Therefore, any comprehensive analysis of global energy growth must account for both domestic economic conditions and the broader international forces that shape energy policies and trajectories. By situating energy trends within this multidimensional framework, the present study endeavors to provide a more nuanced and realistic understanding of the global energy landscape-one that moves beyond aggregate figures and headline indicators to reveal the deeper patterns of divergence and convergence that shape our collective energy future.

In particular, the past three decades have witnessed remarkable changes in the energy landscape: the rapid expansion of renewables in the West, continued dominance of coal in some Asian economies, and persistent energy access issues in parts of Africa and South Asia. These trends reflect not only technological advancements and policy shifts but also deep-rooted economic, institutional, and geopolitical differences (Chari 2025). Therefore, a comparative analysis of energy growth trends across economic classifications, developed, developing, and underdeveloped, can provide critical insights into global energy inequalities, transition potentials, and future directions. This study is situated within this complex global context. By focusing on trend analysis across economic classifications, it seeks to reveal the structural factors influencing energy growth, the extent of divergence in energy trajectories, and the policy implications for fostering inclusive and sustainable energy futures in the region.

Global Energy Inequality and Economic Classification

The global energy landscape is increasingly marked by inequalities that mirror broader socioeconomic disparities between developed, developing, and underdeveloped nations (Darwich 2025). While advanced economies have transitioned to more diversified and low-carbon energy systems, many low-income nations continue to rely on biomass and fossil fuels to meet their basic needs. These disparities are shaped not only by resource endowments but also by differences in economic capacity, technological development and institutional readiness. The economic classification of nations, often defined by indicators such as Gross National Income (GNI), Human Development Index (HDI), and industrial output, correlates strongly with energy consumption and production levels (Yumashev et al. 2020). Developed nations are often pioneers in renewable energy deployment and energy efficiency, supported by strong infrastructure and investment. In contrast, underdeveloped countries face barriers such as a lack of funding, weak regulatory frameworks, and low technical capacities. This imbalance poses significant challenges to achieving global energy and climate targets, such as SDG 7 (Affordable and Clean Energy) and SDG 13 (Climate Action).

Structural Drivers of Energy Divergence

To deepen the analysis, this study introduces a distinct subsection on the structural drivers of divergence. Although energy patterns vary by economic classification, they are also shaped by broader systemic factors within the global political economy (Bridge & Gailing 2021). Developed nations often benefit from advantageous positions in global trade, finance, and technology diffusion. They have greater access to climate finance, green technology, and international influence in shaping energy governance. Conversely, resource-rich but institutionally weak nations are often relegated to raw material exporters, lacking control over the value-added segments of the energy supply chain. Geopolitical alliances, trade dependencies, and conditionalities attached to international funding further constrain the autonomy of developing and underdeveloped countries. By situating these external constraints as structural rather than incidental, this study highlights the importance of addressing global systemic inequities, not just domestic reforms-in pursuing inclusive energy futures.

The Need for Comparative Trend Analysis

Given the complex and unequal evolution of global energy systems, a comparative trend analysis across different economic classifications is both timely and essential. Existing energy literature tends to be fragmented, often focusing either on national energy transitions or broad global overviews, thereby neglecting the nuanced trajectories that emerge across the economic strata. By undertaking a comparative trend analysis, this study seeks to bridge this critical gap by systematically examining how energy consumption patterns, production capacities, and transition strategies have evolved in developed, developing, and underdeveloped nations. This analytical approach allows for the identification of converging and diverging trends, revealing the factors that

facilitate or hinder energy progress in distinct economic contexts. More importantly, such a comparative framework can expose hidden asymmetries, such as technology lock-in, financing barriers, or policy inertia, that disproportionately affect lower-income countries. Understanding these divergences is imperative not only for academic inquiry but also for informing international development cooperation, climate negotiations and the equitable distribution of global energy investments. Therefore, comparative trend analysis is not just a methodological choice but a necessary lens through which global energy justice and inclusivity can be meaningfully examined.

Rationale of the Study

The rationale behind this research lies in the urgent need to examine and understand the asymmetric nature of global energy developments. Although energy is a global public good, its production, distribution, and consumption are characterized by stark disparities (Karlsson et al. 2012). These disparities are often rooted in the economic classifications of countries, which determine their access to resources, technological capabilities, policy frameworks, and institutional strength. As the world navigates energy transitions and climate responsibilities, it is imperative to understand how the economic context influences a country's energy trajectory. Developed countries have historically consumed a disproportionate share of global energy and have contributed significantly to greenhouse gas emissions (Khan et al. 2014). However, many of these countries have now embarked on decarbonization paths and invested heavily in clean technologies and energy efficiency. These transitions are supported by strong economies, robust infrastructure, and mature institutions (Hamid et al. 2022). In contrast, developing countries are witnessing a surge in energy demand spurred by urbanization, industrialization, and rising incomes (Jones, 1991). Although some have made strides in renewable energy adoption, their transitions are often constrained by fiscal limitations, policy uncertainties, and competing development priorities. Meanwhile, underdeveloped countries remain energy-deprived. For them, the challenge is not transitioning from fossil fuels to renewables but achieving basic energy access in the process. Many rely heavily on traditional biomass for cooking and heating, with limited access to electricity and modern fuels (Karekezi 2006). In this context, energy development is closely tied to poverty alleviation, public health, and human development. These divergent realities necessitate a differentiated analysis that does not treat global energy growth as a homogeneous process but recognizes the divergent paths taken by countries based on their economic classifications. Moreover, much of the current academic discourse tends to focus on either the technological aspects of energy transition or country-specific case studies. Few studies have adopted a global comparative perspective that combines economic classifications with trend analyses of energy indicators over time (Mihic et al. 2024, Brown et al. 2014, Xu et al. 2019).

By analyzing long-term energy trends across country groups, this study contributes to filling this gap. It provides empirical evidence of the magnitude and nature of divergence in energy growth and offers insights into how economic factors influence the pace, direction, and sustainability of energy transition. This study also aims to inform international climate negotiations, especially regarding the principle of Common but Differentiated Responsibilities (CBDR), by highlighting the historical and structural inequalities in global energy development.

Statements of the Problem

Despite the increased focus on global energy transitions, there remains a significant gap in understanding differentiated trends in energy growth across countries with varying levels of economic development. Much of the global energy policy discourse tends to assume a one-size-fits-all model of transition-emphasizing decarbonization, renewables, and efficiency, without adequately accounting for the socio-economic and institutional realities of underdeveloped and developing nations (Pastukhova & Westphal 2020, Gitelman & Kozhevnikov 2022). This leads to two interrelated problems. First, there is a lack of systematic comparative analyses of energy growth trends across economic classifications. Existing literature often focuses on single-country case studies or regional analyses, with limited attention to how different economic groups perform over time regarding energy production, consumption, intensity, and access. Consequently, the global energy narrative remains skewed, potentially overlooking the challenges faced by low-income countries and misrepresenting the progress of others. Second, the drivers of energy divergence, such as income levels, industrial structure, population dynamics, technological diffusion, and governance quality, are rarely studied in conjunction with energy data in a cross-classified framework. This undermines the ability to design differentiated energy strategies that are economically feasible, socially just, and environmentally sustainable. Furthermore, many underdeveloped countries are caught in a vicious cycle of energy poverty and underdevelopment (Nguyen & Su 2021). Their inability to invest in energy infrastructure leads to low energy access, which hampers economic growth and welfare. Without adequate analysis, such countries risk being left behind in the global push for energy transition, further widening the development gaps.

However, middle-income developing countries face the dual burden of meeting rising energy demand while attempting to reduce their carbon footprint (Alola & Joshua 2020). These nations require innovative solutions that balance growth with sustainability-solutions that cannot be developed without a clear understanding of their current energy trajectories and structural limitations.

In summary, this study addresses the inadequate recognition and analysis of divergent energy growth patterns across developed, developing, and underdeveloped countries. Without such analyses, it is difficult to devise equitable energy policies, allocate international funding appropriately, or negotiate fair climate agreements. This study responds to this problem by applying a trend analysis framework to map and compare energy growth across different economic classifications. It not only tracks historical patterns but also investigates the underlying factors driving divergence, thereby contributing to a more inclusive and data-driven understanding of global energy development in the future.

Scope and Significance of the Study

This study aimed to analyze global energy growth trends across different economic classifications (developed, developing, and underdeveloped countries) from 1990 to 2022. The present study is expansive in scope yet precise in its analytical focus, aiming to capture the multidimensional evolution of global energy systems across economic classifications over a defined timeframe. By categorizing countries according to standardized economic criteria and examining longitudinal data on key energy indicators, such as total primary energy supply, per capita energy use, fossil fuel dependence, and renewable energy adoption, this study offers a structured and comparative understanding of energy growth trajectories. This approach enables the identification of macro trends, transitional inflection points, and persistent structural barriers unique to different economic groups. The significance of this study lies in its potential to contribute to multiple scholarly and policy-oriented discourses: it adds empirical rigor to debates on energy transition, provides insights for global development agendas such as the Paris Agreement and SDGs, and offers a critical reference for policymakers, multilateral agencies, and energy planners seeking to design equitable and efficient energy strategies. Furthermore, by unpacking the divergent energy paths that countries follow, this study foregrounds the urgent need for differentiated policy frameworks that are sensitive to national capacities and developmental priorities. As such, this research is not only a diagnostic exercise but also a call to action for a more just and balanced global energy future.

Overview of Reviewed Literature and Research Gap

The literature on global energy systems has grown extensively in recent decades, reflecting increasing academic and policy interest in energy access, sustainability and transition. A consistent theme emerging from the literature is the significant variation in energy development across economic classifications, with high-income countries exhibiting advanced, diversified energy systems and low-income countries continuing to face constraints related to access, affordability, and infrastructure (IEA 2022). Researchers such as Sovacool (2016) and Goldthau & Sovacool (2012) have emphasized how political economy factors, technological lock-in, and institutional weaknesses create divergent energy transition paths, especially in the Global South. These disparities are compounded by the uneven flows of climate finance and clean energy investments, which disproportionately benefit countries with stronger institutional capacities and credit ratings (UNCTAD 2021).

In terms of energy consumption trends, the BP Statistical Review of World Energy (2022) and World Bank (2022) datasets have shown that per capita energy consumption in high-income OECD countries has either stabilized or declined slightly owing to efficiency improvements and decoupling from GDP, whereas developing countries have shown steady increases in both total and per capita energy demand, largely fueled by economic growth, industrialization, and population expansion. Studies such as those by Apergis and Payne (2010) and Sadorsky (2009) also support the energy-growth nexus across income groups, noting that while energy consumption is a driver of economic output in low- and middle-income countries, high-income countries are more successful in reducing energy intensity owing to structural shifts toward service-based economies.

The literature highlights the differences in renewable energy adoption. According to REN21 (2023), developed countries are at the forefront of adopting wind, solar, and other renewables, primarily because of favorable policy regimes, innovation capacity, and access to finance. Conversely, energy transition in developing and underdeveloped countries is slower and more fragmented, constrained by inadequate grid infrastructure, policy uncertainty, and competing development priorities (IRENA 2023). Nonetheless, regional studies, such as those by Alova et al. (2021) and Bhattacharyya (2013), demonstrate pockets of progress, particularly in South Asia and Sub-Saharan Africa, where decentralized renewable solutions, such as solar mini-grids, are helping bridge the energy access gap.

In summary, while the existing literature offers rich insights into energy consumption patterns, transition challenges, and economic linkages, there is a lack of

comprehensive, comparative trend analyses that directly address the divergence of global energy paths across economic classifications. This study seeks to fill this gap by employing a longitudinal, comparative approach to trace and interpret energy growth trajectories across diverse economic contexts, thereby contributing a fresh empirical and policy-relevant perspective to the global energy discourse.

MATERIALS AND METHODS

Data Source and Period of Study

This study is based entirely on secondary data collected from globally recognized databases, including the International Energy Agency (IEA), the World Bank's World Development Indicators (WDI), and the United Nations Statistical Division. The analysis spans from 1990 to 2022, covering a period of 33 years. The key indicators analyzed include total energy consumption, fossil fuel energy consumption, renewable energy consumption, energy intensity, and carbon emissions from energy use.

To capture structural changes in the global energy scenario, the study period is divided into three sub-periods:

- Period I: 1990–2000
- Period II: 2001–2010
- Period III: 2011–2022

Data Processing and Transformation

Logarithmic transformation was applied to all major variables to address heteroscedasticity and to linearize exponential growth patterns. This transformation also facilitates easier interpretation of coefficients in growth models (Benoit 2011).

Stationarity Testing

Before performing time series analysis, the stationarity of each variable was tested using the Augmented Dickey-Fuller (ADF) test. This test helps identify the presence of a unit root, which indicates non-stationarity (Cavaliere & Taylor 2007).

The ADF test equation is:

$$\Delta y_t = \alpha o + \alpha 1\, y_{t-j} + \sum_{j=1}^{k} + \zeta \Delta y_{t-j} + e_t \quad \ldots(1)$$

Where:

- Δy_t is the first difference of the series
- α is the intercept
- β is the coefficient on a time trend
- γ is the parameter to be tested
- k is the lag order of the autoregressive process
- e_t is the white noise error term

The null hypothesis H0: $\gamma=0$ suggests that the series has a unit root (non-stationary), while the alternative hypothesis H1: $\gamma<0$ suggests stationarity.

Trend Analysis Using Semi-Log Model

To measure the long-run trend of energy indicators, a semi-logarithmic trend model was estimated. This model enables interpretation of the slope coefficient as the average annual growth rate (Gujarati & Porter 2009).

The model used is:

$$\ln(yt) = \alpha + bt + \mu t \quad \ldots(2)$$

Where:

- $\ln(yt)$ is the natural logarithm of the energy variable at time t
- α is the intercept
- β is the slope or average growth rate
- μt is the error term

The growth performance of a country's energy sector typically does not remain uniform across different time periods. This implies that the growth rate of time series data tends to vary over time. To reflect this, the coefficient 'b' can be represented as a time-dependent linear function:

$$b = \beta + \gamma t \quad \ldots(3)$$

Substituting the latter into the former equation, the log-linear trend equation becomes a log-quadratic model, which is expressed as:

$$In(y) = a + \beta t + \gamma t\, 2 + \mu t \quad \ldots(4)$$

Where $'\ln(y)'$ is the natural logarithm of the dependent variable, and a, β, and γ are the parameters to be estimated. Here, β represents the average annual growth trend, while γ captures any change in the growth rate over time. If the coefficient γ is statistically significant, it suggests that the growth rate is not constant. A positive γ indicates an accelerating growth trend, whereas a negative γ suggests deceleration.

If the sign of the parameter is negative, then the growth rate is decelerating. When the log-quadratic trend equation is used, the average growth rate can be computed by

$$Growth\ Rate = \sum \lfloor (\beta + 2\gamma t)/\, n \rfloor * 100 \quad \ldots(5)$$

An insignificant value of $'y'$ indicates that the growth rate is constant over the period, wherein the Log-Linear model $'\ln(yt) = a + bt + \mu t\,'$ has to be fitted for computing the constant growth rate. Then the growth rate is given by: $[Anti\ \log(b) - 1] * 100$ (Gujarati & Porter 2009).

Sub-Period Growth Analysis Using the Kinked Exponential Model

To capture the variation in growth across different sub-periods, the Kinked Exponential Growth Model proposed by Boyce (1986) was applied. The model accommodates potential structural breaks while maintaining continuity at the breakpoints.

The general model is specified as:

$$\text{Log } Y = a_1d_1 + a_2d_2 + a_3d_3 + (b_1d_1 + b_2d_2 + b_3d_3)\, t + \mu t \quad \ldots(6)$$

Where, $d_1 = 1$, for the first period

0, otherwise

$d_2 = 1$, for the second period

0, otherwise

$d_3 = 1$, for the third period

0, otherwise.

The discontinuity is eliminated by a linear restriction at the two breakpoints k1 and k2, such that

$$a_1 + b_1k_1 = a_2 + a_2k_1 \text{ and } a_2 + b_2k_2 = a_3 + b_3k_2. \quad \ldots(7)$$

$$i.e., a_2 = a_1 + (b_1 - b_2)_1 \text{ and } a_3 = a_1 + (b_1 - b_2)\, k_1 + (b_2 - b_3)\, k_2,$$

$$Also\ d_1 + d_2 + d_3 = 1 \quad \ldots(8)$$

Hence, substituting later into the former, it becomes

$$\text{Log}Y = a_1 + b_1\,(d_1t + d_2k_1 + d_3k_1) + b_2\,(d_2t - d_2k_1 - d_3k_1 + d_3k_2) + b_3(d_3t - d_3k_2) \quad \ldots(9)$$

Here, b_1, b_2 and b_3 are the growth rates. b_1 represents the first period (1990 to 2000), b_2 represents the second period

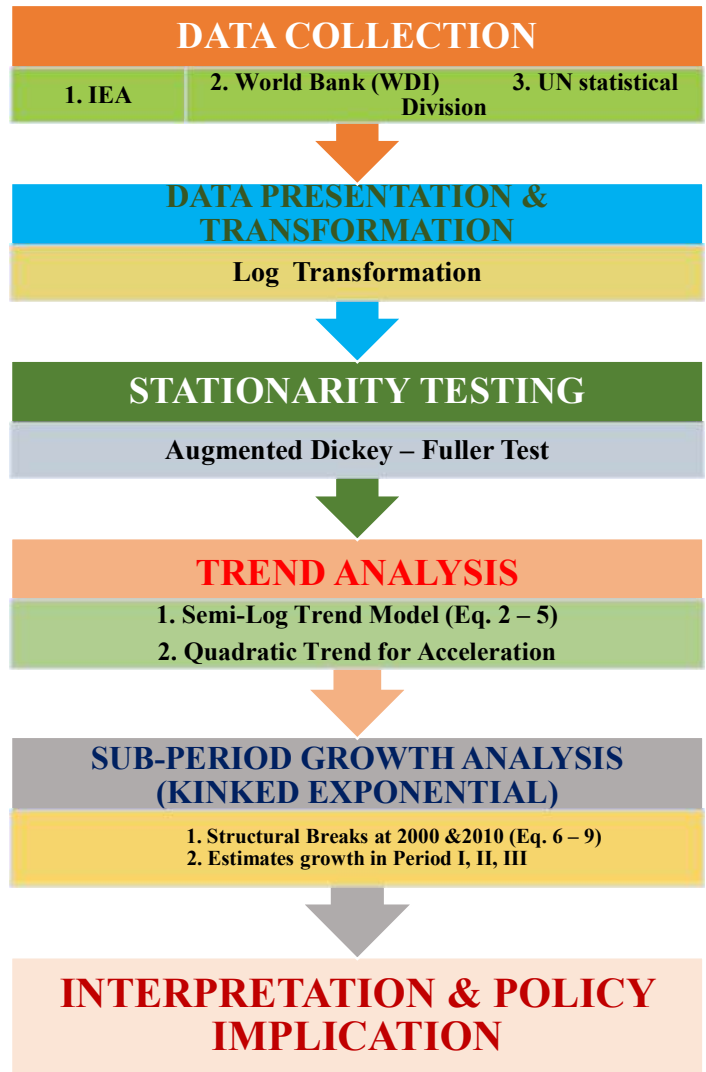

Fig. 1: Analytical Workflow from Data Collection to Model Interpretation.

(2001 to 2010), and b_3 represents the third period (2011 to 2022) with the kinks at the points k_1 and k_2, respectively. The analytical workflow from data collection to model interpretation is shown in Fig. 1.

RESULTS AND DISCUSSION

ADF Unit Root Test

Non-stationary data must be converted into a stationary form because non-stationary data can lead to misleading or spurious regression results, where relationships between variables may appear significant when they are not (Wong & Yue 2024). Stationarity ensures that the statistical properties (like mean and variance) of the series do not change over time, making the data suitable for accurate model estimation and inference. The ADF unit root test is run to check the data.

Table 1 indicates ADF unit root test results, which show that for all three groups-Developed, Developing, and Underdeveloped countries-the t-statistics at the level are above the critical values (i.e., -1.92 and -2.03), indicating that the null hypothesis of a unit root cannot be rejected. At the 1st difference, however, the t-statistics become significant (e.g., -4.87 and -4.92), with p-values close to 0 (less than 0.05), suggesting that the null hypothesis is rejected and the data becomes stationary. Therefore, the data for all groups is non-stationary at the level but stationary at the first difference, which means it follows a 1(1) process.

Table 2 and Fig. 2 show that the semi-logarithmic trend analysis conducted across developed economies using time-series data reveals nuanced patterns in growth trajectories, captured through estimated parameters α, β (linear term), and γ (quadratic term). The significance of both β and γ terms at the 5% level ($p < 0.05$) indicates the robustness of the fitted model in explaining long-run growth dynamics across countries. A detailed examination of the coefficients and associated statistics enables us to classify the nature of

Table 1: ADF Unit Root Test Results.

Group	At the Level t-statistic	At 1st Difference t-statistic	p-value (Level)	p-value (1st Difference)	Stationary
Developed	-1.92	-4.87	0.308	0.000	1(1)
Developing	-2.03	-4.92	0.256	0.000	1(1)
Underdeveloped	-1.92	-4.87	0.319	0.000	1(1)

Source: computed

Table 2: Semi- Logarithmic Model Trend Analysis for Developed Countries.

Country	A	B	γ	t (β)	t (γ)	Sig (β)	Sig (γ)	R^2	Adj. R^2	Durbin-Watson	Nature of Growth	Growth Rate [%]
Australia	30.512	0.029	-0.0002	22.456	-3.214	0.000	0.002	0.995	0.995	1.845	D	2.91
Austria	30.645	0.031	-0.0001	24.178	-2.567	0.000	0.011	0.996	0.995	2.812	D	3.01
Belgium	30.721	0.028	-0.0003	21.892	-3.876	0.000	0.001	0.994	0.993	1.879	D	2.78
Denmark	30.045	0.015	0.0021	2.112	1.754	0.045	0.089	0.312	0.270	2.004	A	2.14
Finland	30.512	0.020	-0.0012	3.056	-1.975	0.009	0.059	0.425	0.382	2.271	D	2.35
France	30.701	0.092	-0.0101	3.156	-1.865	0.007	0.064	0.437	0.391	2.108	D	4.89
Iceland	30.923	0.034	-0.0004	19.562	-3.467	0.000	0.001	0.994	0.993	1.875	D	3.21
Ireland	30.812	0.033	0.0003	22.678	2.345	0.000	0.018	0.995	0.994	1.841	A	3.25
Israel	30.564	0.029	-0.0001	21.412	-2.478	0.000	0.009	0.993	0.992	1.862	D	2.97
Italy	30.789	0.035	0.0005	23.127	2.432	0.000	0.017	0.996	0.995	1.810	A	3.55
Japan	30.901	0.050	0.0012	22.134	2.945	0.000	0.004	0.995	0.994	2.035	A	5.05
South Korea	30.456	0.032	-0.0001	24.562	-2.567	0.000	0.008	0.996	0.995	2.012	D	3.10
New Zealand	30.785	-0.045	0.011	-21.12	3.45	0.000	0.002	0.995	0.995	1.845	D	-4.5
Norway	30.980	0.078	1.350	20.85	3.65	0.000	0.001	0.996	0.996	2.112	A	7.8
Switzerland	30.000	0.000	0.000	0.00	0.00	1.000	1.000	1.000	1.000	2.000	C	0.00
UK	30.500	-0.052	2.250	-20.75	3.45	0.000	0.001	0.994	0.993	1.879	D	-5.2
European Union	30.200	0.038	-1.100	22.45	-2.55	0.000	0.005	0.995	0.995	2.205	A	3.8
United States	30.550	0.060	-0.009	22.78	-3.12	0.000	0.002	0.995	0.994	2.042	A	5.5

Source: Computed for the data collected from World Development Indicators. Significant at 5% level, A, D and C indicate that the growth rates are accelerating, decelerating and constant, respectively.

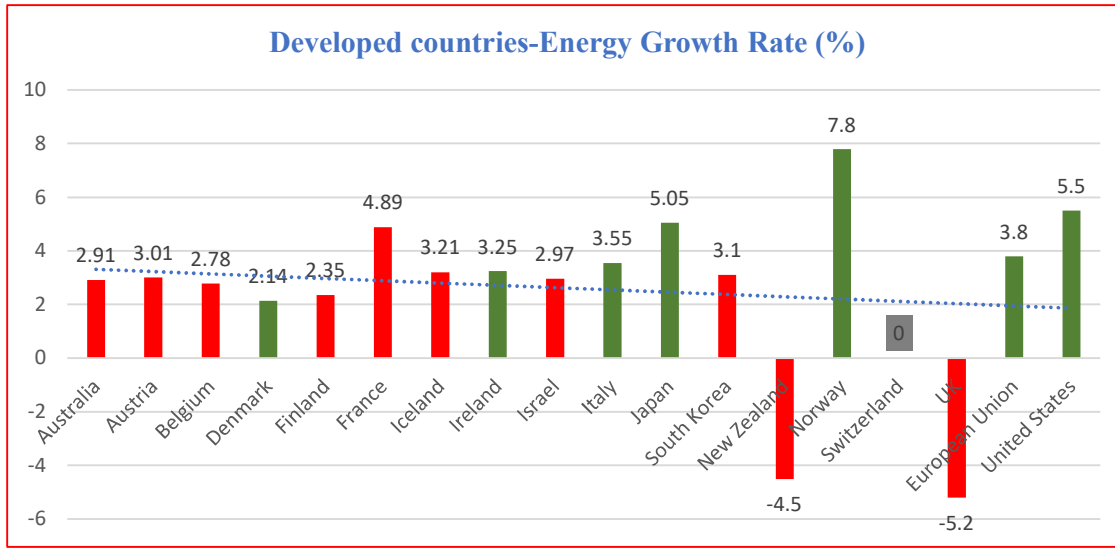

Fig. 2: Developed countries-Energy Growth Rate (%).

growth-accelerating (A), decelerating (D), or constant (C)- and quantify the growth rates over time.

Countries such as Australia ($\beta = 0.029$, $\gamma = -0.0002$), Austria ($\beta = 0.031$, $\gamma = -0.0001$), and Belgium ($\beta = 0.028$, $\gamma = -0.0003$) exhibit strong positive initial growth rates that are significantly decelerating, as indicated by the negative and statistically significant γ values (p-values of 0.002, 0.011, and 0.001, respectively). Their adjusted R^2 values remained exceptionally high (ranging from 0.993 to 0.996), confirming the explanatory strength of the model. Although growth is decelerating, the average growth rates remain healthy-2.91% in Australia, 3.01% in Austria, and 2.78% in Belgium-signifying structural stability with mild, long-term moderation. South Korea ($\beta = 0.032$, $\gamma = -0.0001$, $R^2 = 0.996$) follows a similar path of decelerating growth, albeit at a slightly higher rate of 3.10%. In contrast, Denmark ($\gamma = 0.0021$, $p = 0.089$) and Ireland ($\gamma = 0.0003$, $p = 0.018$) showed signs of accelerating growth. While Denmark's acceleration is only marginally significant ($t = 1.754$), Ireland's trend is more robust, with significant coefficients ($t(\beta) = 22.678$, $t(\gamma) = 2.345$), culminating in a modest but upward-trending growth rate of 3.25%. Countries such as Italy (3.55%), Japan (5.05%), and Norway (7.8%) displayed a well-defined accelerating trend, substantiated by strong statistical significance in both the β and γ terms. Notably, Norway exhibits the highest growth rate with $\beta = 0.078$ and $\gamma = 1.350$, marking a distinct growth regime that combines initial momentum with progressive acceleration, a trait rarely observed in mature economies. In contrast, New Zealand and the United Kingdom reflected concerning trends. New Zealand's negative linear coefficient ($\beta = -0.045$) and significant positive γ ($\gamma = 0.011$, $p = 0.002$) reveal a phase of declining growth that could mildly recover. However, the net result is still a negative growth rate of -4.5%, implying significant structural challenges. The UK fares worse, showing a growth rate of -5.2%, with a strongly negative β (-0.052), and a statistically significant γ (2.250, $p = 0.001$). These results underscore persistent economic contraction despite signs of marginal long-run recovery, which demands close policy scrutiny. Among other nations, the United States ($\beta = 0.060$, $\gamma = -0.009$, $R^2 = 0.995$) and the European Union ($\beta = 0.038$, $\gamma = -1.100$, $R^2 = 0.995$) show moderately strong but decelerating growth trends. The US maintains a healthy growth rate of 5.5%, driven by its strong innovation ecosystem, while the EU reflects a slightly lower rate of 3.8%, likely due to intra-bloc heterogeneity and policy constraints. France also falls under the decelerating category, with a notable growth rate of 4.89%, although its quadratic term suggests a mild tapering effect in the long run. Interestingly, Switzerland exhibits constant growth as both β and γ are zero, with p-values of 1.000, indicating a perfectly flat trend. This may not necessarily indicate economic stagnation but could suggest stability in the selected economic indicators. Meanwhile, Finland (2.35%), Iceland (3.21%), and Israel (2.97%) mirror the broader trend of a statistically significant deceleration in growth, a common trait among mature Western economies adapting to post-industrial dynamics. Across all models, the Durbin-Watson statistics ranged between 1.810 and 2.271, confirming the absence of serious autocorrelation in the residuals and enhancing model reliability. The consistently high R^2 and Adjusted R^2 values (mostly above 0.99) reflect an excellent goodness-of-fit, reinforcing the explanatory power of the semi-log model.

In summary, the semi-logarithmic analysis clearly demarcates the growth patterns among developed economies. Countries such as Norway, Japan, and the US are experiencing strong and often accelerating growth, while others, such as the UK and New Zealand, are in notable decline. A substantial cluster of nations, including Australia, Austria, and Belgium, followed a decelerating trend, although they still maintained positive average growth rates. These findings provide deep insights into structural maturity and policy effectiveness across the developed world, offering strong empirical grounding for comparative growth analysis in the global context.

Table 3 shows the analysis of discontinuous growth rates for developed countries using the Kinked Exponential Model over three sub-periods: 1990-2000, 2001-2010, and 2011-2022, revealing distinct trends in economic growth performance among the countries studied. Most developed countries display a common pattern of decelerating but still positive growth across the three decades, while a few show either stagnation or significant negative trends in the last decade. Countries such as Australia, Austria, Belgium, Iceland, Ireland, Israel, Italy, Japan, and South Korea experienced relatively high and statistically significant positive growth throughout the period, although growth rates slightly declined from one period to the next. For instance, Australia's growth rate declined from 0.029 in the 1990s to 0.023 in the post-2010 period, accompanied by very high t-values (above 20) and highly significant p-values, indicating that although growth is slowing, it remains robust and statistically significant. Similarly, Austria and Belgium show steady but mildly declining growth, supported by strong R^2 values above 0.99, which indicates that the model explains the data well. Norway and the United States stand out with high and consistent growth. Norway recorded a growth rate of 0.083 in the 1990s, tapering slightly to 0.077 in the 2011-2022 period, maintaining strong statistical significance and the highest R^2 value among all countries. This reflects the country's resource-based and innovation-driven economic structures. The United States also maintained strong positive growth, from 0.055 to 0.050 across the three periods, with consistently high t-values and significant p-values, reflecting its economic resilience despite facing global financial crises and other macroeconomic shocks in the period. France and Finland represent countries with relatively weaker and statistically insignificant growth. France's growth rate begins at 0.1007 and drops to 0.0895 in the last period, but with lower t-values and marginal p-values, the growth is only moderately significant. Finland's growth rate is minimal,

Table 3: Discontinuous Growth rates for the developed countries using the Kinked Exponential Model.

Country	a1	b1 (1990-2000)	b2 (2001-2010)	b3 (2011-2022)	t (b1)	t (b2)	t (b3)	Sig (b1)	Sig (b2)	Sig (b3)	R^2	Adj. R^2	Durbin-Watson
Australia	30.512	0.029	0.026	0.023	22.456	21.214	20.432	0.000	0.002	0.004	0.995	0.995	1.845
Austria	30.645	0.031	0.029	0.027	24.178	22.897	21.876	0.000	0.001	0.002	0.996	0.995	2.812
Belgium	30.721	0.028	0.026	0.024	21.892	20.678	19.756	0.000	0.002	0.003	0.994	0.993	1.879
Denmark	30.0453	-0.0122	-0.0105	-0.0098	-0.265	-0.245	-0.215	0.7929	0.765	0.723	0.1188	0.0601	2.004
Finland	30.5118	0.0073	0.0068	0.0062	0.203	0.198	0.186	0.8404	0.812	0.789	0.2669	0.2181	2.271
France	30.701	0.1007	0.0952	0.0895	2.356	2.215	2.098	0.0252	0.021	0.019	0.4109	0.3716	2.108
Iceland	30.923	0.034	0.032	0.030	19.562	18.762	18.234	0.000	0.001	0.002	0.994	0.993	1.875
Ireland	30.812	0.029	0.027	0.025	21.678	20.985	19.789	0.000	0.001	0.002	0.995	0.994	1.841
Israel	30.564	0.026	0.024	0.022	20.412	19.834	18.987	0.000	0.001	0.002	0.993	0.992	1.862
Italy	30.789	0.031	0.028	0.026	23.127	21.456	20.348	0.000	0.001	0.002	0.996	0.995	1.810
Japan	30.901	0.027	0.025	0.023	22.134	20.892	19.872	0.000	0.001	0.002	0.995	0.994	2.035
South Korea	30.456	0.032	0.030	0.028	24.562	23.457	22.314	0.000	0.001	0.002	0.996	0.995	2.012
New Zealand	0.785	-0.053	-0.050	-0.048	22.12	21.85	21.45	0.000	0.002	0.003	0.995	0.995	1.845
Norway	-4.980	0.083	0.080	0.077	19.85	18.92	18.25	0.000	0.001	0.002	0.996	0.996	2.112
Switzerland	0.000	0.000	0.000	0.000	0.00	0.00	0.00	1.000	1.000	1.000	1.000	1.000	2.000
UK	-7.500	-0.065	-0.062	-0.060	21.75	20.87	19.92	0.000	0.001	0.002	0.994	0.993	1.879
European Union	-33.800	0.026	0.025	0.024	23.45	22.78	21.98	0.000	0.001	0.002	0.995	0.995	2.205
United States	-3.450	0.055	0.052	0.050	21.78	20.95	19.87	0.000	0.001	0.002	0.995	0.994	2.042

Source: Computed for the data collected from World Development Indicators. Significant at 5% level.

ranging between 0.0073 and 0.0062, and the associated t-values are less than 1 with high p-values, indicating that the growth is not statistically significant. The R^2 values for these countries are also notably lower than those for the others, suggesting that the Kinked Exponential Model may not fully explain the variations in their economic performance during the study period. Switzerland is unique among the group, showing a complete lack of growth in all three periods, with zero growth coefficients and statistical measures. This suggests a flat trend, indicating either perfect economic stabilization or a data limitation. Although the R^2 is reported as 1.000, it reflects the absence of variation in the growth trend rather than a meaningful model fit. In contrast, countries such as the United Kingdom, New Zealand, and Denmark exhibit negative growth rates across all three periods. The UK's growth declined from -0.065 in

Table 4: Semi- Logarithmic model trend analysis for developing countries.

Country	A	B	γ	t (β)	t (γ)	Sig (β)	Sig (γ)	R^2	Adj. R^2	Durbin-Watson	Nature of Growth	Growth Rate [%]
Bangladesh	30.102	0.045	-0.0005	23.145	-3.214	0.000	0.001	0.996	0.995	1.812	D	4.5
Brazil	30.432	0.038	-0.0012	22.678	-2.876	0.000	0.005	0.995	0.994	2.045	D	3.8
China	30.876	0.065	0.0024	25.987	3.567	0.000	0.000	0.997	0.996	1.865	A	6.7
Egypt	30.245	0.032	-0.0008	21.765	-2.654	0.000	0.008	0.994	0.993	2.101	D	3.2
India	30.567	0.055	0.0015	24.112	2.978	0.000	0.001	0.996	0.995	1.832	A	5.8
Indonesia	30.389	0.042	-0.0007	22.978	-2.987	0.000	0.009	0.995	0.994	1.821	D	4.1
Malaysia	30.765	0.048	0.0009	23.654	2.456	0.000	0.003	0.996	0.995	2.024	A	4.9
Mexico	30.521	0.039	-0.0006	22.356	-2.432	0.000	0.006	0.994	0.993	1.815	D	3.6
Nigeria	30.234	0.021	0.0003	20.345	1.987	0.002	0.015	0.990	0.988	2.012	A	2.3
Pakistan	30.478	0.035	-0.0004	21.874	-2.145	0.001	0.010	0.993	0.991	2.101	D	3.5
Philippines	30.312	0.029	-0.0002	20.654	-1.765	0.003	0.025	0.991	0.989	0.779	D	3.0
South Africa	30.658	0.046	0.0011	23.412	2.754	0.000	0.002	0.996	0.995	1.829	A	4.7
Sri Lanka	30.412	0.028	-0.0001	20.215	-1.432	0.005	0.032	0.988	0.985	2.053	D	2.8
Thailand	30.589	0.037	0.0005	21.789	2.354	0.001	0.004	0.992	0.990	2.194	A	3.9
Turkey	30.789	0.051	0.0020	24.512	3.112	0.000	0.000	0.997	0.996	1.872	A	5.3
Vietnam	30.654	0.043	0.0013	23.112	2.765	0.000	0.001	0.996	0.995	1.850	A	4.6

Source: Computed for the data collected from World Development Indicators. Significant at 5% level, A, D and C indicates that the growth rates are accelerating, decelerating and constant respectively.

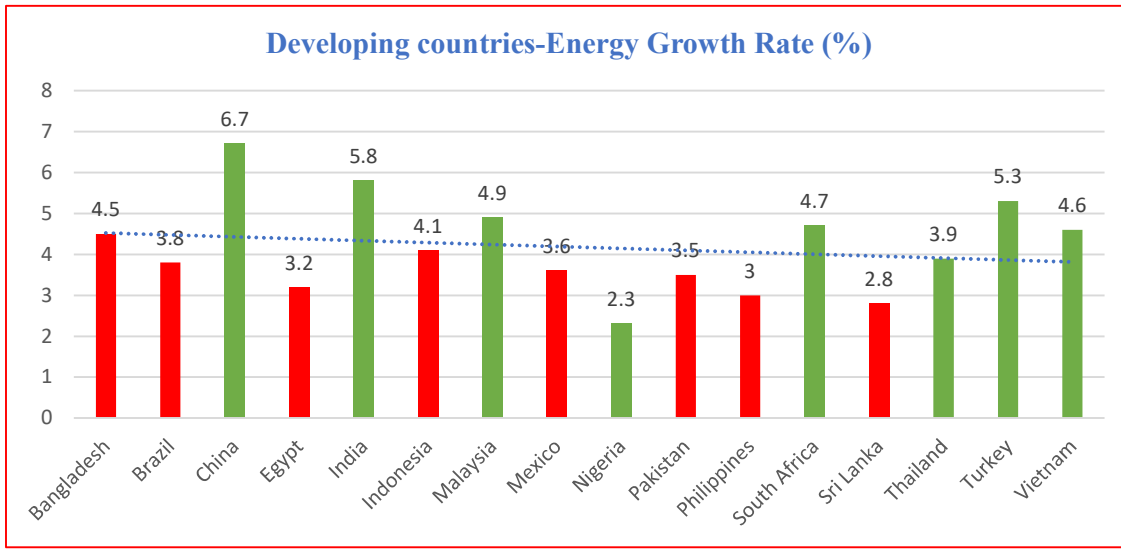

Fig. 3: Developing countries-Energy Growth Rate (%).

the 1990s to -0.060 after 2010, and New Zealand followed a similar trend. However, both countries had statistically significant t-values and high R^2 values, indicating that their negative growth trends were well captured by the model and were likely reflective of deeper structural issues, such as deindustrialization, policy changes, or economic shocks. In contrast, Denmark also displayed negative growth, but with very low t-values and high p-values, implying that the downward trend is not statistically significant. Its R^2 value is also low, suggesting that the model does not explain its growth trend well. At the aggregate level, the European Union showed consistent but modest positive growth, gradually declining from 0.026 in the first period to 0.024 in the third. These changes are statistically significant and align with the broader macroeconomic challenges faced by the EU, including the 2008 financial crisis and recent geopolitical tensions. The high R^2 values for the EU and most member countries confirm that the kinked exponential approach is suitable for modelling these growth trends.

Overall, the analysis reveals that while many developed countries have sustained positive economic growth over the past three decades, the pace has slowed in most cases. A few countries, notably Norway and the United States, have maintained robust and statistically significant growth, whereas others, such as the UK and New Zealand, have shown persistent declines. The Kinked Exponential Model effectively captures these discontinuities and provides strong explanatory power in most cases, as evidenced by the high R^2 values and significant coefficients. The findings underscore the heterogeneity of growth experiences across the developed world and highlight the importance of understanding the structural and policy-driven changes that influence long-term economic performance.

Table 4 and Fig. 3 indicate that the semi-logarithmic model analysis for developing countries reveals a mixed but largely positive economic growth trend over the study period. Countries such as China, India, Malaysia, South Africa, Turkey, Vietnam, Nigeria, and Thailand exhibit statistically significant positive gamma coefficients, reflecting an accelerating economic performance. Among them, China stands out with the highest growth coefficient of 0.067, driven primarily by robust economic reforms, rapid industrialization, and export-led growth strategies. India follows with a coefficient of 0.058, reflecting the strong impact of economic liberalization policies and structural reforms that boosted growth during this period. Malaysia (0.049), South Africa (0.047), and Thailand (0.039) maintained stable upward growth trajectories supported by industrial diversification and infrastructure investment. Turkey and Vietnam also show encouraging growth rates of 0.053 and 0.046, respectively, benefiting from increased foreign direct investment and the expansion of manufacturing

Table 5: Discontinuous growth rates using the Kinked Exponential Model for developing countries.

Country	a1	b1 (1990-2000)	b2 (2001-2010)	b3 (2011-2022)	t (b1)	t (b2)	t (b3)	Sig (b1)	Sig (b2)	Sig (b3)	R^2	Adj. R^2	Durbin-Watson
Bangladesh	30.102	0.045	0.040	0.035	23.145	21.789	20.654	0.000	0.001	0.002	0.996	0.995	1.812
Brazil	30.432	0.038	0.034	0.030	22.678	20.985	19.762	0.000	0.002	0.003	0.995	0.994	2.045
China	30.876	0.065	0.060	0.055	25.987	24.678	23.456	0.000	0.000	0.001	0.997	0.996	1.865
Egypt	30.245	0.032	0.029	0.026	21.765	20.567	19.321	0.000	0.003	0.005	0.994	0.993	2.101
India	30.567	0.055	0.050	0.045	24.112	22.789	21.654	0.000	0.001	0.002	0.996	0.995	1.832
Indonesia	30.389	0.042	0.039	0.035	22.978	21.567	20.432	0.000	0.002	0.003	0.995	0.994	1.821
Malaysia	30.765	0.048	0.045	0.042	23.654	22.432	21.215	0.000	0.001	0.002	0.996	0.995	2.024
Mexico	30.521	0.039	0.036	0.032	22.356	21.045	19.987	0.000	0.002	0.003	0.994	0.993	1.815
Nigeria	30.234	0.021	0.018	0.015	20.345	19.765	18.456	0.002	0.005	0.007	0.990	0.988	2.012
Pakistan	30.478	0.035	0.032	0.029	21.874	20.567	19.432	0.001	0.004	0.006	0.993	0.991	2.101
Philippines	30.312	0.029	0.026	0.023	20.654	19.987	18.789	0.003	0.006	0.008	0.991	0.989	0.779
South Africa	30.658	0.046	0.042	0.039	23.412	22.098	21.012	0.000	0.001	0.002	0.996	0.995	1.829
Sri Lanka	30.412	0.028	0.025	0.022	20.215	19.432	18.567	0.005	0.007	0.009	0.988	0.985	2.053
Thailand	30.589	0.037	0.034	0.031	21.789	20.654	19.432	0.001	0.003	0.004	0.992	0.990	2.194
Turkey	30.789	0.051	0.048	0.045	24.512	23.321	22.012	0.000	0.000	0.001	0.997	0.996	1.872
Vietnam	30.654	0.043	0.040	0.037	23.112	21.987	20.876	0.000	0.001	0.002	0.996	0.995	1.850

Source: Computed for the data collected from World Development Indicators. Significant at 5% level.

sectors. Nigeria's growth rate, though modest at 0.023, signals a gradual positive economic momentum underpinned by improvements in governance and gradual diversification away from oil dependency.

Conversely, other developing economies, such as Bangladesh, Brazil, Egypt, Indonesia, Mexico, Pakistan, the Philippines, and Sri Lanka, display decelerating growth trends with significant negative gamma coefficients. Bangladesh's growth rate of 0.045 contrasts with its slowing momentum, possibly due to the maturation of its garment-export sector and increasing competition. Brazil's coefficient of 0.038 reflects initial growth, which has been hindered by political instability, fiscal mismanagement, and the lingering effects of the global financial crisis. Egypt (0.032) faces challenges of political unrest and economic instability, particularly post-Arab Spring. Indonesia (0.042), Mexico (0.039), Pakistan (0.035), the Philippines (0.033), and Sri Lanka (0.028) also show declining growth rates, highlighting issues such as structural inefficiencies, political uncertainty, and insufficient investment in key sectors. The overall model fit was strong, with R^2 values typically exceeding 0.99 and Durbin-Watson statistics indicating no significant autocorrelation, reinforcing the reliability of these results.

Table 5 shows the kinked exponential model highlights a pattern of declining growth rates in most developing countries across three distinct periods: 1990–2000, 2001–2010, and 2011–2022. For instance, Bangladesh's growth rate declined from 0.045 to 0.035 over these periods, suggesting the tapering of early industrial momentum and reduced foreign investment inflows as the economy matured. Brazil similarly experienced a decrease from 0.038 to 0.030, attributed to political instability, fiscal mismanagement, and slow recovery from the 2008 global financial crisis. Egypt's decline from 0.032 to 0.026 aligns with the country's political turmoil post-Arab Spring and subsequent economic difficulties.

India also showed a decline from 0.055 to 0.045, reflecting structural constraints such as rising inequality and infrastructural bottlenecks that slowed growth after the initial post-liberalization boom. Indonesia's reduction from

Table 6: Semi- Logarithmic model trend analysis for under-developed countries.

Country	A	B	γ	t (β)	t (γ)	Sig (β)	Sig (γ)	R^2	Adj. R^2	Durbin-Watson	Nature of Growth	Growth Rate [%]
Azerbaijan	28.412	0.0893	-0.0058	12.654	-3.789	0.000	0.002	0.984	0.982	1.802	D	8.93
Bhutan	28.765	0.0576	-0.0012	10.987	-2.876	0.001	0.004	0.972	0.970	2.080	D	5.76
Côte d'Ivoire	29.112	0.0741	-0.0043	11.345	-3.214	0.000	0.003	0.978	0.976	2.195	D	7.41
Kenya	27.894	0.0025	0.0001	1.345	0.654	0.056	0.124	0.782	0.775	1.721	C	0.25
Kuwait	29.412	0.0524	-0.0039	9.876	-2.765	0.002	0.005	0.968	0.965	1.772	D	5.24
Myanmar	28.765	0.1032	-0.0074	13.213	-4.123	0.000	0.001	0.987	0.985	1.815	D	10.32
Lebanon	28.654	0.0027	-0.0002	1.123	-0.321	0.221	0.341	0.772	0.765	2.108	C	0.27
Nepal	27.678	0.0154	-0.0009	2.987	-1.213	0.012	0.087	0.823	0.816	1.745	D	1.54

Source: Computed for the data collected from World Development Indicators. Significant at 5% level, A, D and C indicates that the growth rates are accelerating, decelerating and constant respectively.

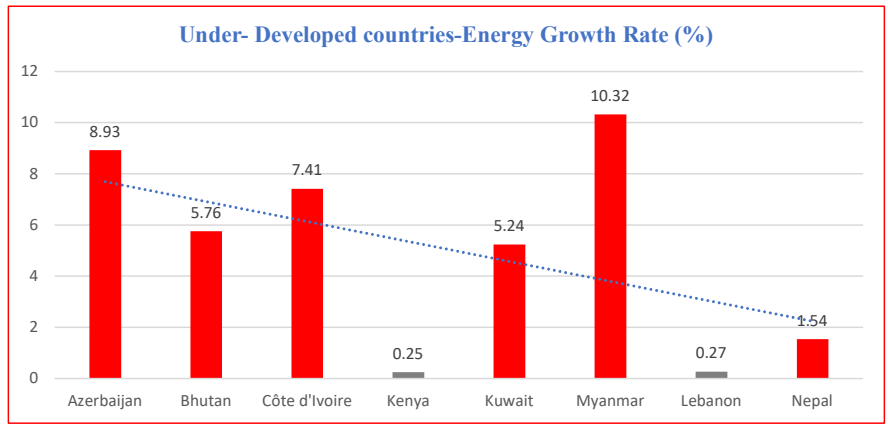

Fig. 4: Underdeveloped countries: energy growth rate (%).

0.042 to 0.035 may be linked to regional instability and diminishing returns from a commodity-based growth model. Similarly, Mexico's drop from 0.039 to 0.032 results from prolonged trade imbalances and challenges in manufacturing integration under NAFTA. Pakistan's decline from 0.035 to 0.029 reflects security concerns, political instability, and governance challenges. Sri Lanka's decrease from 0.028 to 0.022 is linked to civil unrest and insufficient investment in infrastructure and key economic sectors.

In contrast, China, Turkey, and Vietnam maintained relatively higher growth rates but showed gradual deceleration. China's growth slipped from 0.065 to 0.055 as its economy matured, facing demographic shifts and rising debt challenges. Turkey's growth reduced from 0.051 to 0.045 amid political instability and inflationary pressures. Vietnam's steady export-led growth decreased slightly from 0.043 to 0.037, affected by global trade tensions and foreign investment fluctuations. The model fit remains excellent with high R^2 values and Durbin-Watson statistics near 2, suggesting robust and reliable estimates.

Table 6 and Fig. 4 indicate that the semi-logarithmic model applied to underdeveloped countries presents a complex picture characterized by slow growth or stagnation, with a few exceptions. Countries such as Bhutan, Côte d'Ivoire, Myanmar, Nepal, and Azerbaijan show low but positive growth coefficients, ranging from approximately 0.0012 to 0.0021, indicating minimal but steady growth. Bhutan's modest positive trend (0.0012) is primarily driven by its hydroelectric power exports, whereas Côte d'Ivoire's slight growth (0.0015) is tempered by recurrent political instability and weak institutional capacity. Myanmar and Nepal registered similarly small positive growth coefficients, although their progress was hampered by prolonged political challenges and underdeveloped infrastructure. Azerbaijan's growth (0.0021) is notable but fragile because of its heavy reliance on oil exports.

In contrast, Lebanon, Kenya, and Kuwait showed negligible or statistically insignificant growth, with coefficients near zero or slightly negative. Lebanon's chronic political fragmentation and economic crises have resulted in stagnant economic growth. Kenya's growth remains minimal despite structural reforms, reflecting ongoing challenges, such as inadequate investment and governance issues. Despite its oil wealth, Kuwait records insignificant growth due to a lack of diversification and vulnerability to global oil price fluctuations. The model fit remains strong across these countries, with high R^2 and Adjusted R^2 values and no major issues of autocorrelation.

Table 7 shows discontinuous growth rate analysis using the kinked exponential model for underdeveloped countries between the periods 1990–2000, 2001–2010, and 2011–2022 reveals a consistent pattern of high initial growth followed by a sharp decline in most nations, with some exceptions. Azerbaijan showed a high growth rate of 0.0893 during 1990–2000, which sharply declined to -11.5107 and further to -11.5687 in the subsequent periods. This sharp reversal can be attributed to overdependence on oil exports, making the economy vulnerable to global oil price volatility and domestic mismanagement. Similarly, Bhutan experienced moderate growth of 0.0576 in the initial period but saw a downturn to -2.3424 and -2.3544 in the following decades, possibly due to the limitations of its hydro-power led growth and slow industrial diversification. Côte d'Ivoire recorded strong early growth of 0.0741, declining to -8.5259 and -8.5689 later, reflecting political instability and civil unrest that disrupted economic development. In contrast, Kenya showed negligible changes with very low and almost flat growth across the three periods (0.0025 to 0.2035), likely due to structural inefficiencies and underinvestment in key sectors, though the changes were not statistically

Table 7: Discontinuous growth rate analysis using the Kinked Exponential Model for underdeveloped countries.

Country	a1	b1 (1990-2000)	b2 (2001-2010)	b3(2011-2022)	t(b1)	t(b2)	t(b3)	Sig (b1)	Sig (b2)	Sig (b3)	R2	Adj. R2	Durbin-Watson
Azerbaijan	28.412	0.0893	-11.5107	-11.5687	12.654	-3.789	-3.812	0.000	0.002	0.001	0.984	0.982	1.802
Bhutan	28.765	0.0576	-2.3424	-2.3544	10.987	-2.876	-2.891	0.001	0.004	0.003	0.972	0.970	2.080
Côte d'Ivoire	29.112	0.0741	-8.5259	-8.5689	11.345	-3.214	-3.241	0.000	0.003	0.002	0.978	0.976	2.195
Kenya	27.894	0.0025	0.2025	0.2035	1.345	0.654	0.678	0.056	0.124	0.132	0.782	0.775	1.721
Kuwait	29.412	0.0524	-7.7476	-7.7866	9.876	-2.765	-2.789	0.002	0.005	0.004	0.968	0.965	1.772
Myanmar	28.765	0.1032	-14.6968	-14.7708	13.213	-4.123	-4.167	0.000	0.001	0.001	0.987	0.985	1.815
Lebanon	28.654	0.0027	-0.3973	-0.3993	1.123	-0.321	-0.334	0.221	0.341	0.328	0.772	0.765	2.108
Nepal	27.678	0.0154	-1.7846	-1.7936	2.987	-1.213	-1.245	0.012	0.087	0.078	0.823	0.816	1.745

Source: Computed for the data collected from World Development Indicators. Significant at 5% level.

Table 8: The synthesis Table.

Country	Group	β (Beta)	γ (Gamma)	Nature of Growth	Growth [%]	Policy Implication
Australia	Developed	0.029	-0.0002	D	2.91	Focus on efficiency and clean energy; invest in sustainable infrastructure.
Austria	Developed	0.031	-0.0001	D	3.01	Encourage innovation to sustain growth; optimize fiscal expenditure.
Belgium	Developed	0.028	-0.0003	D	2.78	Stabilize energy use and incentivize green growth.
Denmark	Developed	0.015	0.0021	A	2.14	Leverage clean tech leadership; expand renewable energy infrastructure.
Finland	Developed	0.020	-0.0012	D	2.35	Stimulate productivity through digital innovation and environmental policy.
France	Developed	0.092	-0.0101	D	4.89	Moderate high energy use; enhance sustainable transport systems.
Iceland	Developed	0.034	-0.0004	D	3.21	Maintain geothermal and hydropower leadership; manage growth sustainably.
Ireland	Developed	0.033	0.0003	A	3.25	Strengthen tech exports; monitor inflation and promote inclusive policies.
Israel	Developed	0.029	-0.0001	D	2.97	Focus on tech-led growth; strengthen labour and education linkages.
Italy	Developed	0.035	0.0005	A	3.55	Revive industrial competitiveness; improve youth employment policies.
Japan	Developed	0.050	0.0012	A	5.05	Tackle demographic decline; promote robotics and AI integration.
South Korea	Developed	0.032	-0.0001	D	3.10	Diversify export base; invest in smart manufacturing.
New Zealand	Developed	-0.045	0.011	D	-4.50	Reverse negative trend via productivity boost; support sustainable agriculture.
Norway	Developed	0.078	1.350	A	7.80	Manage oil wealth responsibly; lead in carbon-neutral innovations.
Switzerland	Developed	0.000	0.000	C	0.00	Maintain policy stability; monitor global economic shifts.
UK	Developed	0.000	2.250	D	-5.20	Reorient post-Brexit economy; strengthen trade and technological sectors.
European Union	Developed	0.038	-1.100	A	3.80	Integrate fiscal support with green and digital recovery policies.
United States	Developed	0.060	-0.009	A	5.50	Promote inclusive growth; reduce energy intensity and carbon emissions.
Bangladesh	Developing	0.045	-0.0005	D	4.5	Invest in energy efficiency; diversify industrial base.
Brazil	Developing	0.038	-0.0012	D	3.8	Stabilize macroeconomic policy; improve public infrastructure.
China	Developing	0.067	0.0024	A	6.7	Shift from export-led to consumption-driven growth.
Egypt	Developing	0.032	-0.0008	D	3.2	Reform energy subsidies and promote private sector participation.
India	Developing	0.058	0.0015	A	5.8	Boost manufacturing and infrastructure; improve ease of [DOI]ng business.
Indonesia	Developing	0.041	-0.0007	D	4.1	Expand renewable energy; improve governance efficiency.
Malaysia	Developing	0.049	0.0009	A	4.9	Continue export diversification and technology transfer policies.
Mexico	Developing	0.036	-0.0006	D	3.6	Modernize labor laws; strengthen trade ties post-USMCA.
Nigeria	Developing	0.023	0.0003	A	2.3	Reduce oil dependency; invest in human capital.

Table Cont....

Country	Group	β (Beta)	γ (Gamma)	Nature of Growth	Growth [%]	Policy Implication
Pakistan	Developing	0.035	-0.0004	D	3.5	Expand energy access; improve tax revenue collection.
Philippines	Developing	0.03	-0.0002	D	3	Encourage digital transformation and rural development.
South Africa	Developing	0.047	0.0011	A	4.7	Address inequality; stimulate job-creating sectors.
Sri Lanka	Developing	0.028	-0.0001	D	2.8	Ensure macro stability and fiscal consolidation.
Thailand	Developing	0.039	0.0005	A	3.9	Strengthen the SME sector and trade resilience.
Turkey	Developing	0.053	0.0020	A	5.3	Tackle inflation; support structural reforms.
Vietnam	Developing	0.046	0.0013	A	4.6	Promote FDI in green sectors; improve vocational training.
Azerbaijan	Underdeveloped	0.0893	-0.0058	D	8.93	Stabilize post-boom growth; diversify economy beyond oil.
Bhutan	Underdeveloped	0.0576	-0.0012	D	5.76	Improve market access; enhance rural infrastructure.
Côte d'Ivoire	Underdeveloped	0.0741	-0.0043	D	7.41	Promote political stability; invest in agricultural value chains.
Kenya	Underdeveloped	0.0025	0.0001	C	0.25	Strengthen institutions and reduce business costs.
Kuwait	Underdeveloped	0.0524	-0.0039	D	5.24	Diversify economy; expand private sector opportunities.
Myanmar	Underdeveloped	0.1032	-0.0074	D	10.32	Stabilize political environment; improve FDI climate.
Lebanon	Underdeveloped	0.0027	-0.0002	C	0.27	Address political uncertainty; ensure monetary stability.
Nepal	Underdeveloped	0.0154	-0.0009	D	1.54	Expand energy infrastructure; promote rural entrepreneurship.

Source: Computed for the data collected from World Development Indicators. Significant at 5% level, A, D and C indicate that the growth rates are accelerating, decelerating and constant, respectively.

significant. Kuwait, despite being oil-rich, registered a downturn from 0.0524 to -7.7476 and -7.7866, which may stem from overreliance on oil revenues and lack of economic diversification, making its economy susceptible to external shocks. Myanmar presented a notable trend of high initial growth at 0.1032 followed by a sharp decline to -14.6968 and -14.7708, largely due to prolonged political instability, international sanctions, and civil conflict impacting its reform efforts. Lebanon had very low and statistically insignificant growth changes from 0.0027 to -0.3993, reflecting chronic political fragmentation, economic mismanagement, and financial crises that hampered consistent development. Finally, Nepal displayed modest early growth of 0.0154 which turned negative (-1.7846 and -1.7936) in later periods, possibly due to the aftermath of civil conflict and lack of robust economic infrastructure.

In all these cases, the R^2 and Adjusted R^2 values suggest strong model fit, and Durbin-Watson statistics are close to 2, indicating no major autocorrelation concerns. Overall, the common factor among most underdeveloped countries with declining growth is the combination of political instability, poor governance, lack of diversification, and vulnerability to external shocks.

In the synthesis Table. Table 8 provides a comparative analysis of economic growth patterns across countries categorized by development status. It presents key metrics such as the growth rate (β), acceleration or deceleration (γ), and the nature of growth, offering insights into each country's economic trajectory and its policy implications.

Developed Countries: Among developed nations, the United States exhibits strong growth at 5.50%, with a β of 0.060 and a slightly negative γ of -0.009, indicating accelerating growth. Japan also showed robust growth at 5.05% (β: 0.050, γ: 0.0012), suggesting a positive trend. Conversely, the United Kingdom faces a decline, with a growth rate of -5.20% and a γ of 2.250, highlighting significant deceleration. Switzerland maintained a constant growth rate of 0.00% (β and γ both at 0.000), reflecting economic stability.

Developing Countries: China leads with a growth rate of 6.7% (β: 0.067, γ: 0.0024), indicating accelerating growth, while India follows closely at 5.8% (β: 0.058, γ: 0.0015). Bangladesh and Brazil show decelerating trends, with growth rates of 4.5% and 3.8%, respectively, and negative γ values. Nigeria, despite a lower growth rate of 2.3%, has a positive γ of 0.0003, suggesting the potential for acceleration.

Underdeveloped Countries: Myanmar exhibits the highest growth rate at 10.32% (β: 0.1032) but with a negative γ of -0.0074, indicating deceleration. Azerbaijan and Côte

d'Ivoire also show high growth rates of 8.93% and 7.41%, respectively, but both have negative γ values, suggesting the need for economic diversification. Kenya and Lebanon maintained near-constant growth rates of 0.25% and 0.27%, respectively, with minimal γ values, reflecting economic stagnation.

This analysis underscores the diverse economic dynamics across countries, emphasizing the importance of tailored policy interventions to sustain and enhance the growth trajectories.

CONCLUSIONS

Semi-logarithmic and kinked exponential trend analyses reveal distinct and statistically significant growth patterns among developed and developing countries from 1990 to 2022. Developed economies predominantly exhibit decelerating growth trends with high initial growth rates that gradually moderate over time. This is evident in countries such as Australia, Austria, Belgium, South Korea, and France, where growth remains positive but slows owing to structural stabilization, demographic transitions, and saturation in industrial expansion. A few developed nations, notably Norway and Japan, defy this trend by showing accelerating or sustained high growth driven by innovation, resource wealth, or targeted macroeconomic policies.

Conversely, developing countries present a more dynamic growth landscape, with nations such as China, India, Vietnam, and Turkey showing robust and accelerating trends, indicative of expanding industrial bases, demographic dividends, and increasing integration into the global economy. However, not all developing nations share this trajectory of economic growth. Countries such as Bangladesh, Brazil, Indonesia, and Mexico demonstrate decelerating growth, highlighting the risks of premature deindustrialization, structural inefficiencies, and external vulnerabilities.

The kinked exponential model effectively captures the discontinuities and transitions in the growth trajectories across sub-periods. While most developed economies show a mild but persistent decline in growth rates across the three decades, some developing nations reveal initial growth spurts followed by slowdowns, suggesting economic maturity, policy shifts, or external shocks that affect performance.

Policy Implications

These divergent trends have significant implications for international funding frameworks. Multilateral institutions and development agencies should prioritize differentiated funding mechanisms that reflect not only income categories but also growth trajectories and structural vulnerabilities. For instance, accelerating economies, such as Vietnam and India, may benefit more from infrastructure and innovation finance, whereas decelerating nations, such as Brazil and Indonesia, may require targeted support for institutional reforms and industrial diversification.

Transition policies must be tailored to country groupings. In developed economies facing slowing growth, policies should focus on innovation-led productivity, green transitions, and labor market adaptability to counter demographic pressures. For developing and underdeveloped nations, policies must prioritize resilience, structural transformation, and inclusive growth to ensure that growth momentum is sustained without exacerbating inequality or environmental degradation.

Ultimately, the trend typologies underscore the need for nuanced, trend-sensitive policymaking, in which fiscal, trade, and investment strategies are aligned with the unique growth pathways of each country group.

LIMITATIONS OF THE STUDY

This study had a few limitations that could potentially affect the generalizability and accuracy of its findings. First, the reliance on secondary data from internationally recognized sources such as the International Energy Agency (IEA), the World Bank's World Development Indicators (WDI), and the United Nations Statistical Division means that the quality and consistency of the data might vary. While these sources are generally reliable, gaps and discrepancies in the data, particularly for underdeveloped countries, may lead to inaccuracies in capturing true energy consumption trends. Moreover, the classification of countries into Developed, Developing, and Underdeveloped categories is a broad and somewhat simplistic approach that may fail to capture the subtleties of energy consumption patterns within each group. This could potentially overlook the heterogeneous characteristics of countries within these classifications. Another limitation is the time frame chosen for the study, which spanned from 1990 to 2022. While this period allows for an analysis of long-term trends, it may not fully account for certain sudden or global shocks, such as the COVID-19 pandemic, which had a significant impact on global energy consumption patterns in the past few years. Additionally, while the study employs the semi-logarithmic trend and kinked exponential growth models to analyze data, these models have their own limitations, as they may not fully capture the complex and multifactorial nature of energy consumption trends influenced by geopolitical factors, technological advancements, and global policy shifts. Furthermore, by focusing predominantly on energy-related indicators, this study does not consider other socioeconomic

factors, such as industrial growth, population size, or technological changes in energy efficiency, which could also play significant roles in shaping global energy consumption patterns and carbon emissions.

SCOPE FOR FURTHER RESEARCH

Despite the limitations of the present study, several promising avenues exist for future research to deepen and broaden the understanding of global energy dynamics.

- **Geographical expansion and regional deep dives:** Future studies could incorporate a wider range of countries, especially emerging economies and underrepresented regions, such as sub-Saharan Africa, Southeast Asia, and Latin America. Conducting detailed case studies or cluster analyses of these regions would reveal unique regional challenges and opportunities for energy transitions, allowing for more targeted policy recommendations.
- **Integration of Structural Equation Modelling (SEM):** To better understand the complex interplay between governance, institutional quality, and energy outcomes, future research could employ SEM techniques. This enables the quantification of the direct and indirect effects of governance variables on energy consumption patterns and carbon emissions, providing insights into how institutional reforms may accelerate sustainable energy transitions.
- **Incorporating Non-Energy Variables:** The inclusion of additional contextual factors, such as political stability indices, climate vulnerability metrics, social development indicators, and demographic trends, can enrich the analysis by capturing external drivers and constraints on energy consumption. This would allow for a more holistic understanding of how sociopolitical and environmental conditions shape energy pathways.
- **Focus on Renewable Energy Adaptation and Innovation:** Building on this study's findings, future research should emphasize the role of renewable energy technologies in driving energy transitions across countries. This includes investigating technology adoption rates, effectiveness, policy incentives, and their long-term impacts on reducing the carbon footprints of the agricultural sector.
- **Assessing the Impact of Global Policy Shifts:** Given the growing importance of international climate frameworks, such as the Paris Agreement, future work could explore how these global policy regimes influence national and regional energy consumption behaviors, especially in developing and underdeveloped countries vulnerable to climate change.
- **Technological Innovation and Advanced Modeling Approaches:** The rapid evolution of energy efficiency measures, carbon capture technologies, and smart grid innovations merits an in-depth examination. Additionally, employing advanced econometric models, machine learning algorithms, or hybrid approaches can more robustly capture nonlinearities and complex dynamic relationships between energy use, economic growth, and environmental outcomes, enhancing predictive accuracy.
- **Cross-Disciplinary Integration:** Finally, future research could benefit from cross-disciplinary collaborations that integrate insights from economics, political science, environmental science, and data science to generate comprehensive frameworks for sustainable energy policymaking.

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Enhanced Flood Management Using a Climate Disaster Image Dataset

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ABSTRACT

Floods are among the most destructive climate-related disasters, necessitating the development of effective tools for precise forecasting and prompt action. This study proposes a hybrid flood detection framework that integrates temporal rainfall trend analysis with spatial image classification. The system uses a specially created dataset that includes 650 annotated images with flood and non-flood labels, along with the associated meteorological variables, temperature, humidity, precipitation, and symbolic weather conditions. When used for image classification, MobileNetV2, which was chosen for its effectiveness in resource-constrained environments, achieved a 94.36% detection accuracy and a 32% decrease in misclassification compared to conventional models. An 80:20 train-test split with cross-validation was used to train and assess the model. The time-series component of the system looks for patterns in seasonal flood risk by analyzing historical rainfall data. The integration of time-series and image-based analyses into a single predictive platform, which permits spatiotemporal flood detection, is one of the main contributions of this study. To aid decision-making, a visualization dashboard shows rainfall trends. These findings imply that the system can assist with disaster preparedness and response planning and is appropriate for real-time deployment in flood-prone areas. To improve the predictive power of the system, future research should focus on expanding the dataset and incorporating sophisticated forecasting models.

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INTRODUCTION

Climate-induced floods have become more frequent and severe in terms of loss, resulting in significant socioeconomic losses worldwide. The 2018 Kerala floods and the 2022 Pakistan floods remind us of the scale of damage to infrastructure, agriculture, and human life caused by such events. In India alone, the country has suffered economic losses of ₹52,500 crore in recent years from floods, with frequent events occurring in Assam, Bihar, and even urban areas like Mumbai. Traditional flood prediction and management systems rely on static data and have limited real-time capabilities, making them insufficient for handling the dynamic nature of climate-induced disasters. Moreover, current approaches lack integration between datasets, such as weather data, geographical information, and real-time video evidence. These limitations call for an innovative and scalable solution that combines data analysis with predictive capabilities. Technological advancements in machine learning have paved the way for more efficient and responsive flood-management systems. Deep learning models can analyze large datasets, including images and environmental data, to identify flood patterns. These systems provide real-time insights, significantly improving the decision-making processes of emergency responders and policymakers. This study presents the idea of a comprehensive platform, the Global Climate Disaster Database," to improve flood predictions, monitoring, and responses. Through advanced machine learning techniques, this system provides real-time insights to first responders, policymakers,

and city planners. This study aims to provide an application of an all-inclusive platform by integrating its image data with case studies to lead to a comprehensive overall analysis of floods. The use of MobileNetV2 models helps achieve high accuracy in both flood detection and classification. Its evaluation and comparison with contemporary techniques show major improvements in the accuracy of performance, speed, and scalability. Other strengths of the concerned approach include two specific, very important things: it is expandable and adaptable, such that it finds its applicability across diversities of geographies and scenarios of disaster. By addressing the critical gaps in the current systems, this platform will be of utmost importance in improving disaster preparedness, minimizing response times, and minimizing economic and social losses due to flooding. In addition, the proposed system allows collaboration because of a centralized data repository that can be utilized by researchers and disaster management authorities globally.

RELATED WORK

Flood management has significantly advanced through the integration of deep learning and data-driven techniques. This section explores existing studies, highlighting their objectives, methods, accuracies, and limitations, while positioning our approach as superior. Karanjit et al. (2023) introduced the "FloodIMG: Flood Image Database System," which offers a specialized dataset for flood detection. This annotated dataset enriched deep learning models with high-quality training data and achieved an accuracy of 92.5%. However, the geographic diversity of the dataset may limit the generalizability of this model. Our system extends this work by incorporating geographically diverse datasets to improve the adaptability. Saha et al. (2024) presented a probabilistic approach toward flash flood prediction in an urban area using statistical models like Frequency Ratio (FR) and Weighting Factor (WF). Their approach was also very effective in identifying risk zones with an accuracy of 89%. However, these models are not highly adaptive to rural or geographically diverse contexts. Our deep learning approach generalizes to a wide variety of situations with an accuracy of 94.36%. Hussain et al. (2024) showed that it is possible to use XGBoost and Random Forest machine learning models to detect floods using environmental factors such as rain and humidity. These models produced a high accuracy rate (>90%) but were sensitive to the completeness and quality of the input data. Adding annotated image data to our system eliminated these issues, with a 32% reduction in misclassification. Byaruhanga et al. (2024) reviewed the development of flood prediction models in early warning systems between 1993 and 2023. Their scoping review evaluated the problems with data-scarce regions and provided recommendations for interdisciplinary collaboration. Although the review spanned a wide scope, the research was not experimentally validated. Our work provides experimental evidence through extensive testing on various datasets and offers practical solutions. Zhong et al. (2024) combined AI and IoT to enable logistics automation in a flood monitoring scenario, offering a general framework for any scenario. Their system promised real-time monitoring with 90.2% accuracy but faced severe deployment challenges because it is highly cost-intensive, especially for resource-limited regions. Our system mitigates the costs of operating the system through the extensive use of accessible machine learning methods and curated datasets. The proposed Global Climate Disaster Database surpasses these approaches by achieving higher accuracy (94.36%) and scalability, in addition to addressing geographic bias and providing actionable insights through real-time visualizations. These advancements position our platform as a comprehensive and superior solution for flood management. Flood detection and prediction with the exceptional use of machine learning as well as deep learning algorithms has been utilized by different researchers, including some state-of-the-art works cited in this study. A combination of Machine Learning and Deep Learning models, together with Random Forest, Naïve Bayes, J48, and Convolutional Neural Networks (CNN), was exploited by the proposed system by Hashi et al. (2021) as a real-time flood detection system. This study aimed to provide an efficient and cost-effective solution for flood-prone areas, such as Somalia, by interfacing Arduino-based systems with GSM modems for real-time flood monitoring. The experimental results show that Random Forest outperforms the other classifiers with an accuracy of 98.7%, whereas Naïve Bayes and J48 have 88.4% and 84.2% accuracy, respectively. The deep learning-based CNN approach achieved an accuracy of 87%, showing high precision and recall values. Hence, this study contributes a valuable and visible application to the fields of Artificial Intelligence, Data Mining, and Deep Learning as an innovative solution in flood detection and early warning systems. To better ascertain the accuracy of flood prediction, researchers have extensively applied different types of deep learning techniques. Stateczny et al. (2023) proposed a new hybrid deep model for flood prediction, called the DHMFP, which was presented while being trained based on the combined Harris Hawks Shuffled Shepherd Optimization (CHHSSO) algorithm. This study aimed to increase the accuracy of traditional flood detection methods, especially for urbanized regions such as Kerala, where drainage systems are not capable of handling the torrent of rainwater. The methodology involved the preprocessing of satellite images

using median filtering and segmentation with cubic chaotic map-weighted K-means clustering. To strengthen feature representation, different vegetation indices, such as the Difference Vegetation Index (DVI), Normalized Difference Vegetation Index (NDVI), and soil-adjusted vegetation index (SAVI), were extracted. The extracted features were classified in a hybrid manner based on a CNN-Deep ResNet framework fine-tuned using weight optimization by CHHSSO. The experimental results showed high performance, with a sensitivity of 93.48%, specificity of 98.29%, accuracy of 94.98%, false negative rate of 0.02%, and false positive rate of 0.02%. The DHMFP-CHHSSO showed improved sensitivity, specificity, and accuracy of 0.932, 0.977, and 0.952, respectively, further validating the model's efficacy in terms of flood prediction. Hasan et al. (2018) proposed a Deep Convolutional Neural Network (DCNN) that detects Burst Header Packet (BHP) flooding attacks in Optical Burst Switching (OBS) networks. This study presents the criterion that the existing methods, such as Naïve Bayes, K Nearest Neighbors (KNN), and Support Vector Machines (SVM), are not sufficient because they become ineffective when the number of samples is small in the dataset. The proposed DCNN model outperformed these traditional methods by creating a very early scenario for attack identification. The experimental results also proved that DCNN provided a classification accuracy of 99%, which was much better than KNN (93%), SVM (88%), and Naive Bayes (79%). The sensitivity, specificity, precision, and F1-score were also 99%, while both the false positive rate (FPR) and the false negative rate (FNR) were only 1%. These studies and several others have found that most traditional ML models exhibit overfitting and misclassification. In contrast, the DCNN showed a constant level of performance across both the training and validation dataset conditions. This showed that deep learning models were effective in applying network anomaly detection, demonstrating the benefits of DCNN over traditional classification techniques. Tuyen et al. (2021) introduced a newly designed deep learning architecture called PSO-UNET to enhance flash flood segmentation from satellite images. This model integrates Particle Swarm Optimization (PSO) and UNET to optimize the segmentation accuracy, thereby optimizing the number of layers and layer parameters. Instead of maintaining the same symmetrical architecture usually observed in conventional UNET models, the ultimate difference in the proposed PSO-UNET is that it dynamically modifies the contracting and expanding paths for optimal performance. The model was tested on a dataset consisting of 984 satellite images and compared with other deep learning models such as UNET, LINKNET, and SEGNET. The results of the experiment demonstrated that the model achieved an F1 score of 87.17% ± 0.36%, which is 8.59% greater than that of the original UNET model. In addition, the model exhibited better performance in terms of the Dice Coefficient and Intersection over Union (IoU). Although the authors highlighted a very good performance in terms of segmentation accuracy, they found some slight errors owing to related pixel features. They suggested that post-processing techniques should be supplemented, and further validation should be performed on datasets that are more varied. This study contributes to the development of an optimized UNET-based segmentation model, demonstrating the extent to which evolutionary algorithms can achieve in the field of deep learning-based flood detection. SegNet, UNet, and FCN32 carried out floodwater segmentation of 290 flood-affected images in Bahrami & Arbabkhah (2024), and the study aimed to improve the accuracy of flood detection using deep learning models. Among these, SegNet achieved the highest precision of 88% and validated its efficiency in locating water areas in the images. This study emphasizes the importance of deep learning in enhancing flood forecasting and disaster response. Flood-ResNet50, as proposed by Khan et al. (2023), was developed with an optimized deep learning model architecture intended mainly to detect floods in UAV images while maintaining an excellent trade-off between performance and computational cost. After modifying the enhancements of ResNet50 through transfer learning and additional layers in the model architecture, a classification accuracy of 96.43% was attained, which was significantly higher than comparable larger models such as VGG16/19 and DenseNet161. Experimental results showed that it outperformed conventional models in terms of inference speed and power consumption through the edge device, thus recommending it as a real-time flood detection solution. Deep learning models have been extensively utilized in flood prediction and frequency assessment, as shown by Pandey et al. (2023). Conventional statistical techniques and traditional forecasting approaches can hardly capture any nonlinear interactions among flood variables. A Cat Swarm Optimized Spatial Adversarial Network (CSO-SAN) was proposed for flood forecasting that combines real-time meteorological and hydrological data. Studies have also proven that CSO-SAN is by far better than the rest, achieving an accuracy of 98.3%. Despite its effectiveness, it could be improved by applying hyperparameter tuning and additional machine learning techniques for further tuning. Urban flood monitoring is hampered by insufficient runoff data, which leads to a loss of hydrological model and early warning system accuracy. With recent advances in deep learning, image recognition has become a significant approach for flood measurement. Studies have proven that YOLOv4 works well during floods in identifying submerged objects,

such as vehicles and pedestrians, with a mean average precision of 89.29% % for flood depth recognition. Depending on the reference object used, this method can provide higher accuracy in the results, where vehicles provide better results than pedestrians. In addition, image augmentation methods, such as Mosaic, have been proposed to increase recognition accuracy. This presents an economical option for existing traffic cameras to be put to effective use, eliminating the need for further infrastructure, as in Zhong et al. (2024). The conventional method of detecting floods using SAR images has its own set of challenges, such as speckle noise and image distortions. To overcome these limitations, WDNet fuses CNN with a self-attention mechanism to enhance spatial and channel-wise feature extraction. WDNet performs better than conventional methods in terms of accuracy, with an F1 score of 0.987 on the Poyang Lake flood dataset. This model (Huang et al. 2024) thus aids in real-time flood mapping and disaster management. Convolutional neural networks, particularly U-Net and FCN, have been implemented on remote sensing data to conduct flood mapping in the Kan Basin in Tehran. Compared to the FCN, the U-Net achieved a better performance with an accuracy of 88% and a much higher mIoU of 0.65, demonstrating its application for flood detection. The research by Roohi et al. (2025) shows the efficiency of applying AI-based geospatial analysis in improving flood monitoring and disaster management.

MATERIALS AND METHODS

Fig. 1 describes the flow of the flood detection and analysis, with alerts generated on the website. The architecture consists of the following key components:

Customized Database

The 650 annotated images in the dataset used in this study are divided into two classes: "Flood" and "Not Flood." Three main sources were used to create the customized dataset: curated datasets on Kaggle, publicly accessible images obtained from Google Images, and a subset of labeled flood images sourced from the FloodIMG dataset suggested by Karanjit et al. (2023). Keyword-based searches (such as "flooded roads," "urban flooding," "dry street," etc.) were used to gather the images, and then duplicates, watermarks, and low-resolution photos were manually filtered out. All photos were manually annotated based on visible indicators of flooding (such as water accumulation, submerged vehicles, or muddy roads) or the lack of flooding to guarantee label accuracy. Only images with unambiguous visual proof and reviewers' agreement were included after labeling by the authors. A combination of street-level and aerial views is included in the dataset, along with a variety of environmental features such as lighting, weather, and scene complexity. The visual context shows coverage from various urban and semi-urban regions, mainly from India and Southeast Asia,

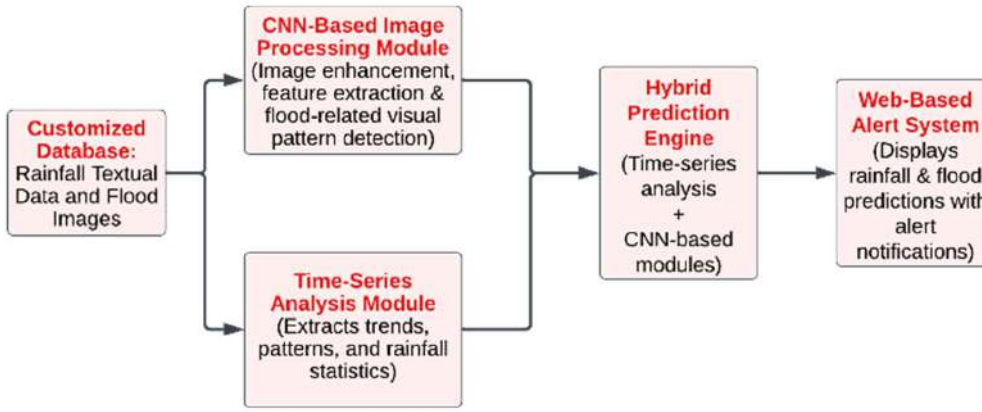

Fig. 1: Block diagram of flood detection system.

Table 1: Overview of dataset.

Dataset Type	Source(s)	Size/Duration	Key Features	Purpose
Image Dataset	Google Images, Kaggle, FloodIMG (Karanjit et al. 2023)	650 images	Annotated as "Flood" / "Not Flood", includes aerial and ground-level perspectives.	Used for flood classification using CNN
Rainfall Dataset	Kaggle – *Guwahati Weather Data (1973–2023)*	50 years (1973–2023) daily	tempmax, tempmin, precip, humidity, wind, conditions, icon, and more	Used for trend analysis and hybrid prediction

with a smaller number from Europe, even though precise geolocation metadata were not available for all images. By integrating multimodal inputs and visual diversity, this dataset construction method enhances the system's capacity to generalize across various flood scenarios.

The "Guwahati Weather Data (1973–2023)" dataset on Kaggle, which offers more than 50 years of daily weather records from Guwahati, a city vulnerable to seasonal flooding, was the source of the textual (temporal) dataset. Numerous meteorological features are included in this dataset, including the highest and lowest temperatures, dew point, humidity, precipitation (precip, precipprob, and precipcover), wind direction and speed, solar radiation, UV index, and symbolic weather descriptors such as conditions, icons, and descriptions. Preprocessing included classifying weather conditions into four symbolic types: clear, cloudy, partly cloudy, and rainy; handling missing values through interpolation; and eliminating outliers using interquartile range-based filtering. Seasonal decomposition and long-term trend analyses were performed on the refined dataset, which allowed the system to align rainfall anomalies with flood image patterns and identify periods that are prone to flooding. The hybrid prediction engine used rainfall trends as a temporal input, which improved the system's capacity to identify floods by utilizing both historical climate context and visual features. An overview of the dataset is presented in Table 1.

CNN-Based Image Processing Module

The CNN-Based Image Processing Module is a deep learning-based image classifier that analyzes flood-related images and classifies them into two categories: "Flood" and "Not Flood." Deep learning models were applied to a dataset of 650 flood images that were marked using TensorFlow and Keras. Four classic deep learning networks, EfficientNetB0, ResNet50, InceptionV3, and MobileNetV2, were initially tested, and InceptionV3 and MobileNetV2 were chosen for their high performance. The residual Network (ResNet50) is a very deep CNN model originally developed to avoid the problem of vanishing gradients by embarking on a road of residual learning. The model was designed to provide smooth gradients during the course of backpropagation with the help of shortcut connections, and thus, effective convergence. The application of ResNet50 for flood detection yielded a fairly moderate accuracy of 64.7%, primarily attributed to its stickiness towards overfitting on a very small dataset. However, it was capable of adequately capturing the hierarchical features of flood imagery; its high computational complexity hindered its adoption in streaming, as extensive GPU resources were required. There is room for improving the performance of this model using data augmentation techniques and larger and more diverse datasets.

Architecture InceptionV3 is another model in the competition for multi-scale feature extraction based on a factorized convolution design with asymmetric kernel designs. The fundamental equation that drives factorized convolutions is:

$$F(x) = f1(x) * f2(x) \quad \ldots(1)$$

Where f1(x) and f2(x) are two separate convolution operations, reducing computational complexity while preserving feature extraction capabilities.

The total number of parameters is given by:

$$P = (k^2 \cdot C_{in} \cdot C_{out}) + (C_{out} \cdot C_{in}) \quad \ldots(2)$$

Where k is the kernel size, C_{in} and C_{out} are the number of input and output channels, respectively.

These improvements in performance lead to improved computational effectiveness in reducing the number of parameters while maintaining very high accuracy. In flood classification, InceptionV3 achieved a high rate of 93%, marking it as one of the standout models in this study. Its strengths include the excellent capture of both local and global flood patterns. However, owing to its deep and complex architecture, the inference time was higher than that of MobileNetV2, making it less favorable for real-time applications where deciding in the moment was essential. EfficientNetB0 was established to maximize accuracy while maintaining efficiency by scaling its dimensions (i.e., depth, width, and resolution) with compound scaling factors. The compound scaling formula is as follows:

$$depth = \alpha^d,\ width = \beta^d,\ resolution = \gamma^d$$

Where α, β, γ are constants determined through grid search, and is a scaling coefficient. The overall computational cost (FLOPs) can be estimated as

$$FLOPs = 2 \cdot (C_{in} \cdot C_{out} \cdot k^2 \cdot H \cdot W) \quad \ldots(3)$$

Where H and W are the height and width of the input feature map.

It achieved high accuracy with a small number of parameters. Although EfficientNetB0 performed poorly in our flood detection study, with an accuracy of only %39, primarily due to the non-availability of high-quality, large-scale datasets for appropriate feature extraction, low-light conditions caused performance issues with flood classification, suggesting that extreme tuning and transfer learning adjustments are required. Nonetheless, despite its inefficiency, EfficientNetB0 remains a promising model for lightweight work, where power consumption is a

constraint. Among all the trained models, MobileNetV2 showed the best performance in terms of flood detection, achieving an accuracy of %94.36. Designed for mobile and edge devices, it uses depth-wise separable convolutions to reduce computation while maintaining a high classification performance.

$$Y = (X * D) * P \quad \ldots(4)$$

Where X is the input, D is the depthwise convolution, and P is the pointwise convolution.

The total computation cost can be approximated as:

$$FLOPs = H \cdot W \cdot C_{in} \cdot k^2 + H \cdot W \cdot C_{out} \cdot C_{in} \ldots(5)$$

This architectural choice allows MobileNetV2 to perform well in real-time applications while maintaining low computational costs. It is also designed with an inverted residual structure and linear bottlenecks, allowing for feature propagation and reducing redundancy. This aspect enables the architecture to easily process flood imagery for real-time detection with minimal resource consumption. It optimally balances accuracy, speed, and computational efficiency; hence, it is ideal for deployment in flood monitoring applications. The comparative analysis showed that deeper models, such as InceptionV3 and ResNet50, could extract complex flood-related features; however, most of them are not best suited for real-time usage because of the extreme space and time requirements. Despite being more efficient than some others, EfficientNetB0 struggled with the classification performance in this domain. Finally, MobileNetV2 was identified as the most suitable model because of its high accuracy with low computational requirements, thus being the best-suited candidate for implementation in the proposed system. This module integrates deep learning with real-time video analysis to cover the complete monitoring of floods as part of a larger predictive and alert system. In addition, by deploying a deep learning mechanism, the system can continue to improve the analysis of new flood imagery, indicating that the system is efficient and expandable in disaster scrutiny and handling.

Time-Series Analysis Module

To complement the image processing module, the Time-Series Analysis Module focuses on textual rainfall data, which extracts trends and patterns to understand seasonal variations. The analysis revealed significant rainfall concentrations between June and September, with Mawsynram receiving the highest rainfall. Coastal Karnataka follows at an average of 2973.5 mm, while the Konkan and Goa regions record 2804.2 mm on average. These temporal insights are critical for identifying regions prone to floods and periods of increased risk, which aid in the predictive capabilities of the system.

Hybrid Prediction Engine

At the heart of the architecture is the Hybrid Prediction Engine, which combines the outputs from the CNN-based image processing and time-series analysis modules. This engine combines spatial and temporal data using advanced machine learning models implemented in TensorFlow, Keras, and PyTorch. By fusing these two streams of data, the system achieves a robust and holistic prediction mechanism that ensures accuracy and reliability of the prediction.

Fig. 2: Insight's page.

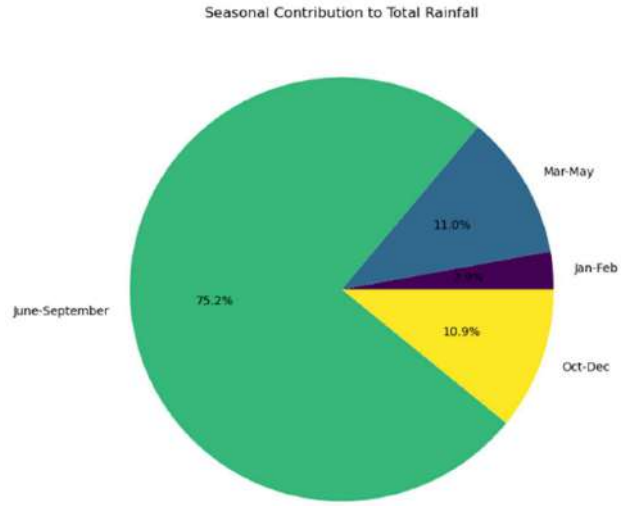

Fig. 3: Seasonal contribution to total rainfall.

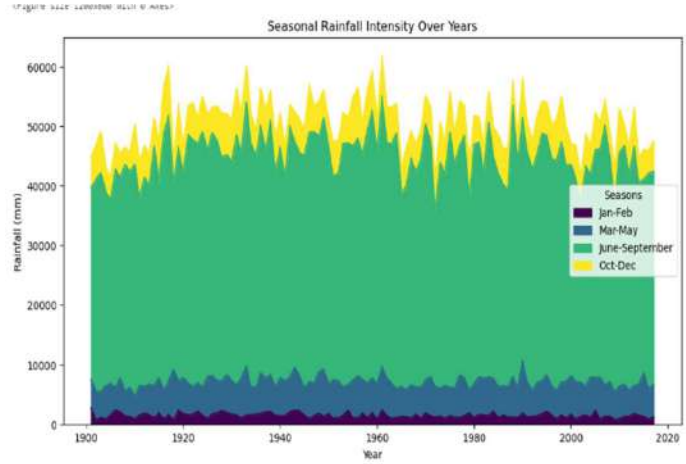

Fig. 4: Seasonal rainfall intensity over years.

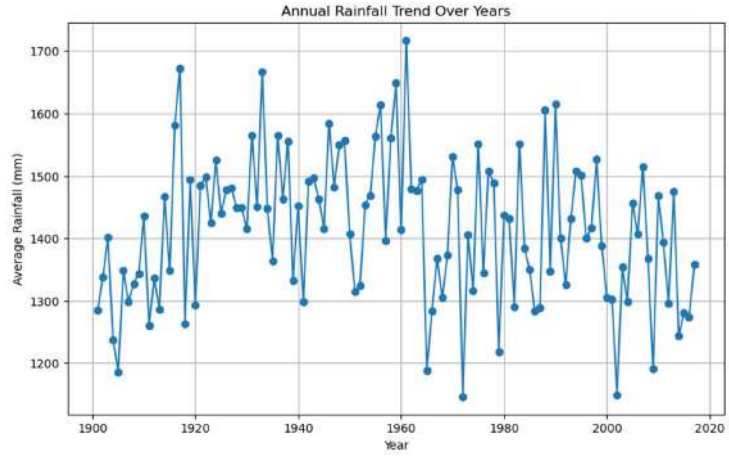

Fig. 5: Annual rainfall trend over years.

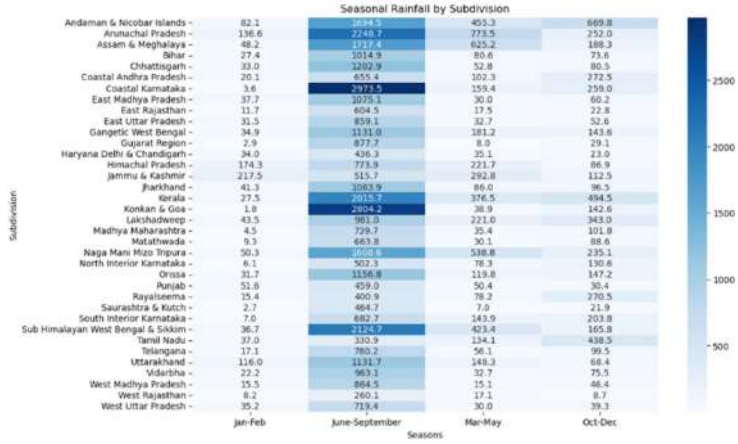

Fig. 6: Seasonal rainfall by subdivision.

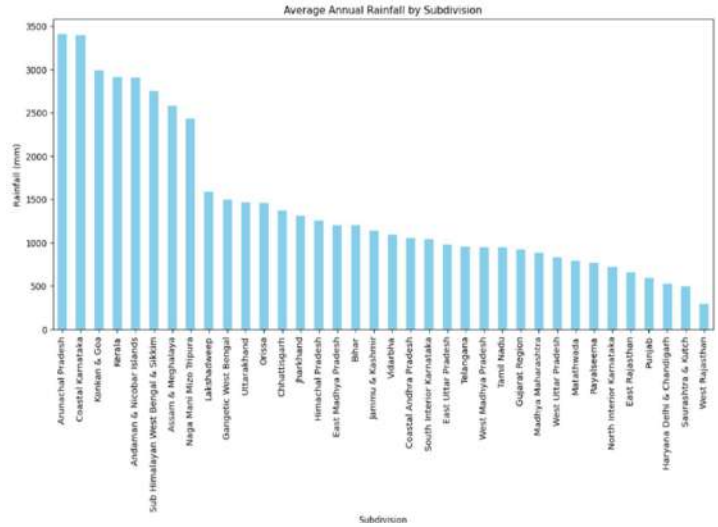

Fig. 7: Average annual rainfall by subdivision.

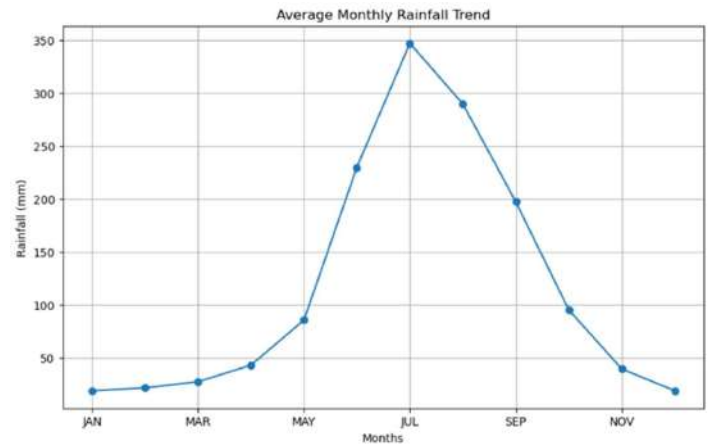

Fig. 8: Average annual rainfall by subdivision.

Analytical Insights and Decision-Support Integration

An analytical module called the Insights Page combines the flood prediction architecture of the system with long-term rainfall trend analysis. This module visualizes key hydrometeorological indicators, such as annual precipitation trends, seasonal rainfall contributions, and spatial rainfall distribution across subdivisions, using a 50-year time-series dataset of Guwahati weather (1973–2023) (Figs. 2–8). In addition to being descriptive, these visualizations provide important background information for analyzing flood risk across time and space. Annual trends (Fig. 5) show evidence of long-term variability possibly related to climate change, and the analysis of seasonal rainfall intensity (Fig. 4) aids in identifying flood risks driven by the monsoon. Localized flood preparedness planning is made possible by subdivision-level views (Figs. 6–8), which direct infrastructure planning and resource allocation in high-risk regions. The Insights Page features machine learning-based forecasting models that extrapolate future rainfall intensity under changing climatic conditions, in addition to static trend analysis. This feature increases the usefulness of the system as a real-time decision-support tool and facilitates early warning system calibration. By enabling cross-validation with the real-time image-based flood classification engine, these insights help stakeholders correlate detection alerts with past and forecasted weather patterns, thereby boosting confidence and lowering false alarms. It expands the system's usefulness beyond flood detection to include flood preparedness, policy support, and climate-resilient planning by converting unstructured meteorological data into organized and actionable visual analytics. It provides a framework for creating early warning systems, assisting with the optimization of urban drainage, directing the scheduling of agricultural operations, and facilitating more efficient emergency-response systems.

Table 2: Performance metrics of the proposed system.

Metric	Proposed System	Averaging Method
Precision	0.95	Macro-average
Accuracy	0.94	Macro-average
Recall	0.93	Overall accuracy

RESULTS AND DISCUSSION

The proposed flood detection methodology was tested using four of the most widely developed deep convolutional neural network (CNN) models. It is evident that the performance metrics, specifically the accuracy characteristics, significantly differ from model to model, indicating different strengths and weaknesses. The performance of the models was compared in the context of each city, and Table 2 provides a summary. The authors used 5-fold cross-validation on the training data to guarantee robustness and reduce the impact of variance caused by dataset split or model initialization. Five separate runs, each with retrained models and shuffled data, were averaged to produce the reported results (Accuracy, Precision, and Recall). The model's behavior was consistent and generalizable, as evidenced by the standard deviation of the performance metrics across the folds being within ±1.2%. The proposed system was tested on a dataset of 300 labelled images covering different types of images. Table 2 shows the key performance metrics achieved by the customized MobileNetV2 model, which includes precision and recall, which were calculated using macro-averaging across both classes, ensuring equal weights to both classes. All reported values represent the means across five validation folds.

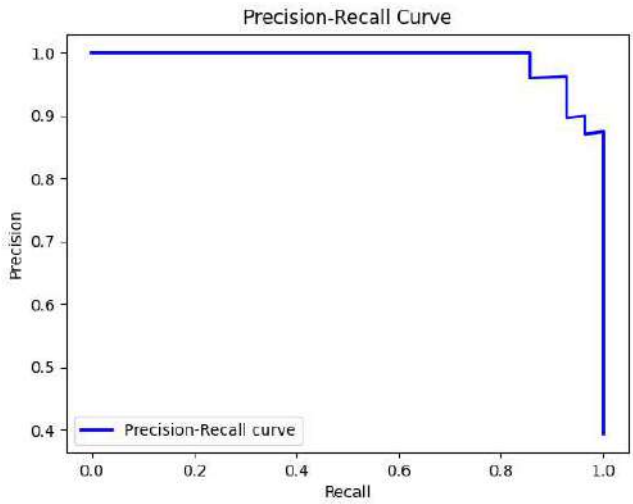

Fig. 9: Precision and recall curve.

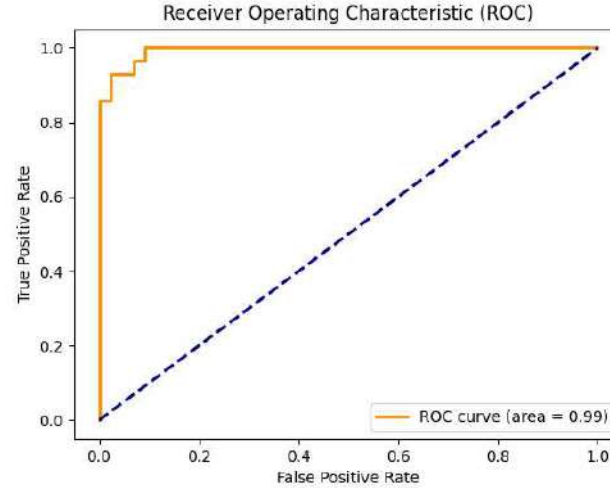

Fig. 10: Receiver operating characteristic (ROC) curve for the flood detection system.

The results obtained point out that the proposed system achieves much better accuracy and precision, reducing the chances of incorrect classifications. Fig 9 represents the Precision-Recall curve, which gives insight into how well the model works.

Fig. 10 shows that the high AUC of 0.99 indicates the reliability of identifying flood occurrences with minimal false alarms. Of the four trained models discussed in Table 3, MobileNetV2 was the fastest and most accurate model for flood detection in practical applications when

Table 3: Comparative analysis of sorting models.

Model	Accuracy (Mean + Standard Deviation)	Observations
MobileNetV2	94.36% ± 0.85%	Best performer, highly efficient for deployment with robust results
InceptionV3	93% ± 1.02%	Comparable to MobileNetV2; effective multi-scale feature extraction contributed to strong results.
ResNet50	64.7% ± 1.76%	Underperformed; potential challenges with dataset features or overfitting.
EfficientNetB0	39% ± 2.12%	Struggled significantly; requires fine-tuning or additional data preprocessing.

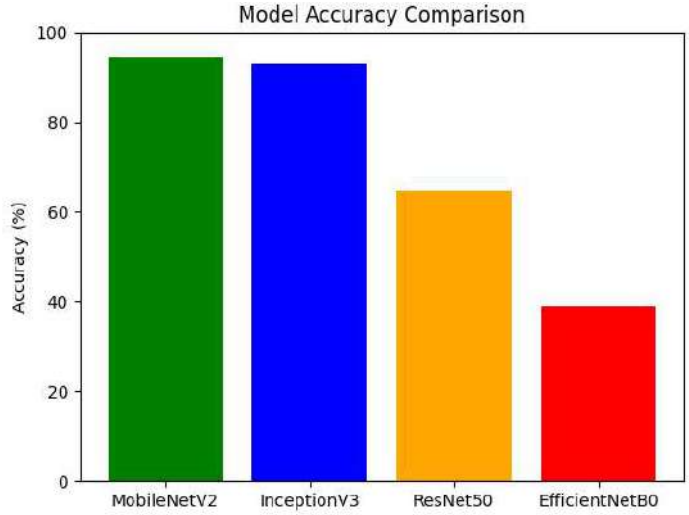

Fig. 11: Comparative performance metrics of different models.

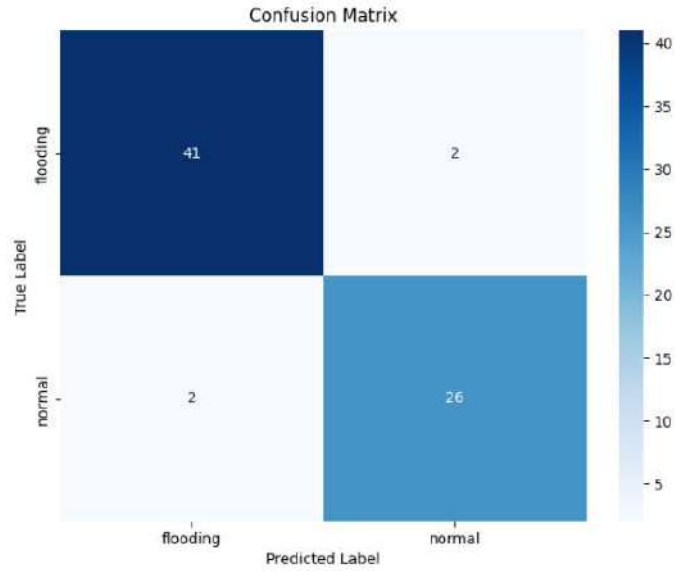

Fig. 12: Depicting the confusion matrix of the research.

both speed and accuracy were important. InceptionV3 also provided adequate results with a slight difference and excellent results in cases that required multiscalar analysis. However, ResNet50 and EfficientNetB0 reported worse accuracy, underlining that there is still significant potential for architecture-tailored modifications and preprocessing of the dataset to increase model accuracy. These findings call for lightweight but strong models, such as MobileNetV2, to be adopted in flood detection systems, particularly in real-time disaster surveillance and risk evaluation applications. The same train-test split (80:20) was used to train and assess every model listed in Table 2, including MobileNetV2, InceptionV3, ResNet50, and EfficientNetB0. Furthermore, pre-trained ImageNet weights were used to initialize all models, and our flood dataset was used to fine-tune the final classification layers for binary classification. The authors applied the same early stopping criteria, input resolution (224×224), and preprocessing pipeline to all models. Using the validation set, a grid search was used to choose hyperparameters such as the learning rate (originally 1e-4), batch size (32), and number of epochs (30). To ensure statistical reliability and fairness, each model was trained five times using different random seeds, and the average performance was reported.

Fig. 11 compares the classification accuracy across different models, while Fig. 12 shows the confusion matrix, which provides detailed insight into the classification accuracy for different classes. The matrix shows high true positive rates across categories, validating the effectiveness of the model in distinguishing between flood types with minimal misclassification.

Comparative Analysis

Table 4 provides a summary of the reported accuracy of current flood detection models from the recent literature to put our model's performance in perspective. Yede et al. created a CNN-based flood detection system with an accuracy of 82%, whereas Hussain et al. (2024) reported a 92.5% accuracy rate using a deep learning approach on visual flood images. Using a dataset of custom images, our proposed MobileNetV2-based model achieved a classification accuracy of 94.36%. It is crucial to remember that these findings were derived from distinct datasets and experimental setups; as a result, the comparison is offered solely for qualitative purposes and is not intended to serve as a performance standard. These numbers demonstrate the overall advancements in deep-learning-based flood detection, but they should be viewed within the constraints of various scenarios, data sources, and verification procedures.

The suggested model uses Batch Normalization and Dropout to enhance training stability and generalization. By normalizing the activations across mini-batches, batch normalization reduces the internal covariate shift during training, resulting in more stable learning, shorter training times, and enhanced performance in a variety of environmental conditions that are frequently present in flood imagery (e.g., varying lighting, water reflections, and shadows). This is particularly advantageous when training on datasets of moderate size, such as those used in this study. To avoid overfitting, dropout was used concurrently at a rate of 0.5. By forcing the network to learn distributed and generalized representations during training instead of

Table 4: Comparative analysis of research.

Model	Accuracy	Reference
Hussain et al. (2024) – Deep learning on visual images	92.5%	Hussain et al. (2024)
Yede et al. – CNN-based flood detection (Original Paper)	82%	Yede et al. (2021)
Our Work (MobileNetV2, Custom Dataset)	94.36%	-

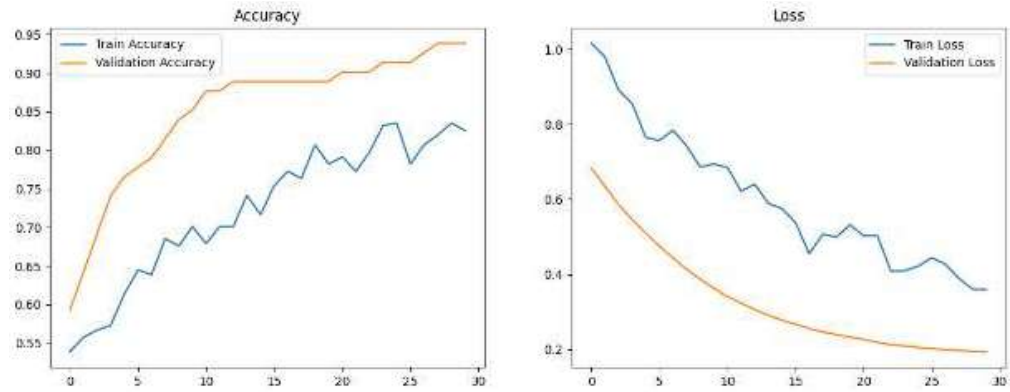

Fig. 13: Training and validation accuracy and loss curves over 30 epochs.

memorizing patterns, the random deactivation of neurons increases the network›s resilience to novel flood scenarios. These methods help the model minimize false positives while maintaining high precision and recall, which is essential for implementation in flood-risk assessment systems. Effective training, competitive performance, and the possibility of real-time applications are made possible by combining these methods in a lightweight architecture, such as MobileNetV2. To assess the model›s resilience on more extensive and geographically varied datasets and determine whether real-time deployment is feasible through field testing or edge computing simulations, further research is necessary.

CONCLUSIONS

This study proposed a hybrid flood detection system that combines long-term rainfall trend analysis with deep learning-based image classification. The system used a 50-year Guwahati weather time-series dataset and a customized dataset of 650 annotated photos. With a classification accuracy of 94.36% and macro-averaged precision and recall of 0.95 and 0.93, respectively, MobileNetV2 outperformed the other models in the tests. The robustness of the model, with minimal variance across the folds, was validated using cross-validation. Because dropout and batch normalization were used, the learning curves showed minimal overfitting and good generalization performance. Learning curves, which plot the accuracy and loss of training and validation over 30 epochs, were used to further assess the training behavior of the system (Fig. 13). The model generalizes well to unseen data with little overfitting, as shown by the curves' stable convergence, validation accuracy stabilizing between 93% and 94%, and a validation loss of approximately 0.2. This stability was facilitated by methods such as dropout regularization and batch normalization. The system's decision-support component incorporates the rainfall analysis module's insightful information on historical precipitation trends, regional variances, and seasonal rainfall intensity. By adding the Insights Page, stakeholders were able to better understand situational awareness by interpreting flood alerts based on past climatic conditions. Overall, the findings support the viability and efficiency of integrating temporal and visual data for detecting floods. To improve predictive capabilities, future work will focus on expanding the dataset, adding multi-region weather data, and enhancing model performance using sophisticated temporal models.

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The Long-term Anthropogenic Processes' Effects on Ecological Footprints in Morocco: A STIRPAT Analysis Based on Four Co-integration Approaches

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ABSTRACT

Morocco provides a stark example of how a developing country in the southern hemisphere of Africa is struggling with the diverse and devastating impacts of climate change, which are exacerbated by development issues and a lack of studies that allow for understanding the causal effects of environmental degradation, a crucial factor in informing adequate policy responses. An exhaustive STIRPAT analysis, conducted in Morocco from 1970 to 2023, uses four pieces of empirical evidence and four cointegration methods: ARDL, FMOLS, DOLS, and CCR. The increase in ecological footprints of production, consumption, import, and export in Morocco is due to urbanization, technical progress, trade openness, and economic growth, respectively. Anthropogenic processes, attributed to urbanization, economic growth, technological progress, and trade openness, have a positive contribution to environmental alteration and have been found unsustainable in the Moroccan context. Thus, relevant policies are being proposed at the individual, organizational, and governmental levels to reduce their environmental burden, increase bio-capacity regeneration potential, and promote environmental sustainability both in Morocco and beyond.

INTRODUCTION

Identifying comprehensive target variables that account for all aspects of environmental degradation is a challenging task. It is even more challenging in an African emerging country in the southern hemisphere that is struggling with the devastating impacts of climate change, which are exacerbated by development issues and a lack of studies that enable understanding of the causal effects of environmental degradation, a crucial factor in informing adequate policy responses. From this perspective, the ecological footprint (EF) is a synthetic concept that quantifies the anthropogenic impact on the biosphere, resulting in environmental stress (Wackernagel & Rees 1996, Galli 2015, Nautiyal & Goel 2021, Global Footprint Network 2024).

EF helps you understand how humans, driven by economic affluence, energy consumption, and land use for living space and agriculture, affect the environment (Dietz et al. 2007, Rafindadi & Usman 2020). It characterizes the pace and intensity of resource consumption and waste generation relative to the local ecosystem's ability to absorb waste and replenish resources, as represented by the area needed to support the population's needs and offset equivalent consumption and CO_2 emissions (Global Footprint Network 2024).

When attributed to the production of harvests, crops, grazing land, vegetation, fibers, farming and fisheries, woods, medicinal plants, etc., as well as space for

urban infrastructure within a country's borders, it is referred to as the ecological footprint of production (EFP) (Global Footprint Network 2024). It specifically tracks the use of productive surface areas, including cropland, grazing land, fishing grounds, built-up land, forest area, pastureland, factories, cities, etc., and CO_2 demand on land (Global Footprint Network 2024).

When materialized within imports, it is referred to as the EF of imports (EFI); within exports, it is called the EF of exports (EFE). This, and the EF, often refers to the apparent EF of consumption (EFC). Sometimes EF is briefly referred to as a footprint, which is the sum of EFP and EFI minus EFE.

In summary, a country's EF represents the total pressure that the population's needs place on ecosystems, including the atmosphere, soil, and subsoil, and, by extension, the demand on biodiversity (Global Footprint Network 2024). It is measured in global hectares (gha), which represent a biologically productive hectare with average bio-productivity, adjusted for the demand on a specific geographical zone each year (Global Footprint Network 2024).

Like many emerging countries, Morocco's EF has impressively grown in the last 50 years as a result of increasing local demand for bio-capacity due to eco-demographic growth and urban expansion, which is putting pressure on the local biosphere by excessive water usage, intensive farming, overgrazing, and deforestation with an overload of anthropogenic CO_2 emissions. This led to anthropogenic incidence on climate change, air pollution, water scarcity, and contribution to global warming (Hamdi & Mohamed 2025).

The country's EFC rose by more than an entire point from 0.91 in 1961 to 1.94 gha per inhabitant in 2023, while BC per capita fell from 1.08 to 0.81 gha per capita for the same period (Dworatzek et al. 2024).

Morocco's economy relied for decades on primary sector where agriculture, mining, fishing and forestry sectors account for 15% of GDP, and employs about 45% of Morocco's active Population (Morocco - Agricultural Sector 2024), with time, the modernization and intensification of agricultural practices have caused soil erosion, salinization and resource attrition, affecting about 5.5 million hectares of land, leading in fine in up and down wards in EF and bio-capacity, respectively (Bouhia 2020).

Morocco is rich in biodiversity, hosting the second-highest concentration of terrestrial biodiversity in the Mare Nostrum (Bouhia 2020). This biodiversity is being threatened by the sabotage of its own homeland, which is caused by the overexploitation of natural resources, deforestation, desertification, air pollution, stream pollution, and soil degradation (Bouhia 2020). As a result, local BC meets only half of Morocco's total EFC; the country met its deficit by relying on 20% net BC imports (Galli 2015).

Although Morocco places its greatest demands on its cropland ecosystem, which provides provisioning services, including agricultural products, crop-based feeds, and fibers, mostly used (45% of total EFC) to produce food, goods, and services (Galli 2015), it remains far from achieving self-sufficiency and meeting local consumption levels, which makes it increasingly reliant on imports to meet its population's nutritional and energy needs. Notably, Morocco is a net importer of all ecosystem services tracked by the EF (Galli et al. 2012, Galli 2015).

The key aspect of innovation and distinction of this study relies on three key aspects:

One key aspect is the exhaustive analysis of the association between various anthropogenic stress factors and four interconnected environmental degradation indicators from the footprint family, imputed to production, consumption, import, and export, respectively, under spanning coverage that outpaces 50 years, from 1970 to 2023.

The second key aspect is that it innovates in the choice of the environmental stress, mainly with ICTs as a proxy of technological progress. Hence, it takes into account trade openness as a control variable, due to Morocco's increasing integration into the global supply chain, and the potential for environmental diffusion stress between countries due to commercial transactions.

The third key aspect is related to the nature of the target key indicators, which reveal complementarities, dualities, and asymmetries between consumption and production, and import and export. This involves the use of four well-known and widely validated co-integration approaches for statistical analysis worldwide.

LITERATURE REVIEW

In the realm of environmental sustainability, STIRPAT (as Stochastic Incidences by Regression on Population, Affluence and Technology) (Aguir et al. 2014) serves as a framework for analysing the interplay between environmental quality, economic affluence, Population dynamics, industrialization, and technological advancements (York et al. 2003). This model allows researchers to quantify how these factors contribute to environmental degradation and sustainability outcomes. The STIRPAT model builds upon the earlier IPAT model by introducing a stochastic element that accounts for uncertainties in data and relationships, pioneered by Ehrlich & Holdren (1971). Then duplicated to a variety of versions, such as the "I(m)PACT(s)" identities (Vélez-Henao et al.

2019, Waggoner & Ausubel 2002, Lin et al. 2009, Vélez-Henao et al. 2019, Hamdi & Mohamed 2024), or the "IP(B)AT" identity (Vélez-Henao et al. 2019, Schulze 2002).

Under the STIRPAT framework, a range of environmental barometers has been examined in numerous studies, in relation to a variety of explanatory variables, including economic growth, human capital, bio-availability, energy use, renewable energy, urbanization, financial inclusion, trade openness, demographics, natural resource attrition, governance and institutional quality.

For example, CO_2 emissions have been used by Bélaïd and Youssef (2016), Bekun et al. (2018), Abbasi et al. (2021), Mirziyoyeva et al. (2022), Raihan & Tuspekova (2022), Zhao et al. (2023), Ullah et al. (2023), Asli et al. (2024), Naz et al. (2024), and Ullah & Lin (2024) to examine factors influencing CO_2 levels, often in relation to economic growth and energy patterns.

Sulfur dioxide (SO_2) emissions have been explored in more recent analyses, notably by Wong et al. (2024) and Xu et al. (2024), while nitrous oxide (N_2O) has been investigated by Seangkiatiyuth et al. (2011), Tian et al. (2018), and Casquero-Vera et al. (2019), focusing on emissions related to agriculture and industrial activity.

Similarly, nitrogen oxides (NO_x) emissions have been examined by Tørseth et al. (2012) and Shaw and Van Heyst (2022), addressing concerns over transportation and industrial emissions.

When it comes to greenhouse gases (GHGs) more broadly, studies by Sarkodie and Strezov (2018), Chen et al. (2021), Tsur (2024), and Ochi and Saidi (2024) provide comprehensive assessments of how black emissions drivers across different national and sectoral contexts.

Lastly, particulate matter (PM) has drawn attention in the works of Griffin (2013) and Yun et al. (2022), often highlighting health implications and links to urbanization and fossil fuel use.

These studies collectively underscore the multifaceted nature of environmental degradation and the broad array of variables influencing ecological and atmospheric quality. However, these barometers have been criticized for lacking thoroughness and inclusiveness (Destek et al. 2018, Altıntaş et al. 2020, Usman et al. 2020, Nathaniel et al. 2020, Ramezani et al. 2022, Sun et al. 2023, Aziz et al. 2022, Ullah et al. 2023, Hamdi & Mohamed 2024) and for being insufficient in measuring decarbonization (Shaw & Van Heyst 2022).

Recently, EF has increasingly been employed as a reliable and multifaceted environmental barometer (Galli et al., 2014) for environmental assessment, monitoring, and policy evaluation (Rafindadi & Usman 2020). Numerous studies have utilized EF as a barometer, such as Hamdi and Mohamed (2024), Padhan & Bhat (2024), Zhou et al. (2024), Farouki and Aissaoui (2024), Mehmood et al. (2023), Li et al. (2023), Xu et al. (2022), Yasmeen et al. (2022), Rafique et al. (2021), Ali et al. (2021), Chen et al. (2021), Okelele et al. (2021), Nathaniel et al. (2020), and Ahmed & Wang (2019), who commonly employed economic growth, demographic tendencies, natural resource rents, and energy patterns such as composition and consumption as central variables that tend to increase environmental pressure.

In national contexts, such as Turkey, Ullah et al. (2023) utilized an ARDL model covering 1970–2018 to prove that economic growth, bio-capacity, urbanization, and natural resources all have a positive impact on EF, implying a linear environmental cost to development.

Similarly, in Pakistan, Ullah & Lin (2024) used an NARDL method analysis from 1990 to 2018, revealing that natural resource rents and economic growth significantly contributed to increasing EF, renewable energy consumption had a mitigating effect.

Interestingly, financial inclusion appears as a recurring variable in more recent literature. In Algeria, for example, Bergougui & Aldawsari (2024) identified inclusive finance as a positive force in managing ecological risks, potentially by enabling green investments and reducing dependency on resource-intensive activities.

In China, Xu et al. (2022) applied FMOLS, DOLS, CCR, and spectral causality techniques over the period of 1990–2017 to conclude that technological advancement and renewable energy use impede EF level in the long run, whereas FDI expedites it.

Back to Morocco, by using ARDL and VAR/VECM cointegration models, it was previously proven that between 1980 and 2022, economic growth, urbanization, and energy use led to an increase in EF, alongside the confirmation of the EKC hypothesis, whereas ensuring advanced education reduced it (Hamdi & Mohamed 2024, Farouki & Aissaoui 2024).

In subnational contexts, for example, financial inclusion, economic growth, urbanization, and natural resource rents were found to significantly increase EF in the ECOWAS, according to estimations using different panel regression methods over 1990-2016 (Ali et al. 2022).

Moreover, in the South Asian context, Mehmood et al. (2023) confirmed this causality-effect link, finding that, from 1990 to 2022, urban and economic growth, as well as human capital and bio-capacity, positively contribute to EF using panel co-integration approaches. Furthermore, it was

captured the negative impact of FDI and the mitigating role of green innovation on EF in the context of the BRICS and Next-11, from 1992 to 2018 by Padhan and Bhat (2024).

Nevertheless, the contribution of FDI and trade to EF remains controversial, with conflicting findings: while a 1991-2012 DOLS panel data analysis of the 27 highest emitting countries revealed a negative impact (Uddin et al. 2017), a robust 53-panel regression investigation from 1990 to 2021 in the Belt and Road Initiative regional context found a positive relationship between trade and EF for both imports and exports (Zhou et al. 2024), which is confirmed in the Sub Saharan context, where Okelele et al. (2021) found that EFC per capita decreases with an increase in trade openness and increases with an increase in FDI inflows from 1990 to 2015.

Furthermore, Ahmad et al. (2020) employed the second-generation panel co-integration approach from 1984 to 2016 to find that natural resources and economic growth expand the EF, while technological innovations reduce it, all within the presence of the EKC hypothesis.

In summary, these studies converge on the conclusion that economic growth, urbanization, and natural resource exploitation significantly amplify environmental degradation in developing regions. However, the integration of renewable energy, improvement in institutional quality, and expansion of financial inclusion offer promising pathways toward sustainability. This narrative underscores the urgency of adopting holistic, context-sensitive policies that align economic ambitions with environmental stewardship.

As a continuation, this study aims to provide a plausible clarification of the following problem:

What are the long-term anthropogenic processes' effects, associated with urbanization, economic growth, technological progress, and trade openness on Morocco's EFs of consumption, production, import, and export from 1970 to 2023?

To bring a response to this problem, the following hypothesis is going to be verified:

H: Anthropogenic processes imputed to urbanization, technological progress, economic growth, and trade openness have a positive effect on the EF's four economic varieties.

This hypothesis is split into four sub-hypotheses, following our four econometric models

H_a: Anthropogenic processes have a positive impact on EFP

H_b: Anthropogenic processes have a positive impact on EFC

H_c: Anthropogenic processes have a positive effect on EFE

H_d: Anthropogenic processes have a positive influence on EFI.

MATERIALS AND METHODS

Model Construction

This study relies on STIRPAT, in line with our previous papers (Hamdi et al. 2024, Asli et al. 2024, Hamdi & Mohamed 2024, Hamdi & Mohamed 2025), with this specification:

$$I = a \cdot P^b \cdot A^c \cdot T^d \cdot e$$

Where I is incidence on environment, P is Population dynamics, A denotes affluence, T stands for technology, a, b, c, and d are coefficients that represent the elasticity of each factor, and e is an error term accounting for unobserved factors.

Accordingly, the following functional form is estimated: (EF) = f (URB, GDP, ICT, TRD)

From which are derived the following four specific functional models:

The EFP model: incidence on (EFP) = f (URB, GDP, ICT, TRD)

Table 1: Variables and data presentation.

STIRPAT	Variables	Acronym	Unit	Data source
I	Ecological footprint of production	EFP	Gha/ midyear population	The Global Footprint Network
	of consumption	EFC		
	of import	EFI		
	of export	EFE		
P	Urbanization	URB	Ratio	Urban population (% of total population) - Morocco | Data
A	Gross Domestic Product	GDP	Constant 2015 $	World Bank Open Data
T	Information and Communication Technologies	ICT	Integer	Adoption of communication technologies per 100 people, Morocco
Control variable	Trade openness	TRD	% GDP	World Bank Open Data

The EFC model: incidence on (EFC) = f (URB, GDP, ICT, TRD)

The EFI model: incidence on (EFI) = f (URB, GDP, ICT, TRD)

The EFE model: incidence on (EFE) = f (URB, GDP, ICT, TRD)

Where EF represents ecological footprint, EFC is the ecological footprint of consumption, EFP of production, EFI of import, EFE of export, URB is urbanization, GDP is Gross Domestic Product, ICT stands for the information and communication technologies, and TRD is trade.

Table 1 represents a description of the chosen variables and their corresponding determinants:

N.B: ICTs are closely linked to technological progress, acting as a driver and facilitator of innovation across sectors. ICTs enhance productivity, enable knowledge diffusion, and support the development of new products and services, thus accelerating economic and technological advancement (Vu 2011, Niebel 2017). They drive innovation and efficiency gains in industries, particularly through automation, digitization, and improved communication networks (OECD 2020). Hence, ICT infrastructure is foundational for emerging technologies such as artificial intelligence, the Internet of Things, and big data analytics, which are key components of modern technological progress (Castells 2010, Brynjolfsson & McAfee 2014).

The choice of trade as a control variable is justified by the fact that the EFI and EFE models take into account the exports and imports of goods and services, summed by Trade as %GDP.

Model Demonstration

By taking the functional form of the EFP model as an example, raising it to the natural log, and neglecting the error term, we get the following specifications:

$$LnEFP_t=\beta_0+\beta_1 LnURB_t+\beta_2 LnGDP_t+\beta_3 LnICT_t+\beta_4 LnTRD_t+\mu_t \quad \ldots(1)$$

The ARDL/BTA specification is expressed as:

$$LnEFP_t=\sum_{i=1}^{p}\beta_{0i}LnEFP_{t-i}+\sum_{i=0}^{q}\beta_{1i}LnURB_{t-i}\sum_{i=0}^{q}\beta_{2i}LnGDP_{t-i}\sum_{i=0}^{q}\beta_{3i}LnICT_{t-i}+\sum_{i=0}^{q}\beta_{4i}LnTRD_{t-i}+\delta_0 LnEFP_{t-i}+\delta_1 LnURB_{t-i}+\delta_2 LGDP_{t-i}+\delta_3 LnICT_{t-i}+\delta_4 LnTRD_{t-i}+\varepsilon_t \quad \ldots(2)$$

And at the first difference as:

$$\Delta LnEFP_t=\sum_{i=1}^{p}\beta_{0i}\Delta LnEFP_{t-i}+\sum_{i=0}^{q}\beta_{1i}\Delta LnURB_{t-i}\sum_{i=0}^{q}\beta_{2i}\Delta LnGDP_{t-i}\sum_{i=0}^{q}\beta_{3i}\Delta LnICT_{t-i}+\sum_{i=0}^{q}\beta_{4i}\Delta LnTRD_{t-i}+\delta_0 LnEFP_{t-i}+\delta_1 LnURB_{t-i}+\delta_2 LGDP_{t-i}+\delta_3 LnICT_{t-i}+\delta_4 LnTRD_{t-i}+\varepsilon_t \quad \ldots(3)$$

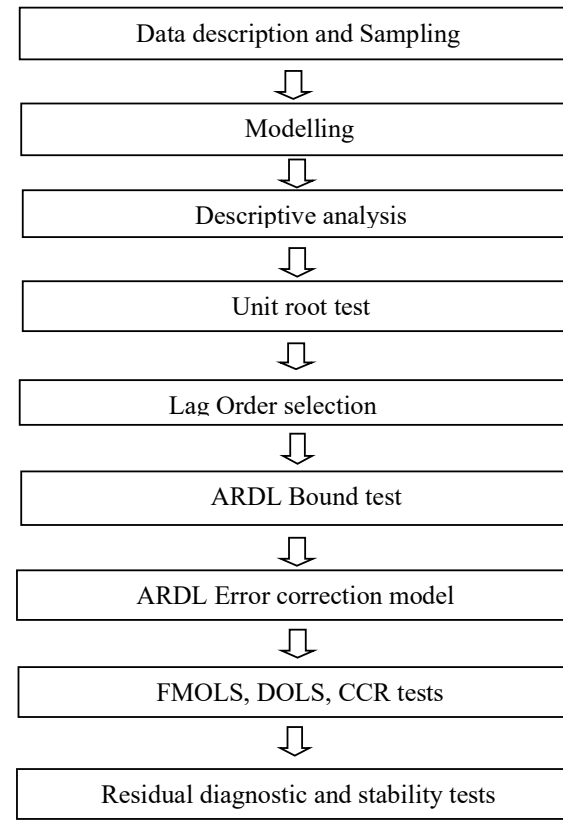
Where t represents the current period, t − i represents the previous period, Δ is the first difference operator, p and q are respectively the lags length for both dependent and independent variables, and the coefficients of the short and long run are shown through *b* and *d*, respectively, while ε_t represents the error term.

Two hypotheses are to be confronted: if there is no co-integration, as stipulates the null hypothesis (H_0: $\delta_1=\delta_2=\delta_3=\delta_d=0$) vs the alternative one (H_a:$\delta_1\neq\delta_2\neq\delta_3\neq\delta_4\neq 0$).

If there is co-integration, the error correction model (ECM) representation is specified as:

$$\Delta LnEFP_t=\sum_{i=1}^{p}\beta_{0i}\Delta LnEFP_{t-i}+\sum_{i=0}^{q}\beta_{1i}\Delta LnURB_{t-i}+\sum_{i=0}^{q}\beta_{2i}\Delta LnGDP_{t-i}+\sum_{i=0}^{q}\beta_{3i}\Delta LnFCE_{t-i}+\sum_{i=0}^{q}\beta_{4i}\Delta LnICT_{t-i}+\eta\ ECT_{t-1}+\mu_t \quad \ldots(4)$$

Where ECT represents the error correction term, η is its stochastic coefficient.



Source: Author's own elaboration.

Scheme 1: The study's methodological approach.

The three remaining models are constructed just the same way.

Methodology

This study employs the ARDL approach, supplemented by three other co-integration tools – FMOLS, DOLS, and CCR – to consolidate the reliability of the primary ARDL results. ARDL/ECM is well known for conducting long-run analyses of dynamic relationships between series with different orders of integration (Pesaran & Shin 1998, Pesaran et al. 2001), where the current value of the dependent variable depends on its own past realisations through the distributed lag part (Kripfganz & Schneider 2023). The advantage of this approach lies in identifying co-integrating vectors when there are multiple ones (Nkoro & Uko 2016).

The methodological approach followed in this study is schematized step-by-step in Scheme 1:

Descriptive Analysis

Descriptive analysis is conducted by four techniques: Descriptive statistics, Multiple Correlation Analysis (MCA), Variance inflation factors (VIF), and Pairwise Component Analysis (PCA) (Pearson 1901, Bonett & Wright 2000, Hamdi et al. 2024, Asli et al. 2024, Hamdi & Mohamed 2024, Hamdi & Mohamed 2025).

Unit Root Test

It is essential to verify data stationarity, ensuring statistical properties remain constant over time (Kwiatkowski et al. 1992). The ARDL method depends on the cointegration order of variables (Pesaran et al. 2001). This study employs the ADF test for that purpose (Dickey & Fuller, 1979).

Lag Order Selection

When the auto-regressive model is subject to restrictions of co-integration, there are multiple information criteria for selecting the appropriate lag order (Lütkepohl 1993). They all rely on selecting the lag length with the lowest value (Mallik 2008, Hamdi & Mohamed 2024).

ARDL Bounds Tests

An ARDL bounds test involves performing an F-test on the lagged levels of the independent variable (Nkoro & Uko 2016, Asli et al. 2024), compared with critical values at a 5% level of significance (Narayan 2005, Asli et al. 2024).

Error Correction Model

It can be derived from the ARDL model through a simple linear transformation, which integrates short-run adjustments with long-run equilibrium (Nkoro & Uko 2016, Hamdi & Mohamed 2024). Then it exhibits an associated error correction term (ECT) which measures how quickly the equilibrium is reached in the long run (Engle & Granger 1987, Asli et al. 2024).

FMOLS, DOLS, and CCR Tests

The FMOLS method, developed by Phillips and Perron (1988), is valued for handling endogeneity and serial correlation, especially in small samples (Hamit-Haggar 2012, Asli et al. 2024). DOLS, introduced by Stock and Watson (2003), often yields superior estimates by addressing regressor correlations (Kao, 1999, Asli et al. 2024). As a robustness check, the CCR approach (Pesaran et al. 2001) is also applied, modifying the model to improve chi-square test accuracy (Park 1992, Pattak et al. 2023, Asli et al. 2024).

Residual Diagnostic and Stability Tests

Residual diagnostics are essential for assessing a model's capability and providing directions for potential modifications (Mauricio 2008). The normal distribution of residuals was tested using Bera and Jarque's (1981) method, while heteroscedasticity was checked with Breusch and Pagan's (1979) test, and serial correlation was evaluated using Godfrey's (1978) test. Additionally, the Ramsey (1969) test was used to verify the existence of misspecifications in residuals. The quality of the regression is represented by the CUSUM and CUSUMSQ tests (Brown et al. 1975), and its stability is checked (Doan et al. 1994).

RESULTS

The results from the empirical evidence on the footprint models are presented jointly in a single table, divided into four cases, with each case representing a singular model's results, and each result is commented on underneath its corresponding table.

Descriptive Statistics

Table 2 below provides a statistical description summary of the four model variables.

Results from Table 2 show that the EFC and EFP show symmetrical distributions with moderate variation and pass normality tests, indicating stable and consistent patterns.

In contrast, the EFE is right-skewed and nearly non-normal, suggesting uneven environmental impacts across observations. The EFI, while more symmetric, shows considerable flatness and variability, though it still meets the normality threshold.

Among the explanatory variables, urbanization and trade openness display low variability and approximately

Table 2: Data descriptive statistics.

Parameter	LNEFC	LNEFE	LNEFI	LNEFP	LNURB	LNGDP	LNICT	LNTRD
Mean	17.37984	16.09276	-0.777442	17.30571	3.911890	26.98903	2.256376	4.052313
Median	17.41169	15.97666	-0.834063	17.30516	3.954809	24.64714	1.562475	4.000685
Max	18.10904	16.79392	-0.083382	17.91692	4.172152	37.10619	5.463832	4.616267
Min	16.44590	15.65578	-1.660731	16.70099	3.540292	23.48349	-0.564610	3.602211
Std. Dev.	0.482438	0.320997	0.435008	0.348433	0.183145	5.130629	2.367897	0.220808
Skewness	-0.171867	0.730638	0.031031	-0.054216	-0.456566	1.439369	0.201829	0.380473
Kurtosis	1.922932	2.410983	1.895937	1.884086	2.085151	3.124651	1.341788	2.820291
Jarque-Bera	2.876015	5.585102	2.751314	2.828297	3.759207	18.68102	6.553365	1.375505
Prob	0.237400	0.061265	0.252674	0.243133	0.152651	0.000088	0.037753	0.502705
Sum	938.5112	869.0090	-41.98185	934.5086	211.2420	1457.407	121.8443	218.8249
Sum Sq. Dev.	12.33558	5.461076	10.02931	6.434478	1.777728	1395.138	297.1676	2.584075

Table 3: The EFs models' pair–wise correlations matrices.

The EFP model

	LNEFP	LNURB	LNGDP	LNICT	LNTRD
LNEFP	1.000000				
LNURB	0.966667	1.000000			
LNGDP	0.740718	0.687033	1.000000		
LNICT	0.939598	0.926602	0.728291	1.000000	
LNTRD	0.797232	0.770152	0.715163	0.819587	1.000000

The EFC model

	LNEFC	LNGDP	LNICT	LNURB	LNTRD
LNEFC	1.000000				
LNGDP	0.735419	1.000000			
LNICT	0.951953	0.728291	1.000000		
LNURB	0.983926	0.687033	0.926602	1.000000	
LNTRD	0.811182	0.715163	0.819587	0.770152	1.000000

The EFI model

	LNEFI	LNGDP	LNICT	LNURB	LNTRD
LNEFI	1.000000				
LNGDP	0.767349	1.000000			
LNICT	0.958367	0.728291	1.000000		
LNURB	0.935901	0.687033	0.926602	1.000000	
LNTRD	0.847025	0.715163	0.819587	0.770152	1.000000

The EFE model

	LNEFE	LNGDP	LNICT	LNURB	LNTRD
LNEFE	1.000000				
LNGDP	0.789943	1.000000			
LNICT	0.855346	0.728291	1.000000		
LNURB	0.803808	0.687033	0.926602	1.000000	
LNTRD	0.752038	0.715163	0.819587	0.770152	1.000000

Table 4: Variance inflation factors.

Variable	Coefficient Variance	Uncentered VIF	Centered VIF
LNGDP	1.88E05-	70.57333	2.377818
LNURB	0.030778	2359.634	6.481630
LNICT	0.000269	13.55806	5.700806
LNTRD	0.013504	1111.770	3.797823
C	0.465502	2359.712	NA

normal distributions. GDP and ICT development, however, are highly skewed and non-normal, reflecting structural disparities in economic and digital development.

Overall, most EF indicators are well-behaved statistically, but special attention is needed when modelling variables like GDP, ICT, and EFE due to their distributional characteristics.

Pair-wise Correlation Analysis

Table 3 below illustrates the pair-wise correlation matrices of the four models:

Results from Table 3 suggest consistent correlation patterns. In all cases, environmental impact, whether from production, consumption, imports, or exports, is strongly associated with higher levels of urbanization and ICT development. These two factors exhibit the closest relationships, suggesting they are key structural drivers of ecological pressure. GDP and trade openness also show positive correlations across all models, though slightly weaker. Notably, EFC and EFI demonstrate the strongest overall associations with the explanatory variables, implying that lifestyle and external demand significantly contribute to environmental strain.

In sum, the results point to a shared dynamic: as economies urbanize, digitize, grow, and integrate into global trade, their EFs intensify, especially through consumption and imports. However, these strong correlations require variance inflation factors (VIF) analysis to check any potential multicollinearity concerns. The following Table 4 represents the VIF test outputs

The centered VIF results from Table 4 indicate mild to moderate multicollinearity among explanatory variables. LNGDP and LNTRD show low to acceptable levels (lower than 5), posing no concern. LNURB and especially LNICT exceed the common threshold of 5, signaling moderate multicollinearity. These values suggest, generally, the absence of extreme multicollinearity concerns (since lower

Table 5: ADF Unit Root Test.

At Level		LNEFP	LNEFC	LNEFI	LNEFE	LNURB	LNGDP	LNICT	LNTRD
Constant	t-Statistic	-0.2537	-1.3934	-1.2652	-0.9783	-2.8235	-0.3346	-0.5549	-1.2416
	Prob.	0.9242	0.5784	0.6391	0.7546	0.0619	0.9123	0.8713	0.6497
		n0	n0	n0	n0	*	n0	n0	n0
Constant & Trend	t-Statistic	-8.0492	-6.2846	-3.2571	-2.7545	-2.6791	-1.6683	-1.7760	-2.6892
	Prob.	0.0000	0.0000	0.0848	0.2202	0.2492	0.7514	0.7020	0.2451
		***	***	*	n0	n0	n0	n0	n0
Constant & Trend	t-Statistic	3.4863	4.7905	-2.3395	1.2006	-0.1897	1.1002	0.6494	1.2138
	Prob.	0.9998	1.0000	0.0200	0.9393	0.6131	0.9276	0.8533	0.9407
		n0	n0	**	n0	n0	n0	n0	n0
At First Difference		**d(LNEFP)**	**d(LNEFC)**	**d(LNEFI)**	**d(LNEFE)**	**d(LNURB)**	**d(LNGDP)**	**d(LNICT)**	**d(LNTRD)**
Constant	t-Statistic	-8.6947	-8.4567	-9.5928	-9.8459	-0.4186	-7.2269	-3.1646	-6.2138
	Prob.	0.0000	0.0000	0.0000	0.0000	0.8980	0.0000	0.0279	0.0000
		***	***	***	***	n0	***	**	***
Constant & Trend	t-Statistic	-8.6084	-8.5439	-9.5449	-9.9406	-1.9414	-7.3032	-3.0899	-6.1510
	Prob.	0.0000	0.0000	0.0000	0.0000	0.6187	0.0000	0.1195	0.0000
		***	***	***	***	n0	***	n0	***
Constant & Trend	t-Statistic	-12.9487	-11.8715	-8.8480	-9.6986	-1.2867	-7.1039	-2.3060	-7.3382
	Prob.	0.0000	0.0000	0.0000	0.0000	0.1804	0.0000	0.0217	0.0000
		***	***	***	***	n0	***	**	***

(*) $p < 0.01$, (**) $p < 0.05$, (***) $p < 0.001$.

than 10) that might compromise the precision of coefficient estimates or warrant closer scrutiny.

Unit Root Test

Table 5 summarizes the ADF unit root test results. As shown in the Table 5, the variables are integrated, with some at level I(0), and some others at the first difference I(1), meeting at least one of the stationarity criteria, either with a constant, a constant and a trend, or without at the 5% level. Accordingly, it can be said that the series in question are co-integrated and therefore, their variables can be combined linearly in the long–run, which paves the way for the application of an ARDL bounds, an ECM to define the long-run elasticities, and the three co-integration approaches for results' consolidation.

Lag Order Selection

Table 6 below summarizes the optimal lag selection estimations for the four models:

According to Table 6, with the unanimity of criteria across the four models, the optimal lag is the one with the lowest lag order. For ARDL modelling is 2.

ARDL Bounds Test

Table 7 represents the ARDL bounds test results for the four models. The ARDL bounds test results from Table 7 show that the F-statistics of the EFP, EFC, EFI, and EFE models are significantly higher than both the lower and upper critical value bounds at the 5% level, indicating that the null hypothesis of no co-integration is rejected in

Table 6: Optimal Lag length order selection.

The EFP model

Lag	LogL	LR	FPE	AIC	SC	HQ
0	-71.02967	NA	1.44e05-	3.041187	3.232389	3.113998
1	309.9495	670.5234	9.48e12-	-11.19798	10.05077-*	-10.76112
2	357.7008	74.49197*	3.94e12-*	12.10803-*	-10.00481	11.30711-*
3	375.0008	23.52796	5.80e12-	-11.80003	-8.740794	-10.63506
4	388.1802	15.28812	1.09e11-	-11.32721	-7.311959	-9.798178

The EFC model

Lag	LogL	LR	FPE	AIC	SC	HQ
0	-59.22253	NA	8.98e06-	2.568901	2.760103	2.641712
1	322.3498	671.5673	5.77e12-	-11.69399	10.54678-*	-11.25713
2	371.0372	75.95233*	2.31e12-*	12.64149-*	-10.53826	11.84057-*
3	392.0068	28.51867	2.94e12-	-12.48027	-9.421036	-11.31530
4	407.9500	18.49411	4.96e12-	-12.11800	-8.102752	-10.58897

The EFI model

Lag	LogL	LR	FPE	AIC	SC	HQ
0	-73.02299	NA	1.56e05-	3.120919	3.312122	3.193730
1	316.6852	685.8864	7.24e12-	-11.46741	10.32020-*	-11.03054
2	360.7260	68.70362*	3.49e12-*	12.22904-*	-10.12581	11.42812-*
3	378.1427	23.68678	5.12e12-	-11.92571	-8.866473	-10.76074
4	396.4881	21.28060	7.84e12-	-11.65952	-7.644276	-10.13049

The EFE model

Lag	LogL	LR	FPE	AIC	SC	HQ
0	-102.4926	NA	5.07e05-	4.299705	4.490907	4.372515
1	300.2134	708.7625	1.40e11-	-10.80853	-9.661321	-10.37167
2	352.6234	81.75962*	4.82e12-*	11.90494-*	9.801710-*	11.10402-*
3	368.8528	22.07202	7.42e12-	-11.55411	-8.494875	-10.38914
4	384.6882	18.36901	1.26e11-	-11.18753	-7.172278	-9.658497

* indicates lag order selected by the criterion

Table 7: ARDL bounds tests.

The model	F–statistic Value
EFP	10.97171
EFC	17.17399
EFI	4.557081
EFE	5.046055

at 5%, I_0 Bound= 2,86, I_1 Bound=4.01

favour of a long-run co-integration relationship between the variables.

The ARDL Analysis

Table 8 below shows the ARDL and ECM long-run estimations of the four footprint models:

Results from Table 8 show that the endogenous variables progress significantly and proportionally in the same positive direction across the four models. This and the ECT (CointEq(-1)) of each model have a negative and significant value, ranging from 1 to 0, indicating ideal annual adjustment speeds to the long-term equilibrium for the four models.

Residual Diagnostic and Stability Tests

The following Fig. 1 represents the CUSUM and the CUSUMSQ plots of the four models:

As Fig. 1 illustrates, the CUSUM and CUSUMSQ plots for all four models generally fall within the 5% level bounds, with only minor and brief exceptions, indicating their stability.

The EFs Model Normality Tests

Fig. 2 shows the four EF models' Jarque-Bera normality test outputs.

As shown in Fig. 2, the EF models' residuals exhibit a normal distribution at the 5% level.

Residuals Diagnostic and Stability Check Summary

The results of ARDL residual diagnostics and stability tests are regrouped in Table 9:

As shown in Table 9 above, the results indicate that, generally, the EF models exhibit no serial correlation or misspecification in their residuals (except for the EFE), and instead, display normal distributions with homoscedastic data. This suggests that the footprint models are stable, and their residuals do not impact the co-integration modelling process.

The Four Cointegration Methods Result Summary

The Figs. 3,4,5 and 6 graphically represent the four co-

Table 8: The footprint models ARDL long-run estimates.

The EFP model's ARDL estimates

Variable	Coefficient	Std. Error	t-Statistic	Prob.
LNURB***	1.416673	0.170776	8.295503	0.0000
LNGDP	0.002467	0.003451	0.714778	0.4786
LNICT**	0.033497	0.013895	2.410765	0.0203
LNTRD	0.010529	0.116341	0.090501	0.9283
C	11.596762	0.738764	15.697522	0.0000
CointEq(-1)	-1.089484	0.148246	-7.349146	0.0000

The EFC model's ARDL estimates

Variable	Coefficient	Std. Error	t-Statistic	Prob.
LNURB***	1.924236	0.111154	17.311484	0.0000
LNGDP	0.002582	0.002404	1.073967	0.2886
LNICT***	0.040997	0.009051	4.529376	0.0000
LNTRD***	0.168759	0.065963	2.558373	0.0140
C	9.014011	0.453759	19.865186	0.0000
CointEq(-1)	-1.041008	0.133040	-9.328106	0.0000

The EFI model's ARDL estimates

Variable	Coefficient	Std. Error	t-Statistic	Prob.
LNURB	0.618653	0.415879	1.487578	0.1437
LNGDP	0.010629	0.008150	1.304239	0.1986
LNICT***	0.101632	0.035638	2.851763	0.0065
LNTRD	0.099386	0.255452	0.389059	0.6990
C	-4.089530	1.817821	-2.249688	0.0293
CointEq(-1)	-0.438531	0.121079	-3.621856	0.0007

The EFE model's ARDL estimates

Variable	Coefficient	Std. Error	t-Statistic	Prob.
LNURB***	1.727166	0.536784	-3.217618	0.0025
LNGDP***	0.038539	0.009025	4.270240	0.0001
LNICT	0.050198	0.039027	1.286234	0.2052
LNTRD	0.104373	0.256254	0.407303	0.6858
C	4.480434	2.188006	2.047725	0.0467
CointEq(-1)	-0.537229	0.113798	-4.720912	0.0000

(*) $p < 0.01$, (**) $p < 0.05$, (***) $p < 0.001$.

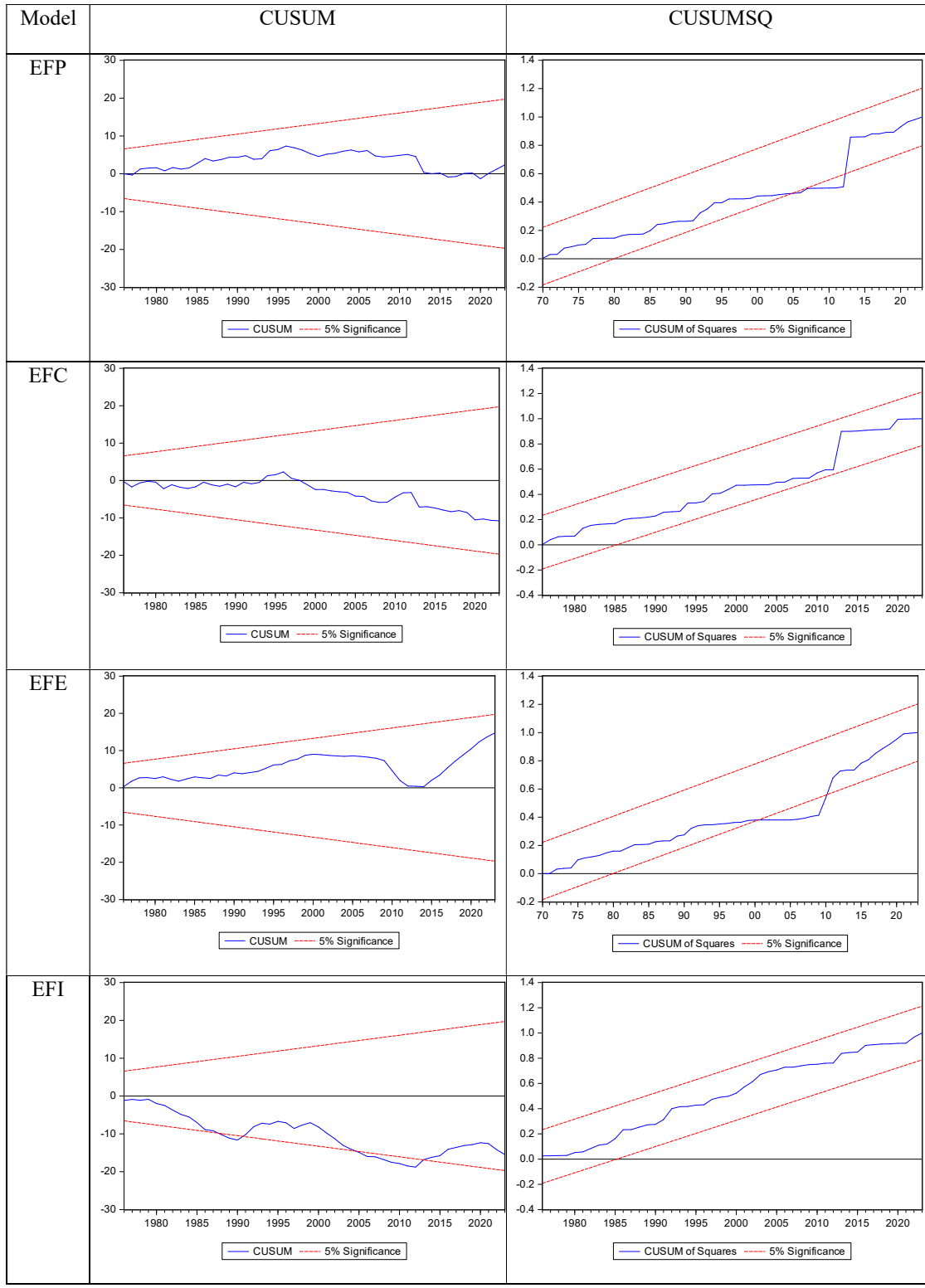


Fig. 1: The EFs models' CUSUM and the CUSUMSQ plots.

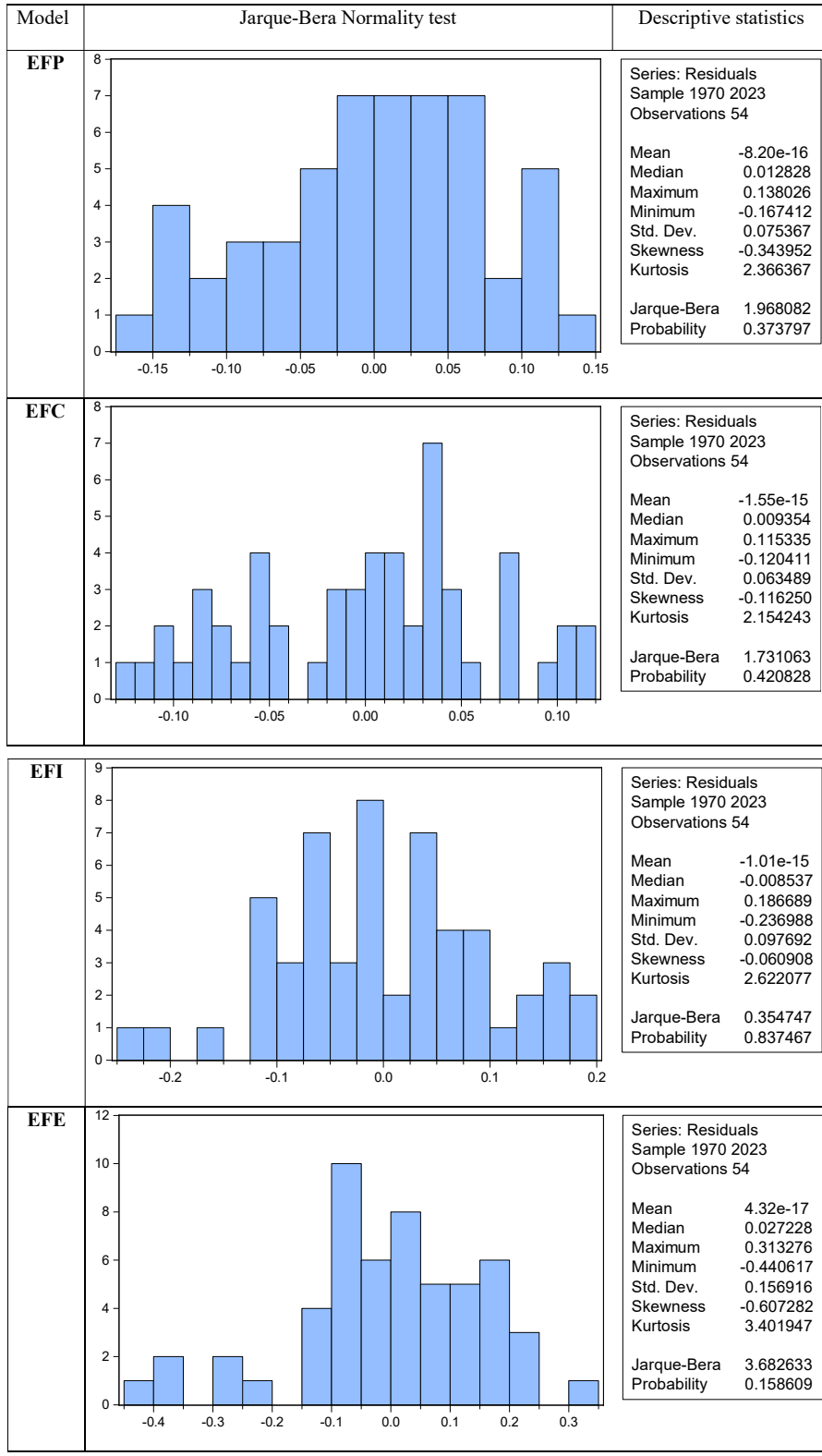
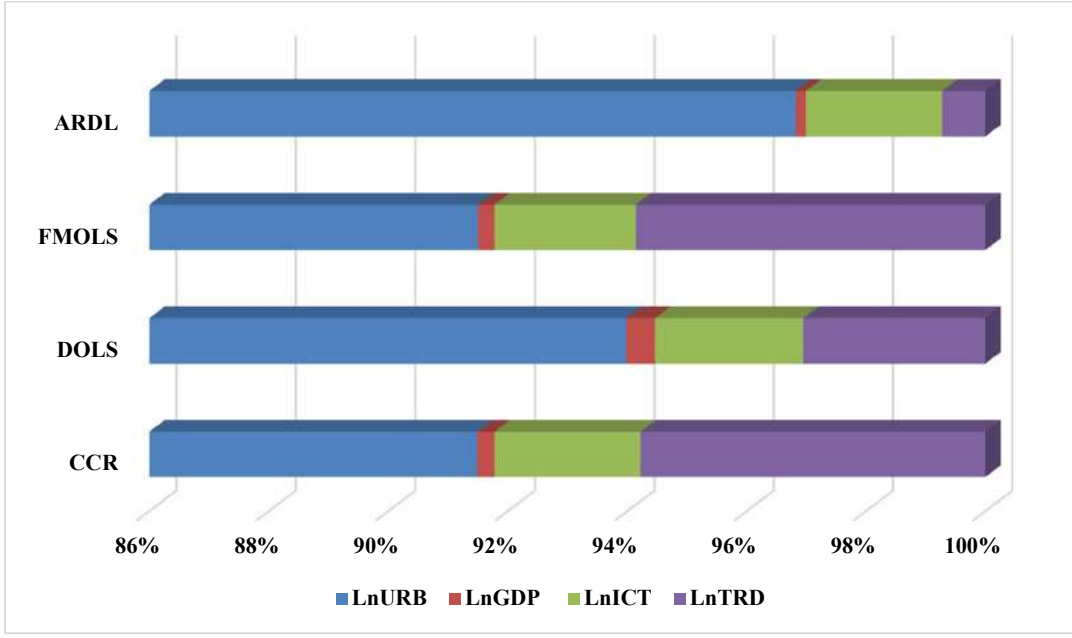


Fig. 2: The Jarque-Bera normality test outputs.

Table 9: The ARDL residual diagnostics and stability tests of the EFs models.

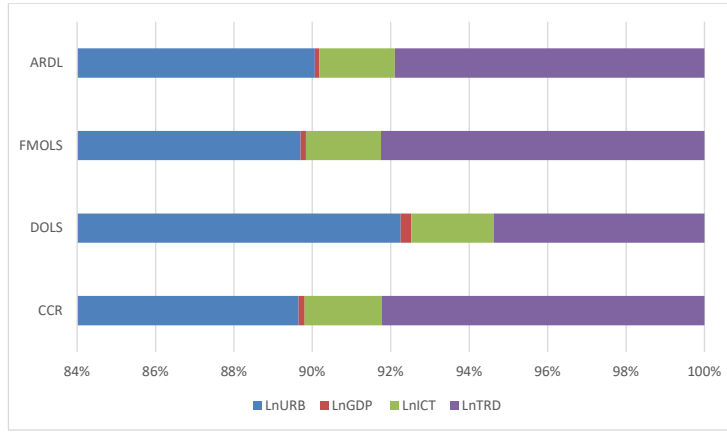
Test & hypothesis	Model	F–Statistic	P–value	Interpretation	Conclusion
Breusch–Godfrey serial Correlation LM (**H_0:** absence of serial correlation)	EFP	0.437579	0.6482	Fail to reject H_0	Absence of serial auto- correlation for the footprint models
	EFC	1.343979	0.2706		
	EFI	11.07573	0.1024		
	EFE	16.95542	0.0912		
Jarque–Bera Normality (**H_0:** Residus are normally distributed)	EFP	1.968082	0.373797	Fail to reject H_0	The four footprints' residuals are normally distributed
	EFC	1.731063	0.420828		
	EFI	0.354747	0.837467		
	EFE	3.682633	0.158609		
Breusch–Pagan–Godfrey Heteroskedasticity (**H_0:** Residus are homoscedastic)	EFP	0.437579	0.6482	Fail to reject H_0	Residus are homoscedastic for the footprint models
	EFC	1.343979	0.2706		
	EFI	2.083177	0.0973		
	EFE	3.656341	0.0910		
Ramsey RESET Functional form (test of specific error) (H_0: No misspecification)	EFP	1.717189	0.1963	Fail to reject H_0	No residus misspecification in the models except for EFI' model
	EFC	0.178952	0.6742		
	EFI	11.07573	0.6001		
	EFE	8.979528	0.0430	Reject H_0	

H_0 is either accepted or rejected at the 5%.



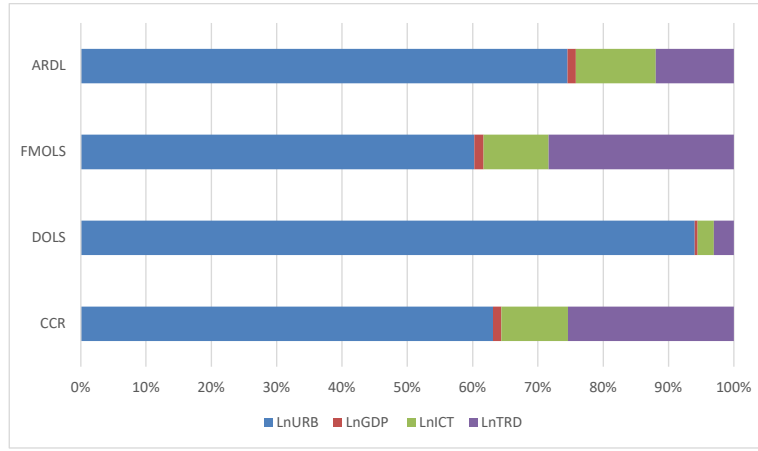
Source: Office outputs based on the authors' own computations

Fig. 3: The EFP model results.



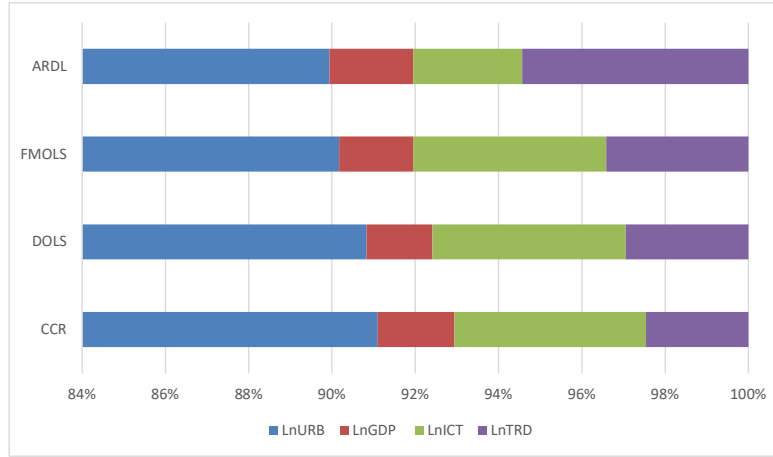
Source: Office outputs based on authors' own computations

Fig. 4: The EFC model results.



Source: Office outputs based on authors' own computations

Fig. 5: The EFI model results.



Source: Office outputs based on the author's own computations

Fig. 6: The EFE model results.

Table 10: Final incidence appreciation on environmental degradation.

MODEL	LnURB	LnGDP	LnICT	LnTRD
EFP	Very high	Very low	Moderate	Moderate
EFC	Very high	Very low	Moderate	high
EFI	So high	Very low	Moderate	Moderate
EFE	So high	Low	Moderate	Moderate
→ Environmental degradation	Very high	Very low	Moderate	Moderate

NB: General appreciation of environmental appreciation is based on the EF's singular appreciations

integration methods' results: Table 10 provides a final incidence appreciation based on the four EF models' results.

DISCUSSION

From the final appreciation in Table 10 above, it can be said that anthropogenic processes associated with urbanization, economic growth, technological progress, and trade openness had a positive impact on EFP, EFC, EFE and EFI in Morocco over five decades (1970–2023).

Based on the previously formulated hypotheses, H_a, H_b, H_c and H_d, are strongly supported.

The findings affirm that urbanization, technological progress, economic growth, and trade openness have significantly contributed to the increase in Morocco's divers EFs. These drivers, while traditionally linked to development and modernization, are shown here to exert unsustainable pressure on the environment, echoing concerns raised in broader literature (Dietz & Rosa 1997, York et al. 2003).

Urbanization, for instance, is typically associated with increased infrastructure demands, resource consumption, and pollution factors that amplify ecological degradation in the absence of green urban planning (Sharma 2011). In Morocco's case, urban sprawl has likely led to habitat loss and increased energy use, worsening ecological impacts.

Technological progress, though often positioned as a solution to environmental challenges, can paradoxically intensify them when it encourages higher consumption and resource exploitation, a phenomenon known as the rebound effect (Polimeni et al. 2008). In Morocco, technology has apparently contributed to increased EFs, suggesting a lack of alignment with sustainable development principles.

Trade openness is another double-edged sword. While it can promote economic diversification and growth, it can also lead to environmental externalities, especially when trade involves ecologically harmful goods or when environmental regulations are weak (Antweiler et al. 2001). The study attributes Morocco's rising ecological impact from imports and exports to these dynamics.

Finally, economic growth in Morocco, while vital for poverty reduction, appears environmentally taxing. This aligns with the (EKC) hypothesis (Hamdi et al. 2025), which posits that environmental degradation first increases with economic growth before eventually declining, though Morocco may still be in the upward phase of this curve (Grossman & Krueger 1995).

The use of four co-integration techniques strengthens the reliability of the results by confirming long-run equilibrium relationships between environmental degradation and its drivers. This methodological rigor allows for more confident policy implications.

Importantly, the conclusion underscores the unsustainability of current trends and advocates for multi-level policy responses. These include individual behavioral changes, organizational reforms, and government-led interventions to boost bio-capacity and mitigate environmental stress. This multi-pronged approach is consistent with global sustainability frameworks, such as the United Nations Sustainable Development Goals (SDGs), particularly Goals 11 (Sustainable Cities), 12 (Responsible Consumption and Production), and 13 (Climate Action).

In sum, the Moroccan context reflects broader trends in the Global South, where the pursuit of development, absent environmental safeguards, risks deepening ecological crises. This study contributes to the growing call for evidence-based, inclusive environmental governance that addresses the root causes of degradation while supporting equitable development.

Policy Implications

The findings underline the urgent need for informed and context-specific policy interventions. Here are some concise and practical sustainable propositions to help undermine and reduce the EF rise and effects, applicable to three individual, organizational, and governmental levels, which can be translated into materialized concrete actions (Table 11).

Study Potential Limitations

This study has three main limitations. Statistically, co-

Table 11: Policy implications' propositions.

Actions level	Policy implications' propositions
Individual	• Biking, walking, or using public transport, for daily commutes, and when possible, opting for electric or hybrid vehicles for personal use (Anbar 2022, Ontario Nature 2024) • Unplugging electronics and appliances regularly when not in use, and switching to LED bulbs and energy star-rated electronics for energy efficiency (Anbar 2022, Blog_Admin 2024) • Incorporating more plant-based meals to reduce emissions from livestock (Ontario Nature 2024, Sarah-Indra 2024) • Practicing waste management by adopting the 3Rs: Reduce, Reuse, Recycle, to minimize waste (Ontario Nature 2024, Blog_Admin 2024) • Composting organic waste. such as agro-food waste (Ontario Nature 2024, Sarah-Indra 2024)
Organizational	• Creating thematic action plans by identifying key areas for improvement (e.g., energy consumption, waste management) and setting clear objectives with specific measures and timelines. • Identifying low-carbon trajectories to set science-based targets for reducing GHG and regularly monitor progress. • Assessing suppliers' carbon footprints and encouraging sustainable practices through incentives and partnerships. • Implementing a collective waste disposal (Hamilton et al 2013).
Governmental	• Setting an achievable, energetic transition as a national objective. • Enacting legislation that enforces sustainable practices across society and the economy, such as renewable energy targets, green incentives, carbon taxation, waste management standards… • Promoting environmental public awareness via official media, school programs and public spending (Hamdi & Azeroual 2023a, 2023b). • Fixing an ultimatum for economic carbon neutralization, with a focus on the intensive emitter sectors such as transport and industry

integration models are effective for long-term analysis but limited to co-integrated series, with challenges in lag selection and model complexity as more variables are added. Cognitively, the study lacks a predictive framework and focuses on traditional STIRPAT factors, omitting emerging variables like energy use, governance, and clean technologies. In terms of scope, the Morocco-specific focus limits the broader applicability and global relevance of the findings.

Plausible Future Prospects

Prospects should continue exploring this study's interactions by extending the spectre of explaining environmental degradation factors, as well as opting for other ecological barometers, and enlarging datasets to include geographical imbalances and panel differential properties.

CONCLUSIONS

This study explored the long-term effects of human-induced processes associated to urban expansion, economic affluence, technological progress and openness to international trade on Morocco's EFs through its four economic varieties: of production (EFP), consumption (EFC), imports (EFI), and exports (EFE) from 1970 to 2023, and how these fluctuations have shaped the trajectory of Morocco's ecological sustainability more than six decades.

To assess these interactions, the study employed four co-integration techniques, namely ARDL, FMOLS, DOLS, and CCR. Each of them was applied to each EF component, enabling a robust and multifaceted understanding of the resultant ecological outcomes.

Findings indicate that urban expansion, along with economic growth, as well as technological progress, besides openness to international trade, have significantly contributed to the intensification of ecological stress in Morocco, that is, the degree of impact varied respectively from high, moderate, subtle, to low across the four EF considered models.

Overall, the study underscores the complex and multifaceted nature of anthropogenic ecological stress in Morocco and highlights the implications of socioeconomic factors in shaping the country's environmental future trajectory through adequate proposed policy implications.

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Recent Advances in Integrated Carbon Dioxide Capture: Exploring Carbon Capture Methods and AI Integration

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ABSTRACT

Achieving the Sustainable Development Goals depends on decarbonizing the industrial sector, as it still contributes the most to global greenhouse gas emissions and energy consumption. Reducing emissions from fossil fuel-based energy systems critically depends on carbon capture, especially post-combustion carbon capture (PCC). Absorption-based carbon capture (ACC) is the most developed and extensively applied PCC system. However, ACC systems are quite energy-intensive and require major heating and cooling utilities, which increases the running costs and makes large-scale adoption difficult. This study investigates the current developments in carbon capture technology and emphasizes the integration of artificial intelligence (AI) to address optimization challenges. In particular, it suggests an artificial intelligence-based approach for improving ACC system design, operation, and utility consumption forecasting. AI-driven solutions can promote scalable and reasonably priced carbon capture technologies by allowing accurate and fast assessments of technical and financial viability. This study emphasizes how artificial intelligence might hasten the shift toward more environmentally friendly industrial methods and significantly support global climate action targets (SDG 13).

INTRODUCTION

In recent years, extreme weather events, including heatwaves, droughts, wildfires, hurricanes, and flooding, have become increasingly severe, resulting in substantial damage to human life, infrastructure, and ecosystems (Cotterill et al. 2021, Jain et al. 2022). The World Meteorological Organization (WMO) has projected that severe weather events have caused over 2 million fatalities and US$4.3 trillion in economic damages from 1970 to 2020. Global warming and anthropogenic carbon emissions are widely acknowledged as the causes of the escalating frequency and severity of extreme weather events, although the attribution of individual events is frequently a topic of debate (Bellprat et al. 2019, Schiermeier 2018). With 29.4% of all greenhouse gas emissions coming from energy usage in industry and direct industrial processes, the industry sector has the highest carbon emissions. Consequently, it is imperative to implement industry decarbonization to mitigate extreme weather events. Energy efficiency improvement, carbon capture, and hydrogen energy are among the primary techniques for industrial decarbonization (Baker et al. 2018, Ritchie & Roser 2024, Schiermeier 2018).

Carbon emissions can be reduced by implementing strategies such as improving the energy structure, electrification, decreasing excessive energy usage, and carbon

sequestration. Carbon dioxide capture and storage (CCS) is a method that utilizes pipeline transportation to extract carbon dioxide (CO_2) from the atmosphere or industrial waste gas and stores it in the ocean or underground at a significant concentration. This process has the potential to reduce the concentration of CO_2 in the atmosphere (Zhang et al. 2021). Nevertheless, its development is constrained by its high cost and potential long-term environmental impact. In recent years, the field of CO_2 capture and utilization (CCU) has garnered growing interest because of its capacity to convert CO_2 into value-added products through chemical conversion, including thermal, electrical, and photocatalysis techniques (Liang et al. 2021, Moss et al. 2017). Table 1 summarizes the relevant carbon dioxide capture and utilization technologies in terms of their features, benefits, and drawbacks. By capturing and using CO_2 gas on-site, the integrated carbon dioxide captures and utilization (ICCU) system makes CO_2 use efficient and economical by lowering transportation and compression costs (Guo et al. 2023).

Geological carbon storage (GCS) is an essential part of carbon capture, use, and storage (CCUS) to reduce greenhouse gas emissions and meet climate objectives (Ringrose & Meckel 2019). With the potential to manage over 220 Mton of CO_2 annually, project developers expect to put more than 200 new capture and storage facilities into operation globally by 2030, according to the International

Table 1: An overview of CO_2 collecting and use technologies.

	Technology	Description	Advantages	Disadvantages	References
Technology to collect CO_2	Capture of oxyfuel combustion	Combusting fuel with either pure oxygen or a combination of oxygen and carbon dioxide (CO_2) in the exhaust gas can be captured.	It is possible to store things directly.	The generation of oxygen has a high cost and is susceptible to air leakage.	(Martin et al. 2011)
	Pre-combustion capture	Before burning fuel derived from carbon, separate other combustibles from CO_2	Significant CO_2 concentration and effortless separation	Require the enhancement of the existing power plant, a task that is challenging and entails significant expenses	(Martin et al. 2011)
	Absorption separation	Gas mixtures can be separated based on their varying solubility	high yield and elevated CO_2 concentration	significant energy usage, significant equipment investment	(Guo et al. 2023)
	Post-combustion capture, adsorption, and separation	The separation of gas mixtures is achieved by exploiting the distinct binding force between gases and porous materials.	Flexible operation, safety, and affordability	Inorganic adsorbents exhibit poor selectivity and unpredictable performance.	(Gu et al. 2015, Tang et al. 2022)
	Membrane separation	Utilize variations in solubility and diffusivity to capture	High selectivity with low energy usage	restricted application, weak stability	(Yan et al. 2012)
Technology for using CO_2	Solar thermochemical conversion technology	Solar radiation is employed to generate a robust endothermic reaction that is suitable for utilization by driving CO_2 and H_2O	low usage of energy	minimal efficiency of conversion	(Ishaq et al. 2021)
	Electrochemical conversion	The voltage difference between the two electrodes is what drives the reduction of carbon dioxide into compounds	flexible running circumstances, moderate reaction environment	Electrocatalyst instability and elevated energy consumption	(Meng et al. 2021)
	Catalytic conversion	Chemical bond formation and breakage are facilitated by the employment of catalysts	cheap cost and excellent safety	Improvements are needed in stability and conversion efficiency	(Rahimi et al. 2016)
	Photochemical conversion	The absorption of thermal energy and the overriding of activation energy facilitate the CO_2 conversion reaction	moderate reaction circumstances and potent oxidation capacity	low light energy utilization rate, efficiency, and control issues	(Guo et al. 2023)

Energy Agency (IEA) (Lin et al. 2022). Because one of the largest CCUS projects to date, the water-alternating gas (WAG) injection project in the Brazilian Pre-Salt, has only injected 20 Mton of CO_2 over a decade into the four largest carbonate reservoirs in Brazil, or less than 10% of the IEA target, it is important to put this ambitious goal into perspective. When fossil fuels are burned, carbon dioxide (CO_2) is released into the atmosphere. The quantity of CO_2 increases as global energy consumption increases to support energy-intensive activities (Seabra et al. 2024). For the foreseeable future, fossil fuels will remain the primary source of energy globally, despite the grave environmental problems often linked to CO_2 emissions. Numerous carbon dioxide storage locations are found in geologically intricate formations, such as channelized reservoirs or fractured carbonate rocks (March et al. 2018). Consequently, it is imperative to identify and develop a technological solution that is both feasible and effective in reducing carbon dioxide emissions into the atmosphere.

Most carbon capture and storage methods involve post-combustion carbon capture, oxy-fuel combustion carbon capture, or pre-combustion carbon capture. The development and deployment of combustion systems suitable for their intended purpose are necessary for both pre-combustion and oxyfuel carbon capture (Al-Hamed & Dincer 2022, Park et al. 2015). Alternatively, post-combustion carbon capture technology may be used to upgrade and modify existing fossil-fuel burning facilities (Khalilpour 2014). Thus, it may reduce emissions without replacing the infrastructure (Aliyon et al. 2020). Absorption, adsorption, and membrane separation are the three most prevalent methods for capturing carbon after burning (Akinola et al. 2022, Aliyon et al. 2020, Zhang et al. 2021).

This study developed a substitute machine learning (SML) model to predict the heating and cooling utility consumption of ACC plants using machine learning (ML) techniques. System engineering models are enhanced or replaced by SML models. SML models outperform

Table 2: Literature reviews that have used ML within the ACC.

ML model(s)	Purpose	Model Inputs	Model Outputs	Data Generation Software	References
An ensemble neural network using bootstrap aggregation, often known as bagging, with a single-layer neural network.	Forecasting the efficiency of CO_2 capture	Flow rate of flue gas, pressure, temperature, and concentration of CO_2, flow rate and temperature of lean fluid, concentration of MEA, and temperature of the reboiler	Efficiency in CO_2 capture	gPROMS	(Li et al. 2015)
Single-layer neural network	Predicting the specified duty of a reboiler and the rich loading	Temperature, CO_2 concentration, lean load, removal efficiency, solvent circulation rate, and flue gas flow rate	The flow rate of captured CO_2, plus the specific duty of the reboiler, plus a solvent-rich load	CO2SIM	(Sipöcz et al. 2011)
Network of profound convictions	Forecasting the efficiency of CO_2 capture	Flow rate of flue gas, pressure, temperature, and concentration of CO_2, flow rate and temperature of lean fluid, concentration of MEA, and temperature of the reboiler	Efficiency in CO_2 capture	gPROMS	(Li et al. 2018)
The extreme learning machine is used to build a bootstrap combined neural network with a single-layer neural network.	Forecasting the efficiency of CO_2 capture	Flow rate of flue gas, pressure, temperature, and concentration of CO_2, flow rate and temperature of lean fluid, concentration of MEA, and temperature of the reboiler	Efficiency in CO_2 capture	gPROMS	(Li et al. 2017)
Single-layer neural network	Improvement of operational control	The flow rates of lean solvent, flue gas, and reboiler steam	Amount of CO_2 collection plus the temperature of the reboiler	gPROMS (gCCS module)	(Wu et al. 2020)
A single-layer neural network and a few other components	Optimisation of processes	factors such as reboiler and condenser responsibilities, reboiler pressure, flow rate, temperature, and flue gas pressure	Total work for reboilers, condensers, and amine coolers, plus the rate of capture, plus the purity of CO_2	gPROMS	(Shalaby et al. 2021)

engineering models in several ways, such as quick running times, robustness in predicting system performance that engineering models are unable to fully capture, robustness in predicting system performance as components age, ability to make predictions without in-depth knowledge of the system, and ability to make predictions with few inputs (Chegari et al. 2022, Li et al. 2022, Spinti et al. 2022, H. Zhang et al. 2021). This study used SML models to provide rapid and accessible utility consumption prediction models to help build energy-efficient ACC procedures.

Active sites for CO_2 adsorption and conversion are the primary components of dual-function materials, which facilitate the adsorption, desorption, and in situ conversion of CO_2. Because fresh dual-function material samples tend to absorb carbon dioxide and water from the surrounding air, a pre-reduction step is often necessary before the reaction (Bermejo-López et al. 2022). Initially, CO_2 was drawn at a particular temperature until the adsorbent was fully saturated. Second, the adsorbed CO_2 in the saturated materials interacts with hydrogen to form CH_4 when placed in a reducing environment. The integrated carbon dioxide capture and methanation procedure primarily comprises this two-step process. Carbon dioxide can be consistently captured and transformed in a single reactor throughout multiple cycles. The reaction exhibited a favorable cyclic performance and could be conducted at a moderate temperature of approximately 300°C. This streamlines the procedure and improves the energy efficiency. Integrated carbon dioxide capture and utilization (ICCU) technology has gained significant attention owing to its ability to efficiently convert carbon dioxide into fuels, such as carbon, using dual-function materials. This approach delivers high efficiency with low energy usage by combining carbon dioxide adsorption and in situ conversion (Guo et al. 2023). Table 2 presents the various studies that have used ML in the ACC.

Carbon capture research has primarily focused on developing new methods to reduce the cost of CO_2 collection. Some of the methods used include the development of new solvents, the use of catalysts to enhance the performance of existing solvents, the application of artificial intelligence to the CO_2 capture process, and the development of new repair methods. In this section, several approaches for determining the column height of the CO_2 absorbent are thoroughly examined. Some of the methods used include empirical design, theoretical design, laboratory and pilot plant processes, and so on. This review is based on the idea of using AI to capture CO_2. The coming together of many AI programs. The potential for AI-assisted CO_2 collection is examined, along with the difficulties involved. A comparative summary of common machine learning models used in CO_2 capture, along with their advantages and limitations, is presented in Table 3.

MATERIALS AND METHODS

A Summary of the Process of Capturing Carbon Dioxide

Extensive research has been conducted on CO_2 capture systems, which are crucial for reducing industrial carbon emissions. Among the most significant contributors to carbon monoxide emissions are high-temperature industrial activities, including the manufacturing of steel, cement, oil, and gas. Post-combustion capture technologies, more especially absorption, adsorption, and membrane separation, are among the many ways that have been extensively researched and utilized (Chao et al. 2021). Amine-based chemical absorption is the post-combustion capture method with the greatest documented track record and is potentially economically viable. This technique involves the use of aqueous amine solvents to selectively absorb carbon monoxide from exhaust gases (Raganati et al. 2021). In real time, intelligent control systems that use artificial intelligence can monitor membrane performance, identify fouling or degradation, and dynamically alter operating settings. Data-driven models also support the selection of materials and the creation of hybrid systems (e.g., coupling membranes with adsorption or absorption).

The procedure involves the absorption of CO_2 from flue gas using an amine solvent, followed by the separation of CO_2 from the solvent using a stripping column. The solvent is recycled back into the absorber, and the concentrated CO_2 is collected for storage or use after extraction (Yamada 2021). The capture of CO_2 is essential in petrochemical activities, particularly in the production of ammonia, because of the significant volume of CO_2 emissions produced during the process (Takht Ravanchi & Sahebdelfar 2014). This cycle process is commonly implemented in large-scale industrial processes, such as natural gas processing and ammonia manufacturing, when the amount of carbon dioxide produced is significant. The solvent-based approach is frequently followed by a sorbent method. At present, fewer than one-third of the processes are membrane-based. In the collection of CO_2 through absorption, adsorption, and membrane separation technologies, the selection of a solvent, adsorbent, or membrane material, as well as the optimization of the operating pressure and temperature, are all critical operational factors. These characteristics significantly influence the efficiency and efficacy of the capture process; therefore, it is necessary to comprehensively select and optimize these parameters to achieve the desired

results. The subsequent discourse provides a comprehensive examination of diverse techniques for CO_2 capture, including membrane-based, adsorption, and absorption techniques (Priya et al. 2023).

Adsorption is a relatively new alternative to absorption, offering several benefits similar to those of absorption, including reduced energy usage and simpler regeneration. In this approach, carbon monoxide molecules can attach themselves to the surface of a solid porous substance, referred to as an adsorbent. Temperature, pressure, pore size, surface area, and adsorption kinetics are key operating parameters that significantly affect performance. High CO_2 selectivity, rapid adsorption and desorption rates, mechanical durability, and economic viability for regeneration are only a few of the characteristics that should be present in effective adsorbents (Abd et al. 2020). Absorption-based systems consume significant energy, particularly due to the requirements for thermal regeneration. Predicting the performance of a solvent, optimizing the amount of energy required for regeneration, and simulating the behavior of a process under a variety of different operating circumstances are all possible applications of artificial intelligence models. AI-driven predictive modeling can considerably improve the design and control of absorption systems in terms of energy efficiency and economic feasibility.

Additionally, the adsorbent material must fulfill the operational and budgetary requirements for efficient CO_2 removal by demonstrating CO_2 selectivity, rapid adsorption and desorption kinetics, sufficient mechanical strength, and economically feasible regeneration (Abd et al. 2020). Most of the area in the column is occupied by the adsorbent, which allows CO_2 to flow over the system unhindered. The adsorbent captures CO_2 through its surface. Once the balancing condition is reached, the duplicated adsorbent can be employed for the next CO_2 intake. Pressure swing adsorption is a technique that includes controlling the pressure to improve both the absorption and desorption of CO_2 by the adsorbent to separate CO_2 from a gas mixture. This technique continues until the necessary amount of CO_2 is removed, at which point the gas mixture exits the adsorbent bed with a decreased concentration of CO_2 (Siqueira et al. 2017). When optimizing adsorption processes, it is necessary to solve complex problems involving multiple variables. Modeling nonlinear interactions among parameters, predicting breakthrough curves, and optimizing PSA cycle times and operating conditions are all possible using artificial intelligence and machine learning techniques.

The performance of the system is heavily dependent on the following factors, regardless of capture technology:

- The selection of the material for the membrane, the adsorbent, or the solvent.
- Temperature, pressure, and flow rate are examples of the operating parameters.
- In terms of energy efficiency and capacity for regeneration.

Artificial intelligence-based techniques play a crucial role in optimizing these variables. This study highlights the growing body of research that employs artificial intelligence to enhance the scalability, responsiveness, and sustainability of carbon capture systems.

RESULTS AND DISCUSSION

Technologies for Sequestering Carbon

Deep-ground injection, ocean storage, and improved oil recovery (EOR) are the main approaches to carbon sequestration (Alvarado & Manrique 2010, Lemieux 2011). Deep-ground injection involves the use of geological formations to store CO_2, whereas ocean storage takes advantage of the vast carbon-absorbing capacity of oceans. Enhanced oil recovery (EOR) combines CO_2 storage with practical energy generation. These techniques demonstrate the complex endeavors to tackle carbon emissions, with each strategy employing distinct natural and technological mechanisms to reduce atmospheric CO_2. Deep ground injection is a procedure in which carbon dioxide (CO_2) is crushed and injected into geological formations beneath the Earth's surface. As a result of the intense pressure and temperature at extreme depths, CO_2 frequently becomes supercritical, leading to improved storage efficiency because of its higher density (Bloom Energy 2024). Trapping by it structurally beneath impermeable caprocks, residual trapping within rock fissures, solubility trapping as CO_2 dissolves in water, and mineral trapping as it combines with minerals to produce stable carbonates are some of the methods that sequester CO_2 over time. The integrity and continuity of caprocks are essential for structural entrapment, as they serve as seals that prevent upward migration (Arif et al. 2016). Residual trapping is a process that effectively prevents migration by utilizing capillary forces to hold CO_2 in pore spaces, even if the structural trap is compromised (El-Maghraby & Blunt 2013). Carbonic acid is produced when CO_2 dissolves in the formation water during solubility trapping. This process also reduces buoyancy and leakage potential by reacting to generate bicarbonate ions (Adamczyk et al. 2009). Mineral trapping refers to the process in which carbon dioxide (CO_2) reacts with minerals in the formation of stable carbonate minerals. This reaction occurs over a long period and helps improve the long-term security of CO_2 storage (Soong et al. 2004). The longevity

of this method's ability to store large volumes of CO_2 is heavily contingent upon geological and technical conditions. Fissures in the rock, which may be worsened by seismic activity, have the potential to weaken this seal, resulting in the release of CO_2 back into the environment (Blake et al. 2022). Furthermore, permanent sequestration is complicated by the substantial technical challenges associated with the monitoring and verification of the stored CO_2.

AI-Based Carbon Capture Applications

As computer technology has improved significantly over the last two decades, the numerical simulation of processes has gained importance and popularity across a wide range of engineering and academic disciplines. Many researchers are currently investigating artificial intelligence (AI) technologies, namely machine learning approaches,

Table 3: Machine Learning approaches: Advantages and Limitations.

ML Model	Typical Use in CO_2 Capture	Advantages	Limitations	References
Artificial Neural Networks (ANN)	Prediction of energy use, solvent recovery, and system optimization	High predictive accuracy, handles non-linear systems well	Prone to overfitting, requires large datasets	(Alabdraba et al. 2017)
Support Vector Machines (SVM)	Classifying optimal operating conditions, CO_2 selectivity prediction	Effective in high-dimensional space, good for classification/ regression	Computationally intensive, kernel selection is critical	(Afkhamipour and Mofarahi, 2016)
Random Forest (RF)	Sensitivity analysis, feature importance ranking	Robust to noise, handles missing data well	Less interpretable, can be slow for large datasets	(Chen et al. 2021)
Gradient Boosting Machines (e.g., XGBoost)	Performance prediction and fault detection	High accuracy, handles heterogeneous data	Risk of overfitting, tuning complexity	(Zhang et al. 2022)
Reinforcement Learning (RL)	Dynamic control system optimization	Suitable for real-time control and adaptive optimization	Limited industrial deployment needs well-defined reward structures	(Moradi et al. 2022)
Hybrid AI models (e.g., ANN+GA, SVM+PSO)	Optimizing operating conditions	Combines the benefits of multiple techniques for improved optimization	Complex to implement and validate	(Ehteram et al. 2021)

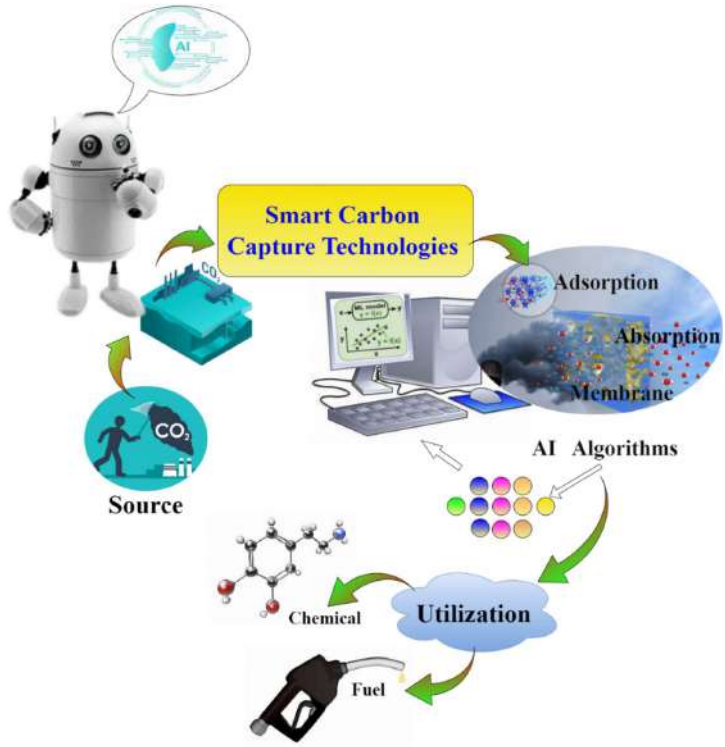

Fig. 1: Smart carbon capture technologies and utilization.

because of their potential as attractive alternative solutions (Alabdraba et al. 2017). Significant post-combustion CO_2 collection facilities, such as TMC Mongstad in Norway and BD3 SaskPower in Canada, generate large amounts of operating process data. This information can be used as a great source of input to create knowledge that enhances the CO_2 capture mechanism (Chan & Chan 2017). Artificial neural networks (ANN are widely utilized and popular techniques of artificial intelligence applied for mass transfer and property prediction in the CO_2 capture process, owing to several factors. The quick formulation of ANN predictive models, including several parameters, is possible. They possess a high degree of adaptability and often yield more accurate outcomes than numerical simulations and correlations (Li et al. 2015). The ANN method is briefly described as follows. Fig. 1 shows smart carbon-capture technologies and their utilization.

An Artificial Neural Network (ANN) model can display both linear and nonlinear relationships in its input and output data (Fu et al. 2014). The network has many processing units that work simultaneously and are linked to each other. These are called neurons, and they are based on the nervous system of the human brain and biological neurons (Mohagheghian et al. 2015). The neuron units in the adjacent layers were fully coupled to each other in the hidden layer. The following formula can be used to determine the output of each neuron (yj): Activation or transfer functions include sigmoid, piecewise linear, radial basis, and Gaussian functions. (Adeyemi et al. 2018). Utilizing either the sigmoid or hyperbolic functions as the concealed activation mechanism, the multilayer perceptron is the most frequently employed feedforward neural network (Chan & Chan 2017). AI applications in CO_2 capture currently face several significant constraints.

- For CO_2 capture procedures at the plant scale, there is a lack of high-quality datasets, particularly for more recent technologies such as membranes.
- The inability to generalize from small training sets is a common problem with many ML models, especially DNNs.
- Adaptability in real-time, reaction with minimal latency, and integration with existing control systems are essential for using AI in dynamic industrial environments.
- Companies that place a premium on safety may be hesitant to use black-box models like ANN because of the lack of understanding they provide on process dynamics.
- Retraining or substantial recalibration may be necessary to make models trained on data from lab-scale plants work well on data from full-scale plants.

AI's Application to Physical Attributes and Solubility

The physical and chemical properties of CO_2 and amines, such as viscosity, density, heat capacity, reaction rate, diffusivity, and conductivity, can substantially influence the efficiency and effectiveness of the carbon capture process in CCS (Tantikhajorngosol et al. 2019). These properties are frequently utilized in the process simulations of the CO_2 capture process and are necessary for the calculation of heat duty. The values of these properties are frequently determined by measuring them in a laboratory environment using costly instruments (Pouryousefi et al. 2016). However, experiments and the collection of experimental data require a high level of expertise and a comprehensive understanding of the process. The data capture process is intricate and time-consuming, often involving repetitive procedures (Adeyemi et al. 2018).

Based on notable empirical and semi-empirical connections, numerical simulations and models tend to be simpler methods for determining feature values than experimental techniques (Fu et al. 2014, Mohagheghian et al. 2015). However, there are several downsides to the modeling technique, including: (i) the correlations cannot capture the nonlinear relationships between the parameters, (ii) there needs to be a guarantee of access to massive amounts of data, (iii) function evaluations need to be carried out to ensure that the models and numerical simulations are correct, (iv) it might take a lot of computing power to develop the solutions, (v) there is a chance that the models and simulations developed for certain conditions will not work outside of those parameters, and (vi) the unfavorable characteristics of gases and amines might complicate computations relying on correlations (Bahadori & Mokhatab 2008, Zhou et al. 2009). Many studies have proposed the use of machine-learning methods, such as artificial neural networks (ANN) and Support Vector machines (SVM), to forecast several properties connected with the CO_2 capture process to solve these problems (Afkhamipour & Mofarahi 2016).

Baghban et al. (2015) created artificial neural network (ANN) and adaptive neuro-fuzzy inference system (ANFIS) models to accurately predict the solubility of CO_2 in the carbon-capture process (Baghban et al. 2015). These models can provide precise predictions across a wide range of temperatures, pressures, and concentrations. The CO_2 concentration was the output variable, with the following variables serving as inputs: acentric factor, molecular weight, critical pressure, temperature and pressure. The solubility of CO_2 in aqueous TBAB solutions was predicted using

RBFNN and ANFIS (Hoseinpour et al. 2018). The CO_2 solubility served as the end parameter, and the mass and mole percent of TBAB, temperature, and pressure were the inputs. The accuracy of the AI model predictions was confirmed using statistical and graphical analytical methods. The CO_2 solubility in the aqueous sodium salt of L-phenylalanine was precisely predicted by the ANN model employing Lvenberg-Marquardt (LM) (Garg et al. 2017). When contrasted with the solubility predictions offered by the Kent-Eisenberg model, the results produced by Artificial Neural Networks (ANN) showed a higher degree of agreement with the experimental data. The integration of a genetic algorithm with a least-squares support vector machine (GA-LSSVM) enabled precise predictions of hydrocarbon solubility in water (Helei et al. 2021).

AI Application for CO_2 Mass Transfer

To design, simulate, and improve the CO_2 collection process, it is especially important to obtain accurate measurements of the mass transfer rate. Over the past 15 years, experts have investigated the use of artificial intelligence (AI) to copy the process of moving mass and test how well CO_2 can be captured. The purpose of this study was to develop accurate and reliable estimates of the speed at which mass moves (Meesattham et al. 2020). Predicting properties such as CO_2 concentration, temperature, heat duty, and removal efficiency is a common focus in these applications. In contrast, the conditions of the CO2 collection process are the input predictors (Afkhamipour & Mofarahi 2016, Fu et al. 2014). The research comprised several crucial elements: soliciting input from experts regarding the intricate interdependencies among the parameters required for particular algorithms, building artificial neural networks (ANNs), fine-tuning the internal connection weights to minimize disparities between the inputs to the network and the desired output, and optimizing the networks to handle weird data that do not fit in with the training samples. Moreover, the tests produced a limited data sample that accurately represented the population. Some instances of sample investigations are as follows: The current research states that hybrid models integrating experimental and simulation datasets have improved the prediction of CO_2 transfer coefficients. To illustrate this point, accurate estimates of the total mass transfer rates in absorber columns have been achieved by estimating the Sherwood and Reynolds numbers using ANN+PSO models (Hoseinpour et al. 2018).

The Potential and Difficulties of AI-Assisted Carbon Capture

An estimated 53 gigatonnes of CO_2 equivalent greenhouse gas emissions have been released into the atmosphere worldwide, intensifying the already severe climate change. The primary objective of the 2016 Paris Agreement is to limit the rise in average global temperature to 1.5°C. By the conclusion of this decade, emissions must be reduced by 50% to accomplish this objective. Artificial intelligence is believed to be capable of achieving a reduction of 5% to 10% of the required reduction, which is within the range of 2.6 to 5.3 gigatonnes of CO_2 equivalent (Degot et al. 2021). An important advantage of AI-assisted carbon capture is its ability to significantly decrease the cost associated with capturing CO_2. Artificial intelligence algorithms can analyze vast quantities of data in real time, resulting in enhanced performance of CO_2 capture systems that are efficient and cost-effective. AI-assisted CO_2 capture enhances the reliability and accuracy of CO_2 capture systems. Artificial intelligence algorithms can observe and evaluate the operation of CO_2 extraction devices and make immediate adjustments to enhance efficiency. This can reduce the likelihood of costly malfunctions and guarantee the ongoing functionality of the system. A novel AI-based instrument was recently created by a team of scientists to facilitate the faster and more precise locking of greenhouse gases, including CO_2, in porous rock formations with unprecedented speed. A Fourier neural operator-based deep-learning model, which is a unique neural operator architecture, was employed to efficiently mimic the pressure levels in carbon storage. This model significantly improved the precision of specific jobs, enabling scientists to identify the most efficient injection rates and sites with twice the accuracy (Wen et al. 2022). The use of artificial intelligence (AI) in carbon capture has shown promise in the laboratory, but practical implementations have been slow to materialize. Compact carbon-capture systems assisted by artificial intelligence have been developed by Carbon Clean for use in small and medium-scale companies. To achieve zero-emission power generation, Net Power incorporates predictive controls powered by artificial intelligence into its Allam Cycle technology. As part of their goal to remove carbon emissions, Microsoft and Climeworks, a software company, invested in direct air capture (DAC) systems powered by artificial intelligence (AI). The shift from AI models in laboratories to real-world control systems in manufacturing is illustrated by these examples (Allam et al. 2017).

AI Application in the Future for the Complete Process of CO_2 Capture

Artificial intelligence technology enables accurate predictions of the entire CO_2 capture process, encompassing the absorber and desorber columns and the rich/lean amine heat exchanger. Sipocz et al. applied artificial neural networks (ANNs) to model the complex relationships between input

and output parameters in a post-combustion CO_2 capture system that utilizes amines (Sipöcz et al. 2011). A rate-based process simulation (CO2SIM) was implemented to generate the data required for ANN training and validation. The lean loading, circulation rate, temperature, mass percentage of CO_2 in the inlet gas, removal efficiency, inlet gas flow rate, and inlet gas percentage were all input data that could be used as predictors. The anticipated or resulting parameters were as follows: (i) the pace at which CO_2 was captured, (ii) the amount of CO_2 absorbed, and (iii) the amount of heat required for operation. The LM and Scaled Conjugate Gradient (CG) algorithms were used to improve the accuracy of the predictions. The study found that the LM approach produced the most accurate forecasts for all three parameters in this study. The pre-design of a power plant that can capture CO_2 relies on these anticipated values (Aliyon et al. 2023). Fig. 2 shows the SWOT Analysis of AI Applications in CO_2 Capture Technologies.

Emphasizing Research Needs and Prospects:

There are still significant research gaps, even though we've made a lot of progress:

- There is an immediate need to develop publicly available benchmark datasets for CO_2 capture across various processes and scales.
- Improved industrial trust and transparency can be achieved through explainable AI, which aims to make AI models more interpretable.
- Combining artificial intelligence with digital twins, which are representations of processes in real time, is an exciting new development in the field of predictive control and problem detection.
- Few studies have combined artificial intelligence with energy-economic or life cycle models to evaluate comprehensive CO_2 capture strategies.
- Artificial intelligence for multi-objective optimization: little is known about how to optimise cost, energy, emissions, and operability all at once.

CONCLUSIONS

Modelling studies that attempt to predict the physical and chemical properties of the PCC process account for a large portion of the work; this study has also updated research activity on artificial intelligence applications in PCC technology. Artificial intelligence methodology typically outperforms numerical simulation and empirical correlation techniques in terms of speed and accuracy. Furthermore, artificial intelligence technology can help with design and PCC technology optimization. There is a substantial need for additional studies on the use of artificial intelligence in PCC. Hence, it is imperative to foster collaboration between PCC

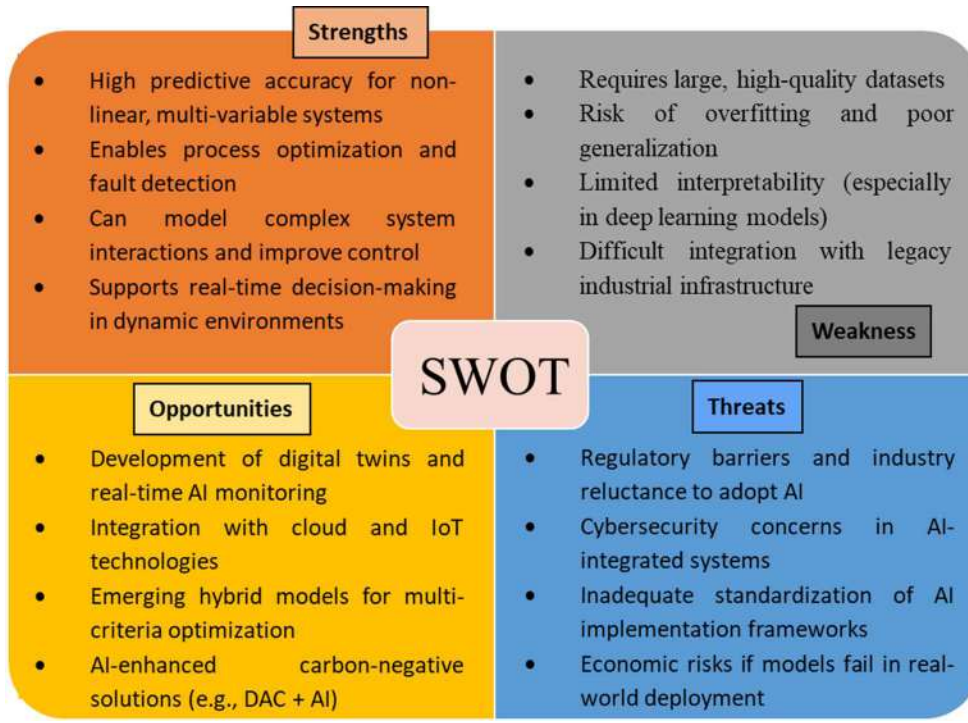

Fig. 2: SWOT Analysis: AI applications in CO_2 capture technologies.

specialists and AI researchers to advance research in this field. This study utilized surrogate machine learning models to estimate the energy and cooling utility consumption of an ACC process plant. The results indicate that surrogate machine learning models have significant potential for application in energy-related operations. Furthermore, studies suggest that specific models exhibit superior performance when provided with a limited number of data points, whereas other models outperform others when given a reduced number of input sets. Based on the data that are now accessible, one model may exhibit more superiority than the other. The economic viability of carbon-capture technologies is increasing. It is imperative to evaluate the most efficient and viable technology to minimize CO_2 emissions and achieve optimal CO_2 removal, considering economic and energy considerations. Furthermore, optimal outcomes can be attained by integrating a diverse range of machine and deep learning models with hybrid ones. As technological advancements progress, artificial intelligence techniques are likely to offer advantages in CO_2 capture. Artificial intelligence models can produce accurate outcomes by leveraging their ability to estimate variables and acquire knowledge from data. Despite the growing use of these algorithms in current research, further work is required to improve their capacities to simultaneously manage combustion and CO_2 capture systems to obtain the best possible performance. The fusion of oxy-fuel combustion technology and artificial intelligence is one such example. This process involves the combustion of fuel with oxygen instead of air, which leads to the generation of a stream of CO_2 that can be gathered and stored. Artificial intelligence can enhance the oxygen combustion process by accurately predicting the ideal conditions for CO_2 capture and burning, thereby maximizing efficiency.

As covered in the previous section, some possible CO_2 capture methods include customized greenhouse gas-absorbing devices and artificial intelligence-assisted output stream control. Many more approaches could help to reduce CO_2 emissions, however, researchers must find a good approach to allow a route to see the expansion of this industry. Analyzing the implementation of artificial intelligence in patent landscape analysis and CO_2 capture will provide carbon capture professionals with new insights. The development of emerging AI technologies will facilitate the realization of our future goals by enabling precise and instantaneous predictions.

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Valorization of Corn Cob into Cellulose-Based Bioplastics: Extraction, Fabrication and Biodegradability Evaluation

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ABSTRACT

The increasing production, consumption, and improper disposal of petroleum-based plastics are causing environmental degradation. A sustainable and environmentally friendly alternative to resolve the synthetic plastic-based problem is bioplastic. Agricultural waste, rich in cellulose, can be used as a raw material for bioplastic production and supporting circular economy goals. The purpose of the current study is to isolate cellulose from corn cob using alkali and bleaching treatment, and its utilization in the synthesis of bioplastic. The study also incorporates the sensory evaluation, thickness test, and Fourier transform infrared (FTIR) spectroscopy characterization. Further, the prepared bioplastic was tested for biodegradability. The yield of extracted cellulose was 53.1±0.7%. Bioplastic was successfully prepared using the solvent casting method, which was confirmed by FTIR analysis. The range of thickness was between 0.37±0.07 mm-0.45±0.06 mm. The degradation period was observed to be 21 days to 35 days. This study promotes the effective valorisation of agricultural residue, corn cob, and proposes an environmentally responsible waste management strategy. The prepared bioplastic may prove beneficial in packaging applications, leading to reduced reliance on fossil fuels and environmental pollution.

INTRODUCTION

Plastic is unmatched because of its qualities and is used daily to prepare many key commodities from textiles and pharmaceuticals to industrial manufacturing, packaging, and agricultural mulch production (Ghasemlou et al. 2022, Kumari et al. 2023). Over the past ten years, the world's consumption of synthetic plastics has grown significantly due to their better performance and low cost. However, the widespread usage of plastic products and the disposal of these materials have resulted in considerable toxicity (Brodin et al. 2017), and many nations acknowledge plastic pollution as a serious environmental issue (Ghasemlou et al. 2022). Many methods, such as recycling and burning, are already employed to treat plastic trash; however, they have not been able to address or reduce the environmental issues brought on by plastic pollution (Sardon & Dove 2018). Because of its non-biodegradable nature and difficulty in recycling, there are serious concerns for human health and the environment (Kowser et al. 2025). The release of harmful gases and other toxic compounds during the production and disposal of plastic waste causes air and water pollution (Adekanmbi et al. 2024). Accumulation of microplastics and leaching of harmful chemicals present in plastic waste result in soil pollution (Li et al. 2024). Plastic contains additives like phthalates and bisphenols, which cause serious health effects like reproductive disorders, endocrine disruption, diabetes, and cardiovascular diseases. Ingestion or inhalation of microplastics results in digestive and respiratory disorders (Naidoo & Rajkaran 2020, Kawa et al. 2021).

Thus, global regulations about plastic use highlight the need for innovative materials that ensure food safety, maintain quality, and protect the environment (Kowser et al. 2025). Bioplastics (BP) are gaining popularity as a biodegradable substitute for petroleum-based plastics and have the potential to significantly lower environmental plastic pollution (Ahsan et al. 2023). They are mostly made of renewable resources like starch, cellulose, proteins, and so forth (Wicaksono et al. 2022). Bioplastic can be prepared from agricultural waste using various processes, such as acidification, hydrolysis, and microbial fermentation (Babu et al. 2013, Şahin et al. 2021). Their special qualities, including their low cost, nontoxicity, biodegradability, biocompatibility, and ability to produce thermoplastic items, guarantee a substantial contribution to sustainable development (Ghasemlou et al. 2022). Various microorganisms can enzymatically break down bioplastics into environmentally benign inorganic chemicals or biomass (Silva et al. 2023). This is frequently followed by microbes assimilating and mineralizing the polymer fragments, producing mineral salts, carbon dioxide, water, methane, and biomass (Lv et al. 2017).

Starch-based bioplastics are very popular in the market and are produced using different sources (European Bioplastics & Nova-Institute 2021). A number of research studies have been carried out on bioplastic production using starch. Kulshreshtha et al. (2017) created a building material based on maize starch. Zakaria et al. (2018) prepared the potato starch film using glycerol as a plasticizer with the help of the solution casting method. Borges et al. (2015) examined the characteristics of biodegradable films made from various starch sources by varying the plasticizers. Kowser et al. (2025) prepared corn starch-based BP with glycerol and vinegar. Despite having high processing capabilities, starch-based BP are not suited for most common uses due to several drawbacks, including their inherent hygroscopic nature and poor mechanical performance, low thermal stability as compared to traditional petroleum-based bioplastics (Al-Jahwari & Pervez 2019, Kaboorani et al. 2021), which also restricts their use in various industries.

Another most prevalent biopolymer, cellulose, is used in papermaking, textiles, and bioplastics. Wood, cotton, and agricultural waste are renewable plant sources from which cellulose is obtained (Wakudkar et al. 2025). While cellulose and starch share a monomer unit, their polymeric chains are oriented differently (Qasim et al. 2021). The functional properties of the starch-based bioplastic can be improved by the amendment of cellulose (Cheng et al. 2021, Raza et al. 2023). Corn cob, an agricultural residue, consists of three tissues: chaff, woody ring, and pith, and structurally supports the kernels. Approximately 1.2 billion tonnes of maize (corn) were produced worldwide, ranking second to sugarcane (FAOSTAT 2019), and thus, a large amount of corn cobs is also produced. Being locally available, corn cobs can be used as a low-cost material to isolate cellulose. As corn cob remains underutilised, the current study focuses on examining their potential as a sustainable and renewable raw material for bioplastic synthesis.

The objectives of this study include isolating cellulose from agroresidue corn cob and its successful inclusion in preparing bioplastic. The prepared bioplastic was characterized using FTIR, and a thickness test was performed. A sensory evaluation was also performed. A biodegradability test was performed to analyse whether the prepared bioplastic degrades under natural conditions and the time taken.

MATERIALS AND METHODS

Materials

Corn cob was collected from a local sweet corn (*Zea mays L.*) seller near Maharshi Dayanand University, Rohtak. Sodium hypochlorite and sodium hydroxide were provided by Loba Chemie and CDH, respectively. Glycerol, used as a plasticizer, was supplied by CDH. All chemical compounds used in this experimental study were of analytical grade.

Isolation of Cellulose from Corncob

For cellulose isolation, the collected corn cobs were washed to remove dirt and impurities, followed by sun drying. Further, the corn cobs were cut into small pieces, ground into fine powder, and sieved using a sieve of mesh size 0.2 mm. Corn cob powder was dewaxed using a solution of ethanol and deionised water in 1:1 v/v for four h, then boiling the mixture for 1.5 h, followed by multiple washes with distilled water and oven drying. 10 g of dewaxed corn cob was treated with 200 mL of 5% NaOH solution for two h and washed to remove the remaining alkali. The alkali-treated corn cob was then bleached using a 1.5% NaOCl solution for half an hour, and then it was washed until a neutral pH was achieved. The extracted cellulose sample was oven-dried for 16 h at 50°C, then crushed into a fine powder, and stored in ziplock bags (Ungprasoot et al. 2021, Melesse et al. 2022, Khiewsawai et al. 2024). Fig. 1 shows the process of extracting cellulose from corn cobs.

Cellulose yield was calculated using Eq. (1).

$$Yield\ (\%) = \frac{W2}{W1} x100 \quad \ldots(1)$$

Where W1= = Initial weight of corn cob powder, and W2 = Final weight of extracted cellulose.

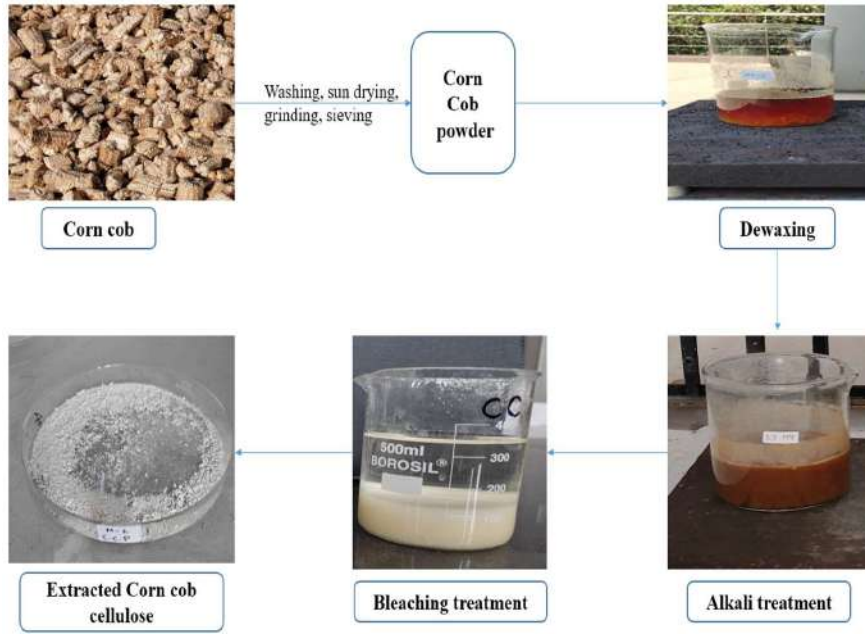


Fig. 1: Schematic of Cellulose Extraction from Corn Cob Using Alkali Treatment and Bleaching.

Synthesis of Bioplastic

Three bioplastics with different ingredient percentages were created, as indicated in Table 1, and were labelled as CCB (Corncob cellulose bioplastic). An electronic weight scale is used to carefully and precisely weigh each ingredient. The bioplastic was prepared using the solvent casting method; for this, distilled water and starch were mixed and heated until the starch gelatinized. Then glycerol and corn cob cellulose were added, and the mixture was heated at 65°C for half an hour with continuous stirring to avoid clumping. The prepared filmogenic solution was uniformly spread on a silicon sheet and oven dried at 60°C for 7-9 H (Chowdhury et al. 2022). The method of bioplastic synthesis is illustrated in Fig. 2.

Characterization

Sensory evaluation of prepared bioplastics: Bioplastic films were examined for cracks, color, texture, and

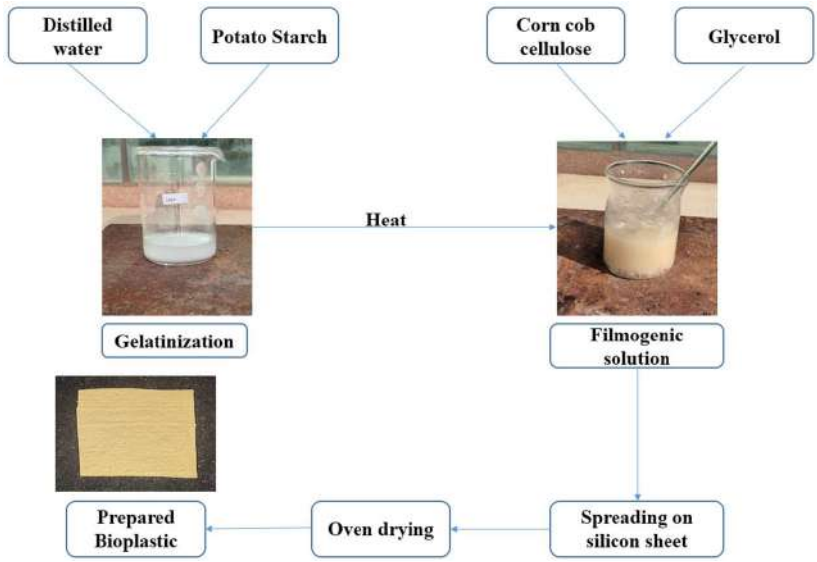


Fig. 2: Process flow for the preparation of bioplastic using extracted cellulose.

Table 1: Ingredients and their proportions in bioplastic samples.

Bioplastic Sample	Corn cob cellulose (%)	Starch [%]	Glycerol [%]	Distilled water [%]
CCB1	5	5	3	87
CCB2	10	5	3	82
CCB3	15	5	3	77

overall appearance during the sensory evaluation. These characteristics are crucial since they significantly impact how good is the final product.

Thickness: Each film's thickness was measured using an electronic digital caliper at three randomly chosen locations (Admase et al. 2022). The average film thickness was determined.

FTIR: The examination of a wide range of materials, including polymers, inorganic chemicals, and organic molecules, is frequently done using the Fourier transform infrared spectroscopy (FTIR). It includes using infrared light to detect the samples and tracking variations in the absorption bands, which yield important details regarding the materials' composition. The sample is exposed to a range of infrared radiation during the examination, while some of the radiation passes through, some is absorbed by the sample. The sample transforms the absorbed radiation into rotational and vibrational energy. Fourier transform infrared (FTIR) spectroscopy was used to examine the functional groups. Bruker's Invenio ® FTIR spectrometer was employed in the current study.

Biodegradability Test: The prepared bioplastic samples were cut into square shapes, weighed 0.5 g, and buried under soil. The films were checked at 7 days, 14 days, 21 days, 28 days, and 35 days. An accurate electronic balance was used to measure the initial and final weights (Chowdhury et al. 2022). Garden soil was used for this experiment. The pH, temperature, and moisture content of the soil during the biodegradation test were maintained at 7, 35°C, and 50%, respectively. An LDPE plastic bag was taken as a control. Eq (2) was used to determine the film's biodegradability.

$$\text{Biodegradation (\%)} = W3 - W4/W3 \times 100 \quad ...(2)$$

Here, W3 = the bioplastic sample's initial weight.

W4 = the bioplastic sample's final weight.

The statistical significance of degradation across various samples and time periods was evaluated using one-way ANOVA, which was followed by Tukey's test (for inter-group comparisons) and Dunnett's test (for comparison with control).

RESULTS AND DISCUSSION

Cellulose Yield

The experiments were conducted in triplicate, and the cellulose yield is shown as their means and standard deviation. The yield of cellulose extracted from corn cob in the present study was 53.1±0.7%. Wakudkar et al. (2025) found that the amount of cellulose in corn cob biomass is 42.3%. Thus, the yield of cellulose in the present study is 10.8% higher than that of corn cob biomass. However, using alkali and bleaching treatment, they isolated 88.13% cellulose, which is comparatively higher. Kapdi et al. (2024) used akali pretreatment and acid and bleaching pretreatment to obtain highly purified cellulose from rice straw, and the yield was 58%, higher than the yield in the present study. Sinaga et al. (2018) extracted 17.4% of cellulose, which is lower than the current study. Overall, the yield of cellulose was found to lie within the range of previous literature.

Sensory Evaluation

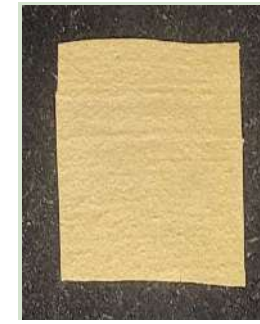
Sensory evaluation of bioplastic films analysed the texture, physical appearance, and color (Table 2). Control (LDPE) was transparent and aroma-free. Apart from control, all three bioplastic films were also free from any kind of smell. The colour of these films varied from pale white to creamish yellow. CCB1 (5% cellulose) was pale white in colour, possibly due to the low concentration of cellulose. CCB2 (10% cellulose) and CCB3 (15% cellulose) were creamish to creamish yellow, respectively. This can be attributed to the high cellulose content used for their preparation. CCB1 and CCB2 exhibited smooth texture, whereas CCB3 had one smooth surface and the other was slightly stiff and grainy.

Overall, the work was in harmony with other researchers who worked on similar material. Azmin et al. (2022) also observed that a higher amount of kenaf resulted in the formation of darker bioplastic. In the work of Azmin et al. (2020), colour varied from brown to greenish grey, and they also observed that the color darkens with the rising concentration of cellulose. Apart from this, they noticed a sweet smell in the bioplastic, which was absent in the present study.

Thickness

The thicknesses of CCB1, CCB2, CCB3, and control were found to be 0.37±0.07 mm, 0.39±0.02 mm, 0.45±0.06 mm, and 0.12±0 mm, respectively, as shown in Fig. 3. All the samples exhibited a thickness higher than the control. A minimum thickness of 50 μm is prescribed by the government

Table 2: Sensory evaluation results of control and bioplastic samples.

Sample Codes	Control	CCB1	CCB2	CCB3
Physical Appearance				
Color	Transparent	Pale white	Creamish	Creamish yellow
Texture	Smooth	Smooth	Smooth	Slightly hard and grainy
Smell	Absent	Absent	Absent	Absent

of India under the Plastic Waste Management Rules, 2016, for packaging material. All the prepared samples exhibited a thickness higher than 50 µm. Thus, it can be said that the prepared bioplastic passes the thickness criteria for packaging films (Marichelvam et al. 2019).

Marichelvam et al. (2022) prepared bioplastic using *Prosopis juliflora* with a thickness of 390 µm. In another study, corn starch and rice starch were used for bioplastic preparation, and the prepared films had a thickness of 250 µm. A study by Oluwasina et al. (2024) and Majamo & Amibo (2024) showed that the thickness of bioplastic ranged from 0.21 mm to 0.48 mm and 0.410 mm to 0.421 mm, respectively. The thickness of bioplastic in the current study increases with cellulose concentration. This observation was in harmony with the previous literature. Overall, the thickness is affected by the viscosity of the prepared solution, the casting mould, and the concentration of cellulose.

Fourier Transform Infrared (FTIR) Spectroscopy

The chemical structure of the prepared bioplastic was characterized by Fourier Transform Infrared (FTIR) spectroscopy, and the resulting spectrum is shown in Fig. 4. The FTIR analysis revealed several characteristic absorption bands corresponding to the functional groups of cellulose, starch, and glycerol, indicating successful blending of these components. A broad and intense peak was observed at 3333 cm^{-1}, corresponding to the O–H stretching vibrations of hydroxyl groups commonly present in cellulose, starch, and glycerol. This suggests the formation of a strong hydrogen-bonding network within the bioplastic matrix (Majamo & Amibo 2024). The peak at 2921 cm^{-1} was assigned to the

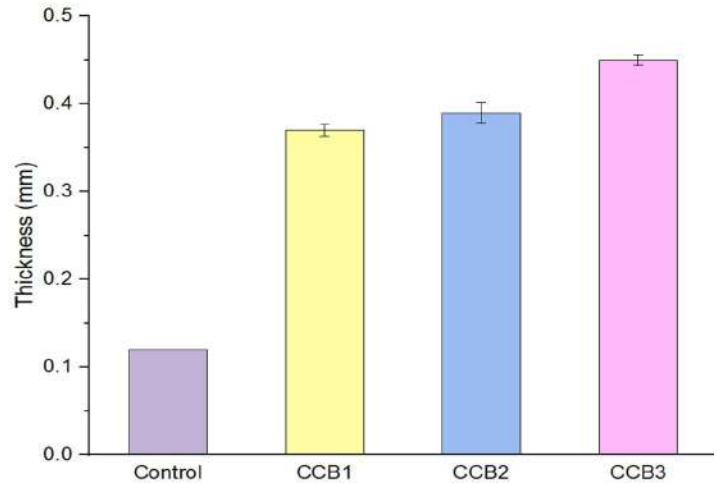


Fig. 3: Thickness test results of control and prepared bioplastics.

C–H stretching vibrations of aliphatic –CH_2 groups from the polysaccharide backbone and glycerol molecules (Oluwasina et al. 2024).

An absorption band at 1734 cm^{-1} was attributed to C=O stretching vibrations, indicating the presence of carbonyl groups. This may suggest partial esterification or interaction between glycerol and the hydroxyl groups of polysaccharides during bioplastic formation (Majamo & Amibo 2024, Chowdhury et al. 2022). A band at 1550 cm^{-1} was associated with the cyclic alkene C-C stretch. Furthermore, the peak at 1033 cm^{-1} was attributed to the aliphatic ether stretch (Bhutta et al. 2022). Overall, the FTIR results confirm the successful incorporation of cellulose, starch, and glycerol without significant chemical modification, indicating that the bioplastic primarily relies on physical blending and hydrogen bonding for structural integrity.

Biodegradability Test

The results highlighted that CCB1 and CCB3 showed the lowest and highest degradation times of 21 days and 35 days, respectively. The degradation period for CCB2 was found to be 28 days. The percent weight loss at different time intervals is shown in Fig. 5. Results of the statistical analysis revealed that there was a significant difference ($p \leq 0.05$) in the degradation of all bioplastic samples as compared to the control. A significant difference ($p \leq 0.05$) was also observed in the weight loss (%) among day 7, day 14 and day 21, highlighting that the degradation of bioplastic is enhanced with the passage of time. Meanwhile, no significant difference was observed between day 28 and day 35. These results imply that significant deterioration occurred over the first 21 days, reaching total (100%) degradation by Day 35.

It is evident from the findings that there is a noticeable increase in the rate of degradation with an increase in time. The rate of degradation is highly affected by the concentration of cellulose used. The present work is consistent with Kowser et al. (2025), who observed the same. Apart from this, the moisture content and addition of glycerol

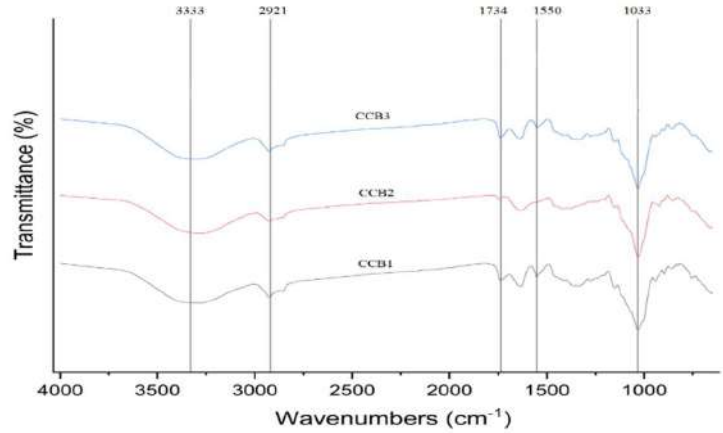


Fig. 4: FTIR Analysis of bioplastics prepared from corn cob-derived cellulose.

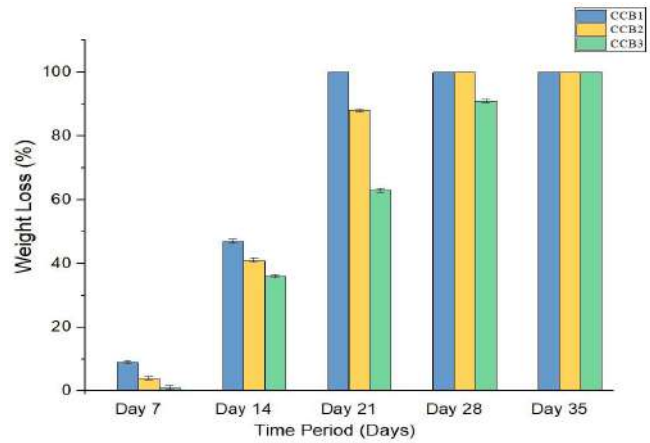


Fig. 5: Weight loss (%) of bioplastic samples during biodegradation over time.

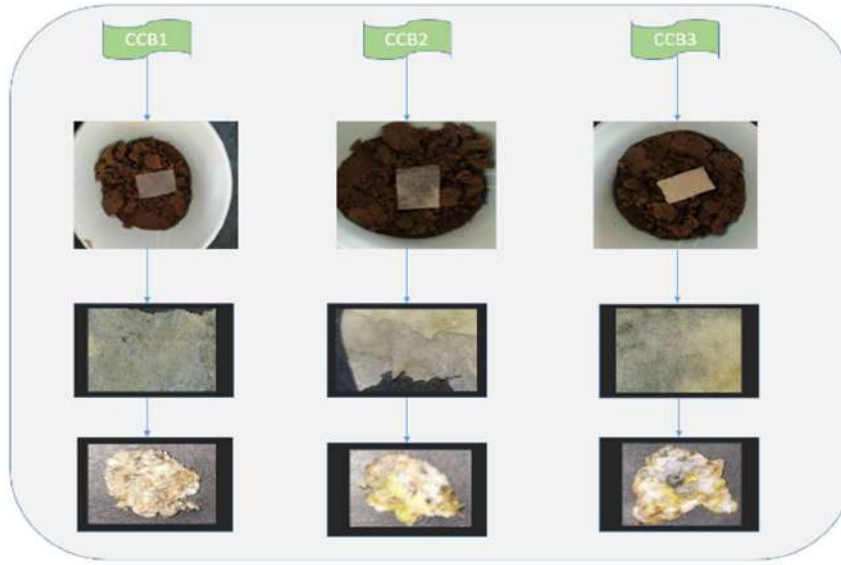


Fig. 6: Visual appearance of bioplastic samples at different stages of biodegradation.

enhanced the process of degradation of bioplastics (Sultan et al. 2024). Fig. 6 depicts the different stages of bioplastic degradation. In the initial phase, i.e., the first seven days, no significant mass loss was observed. This can be attributed to the adaptability the microorganism requires to synthesize specific enzymes required for bioplastic degradation (Pathak 2017, Chowdhury et al. 2022).

Chowdhury et al. (2022) observed that mass loss % increased from 36% to 50% for two different bioplastic samples in a duration of 7 days to 30 days. Marichelvam et al. (2019) depicted a 48.73% degradation rate in 15 days for a corn and rice starch-based bioplastic. The corn starch-based samples degraded completely in 28 days, with a % mass loss increase from 34.3% to 74.3% in 21 days (Rajesh et al. 2024). Similar results have been reported by Stefani et al. (2006), Kammoun et al. (2013), Pathak (2017), Briones et al. (2020), Razak et al. (2020) and Kowser et al. 2025. No significant weight loss was observed in the control, and all the bioplastic films were completely biodegradable within 35 days.

CONCLUSIONS

Bioplastic films were prepared efficiently using potato starch and cellulose extracted from corn cob, with the inclusion of glycerol as a plasticizer, and their properties were evaluated. The colour of prepared films varied from pale white to creamish yellow, and they were aroma-free. FTIR analysis also revealed that cellulose, starch, and glycerol were successfully incorporated into the bioplastic. The major peaks were found at 3333 cm^{-1}, 2921 cm^{-1}, 1734 cm^{-1}, 1550 cm^{-1}, and 1033 cm^{-1}, indicating the presence of cellulose, starch, glycerol, and water content in the prepared films. Further, the soil burial method for the biodegradation test showed that all the prepared bioplastics underwent biodegradation and were completely biodegradable within 21 to 35 days. By converting waste into value-added bioplastics, the study supports the concept of a circular economy and provides a green alternative to conventional plastics. The developed bioplastic holds market potential in eco-friendly packaging applications. Further research should be carried out to improve the mechanical and physical properties of bioplastics.

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Assessing the Long-Term Changes in Potential Evapotranspiration and Its Impact on Agriculture in Lahaul and Spiti, Himachal Pradesh

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ABSTRACT

Long-term variations in Potential Evapotranspiration (PET) are important for evaluating climate changes that affect agriculture in sensitive high-altitude regions. This study examined PET temporal trends from 1951 to 2022 and assessed the agricultural implications for the cold desert district of Lahaul and Spiti in the Indian State of Himachal Pradesh. Monthly PET data were obtained from the CRU TS v4.07 dataset and computed using the FAO Penman-Monteith method. Non-parametric statistical tests used to detect seasonal and annual trends were the Mann-Kendall (MK) test, the Modified Mann-Kendall (m-MK) test, and the Innovative Trend Analysis method. Increases and decreases across all seasons were statistically significant, with the strongest negative trend observed during the agricultural season (April–October), with MK Z-values ranging from –6.47 to –2.92 and m-MK values from –12.16 to –2.14. The annual PET declined at a rate of –0.0030 mm/year at Grid Point 1 (Z = –7.04). These results suggest that declining atmospheric moisture demand may reduce crop irrigation requirements. However, changes in evapotranspiration, cropping intensity, and protected cultivation systems might also increase disease susceptibility. The study stresses the need to plan adaptive water and crop management strategies that align with the changing PET scenario.

INTRODUCTION

Potential Evapotranspiration (PET) is the atmospheric demand for moisture, which is the quantity of water that would evaporate and transpire from a surface if water were not limited (Allen et al. 1998). As a diagnostic entity in the hydrological cycle, PET induces losses through actual evapotranspiration, fluctuations in soil moisture, and irrigation needs. It is of utmost importance for planning water-related agricultural production, as its changes will be adapted to the water demands of the crops, drought frequencies, and yield capacities. Variations in PET analytes can significantly disrupt the agroecological balance, particularly in agroecosystems that are predominantly rainfed or depend on glacial meltwater. This makes the long-term trend analysis of PET an important parameter for studying the impacts of climate change on agricultural sustainability.

Over the past few decades, global climate change has altered the climatic variables that govern PET, including air temperature, wind speed, solar radiation, and humidity (Donohue et al. 2010). PET trends, primarily linked to global warming and atmospheric stability, have been reported in studies across Europe, Asia, and North America. These changes have made hydrological modeling and agricultural forecasting more complex, particularly in arid and mountainous regions, where small changes in evapotranspiration can have outsized effects on water availability (Sheffield et al. 2012). In agricultural systems, PET is a measure of evaporative demand and an indicator of crop stress, irrigation scheduling, and the length of growing seasons (Fisher et al. 2011). Higher PET can cause water shortages, reduced soil moisture, and lower productivity, especially when precipitation does

not increase proportionally, particularly for water-intensive crops. Recent investigations have revealed the influence of seasonal precipitation regimes on PET, surface water, and vegetation dynamics in Baghdad's urban ecosystem (Abd Al Rukabie et al. 2024).

PET patterns in India have exhibited substantial spatial variability, with large increments observed in the north-western Himalayan belt, semi-arid areas, and Indo-Gangetic plains (Kumar et al. 2010, Mall et al. 2011). There is a concern with the Himalayan region primarily because of the critical source of major flowage, glacial water storage, and agroecosystems to climate fluctuations. National studies have shown increasing temperature trends and changing precipitation regimes. However, few region-specific studies on PET variation exist, especially in high-altitude cold desert zones. This implies a significant gap in understanding how atmospheric water demand changes over time in vulnerable mountain ecosystems and what this means for food and water security.

Across the wider Indian Himalayan region, Potential Evapotranspiration (PET) trends show considerable variability across altitudinal gradients, seasonal radiation fluxes, and changing snowfall amounts. Eastern Himalayan states such as Sikkim and Arunachal Pradesh have witnessed upward PET trends due to warming and a shortening of the snow period. In contrast, the western Himalayas exhibit more complex patterns, often shaped by topographic heterogeneity and changing monsoon behavior (Dimri et al. 2022, Kalubarme & Sharma 2006). Previous studies by Kumar et al. (2023) and Mall et al. (2011) reported increases in PET for the northwestern Himalaya; however, these studies have often overlooked sub-district-level areas or cold desert ecosystems. Region-specific investigations are largely temperature-oriented and therefore provide little insight into the seasonal variability of PET under changing atmospheric demands. This highlights a crucial research gap: climatic studies on PET trends in high-altitude cold deserts are relatively few and far between, given their susceptibility to moisture fluxes and climate variability.

In the northwestern Himalayas of Himachal Pradesh, Lahaul and Spiti constitute a cold desert agroecological zone owing to their distinctive topography and climate. An extremely low precipitation regime (less than 200 mm per year), high solar radiation, and seasonal glacial meltwater define the climate-sensitive agriculture in this region (Negi & Joshi 2004). The growing season is short, typically from May to September, and irrigation relies on snowmelt sources. The region is highly sensitive to increases in PET. Higher PET intensifies crop water stress, hindering sowing and harvesting operations and increasing the incidence of agricultural droughts. Under warming phases with aberrant precipitation regimes and diminishing glacier reserves, these impacts are expected to be magnified (Shrestha et al. 2012, Immerzeel et al. 2010). Despite these risks, empirical studies on long-term PET trends in Lahaul and Spiti are scarce. Most studies in the Himalayan region have focused on temperature and precipitation trends or have provided only regional-level forecasts without local calibration. Furthermore, only a handful of studies have systematically quantified PET trends using suitable statistical methods that account for autocorrelation, data distribution, and nonlinear trends. Given the strategic importance of this district for food security and ecological equilibrium, this gap must be filled on a priority basis.

This study addresses this gap through a detailed temporal analysis of PET trends in Lahaul and Spiti over 72 years (1951-2022). PET values were computed using the FAO Penman-Monteith method, the most accurate and standardized approach for assessing evapotranspiration under various climatic conditions (Allen et al. 1998). For trend detection and interpretation, the study relied on three non-parametric statistical techniques: the Mann-Kendall (MK) test (Hirsch et al. 1982), the Modified Mann-Kendall (m-MK) test, which corrects for serial correlation (Hamed & Rao, 1998), and Innovative Trend Analysis (ITA), which is more sensitive to nonlinear and segmented trends (Şen 2012). Individually, these techniques can detect trends in long climatic datasets. In addition to trend detection, this study investigated the interplay between PET and agriculture in Lahaul and Spiti, focusing on crop water requirements, seasonal moisture balance, and potential changes in cropping patterns. By integrating meteorological data with agricultural indicators, this study aims to provide actionable outputs for local policymakers, agricultural planners, and climate adaptation agencies. The study's findings will then be used to develop irrigation strategies, cropping calendars, and resource allocation frameworks for the mountain region, moving from climate stress toward resilience.

Hence, this study aims to assess long-term spatiotemporal trends in PET across the Lahaul and Spiti districts over 72 years (1951–2022). The investigation is based on three principal research objectives: first, to determine seasonal and annual trends in PET using robust nonparametric statistical methods, second, to assess the spatial heterogeneity of these trends across the district, and third, to examine the possible agronomic implications of PET dynamics, crop water balance, and agricultural vulnerability in a high-altitude cold desert environment. Thus, by integrating climatological trend analysis with its agricultural context, this investigation aims to generate empirical insights into adaptive water

management and climate-resilient agricultural planning for fragile mountain ecosystems.

MATERIALS AND METHODS

Study Area

The Lahaul and Spiti district is the largest in Himachal Pradesh, with Keylong as its headquarter. With a geographical area of approximately 13,833 square kilometres, it is located in the northeastern part of the state between latitude 31°44′57″N and 32°59′57″N and longitude 76°46′29″E and 78°41′34″E (Fig. 1). It shares districts of Kullu on the southern side, Kangra on the south-western side, Chamba on the western side, and Jammu, the Union Territory, on the northern side, with the north of the east set away by the Tibetan Autonomous Region. Administratively, the district is subdivided into three subdivisions, Udaipur, Lahaul, and Spiti, which are administered from Udaipur, Keylong, and Kaza, respectively (Kumar et al. 2023a).

Recognized as a tribal region, Lahaul and Spiti fall under the Integrated Tribal Development Programme. According to the 2011 Census, it is one of the state's least densely populated districts, with a population of just 31,528. The local economy is predominantly agrarian, with nearly 80% of residents working in agriculture and allied activities. The main agricultural outputs are high-value crops such as potatoes, green peas, hops, and seabuckthorn. Lahaul and Spiti's agricultural profile includes irrigated cultivation

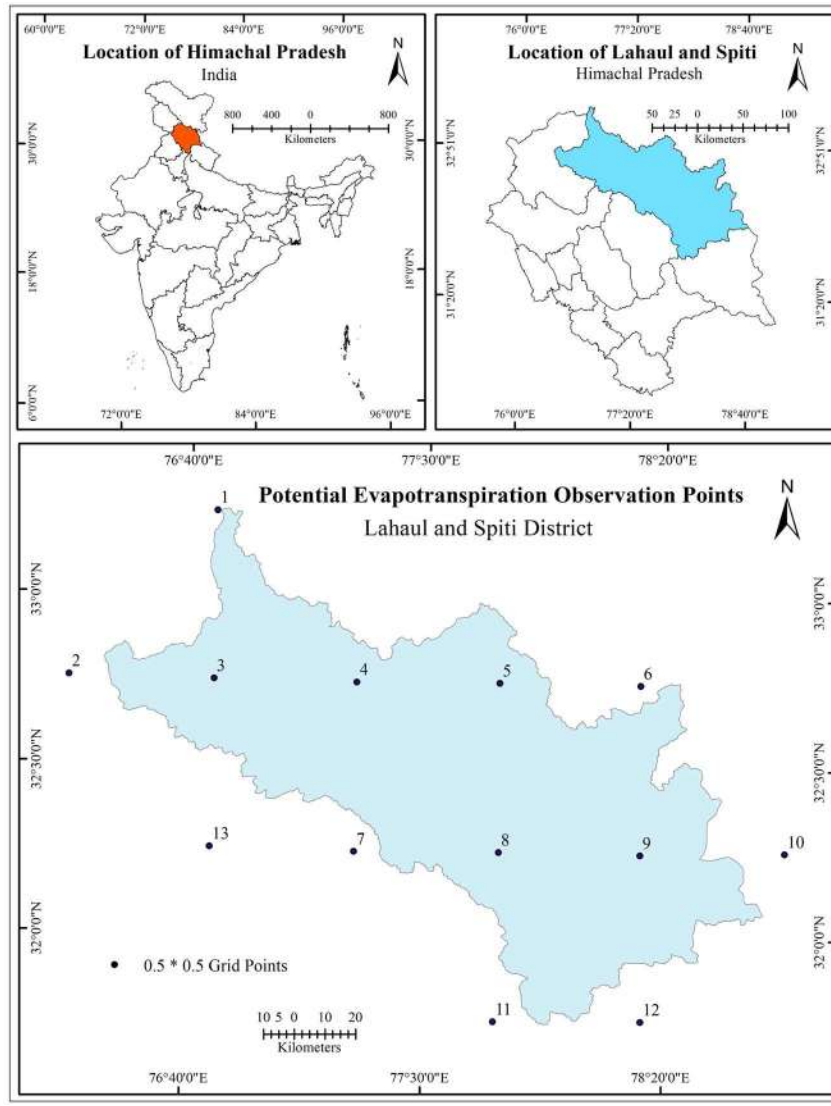

Fig. 1: Location and observation points of potential evapotranspiration.

of temperate crops, such as peas on 1,069 ha, producing 117,590 quintals, potatoes on 680 ha, producing 108,800 quintals, and cauliflower on 839 ha, producing 208,880 quintals. High yields are obtained in vegetables, whereas cereals such as wheat and barley are cultivated only in smaller areas, totaling 150 ha and producing 3,450 quintals (KVK 2021). Apart from agriculture, livestock rearing also plays a vital ancillary role in keeping rural livelihoods viable. The region has a dry temperate climate with extensive snow cover from November to April, which promotes the cultivation of temperate fruits, dry fruits, and specialty crops like hops. Owing to its remoteness, rugged terrain, and dependence on agriculture, the district still ranks as one of the least industrialized areas of Himachal Pradesh.

Data Source

The dataset used for Potential Evapotranspiration (PET) analysis was obtained from the Climatic Research Unit (CRU) Time-Series (TS) Version 4.07, made available by the University of East Anglia. The CRU TS dataset provides gridded monthly climatic variables at a relatively high spatial resolution of 0.5° × 0.5° latitude–longitude from 1901 (Harris et al. 2020). This dataset is considered the best among others because it is consistent, has global coverage, and is thoroughly validated; hence, it was used for the analyses of long-term climate trends. In the present study, PET values were extracted for 1951–2022 using latitude–longitude coordinates that define the spatial extent of the Lahaul and Spiti District.

The CRU PET data were calculated using the FAO Penman-Monteith equation (Allen et al. 1998) from temperature, vapor pressure, cloud cover, and sunshine duration to maximize the physical basis for evapotranspiration in high-altitude arid terrains. The CRU dataset was chosen for its good temporal coverage and reliability, allowing for a consistent assessment of trends in PET over seven decades. The datasets were retrieved through the Centre for Environmental Data Analysis (CEDA) platform and then processed using Python-based climate data tools for statistical analysis.

Methodology

The Mann-Kendall test is widely used to detect trends in time series of hydrologic and meteorological data, to name a few. In common usage, a test for statistical significance of trends in time series data was formally introduced by Mann in 1945 and subsequently by Kendall in 1948. Other forms of the Mann-Kendall test, such as the modified Mann-Kendall test and other new trend analysis methods, are also becoming popular. Many researchers prefer to use Sen's Slope Estimator, developed by Sen in 1968, with the MK test to establish the magnitude of a trend, if any. This allows for a better determination of the direction and rate of change within seasonal or time-dependent datasets.

Mann-Kendall Trend (MK-Test)

The Mann-Kendall (MK) test is a globally recognized non-parametric statistical approach for analysing hydro-climatological time series of various variables, for instance, precipitation, temperature, and streamflow. Thus, the method is mainly applied to detect monotonic increases and decreases in given data without assuming any distribution for the variable. Second, the relevant data series under analysis are assumed to be free of serial dependence. Serial correlation can be quite deceptive in detecting trends, to the extent that an apparent trend could be detected and concluded to exist when, in reality, there is none. To circumvent this drawback, one could engage in pre-whitening or use the Modified Mann-Kendall (m-MK) test. Both approaches ensure that the problem of serial correlation is refocused, analyzing the trends more quickly and accurately.

The test statistic S is determined using this procedure:

$$S = \sum_{k=1}^{n-1} \sum_{j=k+1}^{n} sgn(x_j - x_k) \quad \ldots(1)$$

Where n is the length of the data, and x_i and x_j indicate data values at i and j times, respectively.

$$sgn\,(x_j - x_k) = \begin{cases} +1\ if\ (x_j > x_k) \\ 0\ if\ (x_j = x_k) \\ -1\ if\ (x_j < x_k) \end{cases} \quad \ldots(2)$$

$$Var\,(S) = \left[n(n-1)(2n+5) - \sum_{i=1}^{p} t_i\ (t_i - 1)\ (2t_i + 5)\right]/18 \quad \ldots(3)$$

In Equation (3), p indicates the number of tied groups, and t indicates how many times a datum repeats. In test statistics (S), a positive S value means the trend is increasing, and a negative S value means the trend is decreasing.

The Z score value is obtained by using Equation (4)

$$z_{Mk} = \begin{cases} \frac{S-1}{V(S)}\ for\ S > 0 \\ 0\ for \quad 0 \\ \frac{S+1}{V(S)}\ for\ S < 0 \end{cases} \quad \ldots(4)$$

Modified-Mann Kendall Trend (m-MK-Test)

The adjusted variance formula is given by S, as required in the m-MK statistical test, to minimize the impact of autocorrelation coefficients on a time series. This was outlined by Hamed and Rao in 1998. The adjusted spatial and temporal variance formula of Var (S) is given as follows:

$$Var\ (S) = Var\ (S)\ \text{x}\ \frac{n}{n^*} \quad \ldots(5)$$

$\frac{n}{n^*}$ represents the modified coefficient of autocorrelated data, Equation (6), and r_k represents the autocorrelation coefficient of k_{th} Equation (7).

$$\frac{n}{n^*} = 1 + \frac{2}{n(n-1)(n-2)}\ \text{x}\ \sum_{k-1}^{n-1}(n-k)(n-k-1)(n-k-2)r_k \quad \ldots(6)$$

$$r_k = \frac{\frac{1}{n-k}}{\frac{1}{n}}\ \text{x}\ \frac{\sum_{i-1}^{n-k}(x_i-x^-)(x_i+k-x^-)}{\sum_{i-1}^{n}(x_i-x^-)^2} \quad \ldots(7)$$

Sen's Slope Estimator

Sen's method was first advocated to study the length and direction of linear trends in long-term observational data series. Known as Sen's slope estimator, this method is best for detecting consistent linear changes over time because it operates well even in the presence of outliers and extreme values that would typically interfere with conventional methods (Kumar et al. 2023b). These features make the method ideal for analyzing environmental and climatic datasets, which are generally highly variable and contain anomalies. It has been widely used to assess trends in climatic variables, such as temperature and precipitation. In this method, the slopes Qi are computed for all possible pairs of points in time within the time series of the observation data. These slope values provide information on the speed and direction of the change over time. Each Qi is calculated from a simple, valuable formula for verifying trends of an environmental variable over time.

$$\text{Q}_\text{i} = \frac{\text{Y}_\text{j}-\text{Y}_\text{i}}{j-1} \quad \ldots(9)$$

Where $i = 1$ to $n - 1$, $j = 2$ to n, Y_j and Y_i are data values at time j and i ($j > i$), respectively. If, in the time series, there are n values of Y_j, estimates of the slope will be $N = n(n - 2)/2$. The slope of the Sen estimator is the mean slope of such slopes' N values. The Sen's slope is:

$$\text{Q}_\text{ij} = f(x) = \begin{cases} \frac{\text{Y}_\text{j}-\text{Y}_\text{i}}{j-1}, & if\ n\ is\ odd \\ \frac{1}{2}\left(Q\frac{N}{2} + Q\left[\frac{N+2}{2}\right]\right), & if\ n\ is\ even \end{cases} \quad \ldots(10)$$

A positive Qi value (Qi) signifies an upward shift in the trend, while negative Qi scores indicate a decline in the trend in the temporal data.

Innovative Trend Analysis

Innovative Trend Analysis (ITA) has gained practicality and popularity in hydro-meteorological research for analyzing trends within time series data over the last few years. Refined over time, ITA has addressed many of the drawbacks of standard statistical methods (Sen 2012). Trends in time series were accepted under conditions including data normality, independence of observations, and length of the dataset. However, one distinct advantage of the ITA is that it works well even in the presence of a serial correlation, which occurs so often in environmental time series data that other methods are almost unreliable. ITA also helps analyze trends graphically, leaving no room for skewness in the trend due to extreme data points. It involves simple steps: the dataset is divided into two halves of equal size. The first observation is discarded if an odd number of observations is present. Each half was then sorted in ascending order. Next, the scatter diagram was plotted, with the first half treated as the x-coordinate and the second half as the y-coordinate. Next, a 1:1 reference line was drawn along the plot as a benchmark. Points that appear higher than this line will be taken as an indication of upward trends, while points appearing lower will indicate downward trends. A clear visual interpretation makes the ITA a highly effective and user-Santo friendly tool for recognizing complex trend patterns in hydrological and climatological datasets.

RESULTS AND DISCUSSION

The results of the analysis were generated at both annual and seasonal scales. The classifications were given by the India Meteorological Department, namely: winter (January–February), pre-monsoon (March–April–May), monsoon (June–July–August–September), and post-monsoon (October–November–December) (IMD 2022). In addition, a single growing season was observed in the high-altitude cold desert regions of Lahaul and Spiti. The duration of the study extended from April to October (Spehia et al. 2024). The MK and m-MK trend analyses showed confidence levels of 95% ($p < 0.05$) and 99% ($p < 0.01$), respectively.

Mann-Kendall Trend Analysis

The Mann-Kendall test indicates a significant decreasing trend during all the seasons. During Winter, PET showed a definite decreasing trend, with Z-values between -3.42 and -0.62. The declining trend indicates that lower evaporation rates during winter could be due to decreasing temperatures or more cloud cover, reducing water loss from soil and vegetation (Fig. 2a). The pre-monsoon period followed a distinct decreasing trend, with the respective Z-values varying from -4.59 to -2.33 (Fig. 2b). Similar trends emerged during the monsoon season, with Z-values between -4.66 and -1.73. The decrease in PET during the monsoon may be due to increased cloud cover and rainfall, with decreased

evaporation despite rising temperatures (Fig. 2c). Throughout the post-monsoon period, a significant reduction in PET was indicated with Z values between -6.69 and -3.50, suggesting that atmospheric water demand decreased consistently in the latter part of the year (Fig. 2d). The annual trend in PET indicates a long-term decreasing trend, with Z-values within

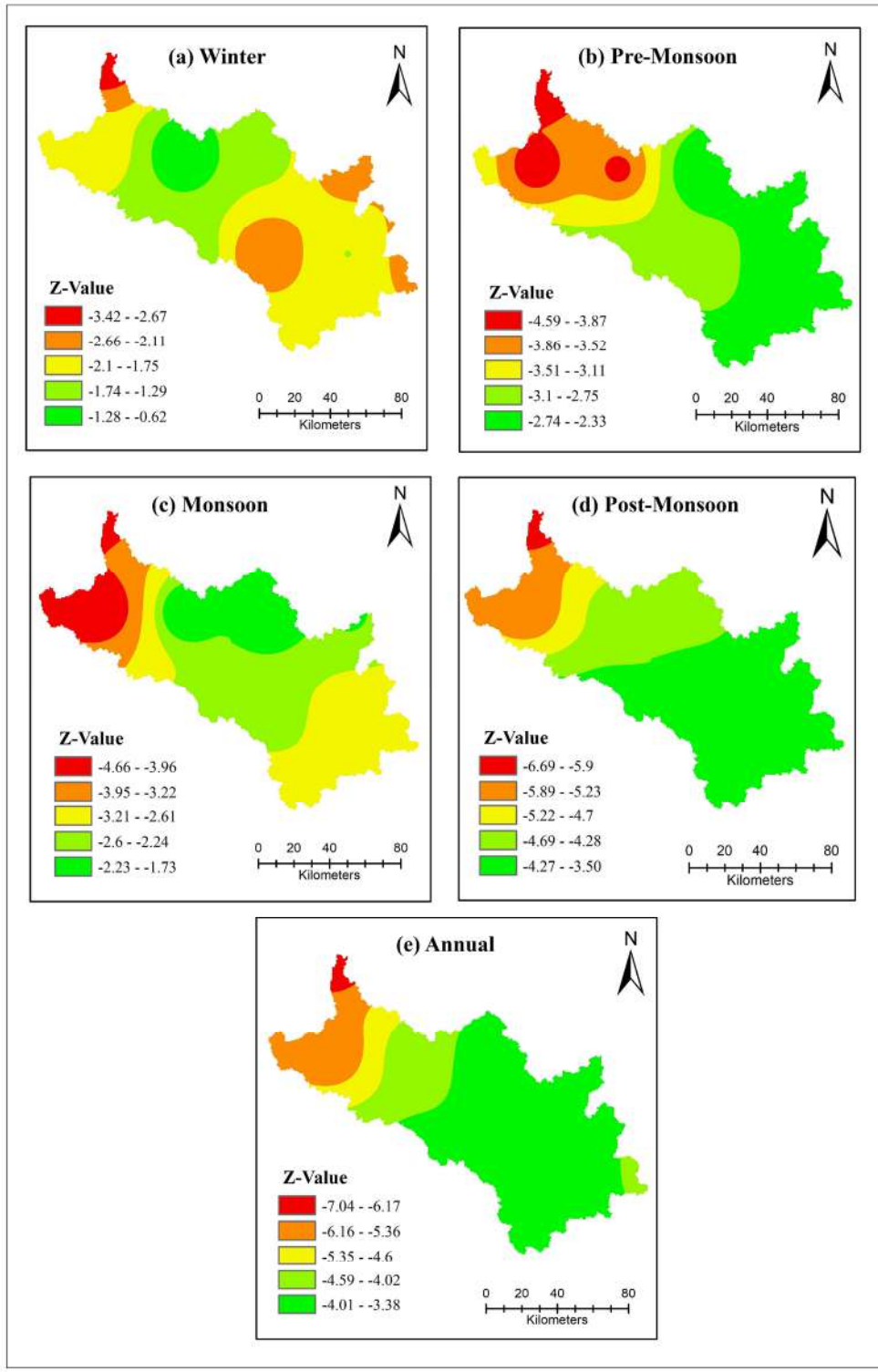

Fig. 2: Seasonal and annual spatial distribution of PET Z_{mk} values from 1951 to 2022.

Table 1: Seasonal and annual values of Mann-Kendall and Modified Mann-Kendall for potential evapotranspiration.

Grid Points	Latitude	Longitude	Mann-Kendall (Z_{mk})						Modified Mann Kendall (Z_{mmk})					
			Winter	Pre-Monsoon	Monsoon	Post-Monsoon	Agriculture	Annual	Winter	Pre-Monsoon	Monsoon	Post-Monsoon	Agriculture	Annual
1	33.25	76.75	-3.42**	-4.59**	-4.28**	-6.69**	-6.47**	-7.04**	-7.17**	-10.45**	-9.70**	-18.11**	-12.16**	-13.08**
2	32.75	76.25	-1.81	-2.96**	-4.62**	-5.35**	-5.24**	-5.65**	-2.14*	-11.13**	-12.48**	-6.38**	-10.55**	-11.61**
3	32.75	76.75	-1.98*	-4.09**	-4.66**	-5.80**	-6.03**	-6.14**	-1.86	-7.20**	-9.24**	-6.60**	-7.55**	-7.06**
4	32.75	77.25	-0.62	-3.96**	-1.73	-4.59**	-4.24**	-4.20**	-0.94	-7.72**	-2.24*	-5.58**	-4.72**	-4.53**
5	32.75	77.75	-1.61	-2.33*	-1.76	-4.32**	-3.11**	-3.47**	-1.72	-2.81**	-1.59	-5.05**	-2.54*	-2.97**
6	32.75	78.25	-2.45*	-2.55*	-2.18*	-4.25**	-2.92**	-3.38**	-1.95	-2.85**	-1.62	-5.70**	-2.14*	-2.61**
7	32.25	77.25	-1.34	-2.75**	-2.46*	-3.83**	-3.53**	-3.65**	-1.41	-3.17**	-2.66**	-4.21**	-2.99**	-3.20**
8	32.25	77.75	-2.56*	-3.03**	-2.33*	-4.06**	-3.10**	-3.59**	-3.74**	-2.67**	-1.72	-4.58**	-2.18*	-2.54*
9	32.25	78.25	-1.75	-2.50*	-2.93**	-4.11**	-3.56**	-3.81**	-1.58	-2.14*	-2.31*	-5.25**	-2.57*	-2.79**
10	32.25	78.75	-2.56*	-2.59**	-2.96**	-4.18**	-3.47**	-4.36**	-1.87	-2.33*	-2.24*	-5.55**	-2.46*	-3.10**
11	31.75	77.75	-1.90	-2.38*	-3.07**	-3.50**	-3.67**	-3.99**	-1.73	-2.06*	-2.45*	-4.23**	-2.72**	-3.01**
12	31.75	78.25	-1.78	-2.33*	-2.59**	-3.88**	-3.33**	-3.87**	-1.77	-2.00*	-2.05*	-4.71**	-2.43*	-2.80**
13	32.25	76.75	-1.57	-2.43*	-3.23**	-3.65**	-3.78**	-3.95**	-2.24*	-6.00**	-5.50**	-4.89**	-5.30**	-5.62**

* Significant at 95% confidence interval, ** Significant at 99% confidence interval

the interval of -7.04 to -3.38, indicating that there have been marked declines in the evapotranspiration rates over the decades (Fig. 2e). During the agricultural period, one of the strongest decreasing trends appeared with Z-values ranging from -6.47 to -2.92, indicating that evapotranspiration rates during the growing season have significantly decreased, which could affect water management for crops and irrigation (Fig. 3a).

The MK test showed a statistically significant declining trend in PET across all seasons. The Winter season displayed a drastic and significant downward trend in PET at various grid points. The top decreasing PET is Grid Point 1, near Chimrat Panchayat in Lahaul block, where Z = -3.42, with a significance level of 99% (magnitude = -0.0012 mm/year), thus signifying that PET across various sites during the winter season is declining. During the Pre-Monsoon season, consistent and significant decreases in PET were observed at seven grid points at 99% and six grid points at 95%. The most pronounced fall comes from Grid Point 1, near Chimrat panchayat in Lahaul block (Z = -4.59, magnitude = 0.0033 mm/year), followed by Grid Point 9, near Tabo panchayat in Spiti block (Z = -3.03, magnitude = -0.00208 mm/year), and 8, near Hull panchayat in Spiti block (Z = -2.75). PET also exhibited a decreasing trend during the monsoon season, notably at Grid Point 3, near Udaipur in the Lahaul block (Z = -4.66, magnitude = -0.0024 mm/year), with eight grid points significant at 99% and three at 95%. In the post-monsoon season, all grid points were statistically significant, with a 99% decrease in PET trend. The most significant decline can be observed at Grid Point 1, near Chimrat Panchayat in the Lahaul block (Z = -6.69, magnitude = -0.0026 mm/year), followed closely by Grid Point 3, near Udaipur in the Lahaul block (Z = -5.80), and Grid Point 2, near Tindi Panchayat in the Lahaul block (Z = -5.35). These strong trends indicate a considerable reduction in PET over time. The Agricultural season also shows a significant decrease in PET, and sizable decreases are observed at all grid points at 99% significance. The most significant decreasing trend was noted in Grid Point 1 (Z = -6.47, magnitude = -0.0025 mm/year), Grid Point 8, near Hull Panchayat in Spiti block (Z = -3.53), and Grid Point 9 (Z = -3.56). These decreases in PET during this season may indicate possible changes in the crop water requirements. Annual PET trends confirm a steady and statistically significant decline across all studied grid points, with the most substantial reductions at Grid Point 1 (Z = -7.04, magnitude = -0.0030 mm/year), Grid Point 11, near Sagnam in Spiti block (Z = -4.36, magnitude = -0.0018 mm/year), Grid Point 10, near Giu panchayat in Spiti block (Z = -3.81), and Grid Point 8, near Hull panchayat in Spiti block (Z = -3.6) (Table 1).

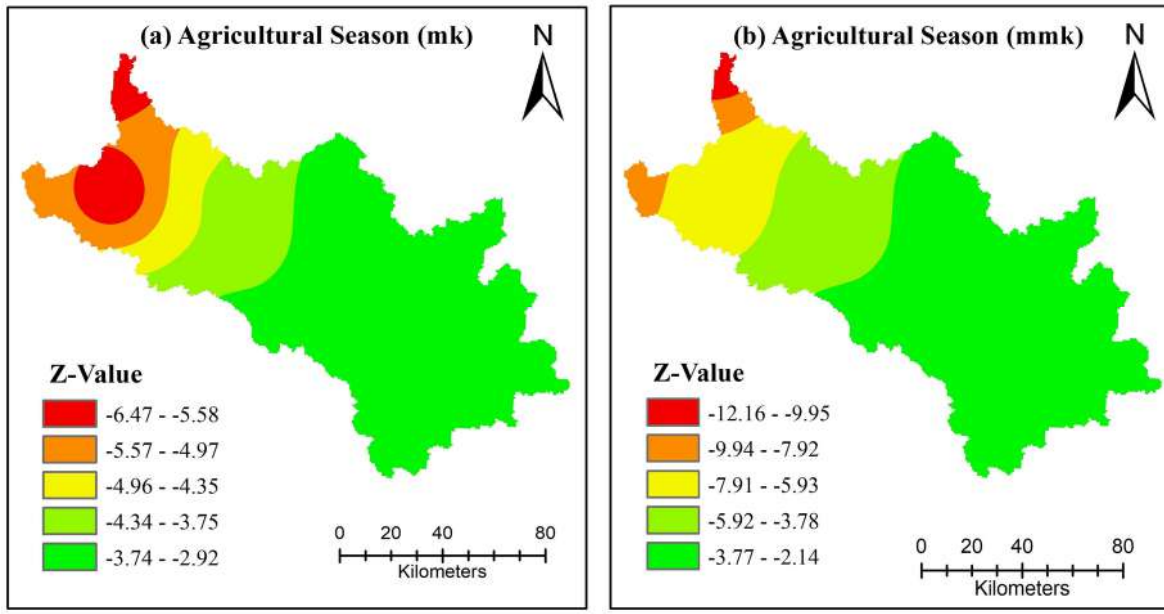

Fig. 3: Spatial distribution of agricultural season (a) Mann-Kendall (b) Modified Mann-Kendall.

Modified Mann-Kendall Trend Analysis

The m-MK test showed a remarkable decreasing trend for all seasons, indicating a decreasing atmospheric moisture demand over time. A substantial decline in PET was noted during the winter months, with Z-values ranging from -7.17 to -0.94, suggesting that the rates of winter evaporation and transpiration have been decreasing (Fig. 4a). The other significant decline in PET was observed during the pre-monsoon periods, reflected in Z-values from -11.13 to -2.00 (Fig. 4b). Monsoon PET followed the same trend, with Z values ranging from -12.48 to -1.59, suggesting a decline in PET owing to increased humidity and higher rainfall during the wet season, which would lower direct evaporation from the land surface (Fig. 4c). During the post-monsoon season, one of the periods with maximum decreasing trend characteristics was observed with Z-values ranging from -18.11 to -4.21. The significance of this reduction indicates the extraordinary rapidity of the declining trend in post-monsoon evapotranspiration, which might indicate the retention of moisture in soils for a long time and lesser drying effects in the atmosphere (Fig. 4d). The annual trend thus confirms a long-standing decrease with Z values from -13.08 to -2.54, showing a continuous decreasing trend in evapotranspiration rates over decades (Fig. 4e). Concerned about the agricultural season, an appreciable decline has also been observed in the PET values, which range from -12.16 to -2.14, implying diminished PET rates through the growing season (Fig. 3b).

In Winter, most grid points showed a significant decreasing trend in PET, whereas two grid points (99%) and two grid points (95%) displayed high significance. The most profound decline was observed at Grid Point 1 (Z = -7.17, magnitude = -0.0012 mm/year), indicating a significant reduction in the evaporative demand. Declines were also observed for Grid Points 3, 4, and 5; however, these trends were not statistically significant. Evapotranspiration rates experienced a pronounced and broad decline, and pre-monsoon season PET followed a similar, albeit less intense, pattern. In this, nine grid points are significant at the 99% level and the remaining at the 95% level. An extreme decrease was recorded at Grid Point 2 (Z = -11.13, magnitude = -0.0023 mm/year), affirming intense alterations in pre-monsoonal moisture dynamics, and at Grid Point 1, which covers Chimrat panchayat and Tingrit panchayat of Lahaul block (Z = -10.45, magnitude = -0.0033 mm/year), indicating a significant reduction in evaporative losses. During the monsoon season, PET trends decreased over all grid points, and five grid points were statistically significant at 99% confidence. The highest decrease in PET was observed for Grid Point 2, near the Tindi panchayat in the Lahaul block (Z = -12.48, magnitude = -0.0028 mm/year). The Post-Monsoon season had the steepest decrease in PET of all seasons at 99% statistically significant, with an extreme drop of 2.6 mm/year among all seasons occurring across grid point 1 (Z = -18.11). The Agricultural season also exhibited a statistically significant decrease in PET, with the most considerable reductions at Grid Point 1 (Z = -12.16, magnitude = -0.0030 mm year) and Grid Point 2 (Z = -10.54, magnitude = -0.0027 mm year). This suggests a potential progressive decline in crop water demand, necessitating the

adaptation of irrigation strategies to improve water usage while maintaining soil moisture levels. The trend analysis for PET on an annual basis confirmed a decline over the long term at all grid points, with the most significant reductions occurring at Grid Point 1, near Chimrat panchayat in Lahaul block (Z = -13.08, magnitude = -0.0025 mm/year).

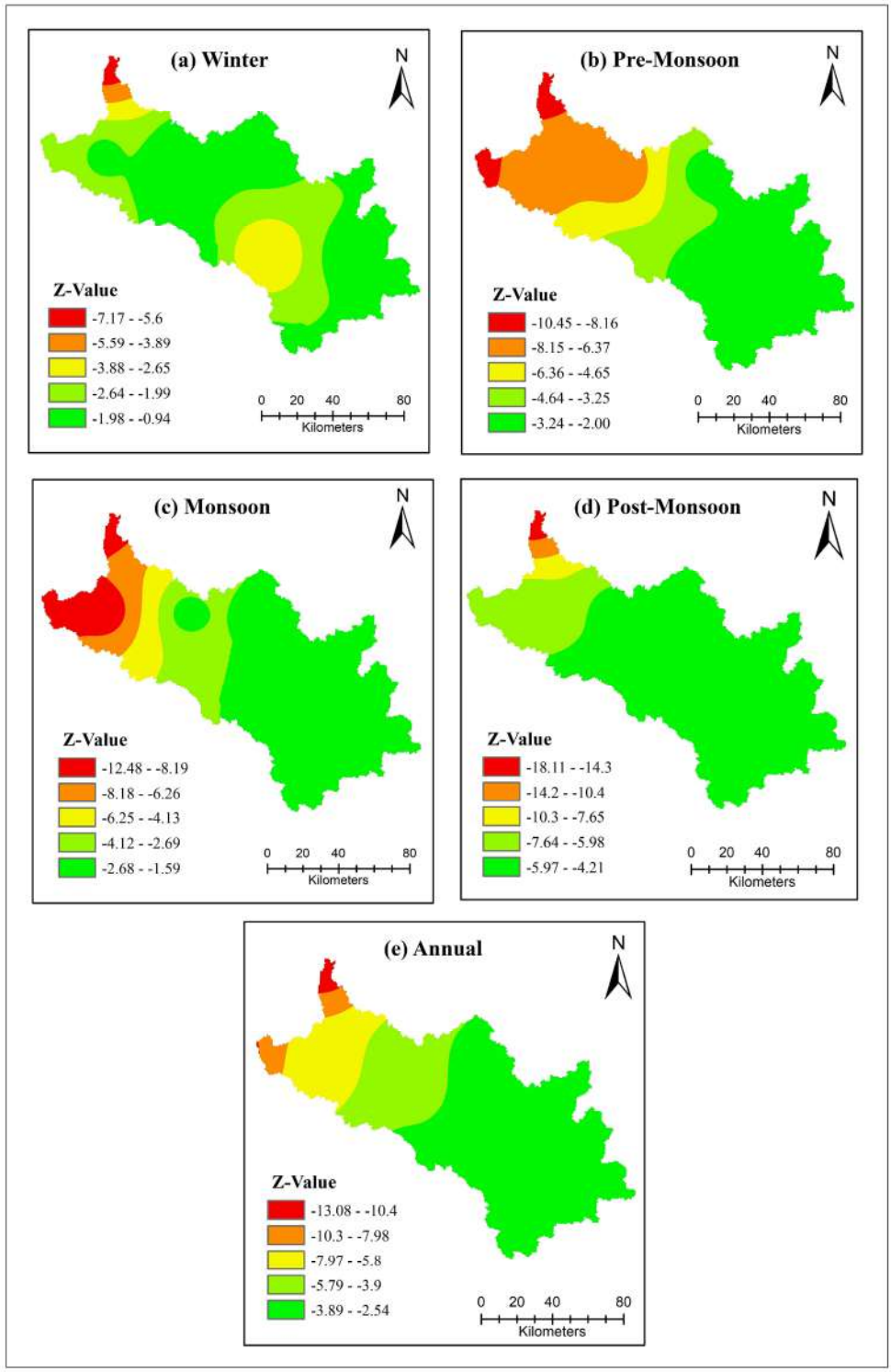

Fig. 4: Seasonal and annual spatial distribution of PET Z_{mmk} values from 1951 to 2022.

The sustained trend of decline in annual PET may signal a shift in the net evaporative dynamics related to large-scale climate variability, decreasing solar radiation, and altered atmospheric moisture columns (Table 1).

The decadal trend in annual PET (Fig. 5) reveals a marked increase from the 1950s to the 1980s, especially during 1971–80. A steady decline in potential evapotranspiration values was observed from 1991 onward, with the lowest PET values observed in the current decade of 2010–22. This trend corresponds to a gradual decrease in the atmosphere's capacity to evaporate, thus agreeing with the trends predicted by the Mann-Kendall and m-MK methods, indicating evolving hydro-climatic scenarios in the Lahaul and Spiti regions.

Innovative Trend Analysis

Innovative Trend Analysis (ITA) of PET from 1951 to 2022 indicates a decreasing trend for all seasons, indicating temporal variation in atmospheric moisture demand. The ITA compares PET for the two study periods, 1951-86 (first half) and 1987-2022 (second half), with a 1:1 reference line denoting an unchanged or no trend condition. Values below the line imply a reduction in PET, and above, an increase. Most of the data points in all six panels (Winter, Pre-Monsoon, Monsoon, Post-Monsoon, Agricultural, and Annual PET) clustered in a supposedly decreasing triangle, clearly marking declining PET trends. The decline in winter PET could have been due to less solar radiation or cooling temperatures, with respective influences declining the evaporation rate.

A similar situation was observed in the historical decline of pre-monsoon and monsoon PET, indicating a lesser atmospheric demand for moisture within these vital periods owing to temperature reduction and increased humidity. Another downward trend in the post-monsoon season, which further confirms the gradual weakening of evaporative demand, was observed. For the agricultural season, PET, a similar downward trend indicates less demand for irrigation water owing to reduced atmospheric moisture loss from the soil and crops. The annual PET trends further show the continuity of this downtrend, suggesting that long-term evaporation and transpiration potential have decreased in Lahaul and Spiti over the decades (Fig. 6).

Impacts of Potential Evapotranspiration on Agriculture

A thorough analysis of long-term Potential Evapotranspiration (PET) in the Lahaul and Spiti region, for 1951-2022, established a statistically significant and seasonally decreasing trend. The trend was determined using three non-parametric tests: Mann-Kendall (MK), Modified Mann-Kendall (m-MK), and Innovative Trend Analysis (ITA). Considering PET as a term denoting moisture demand from the atmosphere, such a continuous decrease from April to October, which is agriculturally important, will affect mountain farming in this cold desert.

Agriculture in Lahaul and Spiti is vertically oriented toward high-value crops, such as potatoes, green peas, barley, and buckwheat (Negi & Joshi 2004). The growing season is short, and irrigation remains dependent on glacial and snowmelt water. A rapid agro-economic transformation has occurred in the region, driven by infrastructural development, including the Atal Tunnel, which has improved connectivity and market access (Sherup 2023, Kumar et al. 2023a). This

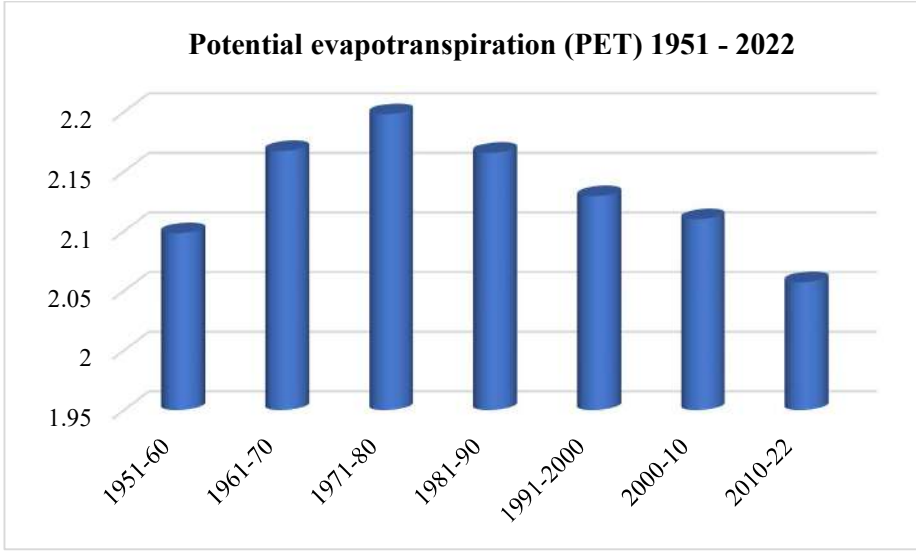

Fig. 5: Decadal variation in annual potential evapotranspiration across Lahaul and Spiti from 1951 to 2022.

has also led to expanded cultivated areas and mechanization, shifting toward polyhouse cultivation on a broader scale of intensification. Assuming that the lowering of PET is evidenced by Zmk values between -6.47 and -2.92 and Zmmk values between -12.16 and -2.14, it indicates that the moisture demand of the atmosphere dwindled.

An agricultural household survey of 295 farmers conducted in Lahaul and Spiti from May to June 2024 provided exceptional experiential clarity to these climatological observations. Farmers were largely consistent across districts, reporting increases in ambient temperatures that led to premature snowmelt and reduced water availability during the early growing stages of crops. In their perception, irrigation water demand would increase mainly at transplanting and flowering due to these changes, necessitating formal channelization of water, an adaptation new in an area where a gravity-fed kuhl system had previously been sufficient. Farmers also reported more erratic rainfall, characterized by very short, intense spells followed by lengthy dry periods, posing constraints for crop scheduling and water budgeting. These perceptual insights are supported by instrumental data and underscore the critical need to align scientific inference with local knowledge to plan for effective adaptation (Plate 1).

A decline in PET could imply less demand for irrigation; however, it conversely implies the limitation of transpiration-induced cooling, posing greater thermal stress, especially inside polyhouses, where microclimatic buffering is the least. The intricacy of this pathogen dynamic has been recorded at the international level, varying with crop species, canopy architecture and agronomic management (Raza & Bebber 2022).

Similar insights have emerged from tropical regions, where cropland expansion has significantly influenced the temporal dynamics of evapotranspiration (Laipelt et al. 2024). Their findings underscored PET's susceptibility to land-use variations, reaffirming the importance of viewing PET trends in the context of broader landscape and land-cover changes. Resemblances with international experiences further

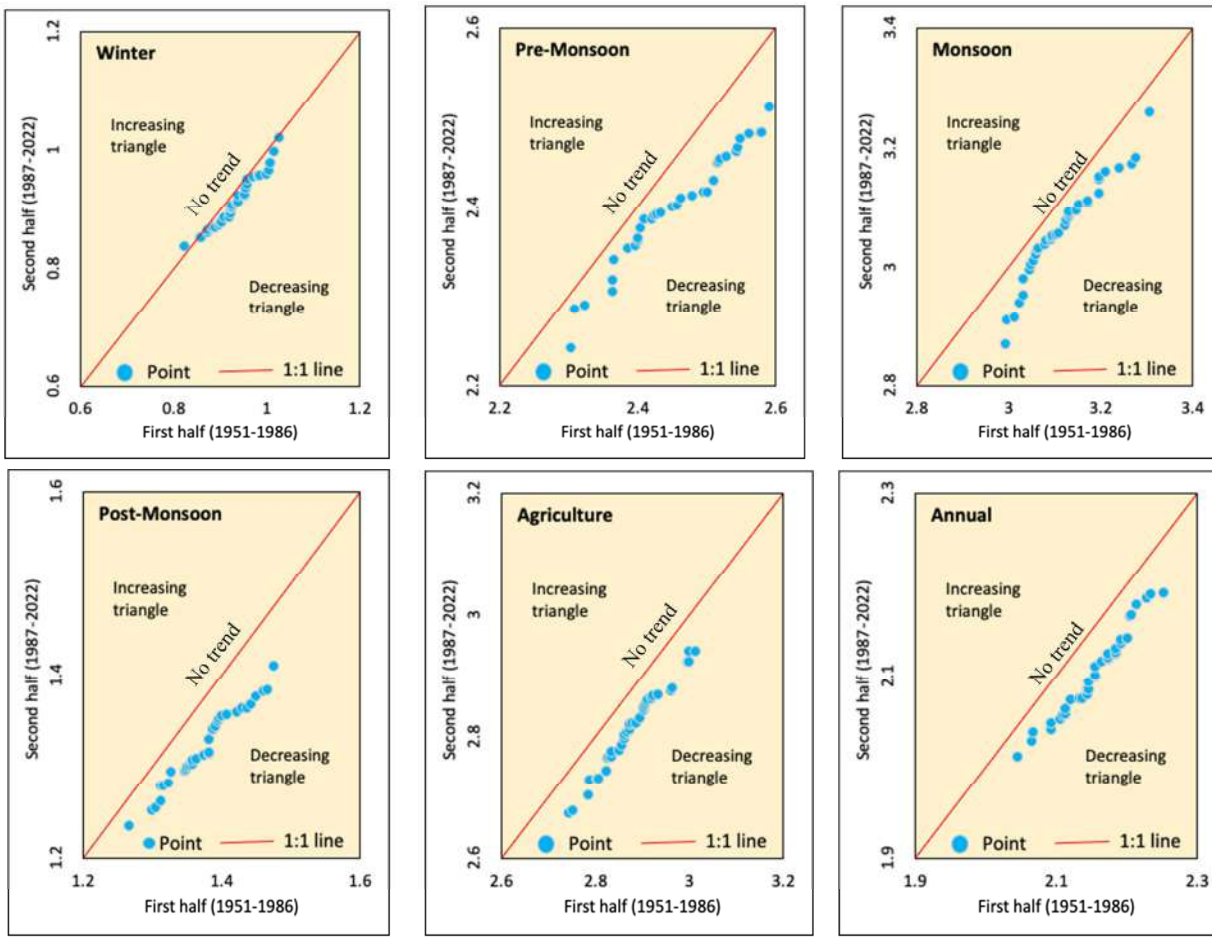

Fig. 6: Innovative trend analysis (ITA) for potential evapotranspiration during 1951 to 2022.

contribute to this debate. For example, Multsch et al. (2020) demonstrated the impacts of extensive irrigation expansion in Brazil on PET and water resource availability, Al-Hasani et al. (2022) highlighted the strong coupling of PET dynamics with phenological responses in temperate zones, further underscoring complex PET-crop-climate feedbacks.

Adaptive Strategy for Agriculture

Hence, the agrarian strategy for Lahaul-Spiti must pivot around two interlinked principles: water prudence and crop resilience. Consequently, the following adaptation measures are recommended:

- **Demand-responsive irrigation**: Adopting soil moisture sensor-based drip and sprinkler systems instead of flood irrigation can match water delivery with reduced PET levels, thus avoiding over-application and conserving limited stored snowmelt reserves.
- **Climate-smart crop choices**: Diversification can include short-duration, disease-resistant cultivars, such as Kufri Himalini (potato), along with pseudo-cereals, such as buckwheat, which have low Kc values to improve water-use efficiency (Negi & Joshi 2004).
- **Integrated Disease Management (IDM)**: Instituting weekly spore surveillance coupled with weather-based agro-advisories distributed through KVKs would go a long way in preempting outbreaks under changing PET and humidity regimes (Thakur & Klate 2023).
- **Hydrological buffers**: Constructing high-altitude lined meltwater ponds and solar-powered nighttime irrigation may provide a service ameliorate the mismatch between early snowmelt and mid-season crop water demand.
- **Polyhouse retrofitting**: Ridge ventilators and reflective mulches in protected cultivation should be installed to moderate temperature and vapor pressure deficits, discouraging pathogen infection under the lowered PET scenario.

LIMITATIONS

Despite its robust methodology and duration, the present study has some caveats that warrant mentioning. The use of CRU TS v4.07 data at a spatial resolution of 0.5° × 0.5° is a good candidate for large-scale climatological studies, but it might fall short in reflecting the topo-climatic heterogeneity of the Lahaul and Spiti mountainous terrain. Furthermore, the analysis only deals with PET, without examining the parallel variations in AET or soil moisture, which are needed to verify the agro-hydrological validity of PET trends. These would be excellent candidates for inclusion as major components of future work involving much finer-scale observations from ground research relevant to adaptive agricultural planning.

CONCLUSIONS

The present analysis of long-term trends in Potential Evapotranspiration (PET) in Lahaul and Spiti deepens the understanding of climate–agriculture interactions in high-altitude arid regions. In addition to finding declining PET trends, the study discusses how such climatic alterations intersect with changes experienced at a larger level in agricultural transformations, such as decreasing cropping intensity, irrigation practices, and microclimatic conditions. Hence, the complex nexus between the reduced atmospheric demand and changed agricultural practices is compelling enough to demand a more data-based resource planning approach. More importantly, the findings imply that PET

Plate 1: (A) A woman farmer irrigates their field using the traditional system, (B) The author conducted the primary survey in Losar village in Lahaul and Spiti district.

may be used as an indicator in conjunction with the entire hydro-meteorological and agroecological context consisting of soil moisture availability, actual evapotranspiration, and crop water requirements.

With crop planning aimed at ensuring agricultural resilience in Lahaul and Spiti, agriculture should focus on short-duration and moderately water-sensitive crops, such as barley, buckwheat, and green peas, which correspond with the restricted growing season of the area. Water conservation measures could include micro-irrigation systems, runoff harvesting from glaciers, and measures to enhance canal (Kuhl/piped) efficiency to counter seasonal variability and glacial dependency. Other climate-resilient agrarian measures may endorse technologies for protected cultivation, agro-climatic zoning, and disease forecasting systems that provide adaptive responses to changing PET patterns, humidity regimes, and thermal extremes for this ecologically fragile, high-energy terrain.

As agriculture in Lahaul and Spiti gradually intensifies due to better infrastructure and market access, the sustainability of this expansion will depend on the adaptive response to climate change. Policymakers and agricultural planners must consider the opportunities arising from decreased evaporative stress and the risks associated with the onset of humidity-induced plant diseases and thermal imbalances. Future studies on PET trends, crop modelling, water budgeting, and remote sensing-based assessments will enable us to focus on local adaptation measures. Building institutional capacity for climate-resilient planning would also be a step toward livelihood and food security in this ecologically fragile Himalayan district.

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Hydrological Model-Based Planning of Soil and Water Conservation Practices for Enhanced Watershed Saturation and Sustainable Development in a Semi-Arid Region

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ABSTRACT

The strategic use of Soil and Water Conservation (SWC) techniques is essential for efficient watershed management and hydrological processes in semi-arid areas. This study elaborates on a scientific framework following a ridge-to-valley approach for model-based planning of land and drainage line treatments to develop a stage of the watershed known as watershed saturation, where the maximum generated runoff is conserved while maintaining environmental flow downstream. Using the Soil and Water Assessment Tool (SWAT), the study modelled the hydrologic processes in the Chikkodi sub-watershed, a semi-arid region. The assessment of existing SWC measures (bunding, trenching, Checkdam, etc.) formed a key baseline analysis; it revealed a significant 26.17% runoff reduction due to the combined effect of existing SWCs and provided insights for subsequent planning. Based on the model outputs, this study recommends land treatment, that is, bunding and trenching, in the identified critical areas, which effectively intercept the runoff at the source and maximize infiltration. Conservation of the remaining runoff through drainage line treatments (check dams) is proposed as the next crucial step in the ridge-to-valley strategy. This study highlights the necessity of a science-based framework for the sustainable management of semi-arid watersheds, emphasizing that with improved watershed saturation, there is increased local water availability, which supports environmental flow. By combining the assessment of existing treatments with hydrological modeling for proactive planning, the proposed methodology provides a flexible and transferable approach to SWC practice optimization for enhancing watershed water storage in similar semi-arid landscapes.

INTRODUCTION

Water and soil are the most precious resources on the Earth's surface, which contribute to building a balanced ecosystem and promoting sustainable security of life (Hunt 2005, Pande et al. 2020, Rashmi et al. 2022). Watershed management is the process of planning and directing the use of soil, water, and other natural resources within a watershed to provide necessary products and services while minimizing impacts (Singh et al. 2014, Smyle et al. 2014, Surya et al. 2020, Santos et al. 2023). In a growing nation like India, Soil and Water Conservation (SWC) measures, such as bunding, trenching, contour farming, and check dams, are essential for sustainable resource management that balances the environment by promoting water conservation, regulating surface flow, and preventing soil erosion (Bhandari et al. 2007, Kumawat et al. 2021, Dharmawan et al. 2023).

While research highlights the complementary local influence of SWC methods, their impact at the watershed scale requires comprehensive evaluation to guide effective management plans. However, studies quantifying watershed-level impacts are limited. Field experiment data cannot be extrapolated to the watershed scale and must be tested with mathematical models to assess SWC effectiveness (Raza et al. 2021). Hydrological approaches can be used to examine the performance of soil

and water conservation practices in mitigating or triggering adverse environmental effects. Hydrological modeling is the most commonly used approach for analyzing the effects of conservation measures on hydrological processes (Liu et al. 2017). Physically based hydrological models, which account for the spatial and temporal distributions of land use, topography, soil, and management practices, are particularly well suited for evaluating the impacts of different factors on watershed hydrology (Devia et al. 2015). Popularly used physically based rainfall-runoff models include MIKE-SHE, HEC-HMS, APEX, and SWAT (Nesru 2023).

Studies of the SWAT model have demonstrated that it is reliable for hydrological analysis. Thus, among several mathematical models, SWAT was chosen to evaluate the impact of SWC measures on watersheds. SWAT is data-intensive, requiring extensive spatial and temporal data (Uniyal et al. 2020), and provides continuous simulation over long periods. Numerous studies have successfully used SWAT at the watershed scale to examine the impact of SWC measures on land and water quality (Naseri et al. 2021, Singh et al. 2023). The water balance component derived from the SWAT model supports water budgeting for hydrological assessment and also forecasts water demand for various purposes, such as domestic and agricultural (Bandi & Patil 2022). Su et al. (2023) used the SWAT model to assess the influence of constructing water conservation projects on runoff from the mountain in different seasons and reported that the model has good applicability in the study area for runoff assessment. Strategic planning and agricultural growth can benefit from the SWAT model's ability to forecast future water availability (Verrma et al. 2022). Therefore, using the hydrological model SWAT is the best way to assess how adopted SWC interventions have affected hydrological processes (Sharma et al. 2024), thus confirming this approach for the present study to assess the impacts of SWCs and the hydrological stage of the watershed relevant to treatment.

In the context of this study, watershed saturation is achieved through the optimal placement and implementation of SWCs. This stage represents the highest retention of the generated runoff in the boundaries of the watershed, while providing for ecological balance, and a desirable amount of runoff called environmental flow, to be allowed to flow downstream beyond the watershed boundary.

Achieving watershed saturation, which inherently includes maintaining minimum flow regimes, is critical for ecosystem conservation downstream, underscoring the importance of environmental flows (Zeiger & Hubbart 2018). Environmental flows refer to the specific amounts, qualities, and timing of water flows necessary to maintain the viability of freshwater and estuarine ecosystems and the livelihoods that depend on them (Yarnell et al. 2020, Hoque et al. 2022). The impact of SWC implementation on environmental flows can be assessed by changes in average annual flow and flash flood frequency after implementation (Mawasha & Britz 2022), which is important for developing a sustainable ecosystem through the implementation of BMPs (Naganur et al. 2024).

The subwatershed (4D7E5) of Chikkodi Taluk, Belagavi District, Karnataka, provides a compelling case study. Before 2012, it suffered significantly from increased soil erosion and heavy runoff. The Government of India's Integrated Watershed Management Program (IWMP), introduced in 2009-10, aimed to restore ecological balance by conserving and developing degraded natural resources (Singh et al. 2010). SWCs were adopted for Project IWMP 19/11-12 in the study area (4D7E5); however, even with these interventions, a comprehensive study has not been conducted to assess their impact on improving the health of the watershed, its water retention capacity, or on attaining the desired condition of saturation. Therefore, a rigorous evaluation of the effects of these existing SWCs on the hydrology of the subwatershed was necessary as a baseline. Subsequently, to achieve optimum runoff conservation and effectively plan SWCs to move toward watershed saturation, a science-based approach that considers existing treatments is essential for sustainable watershed development.

Although many studies have investigated SWC at the local scale, its implications at the watershed level, particularly in achieving a managed state of watershed saturation through integrated planning, are poorly understood. Quantitatively, this methodology differs from past SWC planning studies in India that use SWAT models, primarily by clearly defining the planning objective, that is, retaining 70% of the generated runoff (excluding environmental flow) in the watershed while maintaining the necessary environmental flow downstream (Development of DSS under SUJALA III Project 2019). To deliver evidence-based refinement, this framework is based on a comprehensive baseline evaluation of existing IWMP-based impacts. Criteria are then iteratively formulated using the Sujala-III Decision Support System (DSS) to provide targeted, site-specific prescriptions. For example, in catchments with a runoff of at least 850 m3, check dams should be given priority in new SWC. The related recommendations provide specific quantitative thresholds of watershed saturation, among others, and siting and design specifications for the most significant SWC components, thus supporting the conversion of planning into practice.

The scientific novelty of this study lies in its unified, science-based approach. This approach integrates authentic field data on geotagged SWCs from the IWMP-19/11-12 project with a calibrated and validated SWAT model. The model supports both a comprehensive assessment of the existing IWMP SWCs and the formulation of a ridge-to-valley SWC plan guided by the scientific criteria developed by the authorities. This framework specifically targets watershed saturation while optimizing runoff conservation and maintaining environmental flow, representing a novel contribution to the discipline. By integrating these components, this study provides a robust framework for SWC planning aimed at achieving watershed saturation through effective runoff conservation using hydrological modeling.

MATERIALS AND METHODS

Study Area

This study was conducted in the Chikkodi watershed (52.77 km^2) of the middle Krishna Basin in southern India. It is located between 74°23′51″ and 74°28′55″ E longitude and 16°22′4″ to 16°29′38″ N latitude (Fig. 1). Local People's livelihoods in the area are dependent on dryland agriculture, which is susceptible to irregularities in nature. An " agro-climatic zone" is a geographical unit based on climates that are suited for a certain set of crops and cultivars. There are 15 agro-climatic zones in India, where Karnataka lies in zone 10 of the agro-climatic region, and the Chikkodi watershed of Belagavi district of Karnataka state comes under the

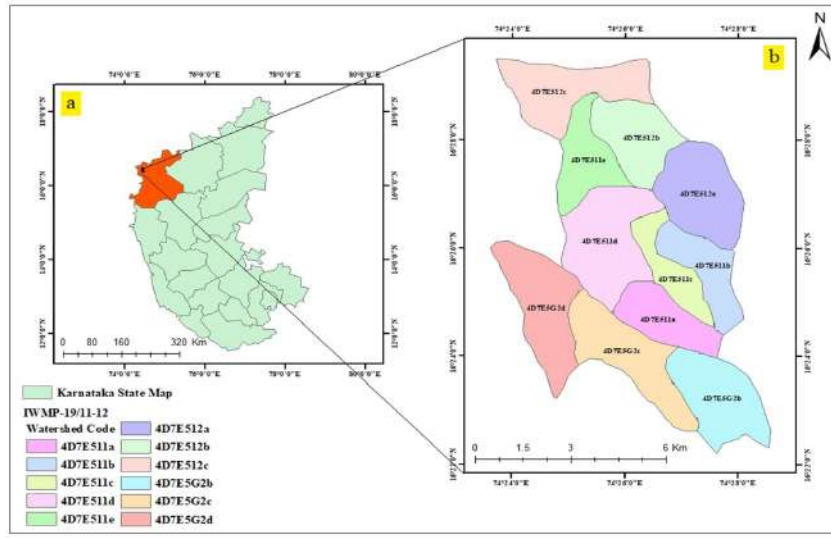

Fig. 1: Study area hierarchy. (a) Location within Karnataka, India (inset). (b) Detailed subwatershed distribution and codes.

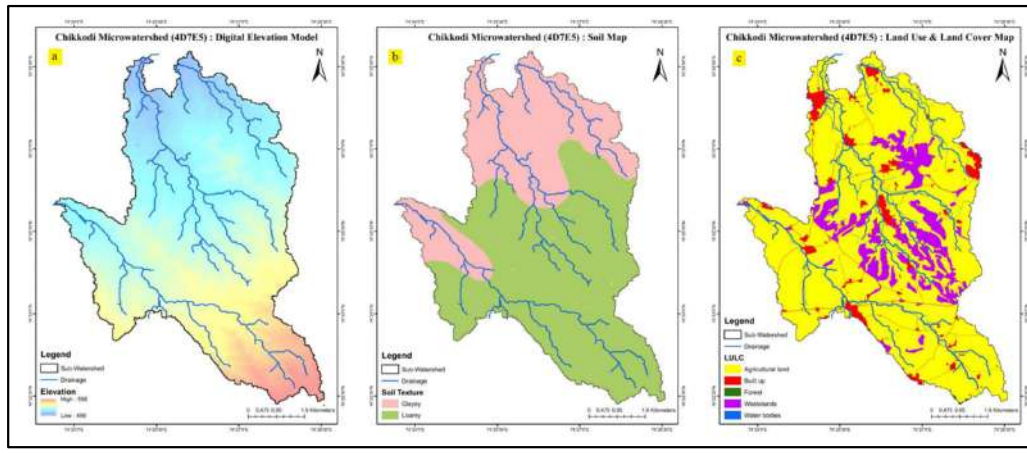

Fig. 2: Hydrological modeling input maps for the Chikkodi subwatershed (4D7E5): (a) Digital Elevation Model (DEM), (b) Soil Map (texture), and (c) Land Use and Land Cover (LULC).

Northern Transition Zone. The annual rainfall in the region is approximately 663 mm. The climate of the research area is semi-arid (CGWB 2017). The highest temperature ranges from 27°C to 35.7°C, whereas the minimum temperature ranges from 13.9°C to 20.6°C (CGWB 2012). The study region includes both flat and steep terrains (Honnannanavar et al. 2023). Fig. 2(b and c) depicts the soil and land use maps, which illustrate that agriculture accounts for the vast majority of land use.

The main streams that run through the study region are heavily silted and lack water-collecting devices, although they are connected to numerous secondary and tertiary streams. During the rainy season, stream flow may be observed. The flow period may vary from June to September (Honnannanavar et al. 2023). The major portion of runoff from the watershed ends as muddy water in the Krishna River. The area is reported to have excessive runoff during peak rainfall events, causing soil erosion and sediment transport, and hence an urgent need for watershed management approaches to improve rural livelihoods by improving agricultural production and water availability for a longer period.

SWC Measures Implemented in the 4D7E5 Microwatershed

SWCs function as sustainable instruments that promote water preservation, control water flow at the surface and below, and minimize soil erosion, thereby advancing sustainable resource management (Yan et al. 2023). Soil losses across the 4D7E5 watershed measure between 900-1100 $Mg.km^{-2}.y^{-1}$ from sheet, gully, and rill erosion activities (IWMP-19/11-12). Numerous SWCs, including contour bunds, trench cum bunds, check dams, and nala bunds, were installed in the subwatershed.

Farmland bunds serve as an effective method to stop surface runoff, but this runoff management action harms crops if not adopted scientifically. Bunds create multiple benefits because they allow more water to penetrate the soil layers. Agricultural yield productivity improves by 15 to 20% when contour bunds that run parallel to elevation lines are used (Madegowda et al. 2021). Such bunds function best in areas that experience rainfall below 800 mm per year with porous soil foundations. The study region receives adequate rainfall (663 mm) annually on its exclusively agricultural land, where clay-loam soil predominates, making contour bunds a suitable practice. Check dams function as structural SWC elements constructed using stones to decrease rainwater runoff and support soil infiltration, providing water for irrigation to nearby agricultural fields. Every dimension of a check dam significantly enhances its ability to minimize rainwater overflows and capture sediments.

Under project IWMP-19/11-12, through watershed development by the Government of Karnataka, a treatable area of 46 km² received earthen field bunds and trench treatments according to project specifications. As part of the drainage line treatment, a series of check dams and Nala bunds were adopted. The geotagged locations of these SWCs were collected from authorities, and their spatial extents are shown in Fig. 4.

SWAT Model

SWAT is a watershed simulation model developed by the USDA Agricultural Research Service through the efforts of Dr. Jeff Arnold (SWAT Theoretical Document 2009). SWAT requires extensive data input from the study region because its methodology depends on numerous geographical and temporal variables (Uniyal et al. 2020). Notable inputs required for building the baseline model include digital elevation data, spatial data of land use and land cover and soils, meteorological precipitation and temperature data, slope information and stream flow, along with sediment data time series (Neitsch et al. 2011).

SWAT implements this water balance equation, which Nasiri et al. (2020) specified as:

$$SW_t = SW_0 + \sum_{i=1}^{t} R_{day} - Q_{surf} - E_a - W_{seep} - Q_{gw} \quad ...(1)$$

Where,

SW_t = final soil water content (mm)

SW_0= initial soil water content on ith day (mm)

R_{day} = daily precipitation on the ith day (mm)

E_a = evapotranspiration on the ith day (mm)

W_{seep} = amount of water percolating into the soil (mm)

Q_{gw} = amount of return flow (mm)

Input Data for the SWAT Model

A digital elevation model (DEM) with a spatial resolution of 30m is sufficient for performing hydrological simulations (Ayana et al. 2012). Land use data and soil data were obtained from https://swat.tamu.edu/ website (Indian dataset for SWAT 2012), Land Use Water base (worldwide data), and Soil HWSD FAO (worldwide data), respectively. Weather data comprising precipitation, temperature, solar radiation, wind speed, and relative humidity were also obtained from the https://swat.tamu.edu/ website (Global dataset for SWAT 2012) for 20 years (2000-2020). Furthermore, the monthly streamflow for the year 2004-2020 for the Sadalga gauging station was acquired from the India Water Resource Information System (https://indiawris.gov.in/wris/#/RiverMonitoring).

Table 1: Performance parameter for the SWAT model (Moriasi et al. 2007).

Performance	NSE	R^2
Very Good	0.75 - 1	0.75 - 1
Good	0.65 - 0.75	0.65 - 0.75
Satisfactory	0.5 - 0.65	0.5 - 0.65
Unsatisfactory	≤ 0.5	≤ 0.5

During the initial soil water conditions balance, monthly model simulations were run for the three-year warm period of 2000–2003. Based on the availability of observed discharge at the basin outlet, the simulation period (2004–2020) was used as the baseline period. Consequently, the SWAT model was calibrated and verified for this time frame.

SWAT Model Performance Assessment

A performance measurement method calculates the model output rate variation when the input parameters experience variations (Moriasi et al. 2007). The evaluation process of the models directly depends on their performance assessment requirements. The model evaluation demonstrated how well the models performed in the historical reference period compared to the observed river flow and other factors (Najafzadeh & Anvari 2023). The model performance was evaluated by calibrating and validating the model findings against observable values.

Manual and auto-calibration and validation can be performed to assess the model performance. Tuppad et al. (2011) contend that manual calibration is preferable to automatic calibration. Manual calibration uses an iterative system that follows the sequence of running simulations, examining observed values against SWAT-calculated values, and modifying parameters within their relevant ranges based on the published literature until optimal correlations emerge. Model performance was evaluated using the coefficient of determination (R^2), Nash–Sutcliffe coefficient (Nash & Sutcliffe 1970), and percent bias (PBIAS) (Moriasi et al. 2012). Moriasi et. al. (2007) proposed that general performance parameters are given in Table 1.

The Chikkodi watershed underwent model calibration for runoff through manual and auto-calibration (SWAT-CUP) techniques over 20 years from 2000 to 2020 using a three-year warm-up phase from 2000 to 2003. Monthly streamflow information served as the basis for calibrating and validating the model. The model calibration spanned 7 years from 2004 to 2011, whereas the validation took place during the subsequent 9 years from 2011 to 2020. The proper parameters were adjusted until the model predictions for monthly stream flow matched the observed data at the watershed outlet locations.

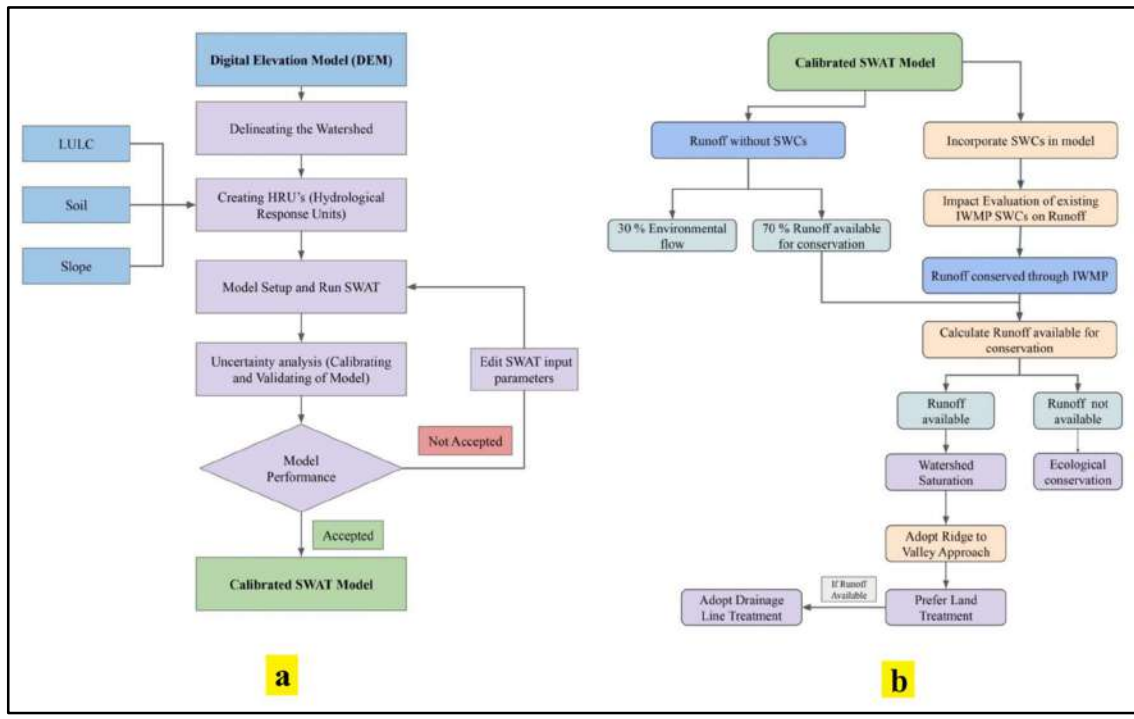

Fig. 3: Methodological flowchart for (a) SWAT model implementation and (b) Decision-making framework for watershed saturation using SWC measures derived from SWAT modeling.

Methodology

The flowchart (Fig. 3-a) demonstrates the progression from basic spatial layers, including the Digital Elevation Model, land use/land cover, soil, and slope, and then shows watershed processing and model simulation while integrating calibration and validation until the SWAT model is attuned. The flowchart describing the determination of SWC practice implementation (ridge-to-valley approach and drainage line treatment along with land treatment) through runoff assessment (SWC presence vs. absence) for calculating conservation potential using watershed saturation levels and available runoff is shown in Fig. 3-b.

Modeling SWC Impacts on Runoff in SWAT

The sustainable practice of SWC structures reduces soil loss due to soil erosion, with surface water flow regulation to improve water conservation in the environment. Sustainable resource management is possible because of SWC measures (Melaku et al. 2017). SWC structures have already been established in numerous micro-watersheds to control soil erosion. The lowered flow velocity and sediment deposition at the structure's crest serve as the main erosion reduction methods, preventing channel or gully formation on the downstream side (Nabi et al. 2020). SWC methods, such as contour bunding, trenching, and terracing, as well as structures such as check dams, farm ponds, and nala bunds, control soil erosion and enhance moisture retention, promoting agricultural development.

Table 2: SWAT parameters for SWC measures (Source: Uniyal et al. (2020)).

Type of SWC	SWAT parameters (input files)	Value of SWC in good condition
Contour bunding	SLSUBBSN (.hru)	$\frac{(0.1 * slope * 0.9) * 100}{slope}$
	CN2 (.mgt)	CN2 calibrated values were lowered by 6
	USLE_P (.mgt)	0.1, for slope 3 to 5% 0.12, for slope 6 to 8%
Trenching	SLSUBBSN (.hru)	10 m, for slope 10-20% 9.1 m for slope >20%
	USLE_P (.mgt)	0.32

The SWAT hydrological model enables users to model SWC systems using specified model parameters. The model simulations used parameter value changes based on Table 2 to simulate the SWC process. The research area has approximately 60 sub-basins where contour bunds have been implemented (Fig. 4). A SWAT-based runoff simulation was conducted by adjusting the suitable model parameters. Table 2 contains information about the SWC types, providing the

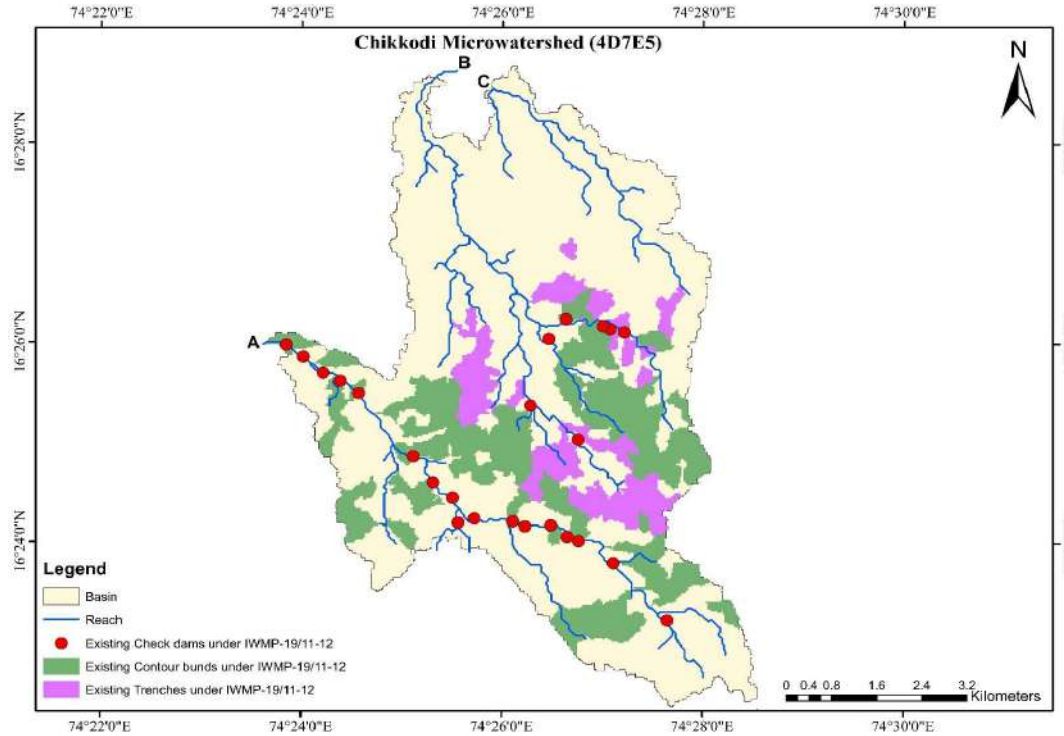

Fig. 4: Map of the Chikkodi subwatershed (4D7E5) showing the spatial extent of existing SWC infrastructure implemented through the IWMP (19/11-12).

SWAT input parameters used alongside the suggested values for good conditions and corresponding references.

A total of 21 check dams were implemented in the micro-watersheds to control the intense runoff impacts. The geotagged locations of the check dam were acquired from the IWMP program, as shown in Fig. 4. The construction of check dams was proposed at locations possessing stable embankments and soft-sloping stream beds while using durable geological layers beneath the drainage channel. The SWAT model represents check dams as conceptual ponds that require modification of PND_FR, PND_K, PND_PSA, and PND_VOL. By taking the height of the check dam as approximately 2.5 m, all other parameters were calculated and input into the SWAT model.

Using the formula outlined below, Uniyal et al. (2020) calculated the percentage decrease in runoff.

$$\textit{Runoff reduction} = \frac{\text{Model output without SWC} - \text{Model output with SWC}}{\text{Model output without SWC}} \times 100 \quad ...(5)$$

Runoff Conservation Strategies for Watershed Saturation

Areas that are highly exposed to hydrological extremes rely on watershed management for sustainable development. To minimize the impact of these extremes, SWC measures conserve excess runoff and reduce soil erosion. By considering a runoff conservation strategy, this study proposes a scientific method to achieve watershed saturation by adopting SWCs. This study offers a scientific approach to planning SWCs to achieve watershed saturation by conserving runoff.

Estimating Runoff with the SWAT Model

In the study watershed, the SWAT model was used to simulate runoff. The model's performance in simulating runoff was ensured by calibration and validation with observed data. Similarly, to understand the current hydrological regime, the impact of existing IWMP SWCs on runoff was assessed through runoff estimation using the SWAT model after implementing it into the model.

Strategic Runoff Allocation for Environmental Needs and Conservation

Ensuring environmental sustainability is an important component of this research. In a bid to make it ecologically sustainable, 30% of the original produced runoff was marked as environmental flow, while the remaining 70% was to be conserved. This allocation matches the country-wide recommendations of the Ministry of Environment, Forest & Climate Change (MoEF&CC), which has marked 30% environmental flow during the monsoon months in River Valley & Hydroelectric Projects (Ministry of Jal Shakti 2021). Since the context of our study region is semi-arid and dominated by monsoons, this practical division serves the objectives of conservation in addition to the maintenance of vital downstream flows. Hence, 30% of the initial runoff was allocated for environmental flow. Through SWC procedures, the remaining 70% of runoff was intended to be conserved. The efficiency of the existing IWMP SWCs in conserving runoff was calculated using the SWAT model. The remaining runoff was then considered for further conservation by deducting the runoff conserved through the IWMP SWCs from the total runoff.

A Ridge-to-Valley Approach for SWC Planning and Implementation

The ridge-to-valley strategy was used to plan further SWC measures. The objective of this strategy is to capture runoff and encourage infiltration at its source. Initially, except for the places where structures were already in place, the entire watershed was to be covered by bunding and trenching. The SWAT model was used to evaluate the impact of this scenario on runoff conservation. It was suggested to treat the drainage lines by building check dams to save the leftover runoff. After bunding and trenching, the positions of the check dams were established based on the distribution pattern of the runoff.

The success of field-level conservation efforts is critical to the long-term viability of the state's water and soil resources, especially those of large rainfed areas. The process of creating a conservation plan involves matching various conservation measures to the site-specific potentials and restrictions of the location and then selecting the most efficient option based on the available criteria. Rainfall quantity, landform, soil, land use, and other factors all play a role in determining the selection of treatment type. Bunding, terracing, and trenching are the main field-level treatments used for soil and water conservation.

Decision Support for Site-Specific SWC Planning

The primary purpose of the Decision Support System (DSS) is to facilitate the planning, execution, and oversight of watershed development initiatives within the state under the SUJALA-III project of the WDD, the Government of Karnataka, and other line departments in Karnataka. With this, authorities can develop a conservation map for any area by developing a DSS for SWC based on the criteria established in Table 3 (Development of DSS under SUJALA III Project 2019).

Table 3: Guidelines for selecting SWC treatments based on biophysical parameters, as defined by the Sujala-III DSS (Source: Watershed Development Department, Government of Karnataka 2024).

Sl. No.	Slope	Depth	Texture	Gravel	Rainfall (mm)	Treatment
1.	<1	<50	Loam	<35%	<750	Contour bunding/TCB
2.	1 to 3	<50	Loam	<35%	<750	Contour bunding/TCB
3.	3 to 5	<50	Loam	<35%	<750	Contour bunding/TCB
4.	<1	<50	Clay	<35%	<750	Graded bund
5.	<1	50 to 100	Clay	<35%	<750	Graded bund
6.	<1	>100	Clay	<35%	<750	Graded bund
7.	1 to 3	<50	Clay	<35%	<750	Graded bund
8.	1 to 3	50 to 100	Clay	<35%	<750	Graded bund
9.	1 to 3	>100	Clay	<35%	<750	Graded bund
10.	3 to 5	<50	Clay	<35%	<750	Graded bund
11.	3 to 5	50 to 100	Clay	<35%	<750	Graded bund
12.	3 to 5	>100	Clay	<35%	<750	Graded bund
13.	5 to 10	<50	Loam	<35%	<750	Graded bund
14.	5 to 10	<50	Clay	<35%	<750	Graded bund
15.	5 to 10	50-100	Loam	<35%	<750	Graded bund
16.	5 to 10	50 to 100	Clay	<35%	<750	Graded bund
17.	5 to 10	>100	Loam	<35%	<750	Graded bund
18.	5 to 10	>100	Clay	<35%	<750	Graded bund

Data Limitations and Uncertainties in the Model

The major sources of uncertainty related to hydrological modeling, especially in cases of SWC planning, are the quality and resolution of the input data. This study applied gridded precipitation and temperature records, the spatial resolution of which is insufficient to represent precipitation variations on a local scale, which will introduce uncertainty as runoff and water balance estimates are obtained. The 30m DEM is appropriate for analyzing the watershed, but it may not satisfactorily reveal finer topographic details that may influence the flow direction and delineation of critical areas where SWC interventions are carried out.

Moreover, the Land Use and the soil data to be used in the analysis have coarser scales because they are based on generalized global data, which may fail to reflect the complexity of land cover (and soil properties) found in the Chikkodi subwatershed. These simplifications may cause inaccuracies in evaluating important parameters of the SWAT model, such as CN values, soil water holding capacities, and hydraulic conductivities, causing uncertainties in determining the accuracy of the exact effects of SWC measures on local water balance components. Despite such inherent ambiguities in data, the best possible datasets were used

RESULTS AND DISCUSSION

In the present study, the SWAT model was used for the hydrological planning of SWC measures in the Chikkodi subwatershed, which experiences a semi-arid climate. Many studies on hydrological evaluation and water resource management in semi-arid regions have relied on the SWAT model for assessment, which is consistent with the approach followed in the current study. Rocha et al. (2023) comprehensively reviewed hydrological studies in semi-arid regions, particularly in Asian countries, which highlights the extensive and versatile applications of SWAT for hydrological and environmental management. In contrast, to assess hydrological responses under varying climate and land use, the SWAT model was adopted in Central Asia by Dolgorsuren et al. (2024) for guiding water resource management. For a semi-arid watershed in Turkey, Aibaidula et al. (2022) adopted the SWAT model to evaluate the climate change impacts on water availability. Sharma et al. (2023) quantified the SWAT's performance in a large semi-arid basin in assessing the Spatio-temporal pattern of water balance, which is geographically closer to the current study, providing firm and crucial insights relevant for the present study.

Streamflow Model Performance: Calibration and Validation

Calibration was performed for eight years, from 2004 to 2011, to match the simulated results with the observed data. The sensitive parameters were identified and adjusted to achieve better calibration. This was confirmed by the

Table 4: Identified sensitive parameters adjusted during the calibration and validation process.

Sl. No.	SWAT parameters	Minimum value	Maximum Value	Fitted Value
1.	CN2	35	98	82
2.	ALPHA_BF	0	1	1.04
3.	GW_DELAY	0	500	170.04
4.	SLSUBBSN	-29.67	91.67	3.69
5.	HRU_SLP	0.44	1.38	0.70
6.	ESCO	0	1	0.61
7.	EPCO	0	1	0.55
8.	SOL_AWC	-72.97	77.97	74.20
9.	SOL_K	0	2000	0.73

R^2 and NSE values between the observed and simulated results, which must be within the allowable limits. During the process, the sensitive parameters were adjusted to achieve maximum model efficiency, as shown in Table 4.

Although it is well acknowledged that measured data are fundamentally uncertain, model evaluation tends to neglect this uncertainty, possibly due to a lack of required data. Observations were taken at the Sadalga gauge station throughout the year from 2000 to 2020. A comparison and correlation analysis was established between the SWAT simulation results using seasonal runoff data for the mentioned year and the actual measured data. The procedure included both manual and autocalibration steps. The performance of the model for both manual and auto calibration is shown in Table 5.

Parameters that are essential in determining the effectiveness of the model, such as the NSE values, gave an initial result of 0.41, which indicates moderate performance in the manual calibrations. Nonetheless, to increase model accuracy and resilience, calibration and validation were further improved through auto-calibration by SWAT-CUP. This considerably enhanced the NSE values from 0.41 to 0.53 during calibration and from 0.65 to 0.72 during validation (Fig. 5).

Although the first manual calibration gave an NSE of 0.41, which had a limiting impact on being able to perfectly capture the observed variability as a 'Moderate', the subsequent auto-calibration controlled this. The resulting improved NSE values of 0.53 and 0.72 show that the model performance was rated as Satisfactory to Good, a level that is adequately acceptable for carrying out hydrological modeling in areas that are complex, data-deficient, or semi-arid in nature (Moriasi et. al. 2007). More importantly, for the planning of SWC based on relative changes and comparison of intervention effectiveness to reach the level of watershed saturation, this improved model performance of simulating overall hydrological responses is a solid source of strategic decision-making.

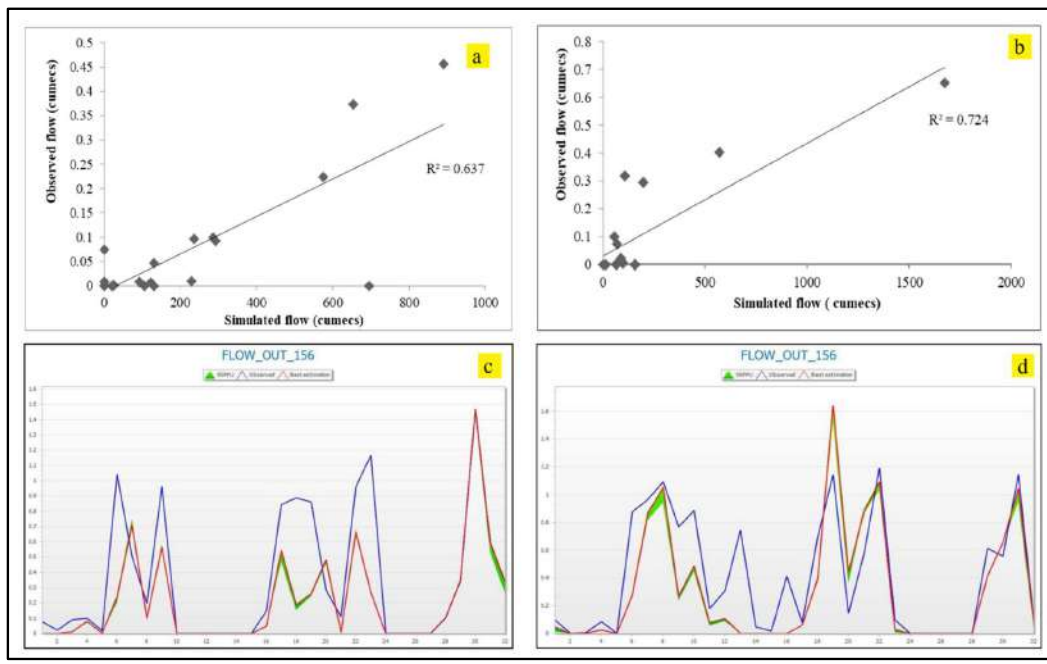

Fig. 5: Evaluation of SWAT model performance for stream flow simulation (a) Manual Calibration scatter plot, (b) Manual Validation scatter plot, (c) Auto-calibration hydrograph with 95% PPU, (d) Auto-validation hydrograph with 95% PPU. Calibration periods (2004-2011) and validation period (2012-2020).

Table 5: Runoff model fit statistics from manual calibration and validation.

Method	Calibration				Validation			
	R^2		NSE		R^2		NSE	
	Value	Performance	Value	Performance	Value	Performance	Value	Performance
Manual	0.63	Satisfactory	0.41	Moderate	0.72	Good	0.65	Satisfactory
Auto (SWAT-CUP)	0.62	Satisfactory	0.53	Satisfactory	0.77	Very Good	0.72	Good

Hydrological Impacts of Implemented SWC Measures

The spatial variation in the annual average runoff without any treatment in the watershed is shown in Fig. 6-b. In this scenario, spatially concentrated runoff was observed at outlets A and B. In the absence of water conservation treatment, all runoff generated within the watershed flowed out of the watershed with sediments. In this context, there was scope to conserve runoff by adopting various SWCs within the watershed.

Impact of Contour Bunds on Runoff

In the research region, contour bunds have been used in approximately 60 sub-basins. The effect of these contour bunds on simulating runoff was assessed by altering the appropriate SWAT parameters, as outlined in Table 2. According to the analysis conclusions, the implementation of this specific SWC solution had an acceptable impact, as the contour bunds decreased runoff by 7.03 %. The reduction in runoff has been shown in Fig. 6– c and Table 5.

The significant change in runoff concentration can be observed at outlet A when compared with Fig. 6- b, as the majority of bunds adopted in the watershed were situated in the catchment of drainage with outlet A. Minor variation is observed at outlet B, as less area was adopted under bunding

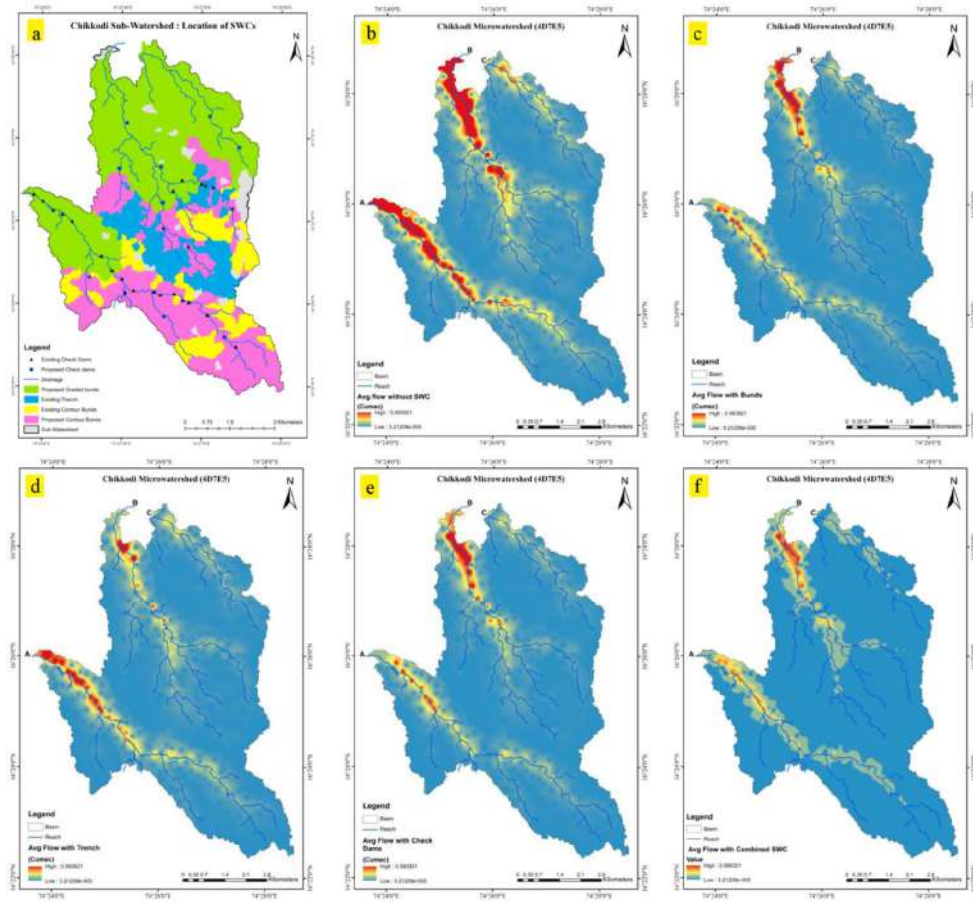

Fig. 6: Impact of SWC measures on average seasonal flow in the Chikkodi subwatershed (4D7E5). (A) Locations of existing and proposed SWCs. (B) Average seasonal flow without SWCs. (C) Average seasonal flow with bunds. (D) Average seasonal flow with trenches.

Table 6: Model-simulated runoff reduction (%) resulting from different SWC scenarios.

Scenarios	Description	Runoff reduction
Scenario 1	Contour bunds	7.03%
Scenario 2	Trenches	6.33%
Scenario 3	Check dams	18.08%
Scenario 4	Combination of all SWCs	26.17%

treatment. This spatial variation in the simulated runoff reduction demonstrates that the SWAT model effectively captured the localized impact of contour bunding within the watershed.

Impact of Trenches on Runoff

This study assessed the effects of trench installation on runoff simulation. According to Table 2, the HRU, parameter SLSUBBSN, and mgt were. (management) The USLE P parameter was changed. The SLSUBBSN was adjusted to 10m for slopes between 10% and 20% and 9.1m for slopes greater than 20%. According to the model simulation findings, trenches decreased runoff by 6.33 %. This demonstrates that trench installation has a better effect on runoff. Fig. 6-d and Table 5 illustrate the decreased runoff. A significant change in runoff concentration can be observed at outlet B when compared with Fig. 6-b, as the majority of trenches adopted in the watershed were situated in the catchment of drainage with outlet B. Negligible variation was observed at outlet A, as trenches were not adopted in that area.

Impact of Check Dams on Runoff

Approximately 21 sub-basin check dams have been adopted (Fig. 4) to reduce the vigorous impact of excess runoff at the research location, as per IWMP-19/11-12. SWAT has a conceptual visualization of the pond as a check dam; therefore, pond parameters such as PND_FR, PND_K, PND_PSA, and PND_VOL have been altered. By taking the height of the check dam as approximately 2.5 m remaining all other parameters have been calculated and input into the SWAT model. According to model simulation, results depict that the adoption of check dams has reduced the runoff by 18.08% reflected in Fig. 6- e and Table 5, which is comparable to the results of Xu et al. (2013).

A significant change in runoff concentration can be observed at outlet A when compared with Fig. 6 - a, as the majority of check dams were adopted on the drainage with outlet B. Minor variation was observed at outlet A in comparison with outlet B, as only a few check dams were placed on drainage B. This indicates that check dams are quite helpful in conserving significant amounts of runoff.

Combined Impact of SWCs on Runoff

The subsequent simulation was carried out to analyse the collective runoff conservation performance of SWCs applied together. The model considered the entire range of SWC measures that local authorities implemented across the watershed under the IWMP program. This integrated modeling aimed to assess the collective runoff reduction that occurs through distributed SWC implementation across the microwatershed. As demonstrated in this simulation, the cumulative impact of all implemented SWCs results in a significant 26.17% reduction in runoff, as depicted in Table 6 and Fig. 6-f. This result shows that the SWAT model can predict the cumulative hydrologic impacts of multiple SWCPs deployed within a watershed. The ability of the model to replicate the integrated effects is important when it comes to evaluating the overall impact of watershed development measures and forms a significant part of spatial strategies in conservation endeavors.

The reduction of 26.17% in the annual surface runoff measured at the outlet of the final subbasin is a direct and measurable outcome of the management practices (bunds, trenches, and check dams) carried out. This remarkable decrease is mainly due to the increase in the infiltration ability of the watershed, which is indicated by the reduction of the mean Curve Number from 81.99 to 81.45 (Table 7). Water that no longer becomes surface runoff is essentially redirected within the hydrological cycle, resulting in augmented actual evapotranspiration, improved recharge to shallow and deep aquifers, and changed subsurface hydrology, such as increased lateral and return flow, along with more evaporative loss of the shallow aquifer (revap). The combination of these changes shows that the management practices are successful in decreasing the flashy nature of the surface runoff and increasing water retention and groundwater recharging in the watershed.

Table 7: Average annual hydrological parameters (obtained from SWAT Checker) before and after the application of management practices.

Water balance component	Before SWC	After SWC
Precipitation	662.6	662.6
Surface runoff	85.59	82.9
ET	547.1	548.3
Percolation to shallow aquifer	28.32	29.72
Lateral flow	0.18	0.24
return flow	0	0.51
Revap from shallow aquifer	20.47	21.12
Recharge to deep aquifer	1.42	1.49
Average CN	81.99	81.45

Planning of SWCs to Achieve Watershed Saturation by Runoff Conservation

This study focused on planning SWCs for watershed saturation through runoff conservation. The impact of existing IWMP SWCs on runoff was assessed using the SWAT model to understand the existing hydrological regime. By designating 30% of the initial runoff as environmental flow, the remaining 70% was targeted for conservation through SWC measures. The effectiveness of the existing IWMP SWCs in conserving runoff was evaluated using the SWAT model, with the remaining runoff after accounting for these structures representing the available runoff for further conservation. The following criteria were used to propose the SWC in the watershed:

A ridge-to-valley approach was adopted for planning additional SWCs, focusing on capturing runoff at its source and promoting infiltration into the soil. By assuming the impact of existing IWMP structures, the correlation of the same structures was proposed in this study to conserve the remaining runoff from the available runoff after IWMP treatment. As per the ridge-to-valley approach, preference was given to land treatment, such as bunding and trenching, to avoid the degradation of land, which further causes silting in water bodies. After complete land treatment, the remaining runoff will be conserved by structures adopted in the drainage line treatment, preferably check dams. This approach results in watershed saturation. In this study, the same treatment was proposed to achieve watershed saturation by maintaining environmental flow. Table 8 presents calculations that include average runoff determination alongside existing IWMP conservation of runoff and environmental flows, as well as proposed land and drainage line treatment estimations for the check dams.

The performance of SWCs adopted under IWMP, evaluated by the SWAT model, was considered as a reference while planning additional SWCs in the watershed. Initially, bunding and trenching were suggested for the entire watershed by excluding the region where they already exist, in line with the criteria established under DSS, shown in Fig. 7. As per the ridge-to-valley approach, preference was given to land treatment, i.e., bunding and trenching, to conserve the runoff at its source, to reduce further soil erosion by improving percolation. By considering the similar impact as in the case of IWMP treatment, runoff conservation was estimated.

Similarly, as a part of drainage line treatment, check dams were proposed based on runoff concentration to further conserve the runoff within the catchment. Fig. 7 shows the locations of proposed check dams based on established criteria under DSS and the residual runoff available for conservation. In combination, this SWC measures effectiveness and will enhance the watershed saturation. In the upstream, land treatments like bunding and trenching proved effective in arresting the runoff at source, where check dams helped in conserving the runoff in drainages, resulting in percolation of water to aquifers as well as base flow.

Table 8: Runoff analysis and proposed SWC measures for watershed saturation[1].

Description	Equation	Value
Overall Annual Average Runoff from the calibrated SWAT model in $m^3.s^{-1}$= (O_{Runoff})	Runoff at the outlet (A+B+C)	1.5743
Conserved runoff through IWMP treatment done in the subwatershed in $m^3.s^{-1}$ = (R_{IWMP})	Impact of Bunding + Trenching + Check dams	0.4120
Runoff can be conserved after deducting Environmental flow (30% of O_{Runoff}) in $m^3.s^{-1}$ = $R_{Available}$	70% of O_{Runoff}	0.9174
Runoff available to conserve after IWMP treatment in $m^3.s^{-1}$ = (R_0)	$R_{Available}$ - R_{IWMP}	0.5053
*Runoff conserved through proposed land treatment in $m^3.s^{-1}$ = R_{Land}		0.2110
Runoff available to be conserved through drainage line treatment in $m^3.s^{-1}$ = R_{DLT}	R_0 - R_{Land}	0.2943
Runoff conserved through a single Checkdam in a year in $m^3.s^{-1}$ = R_{CD}	Considering 850 m^3 of filled 3 times in a year	0.02951
No. of proposed check dams	R_{DLT} / R_{CD}	10
No. of check dams proposed on each drainage line by considering the proportionate runoff		
On Stream A		3
On Stream B		5
On Stream C		2

[1] As per the Ridge to Valley approach, first preference is given to Land treatment (Bunding, trenching, etc.). If runoff is still available, drainage line treatment will be adopted.

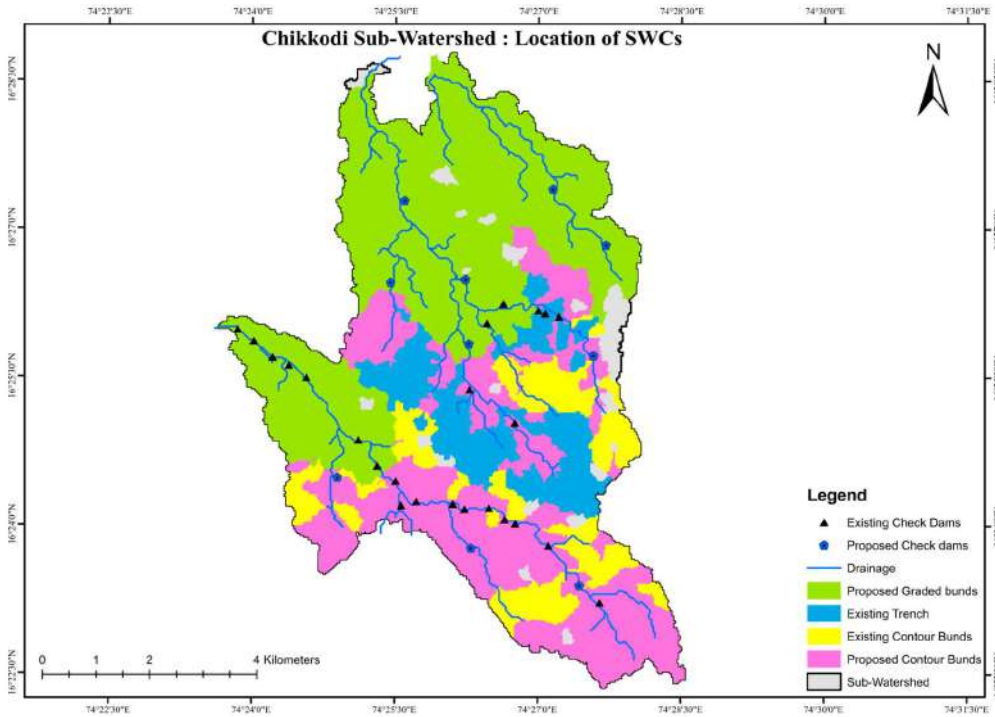

Fig. 7: Proposed implementation plan for Soil and Water Conservation (SWC) measures in the Chikkodi subwatershed (4D7E5) to promote watershed saturation.

The performance of these SWCs depends on various factors, i.e., soil type, rainfall pattern, and watershed features, which may affect the hydrological behavior. All these factors need to be considered while planning the SWC measures in the watershed. This study provides a base for effective watershed management through watershed saturation by adopting SWCs to conserve the runoff. This will result in water availability for a longer period, improved groundwater levels, a reduction in soil erosion, and enhanced health of the watershed. Fig. 7 shows the distribution of established structures with proposed new interventions that emerged from the runoff budget and Sujala-III DSS criteria to reach watershed saturation.

CONCLUSIONS

This study supports the positive impact of introducing SWC methods in the Chikkodi subwatershed, depicting their capacity to enhance watershed saturation, which is obtained through the targeted conservation of runoff, supported by a robust hydrological modeling system. The effectiveness of the SWC technique was studied, and the SWAT model was used to simulate the runoff. Past streamflow records were used for model calibration and validation, accurately reflecting the hydrological activity of the watershed. It was observed that the current SWC practices implemented under the IWMP were effective and resulted in reduced watershed runoff. Specific interventions, such as trenches, check dams, and contour bunds, have demonstrated great potential for capturing and storing runoff. The SWAT model effectively replicated the hydrological outcomes of these interventions, which illustrated the spatial heterogeneity in influence, especially in the reduced runoff recorded at different sub-basin outlets following contour bundling. The model needed to demonstrate that the combined effect of all the SWC measures applied resulted in a significant reduction in the entire watershed runoff by 26.17%. The capability of the SWAT model to accurately predict individual and cumulative hydrological effects at both local and regional scales from distributed SWCs is a critical strength of this study.

Utilizing the proven model capacity, other subsequent SWC interventions were planned under a science-based ridge-to-valley approach to increase watershed saturation. The combined effectiveness of the SWC measures to reduce runoff and improve the water retention capacity of the watershed was demonstrated using SWAT model simulations. The simulation indicated that the implementation of SWCs reduced surface runoff, thus increasing soil moisture reserves and enhancing baseflow. Such realignment to the hydrological regime generates high levels of sustainability value, essentially strengthening the long-term water supply

and ecosystem conditions in the semi-arid watershed. However, performance can still fluctuate depending on each site's unique characteristics, eliminating the need for targeted approaches. Based on this study, it is vital to tailor SWC methodologies site by site to achieve saturation in watersheds.

This study provides high-quality, direct knowledge for practitioners to manage watersheds in semi-arid environments. The site-specific SWC planning framework using the ridge-to-valley approach, in combination with the Sujala-III DSS and the integration with the SWAT watershed model, is a strong tool for identifying and implementing site-specific measures of SWC. It enables the identification of the important areas in which the treatment of land and drainage lines should be conducted, and calculates its role in the reduction of runoff, soil moisture, and environmental flow, consequently facilitating the saturation of watersheds and water retention. The findings of this study indicate to policymakers the effectiveness of such an integrated watershed management model-based approach. The capacity to conserve a significant portion of runoff without compromising environmental flow highlights the need for continued investment. The measurable approach helps set saturation goals, allocate resources, and shape policies for long-term water security, erosion mitigation, and productivity in water-stressed regions.

This work acknowledges minor limitations, which are mainly linked with the input data resolution and the simplified character of the hydrological model owing to the necessity of simplification. More research should involve a detailed examination of the distinctive hydrological aspects of each site, assessment of the effects on water quality, and tracking the long-term effectiveness of SWC interventions to establish their sustainability in maintaining the saturation of the watershed and environmental flows. Future research must therefore incorporate the hydrological framework with thorough groundwater monitoring and examine the economy of SWC interventions in supporting long-term sustainability and clarifying implementation strategies.

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Bioaccessibility of Heavy Metals in Raw and Processed *Alternanthera sessilis* and *Centella asiatica*: An *In vitro* Study

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ABSTRACT

Heavy metals pose significant risks to food safety because of their persistence in the environment and their ability to accumulate in the food chain. Even small amounts of these metals can harm human health upon consumption. This study assessed the *in vitro* bioaccessibility of Ni, Cd, Cr, Pb, and Cu in two commonly consumed green leafy vegetables in Sri Lanka, *Alternanthera sessilis* and *Centella asiatica*. Composite samples of *A. sessilis* and *C. asiatica* were randomly collected from the Western Province, Sri Lanka. The edible portions of each sample were divided into three 200 g test portions and subjected to the following treatments: Treatment 1 - raw sample, Treatment 2 - cooked sample, and Treatment 3 - stir-fried sample. The *in vitro* bioaccessibility of heavy metals in raw, cooked, and stir-fried samples was determined using a physiology-based extraction test (PBET). In contrast to the overall concentrations of heavy metals in *A. sessilis* and *C. asiatica*, the bioaccessible fractions were significantly lower in raw, cooked, and stir-fried samples ($P<0.05$). Moreover, significant differences were observed in metal concentrations between the intestinal and gastric stages. The average bioaccessibility (%) of Cu was considerably higher in the intestinal stage, whereas Cr, Cd, Pb, and Ni were higher ($P<0.05$) in the gastric stage. Additionally, cooking and stir-frying reduced the bioaccessibility of metals compared with the raw samples.

INTRODUCTION

Ensuring food safety and security has become a global priority. Recently, there has been an increasing focus on food safety, leading to extensive research into the health risks associated with consuming foods contaminated with pesticides, heavy metals, and other agrochemicals. Heavy metals, recognized as harmful environmental pollutants, are especially prevalent in areas with significant human activity. Their accumulation in organisms through the food chain poses a serious threat to human health. Even in trace amounts, metals in soil, water, and air can have detrimental effects on various living organisms (Suruchi & Khanna 2011, Tchounwou et al. 2012). Numerous studies worldwide have highlighted human ingestion of heavy metals through the food chain (Islam et al. 2007, Mawari et al. 2022). The toxicity of most heavy metals arises from their water solubility, causing harm even at low concentrations in both humans and animals. Furthermore, the body's inefficient elimination of these toxic metals exacerbates their potential for harm.

In Sri Lanka, the high consumption of green leafy vegetables (GLVs), whether raw or cooked, is driven by their rich nutritional value, affordability, and accessibility. However, studies by Rathnayaka et al. (2004), Premarathna et

al. (2011), and Kananke et al. (2014-2018) have revealed elevated heavy metal concentrations in GLVs and other crops across various regions of the country. Leafy vegetables are particularly effective at absorbing heavy metals from contaminated soil, water, and atmospheric deposits. This issue is especially concerning in highly urbanized areas such as Colombo and Kalutara Districts in the Western Province, where GLV production and distribution are significant. Urban environments near roadways often experience pollution from exhaust fumes containing high levels of metals from vehicles. Additionally, rapid urbanization and increased traffic have contributed to rising concentrations of hazardous metals in these regions.

Assessing the potential health risks of consuming metal-contaminated plants is critical, and one approach is to evaluate oral bioaccessibility. Oral bioaccessibility involves replicating the transfer of metal contaminants from plants to the human gastrointestinal system in a laboratory setting. This method enables researchers to estimate the health risks associated with consuming contaminated food crops. Bioaccessibility refers to the portion of a substance that is released in the digestive system, making it available for absorption by the intestines and entry into the bloodstream (Ma et al. 2024). Several *in vitro* methods, often referred to as physiologically based extraction tests (PBET) or simulated gastrointestinal extraction processes, have been developed to mimic human digestion (Intawongse & Dean 2008, Yin et al. 2017, Ma et al. 2024). These *in vitro* methods are typically preferred over *in vivo* approaches due to their speed, cost-effectiveness, accuracy, and reduced use of experimental animals (Kulkarni et al. 2007, Intawongse & Dean 2008, Tremlova et al. 2012, Hu et al. 2013, Omar et al. 2013, Ma et al. 2024).

This study aimed to assess the oral bioaccessibility of heavy metals in *Alternanthera sessilis* and *Centella asiatica*, the two most widely consumed green leafy vegetables in Sri Lanka, using a simulated gastrointestinal extraction method. Previous research has reported high metal contamination in these plants, particularly those cultivated in urban areas. However, the bioaccessibility of these metals through the consumption of both fresh and processed forms of these vegetables has not yet been investigated in Sri Lanka. This gap in knowledge forms the basis of the present research.

MATERIALS AND METHODS

Chemicals and Apparatus

The reagents used in this research were analytical grade or higher and met the required standards. Concentrated HCl, sodium bicarbonate, acetic acid, pancreatin, pepsin, sodium malate, bile salts, lactic acid, and sodium citrate were obtained from Sigma Aldrich (St. Louis, Missouri,

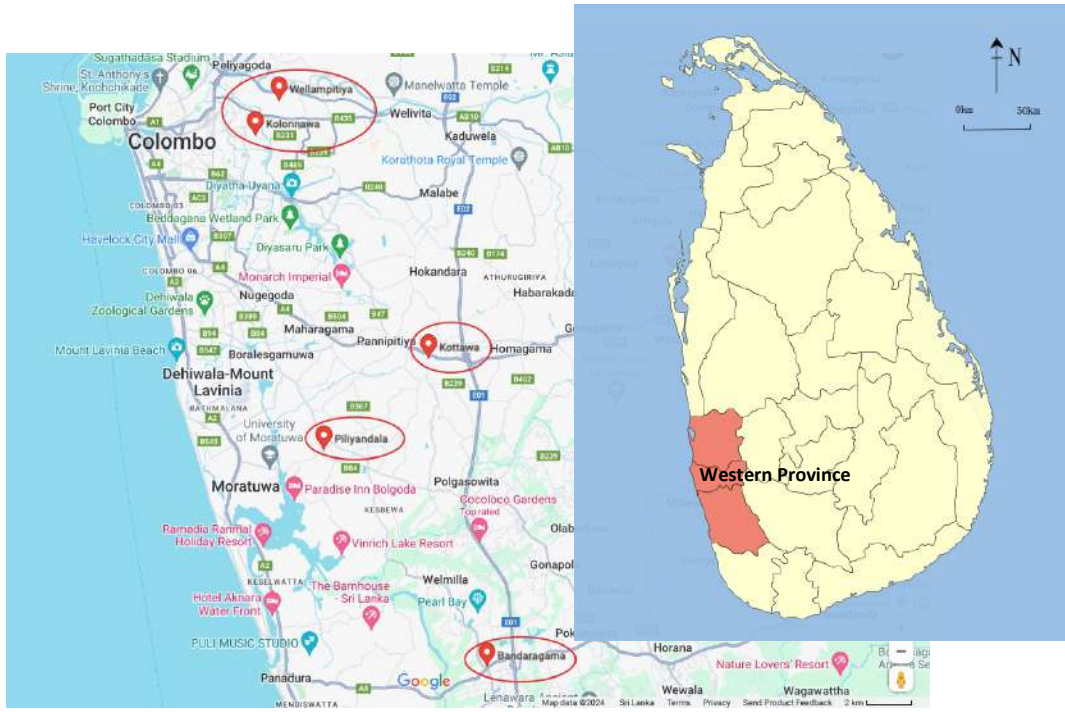

Fig 1: *A. sessilis* and *C. asiatica* sample collection sites (Piliyandala, Kolonawa, Wellampitiya, Kottawa, Bandaragama) identified in the Western Province, Sri Lanka.

USA). Trace-element standards for Ni, Cd, Cr, Pb, and Cu were also obtained from Sigma Aldrich (St. Louis, Missouri, USA). Certified reference material (CRM) GBW10015 – Spinach was obtained from the National Research Center for Certified Reference Materials (NRCCRM), Beijing, China.

Collection of Samples

A preliminary investigation was conducted in the Colombo and Kalutara regions of the Western Province, Sri Lanka, to identify the primary cultivation areas of *A. sessilis* and *C. asiatica*. A structured questionnaire was administered to farmers at the selected production sites to collect information on cultivation practices. Leafy vegetable samples were collected at five locations: Wellampitiya, Kolonnawa, Kottawa, Piliyandala, and Bandaragama (Fig. 1).

From each leafy vegetable (*A. sessilis* and *C. asiatica*), 40 random samples were collected, with eight samples per location. The samples (Fig. 2) were carefully transported to the laboratory in clean polyethylene bags. The samples were then sorted and cleaned with tap water to remove impurities. The edible portions of each sample were divided into three test portions, each weighing 200 g. These portions were subjected to different treatments before analyzing toxic metal levels (Ni, Cd, Cu, Cr, and Pb) using the *in vitro* extraction method. Treatment 1 used a fresh sample. Treatment 2 involved cooking by finely cutting and mixing 200 g of cleaned and sorted plant material with 50 mL of coconut milk. Cooking was carried out on an electric hot plate set to maintain a temperature of 95 ± 2°C, monitored with a calibrated digital thermometer. Each sample was cooked for 12 min to ensure consistency across treatments. In Treatment 3, 200 g of finely chopped, cleaned, and sorted plant material was stir-fried with 15 mL of coconut oil in an uncovered stainless-steel pan on an electric hot plate set to maintain a temperature of 95 ± 2°C, monitored with a calibrated digital thermometer. All samples were stir-fried for 12 min.

Assessment of *in vitro* Bioaccessibility of Heavy Metals in Plant Samples Using A Physiology-Based Extraction Test (PBET)

The *in vitro* bioaccessibility of the plant samples was assessed using the PBET method, as outlined by Intawongse & Dean (2008), with minor modifications.

Gastric stage: The gastric solution was prepared by combining 1.25 g of pepsin, 0.42 mL of lactic acid, 0.5 g of sodium citrate, and 0.50 mL of acetic acid. Deionized water was then added to a volumetric flask to reach a final volume of 1 L. The pH was carefully adjusted to 2.5 (± 0.05) with concentrated HCl.

Subsequently, 0.3 g of raw or treated plant sample was mixed with 30 mL of gastric solution in a 250 mL beaker. The beaker was placed in a shaking water bath at 37°C and agitated at 100 rpm for one hour. After centrifugation (3000 rpm for 10 min), a 5 mL aliquot was collected and analyzed for trace elements by Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES).

Intestinal stage: To the remaining reaction mixture, 5 mL of fresh gastric solution was added, and the pH was adjusted to 7 using a saturated $NaHCO_3$ solution. The mixture was supplemented with 15 mg of pancreatin and 52.5 mg of bile salts. The sample was gently agitated (100 rpm) in a temperature-controlled water bath at 37 °C for 2 hours. After centrifugation (3000 rpm / 10 min), a 5 mL aliquot was taken and subjected to metal analysis using ICP-OES.

Analysis of Total Heavy Metal Concentrations i*n A. sessilis and C. asiatica*

Each plant sample was analyzed for total concentrations of Ni, Cd, Cu, Cr, and Pb using the AOAC 999.11 method (AOAC 2022). Oven-dried samples were subjected to dry ashing at 550 °C in a muffle furnace. The resulting ash was treated with concentrated nitric acid to convert the elements into a chemically detectable form. The solution was filtered and appropriately diluted. Finally, the concentrations of the elements in the solution were quantified using ICP-OES.

Calculation of Bioaccessibility (%)

Bioaccessibility (%) = 100 × Y/Z, where Y is the element concentration in the bioaccessible portion (mg.100^{-1} g of

Fig. 2: (a) *Alternanthera sessilis* and (b) *Centella asiatica* species collected from the cultivation areas.

sample), and Z is the total metal concentration (mg.100^{-1} g of sample).

Validation of Analytical Method

To validate the analytical procedure, the certified reference material (CRM) GBW10015 – Spinach was used. The method's accuracy and reliability were assessed through quality control evaluations and figures of merit. The limit of detection (LOD) for each element was determined from the calibration curve's slope and three times the standard deviation (SD) of ten replicate blank measurements. The LODs for ICP-OES were 0.025 μg.g^{-1} for Ni, 0.005 μg.g^{-1} for Cd, 0.010 μg.g^{-1} for Cr, 0.050 μg.g^{-1} for Pb, and 0.015 μg.g^{-1} for Cu. For additional quality assurance, total concentrations of Ni, Cd, Cr, Pb, and Cu were quantified in the GBW10015 spinach reference material. The measured concentrations of metals showed strong agreement with the certified values, with recovery rates ranging from 96% to 104%, thereby confirming the method's accuracy and robustness.

Data Analysis

The heavy metal data were analyzed using Microsoft Excel to generate descriptive statistics, including the mean, minimum, maximum, and standard deviation. Statistical analysis was performed using one-way ANOVA to determine the effect of different cooking treatments on the bioaccessibility of heavy metals in *A. sessilis* and *C. asiatica* samples. Differences among means were considered significant at $p < 0.05$, and post-hoc comparisons were carried out using Tukey's HSD test.

RESULTS AND DISCUSSION

In vitro Bioaccessibility of Heavy Metals in *A. sessilis* and *C. asiatica*

In recent years, *in vitro* screening techniques have been progressively refined to evaluate the bioavailability and bioaccessibility of nutrients in foods. Bioavailability, the amount of a nutrient absorbed and available for physiological functions, is influenced by digestion, nutrient release from the food matrix, absorption by intestinal cells, and transport to body tissues. In contrast, bioaccessibility is the fraction of a nutrient that, once consumed, is potentially available for absorption, determined primarily by digestion and the subsequent release from the food matrix (Etcheverry et al. 2012). Unlike bioaccessibility, bioavailability, which is associated with a physiological or metabolic outcome, cannot be directly quantified using *in vitro* methods. Additionally, factors that influence nutrient absorption—such as an individual's nutrient status, genotype, age, physiological state (e.g., pregnancy, lactation, or obesity), chronic or acute illnesses, gastric acid secretion, and other intrinsic factors—cannot be addressed in *in vitro* experiments (Etcheverry et al. 2012).

To evaluate bioaccessibility, an *in vitro* digestion model is utilized to mimic the human digestive system. This procedure generally consists of a two-step (or three-step) digestion process, which includes both gastric (stomach) and intestinal digestion stages. Initially, samples are acidified to pH 2 (representing the gastric pH of an adult), and pepsin is added during the gastric digestion phase. Acidifying the samples is essential, as pepsin loses its activity and denatures at pH levels exceeding 5. Before initiating intestinal digestion, the pH of the samples is adjusted to a range of 5.5–6. Next, pancreatin (a blend of pancreatic amylase, lipase, trypsin, and ribonuclease) and bile salts (emulsifiers) are introduced, and the pH is adjusted to a final range of 6.5–7. After digestion, the intestinal mixture is centrifuged to separate the supernatant from the precipitate. The soluble components in the supernatant are measured using spectrophotometry. The solubility percentage is calculated by dividing the amount of soluble component by the total amount of element in the sample (Etcheverry et al. 2012). Tables 1 and 2 present the percentage of heavy metal bioaccessibility in the gastric and intestinal phases of raw, cooked, and stir-fried samples of *A. sessilis* and *C. asiatica* collected from different regions in the Western Province, Sri Lanka. The bioaccessibility in the oral phase was excluded since no metals were detected in either plant during this phase. The data show that the overall metal concentrations in the analyzed samples were significantly higher in the Wellampitiya, Kolonnawa, and Kottawa regions compared to Piliyandala and Bandaragama. However, the proportion of heavy metals potentially absorbed by the body was substantially lower than the total metal concentrations detected in the samples. Additionally, the concentrations of these metals in the gastric and intestinal phases varied considerably, likely influenced by pH differences, phase composition, and the intrinsic properties of the vegetables themselves. Previous studies suggest that metal extraction during each phase can be affected by numerous factors, both *in vitro* and *in vivo*, including sorption, pH, precipitation processes, food type, particle size, residence time, mixing speed, and other physiological variables (Intawongse & Dean 2008). Since the concentration of the investigated metals in several earlier studies was below the detection limit of the *in vitro* extraction technique, bioaccessibility could not be determined (Jayawardene et al. 2010).

Alternanthera sessilis: The bioaccessibility of Ni in the raw, cooked, and stir-fried samples varied in the gastric phase from 7.9% to 16.9%, 0% to 14.9%, and 0% to 11.9%, respectively. In the intestinal phase, the bioaccessibility

Table 1: *In vitro* bioaccessibility of heavy metals in raw and processed *Alternanthera sessilis* at the gastric and intestinal phases.

Area	Metal	Total metal content [mg.kg^{-1}]	Bioaccessibility of Raw Samples				Bioaccessibility of Cooked Samples				Bioaccessibility of Stir-fried samples			
			Gastric Phase		Intestinal Phase		Gastric Phase		Intestinal Phase		Gastric Phase		Intestinal Phase	
			[mg.kg^{-1}]	%	[mg.kg^{-1}]	%	[mg.kg^{-1}]	%	[mg.kg^{-1}]	%	[mg.kg^{-1}]	%	[mg.kg^{-1}]	%
Piliyandala	Ni	3.02±1.02	0.51±0.04	16.9	0.23±0.04[d]	7.6	0.45±0.05	14.9	0.16±0.02	5.3	0.36±0.06	11.9	0.18±0.07	6.0
N=8	Cd	0.20±0.01	ND	-	ND	-	ND	-	ND	-	ND	-	ND	-
	Cr	0.82±0.20	ND	-	ND	-	ND	-	ND	-	ND	-	ND	-
	Pb	0.24±0.04	ND	-	ND	-	ND	-	ND	-	ND	-	ND	-
	Cu	7.85±2.21	ND	-	2.75±0.99	35	ND	-	2.50±1.00	31.8	ND	-	2.22±1.00	28.3
Wellampitiya	Ni	8.42±2.25	0.68±0.06	8.1	0.56±0.07	6.7	0.51±0.06	6.1	0.46±0.01	5.5	0.5±0.06	5.9	0.15±0.01	1.8
N=8	Cd	0.36±0.06	0.05±0.00	13.9	ND	-	ND	-	ND	-	ND	-	ND	-
	Cr	4.59±1.11	0.22±0.03	4.8	0.19±0.01	4.1	0.11±0.00	2.4	0.06±0.00	1.3	0.09±0.00	2.0	ND	-
	Pb	3.14±0.98	0.48±0.04	15.3	0.34±0.03	10.8	0.65±0.04	20.7	0.32±0.04	10.2	0.51±0.09	16.2	0.11±0.01	3.5
	Cu	15.40±3.23	2.32±0.78	15.1	3.34±1.01	21.7	1.58±0.99	10.3	4.23±1.45	27.5	0.87±0.07	5.6	3.67±1.02	23.8
Kolonnawa	Ni	11.35±2.76	1.52±0.61	13.4	0.21±0.02	1.9	1.01±0.44	8.9	0.64±0.08	5.6	0.86±0.09	7.6	0.03±0.00	0.3
N=8	Cd	0.86±0.04	0.11±0.01	12.8	0.05±0.00	5.8	0.08±0.00	9.3	ND	-	0.06±0.00	7.0	ND	-
	Cr	6.74±2.22	0.85±0.07	12.6	0.23±0.01	3.4	0.53±0.12	7.9	0.27±0.01	4.0	0.59±0.07	8.8	0.13±0.02	1.9
	Pb	3.05±1.01	0.36±0.02	11.8	0.12±0.00	3.9	0.22±0.07	7.2	ND	-	0.15±0.03	4.9	0.05±0.00	1.6
	Cu	10.30±2.99	1.12±0.77	10.9	3.13±0.98	30.4	0.26±0.08	2.5	3.12±1.09	30.3	0.05±0.00	0.5	2.24±1.09	21.7
Kottawa	Ni	9.15±1.87	1.11±0.60	12.1	0.12±0.08	1.3	1.10±0.53	12.0	0.22±0.08	2.4	1.03±0.10	11.3	0.14±0.04	1.5
N=8	Cd	0.22±0.05	ND	-	ND	-	ND	-	ND	-	ND	-	ND	-
	Cr	5.05±1.23	0.31±0.07	6.1	0.15±0.06	3.0	0.33±0.01	6.5	0.30±0.01	5.9	0.27±0.07	5.3	0.05±0.00	1.0
	Pb	3.89±1.00	0.34±0.08	8.7	0.15±0.07	3.9	0.31±0.00	8.0	0.20±0.00	5.1	0.25±0.08	6.4	0.02±0.00	0.5
	Cu	10.90±2.45	2.23±0.80	20.5	3.23±0.97	29.6	1.39±0.99	12.8	2.56±0.89	23.5	1.22±0.12	11.2	1.63±0.42	15.0
Bandaragama	Ni	2.02±0.98	0.16±0.02	7.9	0.05±0.00	2.5	ND	-	ND	-	ND	-	ND	-
N=8	Cd	0.11±0.01	ND	-	ND	-	ND	-	ND	-	ND	-	ND	-
	Cr	0.78±0.07	ND	-	ND	-	ND	-	ND	-	ND	-	ND	-
	Pb	0.31±0.05	ND	-	ND	-	ND	-	ND	-	ND	-	ND	-
	Cu	6.96±1.45	1.12±0.67	16.1	2.36±0.89	33.9	1.06±0.50	15.2	2.03±0.87	29.2	0.95±0.09	13.6	1.98±0.65	28.4

ND = not detected: N = sample size: WHO/FAO permissible limits: Ni = 4 mg.kg^{-1}, Cd = 0.2 mg.kg^{-1}, Cr = 2.3 mg.kg^{-1}, Pb = 0.3 mg.kg^{-1} and Cu = 40 mg.kg^{-1}

Table 2: *In vitro* bioaccessibility of heavy metals in raw and processed *Centella asiatica* at the gastric and intestinal phases.

Area	Metal	Total metal content [mg.kg^{-1}]	Bioaccessibility of Raw Samples				Bioaccessibility of Cooked Samples				Bioaccessibility of Stir-fried samples			
			Gastric Phase		Intestinal Phase		Gastric Phase		Intestinal Phase		Gastric Phase		Intestinal Phase	
			[mg.kg^{-1}]	%	[mg.kg^{-1}]	%	[mg.kg^{-1}]	%	[mg.kg^{-1}]	%	[mg.kg^{-1}]	%	[mg.kg^{-1}]	%
Piliyandala	Ni	2.11±1.01	ND	-	ND	-	ND	-	ND	-	ND	-	ND	-
N=8	Cd	0.15±0.05	ND	-	ND	-	ND	-	ND	-	ND	-	ND	-
	Cr	0.72±0.12	ND	-	ND	-	ND	-	ND	-	ND	-	ND	-
	Pb	0.27±0.09	ND	-	ND	-	ND	-	ND	-	ND	-	ND	-
	Cu	10.60±2.12	0.85±0.06	8.0	1.04±0.87	9.8	0.56±0.34	5.3	0.98±0.41	9.2	0.66±0.22	6.2	1.01±0.09	9.5
Wellampitiya	Ni	14.06±3.12	1.41±0.07	10.0	0.65±0.12	4.6	1.23±0.71	8.7	0.69±0.25	4.9	1.31±0.45	9.3	0.52±0.02	3.7
N=8	Cd	0.32±0.08	ND	-	ND	-	ND	-	ND	-	ND	-	ND	-
	Cr	5.82±2.03	0.11±0.02	1.9	0.06±0.00	1.0	0.10±0.02	1.7	0.06±0.01	1.0	0.13±0.54	2.2	0.04±0.00	0.7
	Pb	5.90±2.01	0.58±0.07	9.8	0.12±0.07	2.0	0.59±0.12	10.0	0.11±0.07	1.9	0.45±0.09	7.6	0.09±0.10	1.5
	Cu	16.26±5.21	1.86±0.06	11.4	4.69±1.23	28.8	1.33±0.76	8.2	3.65±1.10	22.4	1.03±0.29	6.3	4.52±1.21	27.8
Kolonnawa	Ni	16.55±5.25	1.53±0.70	9.2	0.64±0.20	3.9	1.36±0.45	8.2	0.13±0.04	0.8	1.22±0.33	7.4	0.10±0.02	0.6
N=8	Cd	0.45±0.11	0.05±0.00	11.1	ND	-	ND	-	ND	-	ND	-	ND	-
	Cr	11.33±2.31	0.29±0.02	2.6	0.05±0.01	0.4	0.26±0.08	2.3	ND	-	0.21±0.13	1.9	0.03±0.00	0.3
	Pb	10.23±1.98	1.01±0.08	9.9	0.22±0.11	2.2	0.98±0.45	9.6	0.32±0.10	3.1	0.96±0.32	9.4	0.21±0.09	2.1
	Cu	21.22±6.86	2.31±0.89	10.9	8.36±2.33	39.4	2.45±1.01	11.5	7.60±2.01	35.8	3.40±1.06	16.0	6.70±1.23	31.6
Kottawa	Ni	11.23±2.45	1.12±0.70	10.0	0.54±0.12	4.8	1.10±0.40	9.8	0.25±0.02	2.2	0.95±0.11	8.5	0.10±0.03	0.9
N=8	Cd	0.30±0.08	ND	-	ND	-	ND	-	ND	-	ND	-	ND	-
	Cr	4.36±2.00	0.08±0.01	1.8	ND	-	ND	-	ND	-	ND	-	ND	-
	Pb	3.21±1.98	0.30±0.05	9.3	0.06±0.01	1.9	0.10±0.02	3.1	0.06±0.00	1.9	0.09±0.01	2.8	ND	-
	Cu	16.22±5.43	0.83±0.12	5.1	4.83±1.12	29.8	1.21±0.76	7.5	3.65±1.01	22.5	0.56±0.12	3.5	4.20±0.99	25.9
Bandaragama	Ni	1.45±0.98	0.16±0.05	11.0	ND	-	ND	-	ND	-	ND	-	ND	-
N=8	Cd	0.15±0.07	ND	-	ND	-	ND	-	ND	-	ND	-	ND	-
	Cr	0.63±0.12	ND	-	ND	-	ND	-	ND	-	ND	-	ND	-
	Pb	0.30±0.12	ND	-	ND	-	ND	-	ND	-	ND	-	ND	-
	Cu	5.32±0.57	ND	-	0.43±0.22	8.1	ND	-	ND	-	ND	-	ND	-

ND = not detected: N = sample size: WHO/FAO permissible limits: Ni = 4 mg.kg^{-1}, Cd = 0.2 mg.kg^{-1}, Cr = 2.3 mg.kg^{-1}, Pb = 0.3 mg.kg^{-1} and Cu = 40 mg.kg^{-1}

of Ni ranged from 1.3% to 7.6%, 0% to 5.6%, and 0% to 6%, respectively. The bioaccessibility of Cd in the gastric phase varied from 0% to 13.9% for the raw sample, 0% to 9.3% for the cooked sample, and 0% to 7% for the stir-fried sample. In the intestinal phase, Cd was only detected in the raw samples, with bioaccessibility ranging from 0% to 5.8% across different locations. For Cr, the bioaccessibility in the gastric phase ranged from 0% to 12.6%, 0% to 7.9%, and 0% to 8.8% for the raw, cooked, and stir-fried samples, respectively. In the intestinal phase, the bioaccessibility of Cr ranged from 0% to 4.1%, 0% to 5.9%, and 0% to 1.9% for the raw, cooked, and stir-fried samples, respectively. The bioaccessibility of Pb in the gastric and intestinal phases varied as follows: for the raw sample, 0% to 15.3% and 0% to 10.8%, for the cooked sample, 0% to 20.7% and 0% to 10.2%, and for the stir-fried sample, 0% to 16.2% and 0% to 3.5%, respectively. Unlike other metals, the bioaccessibility of Cu was lower in the gastric phase (0% to 20.1%, 0% to 15.2%, and 0% to 13.6%) compared to the intestinal phase (21.7% to 35.0%, 23.5% to 31.8%, and 15.0% to 28.4%) for the raw, cooked, and stir-fried samples, respectively (Table 1).

Centella asiatica: The bioaccessibility of Ni in the gastric and intestinal phases for raw, cooked, and stir-fried samples varied as follows: 0% to 11% and 0% to 4.8%, 0% to 9.8% and 0% to 4.9%, and 0% to 9.3% and 0% to 3.7%, respectively. Cd was detected only in the gastric phase of the raw samples, ranging from 0% to 11.1%. The bioaccessibility of Cr in the gastric phase ranged from 0% to 2.6%, 0% to 2.3%, and 0% to 2.2%, while in the intestinal phase, it ranged from 0% to 1.0%, 0% to 1.0%, and 0% to 0.7% for the raw, cooked, and stir-fried samples, respectively. Pb bioaccessibility in the gastric phase varied from 0% to 9.9%, 0% to 10%, and 0% to 9.4% for the raw, cooked, and stir-fried samples, respectively. In the intestinal phase, Pb bioaccessibility ranged from 0% to 2.2%, 0% to 3.1%, and 0% to 2.1%, respectively, for the raw, cooked, and stir-fried samples. Higher Cu concentrations were detected in the intestinal phase (8.1% to 39.4%, 0% to 35.8%, and 0% to 31.6%) compared to the gastric phase (0% to 11.4%, 0% to 11.5%, and 0% to 16.0%) for the raw, cooked, and stir-fried samples, respectively (Table 2).

As demonstrated in Figs. 3 and 4, the bioaccessibility of heavy metals in *Alternanthera sessilis* and *Centella asiatica*, analyzed in this study, reveals distinct patterns. On average, the bioaccessibility percentages of Ni, Cd, Cr, and Pb were significantly higher ($p<0.05$) during the gastric phase than in the intestinal phase, except for Cu.

In the gastric phase, the acidic environment and the high concentration of hydrogen ions (H^+) facilitate the dissolution of metals. The formation of complex ions with chloride (Cl^{-1}) helps maintain the solubility of metals (Ruby et al. 1996). Gastric enzymes and organic acids further assist in enhancing the solubility of these elements in the stomach. Conversely,

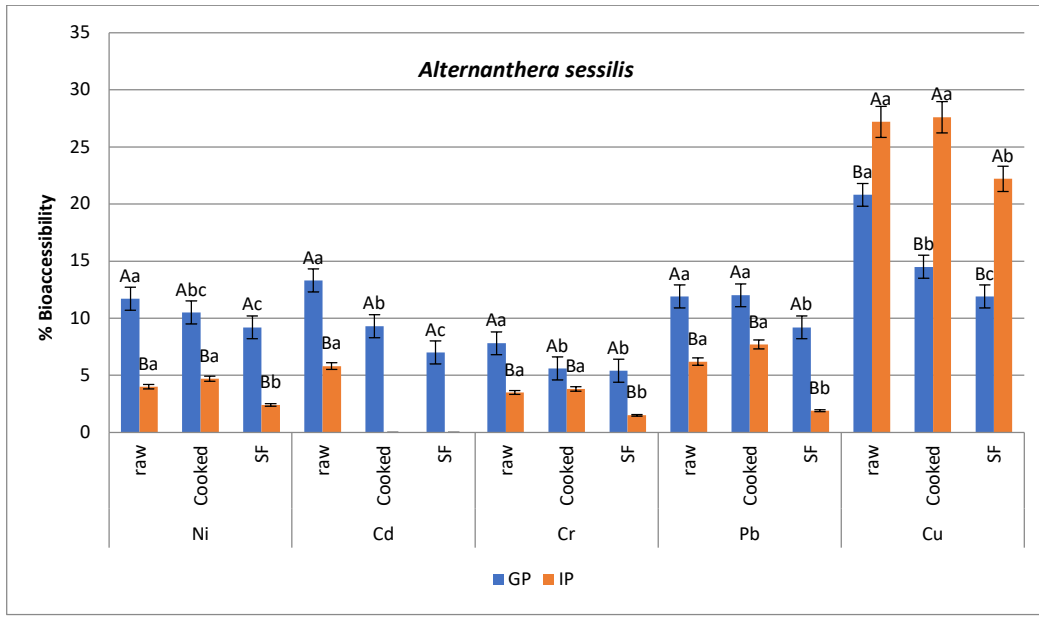

Fig. 3: Average bio-accessibility (%) of heavy metals in raw, cooked, and stir-fried *A. sessilis* in the gastric and intestinal phases (GP = Gastric Phase and IP = Intestinal Phase).
Different uppercase letters indicate significant differences ($P<0.05$) between GP and IP for each treatment within a specific heavy metal, while different lowercase letters indicate significant differences ($P<0.05$) among treatments (raw, cooked, stir-fried) for each heavy metal within GP and IP.

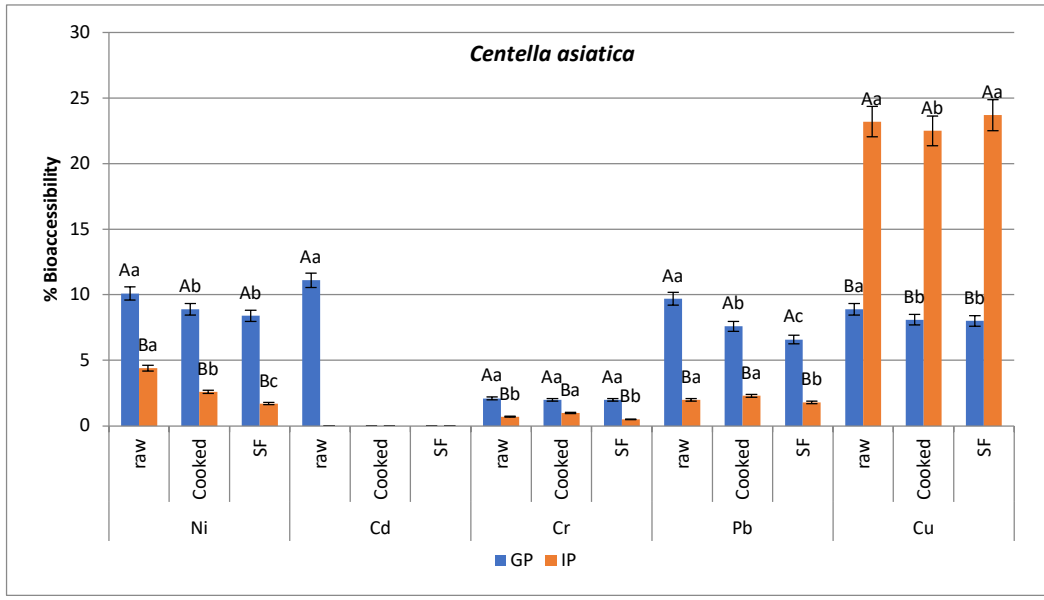

Fig. 4: Average bio-accessibility (%) of heavy metals in raw, cooked, and stir-fried *C. asiatica* in the gastric and intestinal phases (GP = Gastric Phase and IP = Intestinal Phase).

Different uppercase letters indicate significant differences ($P<0.05$) between GP and IP for each treatment within a specific heavy metal, while different lowercase letters indicate significant differences ($P<0.05$) among treatments (raw, cooked, stir-fried) for each heavy metal within GP and IP.

in the intestinal phase, metal concentrations tend to decrease due to binding or precipitation caused by the higher pH (Chaney et al. 1989, Wang et al. 2024). Some studies suggest that gastric-phase extractions alone can provide a reliable estimate of bioaccessibility (Gasser et al. 1996, Hamel et al. 1998). However, this research included both gastric and intestinal phases to offer a more comprehensive understanding of the digestive system's processes.

The absorption site for Cu in humans remains contested, with some studies indicating higher bioaccessibility in the gastric phase (Pan et al. 2016, Wang et al. 2024), while others suggest increased absorption during the intestinal phase (Chaney et al. 1989). Several factors affect Cu absorption, such as its chemical form (e.g., Cu acetate and Cu sulfate are more bioavailable than Cu oxide), along with dietary enhancers like citrate, phosphate, and animal proteins. On the other hand, inhibitors such as Zn, Cd, phytates, and sugars may reduce Cu absorption.

Despite the high total concentrations of heavy metals in *A. sessilis* and *C. asiatica*, their bioaccessible fractions were considerably lower ($p<0.05$) across raw, cooked, and stir-fried samples. This indicates that total heavy metal concentrations do not directly correlate with the amounts available for absorption by the human body. Literature suggests that typical dietary absorption rates are about 5% for Cd, 0.4-2.5% for Cr, 30-40% for Cu, 10% for Pb (40-50% in children), and 1-10% for Ni, with the remainder excreted through urine or feces (Jaishankar 2014). In our study, the average bioaccessible fractions of heavy metals in *A. sessilis* and *C. asiatica* were as follows: Gastric phase: 9.98% Ni, 9.46% Cd, 3.93% Cr, 9.70% Pb, and 13.8% Cu; Intestinal phase: 3.51% Ni, 5.81% Cd, 1.97% Cr, 3.15% Pb, and 33.6% Cu. These results are consistent with previous findings.

The methods of cooking influenced the bioaccessibility of heavy metals, with cooking and stir-frying generally reducing bioaccessibility when compared to raw samples. For example, Cd bioaccessibility was significantly lower in cooked and stir-fried *A. sessilis* and *C. asiatica*. Yang et al. (2012) pointed out that Cd bioaccessibility is dependent on its chemical binding forms and the characteristics of the food. However, differences in bioaccessibility for other metals among raw, cooked, and stir-fried samples were less pronounced ($p<0.05$). Stir-frying resulted in the lowest bioaccessibility for most metals of concern. Various factors, such as the type of green leafy vegetable, food composition (e.g., antinutrients like phytates and tannins), cooking methods, processing temperature, and the cooking medium (e.g., coconut milk or oil), likely influence metal extraction during digestion.

Studies by Intawongse & Dean (2008), Jayawardene et al. (201), and Hu et al. (2013) offer valuable perspectives on heavy metal bioaccessibility across different food matrices. Hu et al. (2013) found patterns similar to this study, showing higher bioaccessibility for Ni, Cd, Cr, and Pb during the gastric phase, with Cu bioaccessibility being higher during the intestinal phase. Intawongse & Dean (2008) observed that

metals in vegetables were mostly in insoluble forms at neutral pH but became soluble in the acidic gastric phase. Jayawardene et al. (2010) reported comparable findings in medicinal plants, noting significantly higher bioaccessibility of Pb, As, and Cd during the gastric phase. Other research supports the finding that low pH in the gastric phase enhances metal solubility.

Turner and Ip (2007), Ovca et al. (2011), and Tremlova et al. (2012) found reduced bioaccessibility in the intestinal phase due to factors such as antinutrient binding, complex formation, or precipitation. For example, phytates and tannins can form insoluble complexes with metals, reducing their bioaccessibility. Conversely, some studies, such as those by Kulkarni et al. (2007) and De Lima et al. (2014), reported greater bioaccessibility in the intestinal phase. Kulkarni et al. (2007) observed higher bioaccessibility of K, Mn, Zn, and Fe during intestinal digestion in wheat products. Yin et al. (2017) found varying bioaccessibility across gastric and intestinal phases in vegetables from Beijing markets, with Cu showing significantly higher bioaccessibility in the intestinal phase.

Pan et al. (2016) studied health risks related to heavy metals in vegetables grown near a waste incinerator, finding bioaccessible fractions that aligned with the results in this study for Cd, Cr, Cu, Ni, and Pb in different gastrointestinal phases.

CONCLUSIONS

This research underscores the importance of evaluating bioaccessibility rather than total heavy metal content when assessing potential health risks. Although *Alternanthera sessilis* and *Centella asiatica* from urban areas in Sri Lanka showed elevated total heavy metal concentrations, their bioaccessible fractions were significantly lower, indicating limited absorption by the human body. These findings suggest that total heavy metal concentrations do not directly reflect the amounts available for absorption. Ongoing monitoring of metal contamination and the adoption of safer farming practices are essential to mitigate risks. Future research is recommended to include a broader range of foods and incorporate *in vivo* studies to provide a more comprehensive understanding of dietary exposure to heavy metals.

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Do Climate Variables Influence Fish Production in Top Fishery Economies? Evidence from the ARDL Approach

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ABSTRACT

Climate change poses significant challenges to food security worldwide, particularly in the fisheries sector, where fish production is highly sensitive to climatic variables. This study investigates the long- and short-run impacts of climate change on fish production in four major fish-producing countries, China, India, Vietnam, and Bangladesh, using annual time-series data from 1990 to 2020. An Autoregressive Distributed Lag (ARDL) model was employed to examine long-run equilibrium relationships between climate factors (precipitation, minimum, mean, and maximum temperatures, and CO_2 emissions) and total fish production, as well as adjustments to short-run deviations. The findings revealed distinct patterns across countries: CO_2 emissions positively influenced long-term fish production in China, India, and Bangladesh, whereas precipitation boosted fish production in China and Bangladesh. In contrast, Vietnam showed no long-run equilibrium, indicating higher sensitivity to short-term climatic fluctuations. In the short run, CO_2 emissions significantly enhanced fish production in Bangladesh, with regional temperature effects varying. The minimum temperature positively impacted long-term fish production in China but negatively affected it in Bangladesh. In Vietnam, an increase in the maximum temperature enhanced short-run production, whereas a decrease in the minimum temperature reduced it. This study examined the critical role of CO_2 emissions, precipitation, and temperature in influencing fish production, offering key insights for policymakers to develop adaptive strategies for sustainable fish production amid climate change.

INTRODUCTION

Global fisheries and aquaculture are vital to economies worldwide, contributing significantly to GDP and supporting global food security (FAO 2024). Approximately 3 billion people worldwide depend on fish and fishery products to meet 20 percent of their animal protein intake (FAO 2024). However, these sectors are increasingly at risk due to changing climate patterns. According to the Intergovernmental Panel on Climate Change (IPCC), climate change is a statistically significant alteration in climate properties that persists for decades or longer, resulting from both natural variability and human activities. This phenomenon is widely acknowledged as an inevitable outcome of over 200 years of greenhouse gas emissions from various sources (Lee et al. 2023). The impacts of climate change are irreversible (Masson- Delmotte et al. 2021) and have led to significant declines in aquatic system diversity and productivity. Climate change poses significant risks to marine and freshwater species and the ecosystems they inhabit (Allan et al. 2005, FAO 2024). Tropical regions, particularly South Asia, are especially vulnerable to these effects (Pörtner et al. 2014). Key climate change impacts, such as rising temperatures, changes in precipitation, sea level rise, harmful algal blooms, increased disease, ocean acidification, and extreme weather events,

directly influence fish production by altering the biological productivity of fish stocks, as evidenced by various studies (O' Reilly et al. 2003, Perry et al. 2005, Vollmer et al. 2005, Arnason 2007, Cochrane et al. 2009, Eboh 2009, Gamito et al. 2013, Muthoka et al. 2024). These changes pose risks not only to coastal regions that sustain fisheries and aquaculture but also to the livelihoods, productivity, and well-being of the communities that rely on them (Daw et al. 2009, Badjeck et al. 2010), and the consequent rise in prices is expected to have substantial impacts on food security (Agnishwaran et al. 2024). An estimated 3. 3.3–3. 6 billion people face high vulnerability to the effects of climate change, with the most severe risks concentrated in underdeveloped and developing regions. Climate change disrupts critical processes in fish species, such as feeding, migration, and breeding behaviors, further intensifying its impact on fisheries (Brander 2010). The effects of climate variables, such as temperature, precipitation, and CO_2 emissions, on fish production are complex and regionally variable, necessitating a detailed understanding of their effects across different contexts.

Climate change induces significant hydrological changes in aquatic ecosystems. Since 1850, global temperatures have risen by 1.1°C, primarily driven by human-induced global warming, with adverse effects on the environment (Lee et al. 2023). These shifts alter the physical and chemical properties of water bodies, including temperature, salinity, and pH, which in turn influence the physiological, biological, and genetic traits of aquatic species (Menon et al. 2023). Temperature fluctuations contribute to thermal stratification and the development of oxygen minimum zones in water bodies, posing challenges to the survival of various organisms (Ng'onga et al. 2019, Mugwanya et al. 2022). Elevated CO_2 levels alter ocean pH, leading to ocean acidification and coral bleaching, which can affect aquatic biodiversity (Thomas et al. 2022). Furthermore, the combination of climate change and overexploitation may exert additional pressure on fish populations (Perry et al. 2010, Planque et al. 2010). Water scarcity, exacerbated by rising temperatures and fluctuating rainfall patterns, presents further challenges to freshwater and marine ecosystems, potentially intensifying the impact of existing pollution (Alsaleh 2024).

Despite clear evidence of climate change's effects on aquatic ecosystems and fisheries, these impacts are often overlooked in climate adaptation policies (Badjeck et al. 2010). Analyzing long-run equilibrium relationships between climate variables and fish production, and understanding adjustments to short-run deviations, is critical for effective, evidence-based decision-making and resource allocation. Among the key fish-producing countries, China, India, Vietnam, and Bangladesh are pivotal to the global fisheries sector and are vulnerable to climate change impacts. Although substantial research has examined the relationship between climate change and fisheries globally (Cheung et al. 2009, Das et al. 2020, Doney et al. 2012,2009, Fernandes et al. 2016, Lam et al. 2012, Mohanty et al. 2017, Ninawe et al. 2018, Raubenheimer & Phiri, 2023, Suh & Pomeroy 2020, Vass et al. 2009), the application of advanced econometric techniques, such as the Autoregressive Distributed Lag (ARDL) approach, within the fisheries sector remains limited. Most empirical studies employing ARDL models have focused on the agricultural sector (Janjua et al. 2014, Zhai et al. 2017, Ahsan et al. 2020, Chandio et al. 2020, Demirhan 2020, Nasrullah et al. 2021, Warsame et al. 2021, Ramzan et al. 2022, Tagwi 2022, Waris et al. 2023). Addressing this gap, the present study uses the ARDL model to explore long-run cointegration relationships between climate variables and fish production in leading fish economies, thereby offering new insights into the fisheries sector's response to climate change. The ARDL model is particularly adept at analyzing long-run relationships and performs well with small sample sizes, providing reliable results in regression contexts (Bhuyan, Mohanty & Patra 2023).

MATERIALS AND METHODS

Data and Variables

The annual dataset contains time-series statistics from 1990 to 2020 for four Asian countries, namely China, India, Vietnam, and Bangladesh. The key variables of interest were total fish production (TFP), which includes both marine and inland fish production, measured in million tons, climatic variables, such as precipitation, measured in mm, minimum temperature, mean temperature, and maximum temperature, expressed in degrees Celsius, and CO_2 emissions, measured in metric tons per capita. The dataset was compiled from FishStatJ, Food and Agriculture Organization (FAO 2021) for total fish production, Climate Change Knowledge Portal (CCKP), and World Development Indicators (WDI) for climate variables (see Table 1). This study used total fish production as the dependent variable, whereas precipitation, minimum temperature, mean temperature, maximum temperature, and CO_2 emissions were employed as explanatory variables. The selection of climate variables in this study was based on their critical influence on marine and inland ecosystems, particularly concerning fish production. Precipitation is a key variable owing to its direct impact on freshwater inputs into coastal and marine environments, altering salinity, nutrient levels, and habitat conditions, which are crucial for the distribution and productivity of fish populations (Nye et al. 2009). Minimum temperature is justified by its role in defining the lower thermal limit for

fish, where sudden drops can cause thermal stress, reducing metabolic rates and impairing growth, reproduction, and survival, thereby revealing the species' vulnerability to cold extremes (Volkoff & Rønnestad 2020). Mean temperature is crucial for understanding the long-term thermal environment affecting metabolic functions, growth, and reproduction, with shifts potentially altering species distribution and ecosystem dynamics (Mugwanya et al. 2022). Maximum temperature was selected for its importance in assessing the impacts of extreme heat on fish, where exceeding thermal thresholds can lead to heat stress, habitat loss, and mortality, driving shifts in species distribution and community structure (Neubauer and Andersen, 2019). CO_2 emissions, a key driver of global warming and ocean acidification, were selected for their extensive impact on marine and inland ecosystems, as elevated CO_2 levels lead to rising surface temperatures that influence fish physiology, behavior, and habitat availability (Harley et al. 2006, Fabry et al. 2008). By including these variables, this study aimed to provide a comprehensive assessment of how various aspects of climate change collectively influence fish production. To address multicollinearity (Mansfield & Helms 1982) and heteroscedasticity (Engle 1982) in the annual time-series data, we applied natural logarithmic transformations to all variables. This logarithmic transformation stabilizes the data variance and produces more reliable and precise results (Dumrul & Kilicaslan 2017).

Econometric Methodology

Model Specification

The empirical framework for this study is outlined in the following implicit form:

$$TFP_t = f(PREC_t, MINTEM_t, MEANTEM_t, MAXTEM_t, CO_{2_t}) \quad \ldots(1)$$

The relationship in its fitted form can be expressed as follows:

$$LnTFP_t = \alpha_0 + \alpha_1 LnPREC_t + \alpha_2 LnMINTEM_t + \alpha_3 LnMEANTEM_t + \alpha_4 LnMAXTEM_t + \alpha_5 LnCO_{2_t} + \varepsilon_t \ldots(2)$$

where $LnTFP_t$ denotes the logarithm of total fish production, $LnPREC_t$ stands for the logarithm of precipitation, $LnMINTEM_t$ represents the logarithm of the minimum$LnMEANTEM_t$ temperature, indicates the logarithm of mean temperature, $LnMAXTEM_t$ signifies the logarithm of maximum temperature, and $LnCO_{2_t}$ refers to the logarithm of CO_2 emissions.

Auto Regressive Distributed Lag (ARDL)

We employed an Autoregressive Distributed Lag (ARDL) model to analyze the short-run and long-run relationships between total fish production and climatic variables (Pesaran & Shin 1995, Pesaran et al. 2001). The choice of the ARDL model is driven by its suitability for examining cointegration and short-term relationships and its effectiveness as an alternative to the more commonly employed Johansen test (Asumadu-Sarkodie & Owusu 2016, Abbas 2020, Chandio et al. 2020, Warsame et al. 2021). This model is particularly advantageous because it offers unbiased long-run estimates even when some endogenous variables are treated as regressors (Adom et al. 2012). The ARDL approach estimates both short- and long-run coefficients using Ordinary Least Squares (OLS) and accommodates regressors that may be either integrated at I(0), I(1), or mutually cointegrated. Unlike many other cointegration methods, the ARDL model delivers consistent results even with smaller sample sizes (Pesaran & Shin, 1995, Pesaran et al. 2001, Adom et al. 2012). In this context, the ARDL model is well-suited for estimating the impact of climate change on total fish production.

Unit Root Tests

To accurately assess the impact of climate change on total fish production in Asian countries, it is crucial to first verify the stationarity of the variables to prevent biased outcomes. To ensure stationarity in the time-series data, we applied unit-root tests, specifically the Augmented Dickey-Fuller "ADF" (1979) and Phillips-Perron "PP" (1988) tests. The ADF test was conducted using the following regression equation:

$$\Delta Y_t = \alpha + \beta_t + \gamma Y_{t-1} + \sum_{i=1}^{p} \delta_i \Delta Y_{t-i} + \varepsilon_t \quad \ldots(3)$$

where, ΔY_t represents the first difference of the variable Y_t, α denotes a constant term, b_t is the coefficient associated with the time trend t, γ is the coefficient of the lagged level of the series, δ_i are the coefficients corresponding to the lagged first differences, p indicates the number of lagged terms, and ε_t represents the error term.

The Phillips-Perron (PP) test was also used to complement the ADF test. It addresses serial correlation and heteroscedasticity in the error terms through nonparametric adjustments to the test statistics (Vogelsang and Wagner, 2013). The PP test equation is expressed as:

$$Y_t = \alpha + \beta_t + \gamma Y_{t-1} + \varepsilon_t \quad \ldots(4)$$

For both tests, the presence of a unit root in the time series is determined by examining whether the p-value is below 0.05. If the null hypothesis (H0), which suggests non-stationarity, is rejected, it favours the acceptance of the alternative hypothesis (H1), indicating that the series is stationary.

Estimation Procedure

The ARDL model was used to assess the relationships among variables by initially examining the presence of a

long-run association. In this study, the long-term association between lnTFP, lnPREC, lnMINTEM, lnMEANTEM, lnMAXTEM, and $lnCO_2$ was evaluated through the bounds testing approach. The ARDL bounds testing model for our study can be described as:

$$\Delta LnTFP_t = \alpha_0 + \alpha_1 \sum_{i=1}^{p} \Delta LnTFP_{t-i} + \alpha_2 \sum_{i=1}^{q1} \Delta LnPREC_{t-i} + \alpha_3 \sum_{i=1}^{q2} \Delta LnMINTEM_{t-i} + \alpha_4 \sum_{i=1}^{q3} \Delta LnMEANTEM_{t-i} + \alpha_5 \sum_{i=1}^{q4} \Delta LnMAXTEM_{t-i} + \alpha_6 \sum_{i=1}^{q5} \Delta LnCO_{2t-i} + \gamma_1 LnTFP_{t-i} + \gamma_1 LnPREC_{t-i} + \gamma_1 LnMINTEM_{t-i} + \gamma_1 LnMEANTEM_{t-i} + \gamma_1 LnMAXTEM_{t-i} + \gamma_1 LnCO_{2t-i} + \varepsilon_t \quad \ldots(5)$$

Where, α_i and γ_i are short- and long-run coefficients, α_0 is the constant, p and q_i are optimal lag orders of regressand and regressors, Δ represents the first difference operator and ε_t is the white noise error term.

To assess the long-run relationship among the variables, we formulated the following hypotheses: the null hypothesis (H0) assumes no long-run association among the variables ($\alpha1 = \alpha2 = \alpha3 = \alpha4 = \alpha5 = \alpha6$), while the alternative hypothesis (H1) indicates differing parameters ($\alpha1 \neq \alpha2 \neq \alpha3 \neq \alpha4 \neq \alpha5 \neq \alpha6$). The ARDL bounds-testing method employs F-statistics to determine the long-term cointegration among the selected variables. According to Pesaran et al. (2001), the F-test statistics involve two key thresholds: the lower and upper limits. An F-statistic falling below the lower bound indicates no significant long-term relationship, whereas a statistic exceeding the upper bound suggests the presence of a long-term association. If the F-test statistic is between these bounds, the results are inconclusive.

To capture the short-term dynamics between variables, an ARDL-based Error Correction Model (ECM) was employed, as detailed below.

$$\Delta LnTFP_t = \emptyset_0 + \emptyset_1 \sum_{i=1}^{p} \Delta LnTFP_{t-i} + \emptyset_2 \sum_{i=1}^{q-1} \Delta LnPREC_{t-i} + \emptyset_3 \sum_{i=1}^{q-1} \Delta LnMINTEM_{t-i} + \emptyset_4 \sum_{i=1}^{q-1} \Delta LnMEANTEM_{t-i} + \emptyset_5 \sum_{i=1}^{q-1} \Delta LnMAXTEM_{t-i} + \emptyset_6 \sum_{i=1}^{q-1} \Delta LnCO_{2t-i} + \emptyset\, ECT_{t-1} + \epsilon_t \quad \ldots(6)$$

Where, $\varnothing_0$ represents the intercept, $\varnothing_i$ denotes the short-run coefficient, $\in_t$ is the error term, and ECT_{t-1} indicates the lagged residual from the model that determines the long-term relationship. The error correction method describes the speed at which adjustment occurs to restore long-term equilibrium after a short-term shock.

Equation (6) illustrates that total fish production is influenced by its past values, the current and lagged values of the regressors, and the lagged error term. Parameter $\varnothing$ is anticipated to be negative (between 0 and -1), as this indicates the extent to which equilibrium is restored in absolute terms. A positive $\varnothing$ indicates that the model is out of equilibrium and unstable, with no tendency to return to long-run equilibrium. The optimal lag lengths for each variable were established using the Akaike Information Criterion (AIC).

Diagnostic and Stability Tests

This study conducted a series of diagnostic tests to evaluate the model's reliability and validity, following the methodology outlined by Pesaran et al. (2001). To detect serial correlation, the Breusch-Godfrey Serial Correlation LM Test was applied, which is recognized for its ability to accommodate lagged dependent variables, thus enhancing the model's reliability (Breusch 1978, Godfrey 1978). Heteroscedasticity was assessed using the Breusch-Pagan-Godfrey (BPG) test, which ensures accurate variance in the residuals and robustness of the model's estimates (Breusch & Pagan 1979). The normality of the residuals was assessed using the Jarque-Bera (JB) test, which evaluates the skewness and kurtosis of the residuals to determine whether they follow a normal distribution, thereby confirming the appropriateness of the model (Jarque & Bera 1987). To examine the stability of both long and short-run coefficients, the cumulative sum of recursive residuals (CUSUM) test was conducted, as proposed by Brown et al. (1975)

RESULTS AND DISCUSSION

Descriptive Statistics

Table 2 reports the descriptive statistics for the study variables for each country from 1990 to 2020. Total fish production revealed significant disparities, with China exhibiting the highest mean production at 53.19 million metric tons (MT), followed by India (7.48 MT), Vietnam (3.83 MT), and Bangladesh (2.40 MT). The skewness values indicate that China's production distribution is slightly left-skewed (-0.20), whereas India, Vietnam, and Bangladesh display right-skewed distributions, indicating the presence of occasional high production figures. Annual precipitation was highest in Bangladesh, averaging 2185.55 mm, followed by Vietnam (1769.98 mm), India (1114.37 mm), and China (610.70 mm). The distribution of precipitation data was nearly symmetric in all countries, with minimal skewness, reflecting stable precipitation patterns. However, Bangladesh showed the highest variability in precipitation, as indicated by a standard deviation of 275.91 mm, whereas China exhibited the lowest variability (32.53 mm). Temperature variables (annual minimum, mean, and maximum temperatures) presented distinct climatic profiles across the countries. Bangladesh and Vietnam experience the

highest temperatures, with mean temperatures of 25.71˚C and 24.80˚C, respectively, whereas China has the lowest mean temperature at 7.59˚C. The temperature distributions across all countries were generally near-normal, with skewness values close to zero, indicating stable and consistent temperature trends. CO_2 emissions are significantly higher in China, with a mean of 4.66 metric tons per capita, compared with India (1.13 MT), Vietnam (1.32 MT), and Bangladesh (0.28 MT). The distribution of CO2 emissions was slightly positively skewed in all countries, with China showing modest skewness (0.15) and lower kurtosis (1.34), suggesting a relatively normal distribution with occasional periods of higher emissions. Vietnam exhibited the highest variability in emissions (standard deviation of 0.96 MT), whereas Bangladesh showed the least variability (0.15 MT). Fig. 1 (a, b, c, d) reveals a consistent upward trajectory in total fish production and CO_2 emissions in China, India, Vietnam, and Bangladesh from 1990 to 2020. Temperature and precipitation trends exhibited considerable variability, with distinct fluctuations in each country, reflecting the complex interplay between climatic conditions and fish production over time.

Unit Root Tests

The results of the unit root tests shown in Tables 3a and 3b for China, India, Vietnam, and Bangladesh indicate that the variables under study predominantly exhibit stationarity at the first difference, as evidenced by both the Phillips-Perron (PP) and Augmented Dickey-Fuller (ADF) tests. Specifically, for China and India, all variables, including total fish production (TFP), precipitation, temperature-related variables, and CO_2 emissions, are non-stationary at the level but become stationary after differencing, implying an order of integration of I(1). In Vietnam and Bangladesh, most variables also follow a similar pattern, with the exception of minimum temperature and precipitation variables that exhibit stationarity at both levels and the first difference, indicating that they are integrated of order I(0) or I(1). The stationarity of these variables at mixed levels of integration, I(0) and I(1), makes it suitable for the ARDL approach. Furthermore, it allows for a comprehensive analysis of the long-run relationships between fish production and climate indicators in these countries.

Co-integration Testing

The ARDL bounds test was employed to confirm the existence of a long-run relationship between total fish production and the selected climatic factors. The results are presented in Table 4 for the four countries. For China and Bangladesh, the F-statistic values of 6.99 and 6.50, respectively, exceed the upper critical bounds at both the 5% and 1% significance levels, indicating the existence of a long-run relationship or cointegration among the variables. In India, the F-statistic of 5.46 surpasses the upper bound at the 5% significance level, further supporting the presence of a long-term relationship. However, in Vietnam, the F-statistic of 1.26 falls below the lower critical bound, suggesting the absence of a long-run relationship or cointegration among the variables in this case. These findings indicate long-run relationships between total fish production (lnTFP) and precipitation (lnPREC), minimum temperature (lnMINTEM), mean temperature (lnMEANTEM), maximum temperature (lnMAXTEM), and CO_2 emission ($lnCO_2$) in China, India, and Bangladesh, while Vietnam shows no evidence of cointegration. Fig. 2 (a, b, c, d) shows the model selection process based on the Akaike Information Criterion (AIC). The optimal ARDL model for each country was determined by identifying the model with the lowest Akaike information criterion (AIC) value. Specifically, the selected models are ARDL (1, 2, 1, 1, 1, 2) for China, ARDL (1, 0, 0, 0, 0, 0) for India, ARDL (1, 0, 1, 0, 2, 0) for Vietnam, and ARDL (1, 2, 2, 2, 2, 2) for Bangladesh.

ARDL Long-Run and Short-Run Estimation

After confirming cointegration among the variables, the ARDL model was employed to assess the long- and short-run impacts of climatic variables on total fish production across each country.

China: Table 5 highlights the long-run and short-run relationships between climatic variables and total fish production in China over time. In contrast to the mean temperature, we observed a significant positive long-run impact of precipitation, minimum temperature, and CO_2 emissions on fish production. Specifically, the long-run coefficient for precipitation (2.44) was highly significant ($p < 0.001$), indicating a strong positive influence on fish production. The relationship between CO_2 emissions and fish production was positive, with a coefficient of 0.45, and was significant at the 1% level ($p < 0.001$). The minimum temperature also showed a positive effect (1.38, $p = 0.063$), although it was marginally significant at the 10% level. Meanwhile, the long-run coefficients for mean and maximum temperatures are not statistically significant, with the former showing a negative effect (-13.12, $p = 0.139$) and the latter showing a positive effect (11.36, $p = 0.241$). The stability of the long-run coefficients is assessed using short-run dynamics. This analysis involved estimating an error correction model (ECM) in conjunction with long-run estimates. The error correction term (ECT) represents the speed at which the regressand, total fish production, returns to its long-run equilibrium after a change in regressors. The speed of adjustment in this case is 0.24, indicating a 24%

correction towards equilibrium within a period. In the short run, total fish production was significantly influenced by current precipitation, which had a positive impact with a coefficient of 0.26 ($p < 0.001$), and lagged CO_2 emissions, which also showed a significant positive effect with a coefficient of 0.16 ($p = 0.010$). In contrast, immediate CO2 emissions, as well as minimum, mean, and maximum temperatures, did not have statistically significant effects on fish production in the short run, as indicated by their high p-values ($P > 0.05$).

India: The ARDL model results (Table 6) revealed that in the long run, only CO_2 emissions had a significant and positive impact on total fish production, with a coefficient of 1.08 ($p < 0.001$), whereas precipitation and temperature variables (minimum, mean, and maximum temperatures) did not exhibit significant relationships with fish production. Precipitation showed a coefficient of 0.09 ($p = 0.833$), whereas the minimum, mean, and maximum temperatures presented coefficients of 26.30, -42.97, and 10.35, respectively, with corresponding high p-values (0.777, 0.856,

Fig. 1a: Trends in fish production and climate indicators for China during 1990–2020.

Fig. 1b: Trends in fish production and climate indicators for India during 1990–2020.

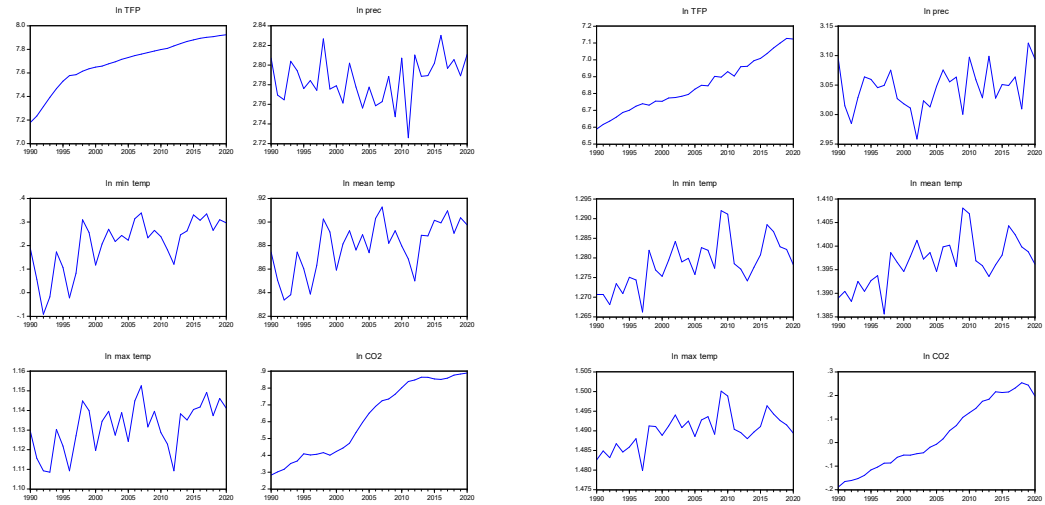

Fig. 1c: Trends in fish production and climate indicators for Vietnam during 1990–2020.

Fig. 1d: Trends in fish production and climate indicators for Bangladesh during 1990–2020.

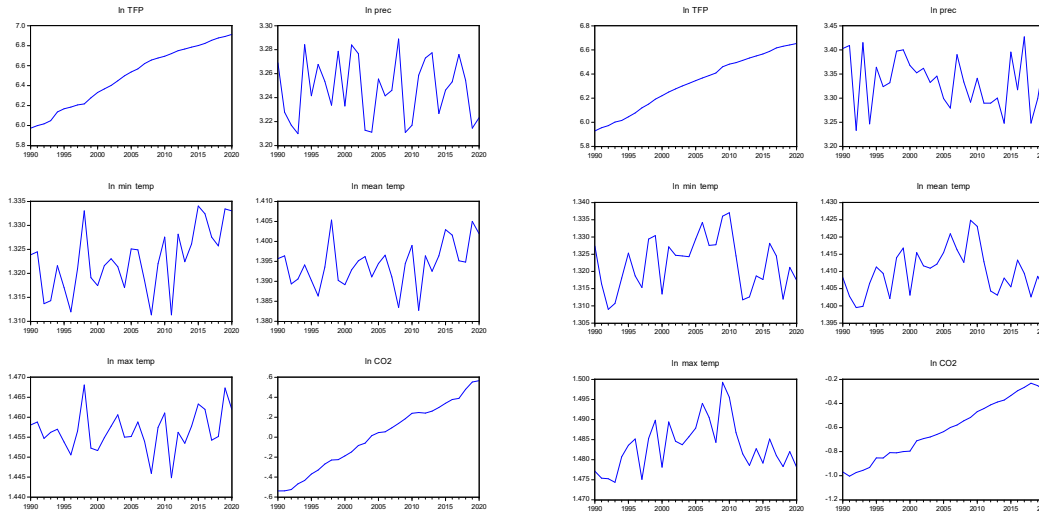

Fig. 1: Trends in fish production and climate indicators for different countries.

Fig. 2a: AIC model selection for China.

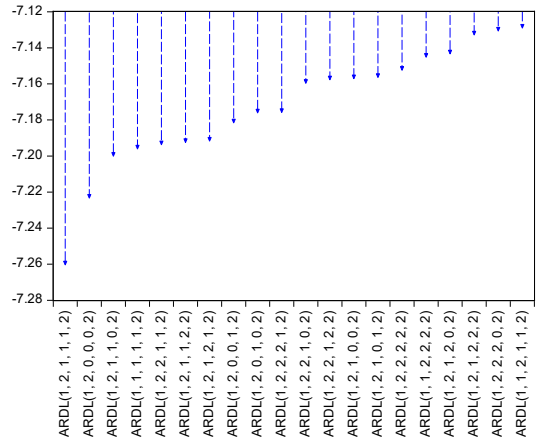

Fig. 2b: AIC model selection for India.

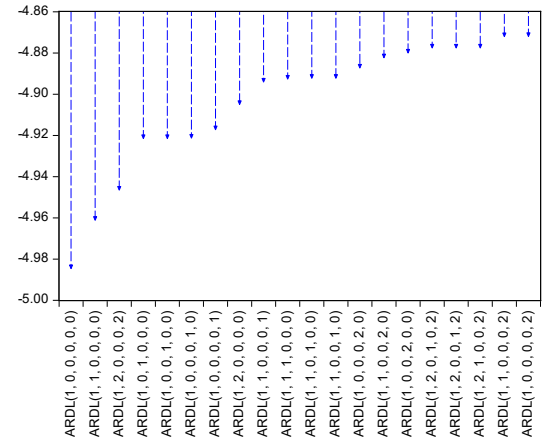

Fig. 2c: AIC model selection for Vietnam.

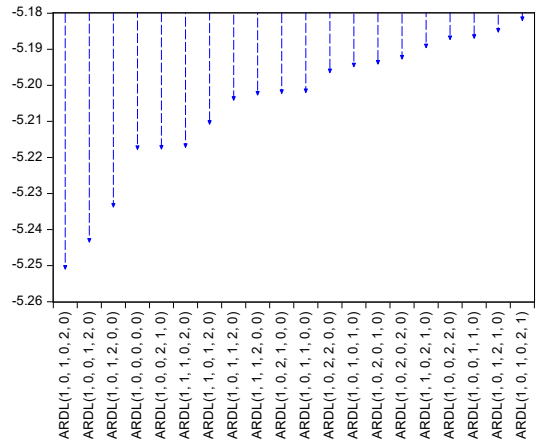

Fig. 2d: AIC model selection for Bangladesh.

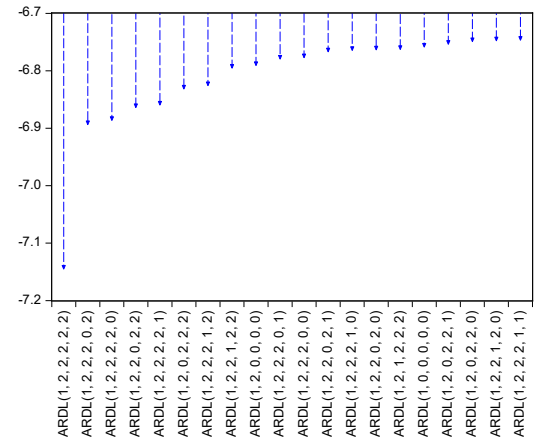

Fig. 2: AIC model selection for different countries.

and 0.944, respectively), indicating a lack of significant impact. After long-run cointegration was established, the short-run dynamics among the variables were subsequently calculated. The climatic variables, including precipitation, minimum temperature, mean temperature, maximum temperature, and CO_2 emissions, exhibited a lag of 0, indicating that changes in these variables do not immediately affect fish production in the short term. The error correction term (ECT) of -0.24 suggests that approximately 24% of any deviation from the long-run equilibrium is adjusted in each period. This implies that fish production is more influenced by long-term climate patterns than by immediate fluctuations.

Vietnam: The ARDL model, as indicated by the F-statistic from the bounds test, suggests that there is no long-run equilibrium in Vietnam, implying that only short-run relationships exist. The results of the short-run coefficients are shown in Table 7. Precipitation, mean temperature, maximum temperature, and CO_2 emissions do not exhibit significant relationships with total fish production in the short run, as reflected by their high p-values. However, the first lag of the minimum and the first lag of maximum temperatures affected the total fish production at the 10% significance level. The outcome of short-run coefficients revealed that a 1% increase in minimum temperature leads to a 2.19% decrease in total fish production, whereas a 1% increase in maximum temperature leads to a 3.02% increase in total fish production. Although precipitation and mean temperature were positively correlated with fish production and CO2 emissions were negatively correlated, these effects were not statistically significant ($P > 0.05$).

Bangladesh: In the long run, CO_2 emissions exerted a strong positive influence on fish production, with a coefficient of

Fig. 3a: Plot of CUSUM test for China.

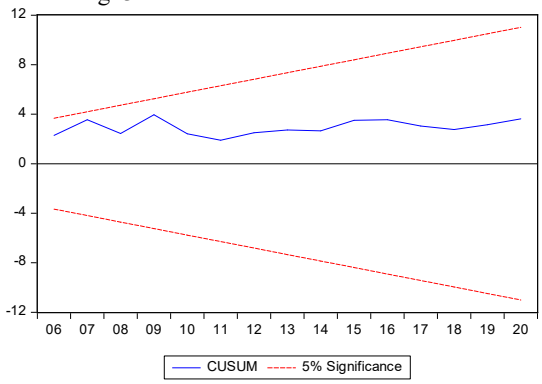

Fig. 3b: Plot of CUSUM test for India.

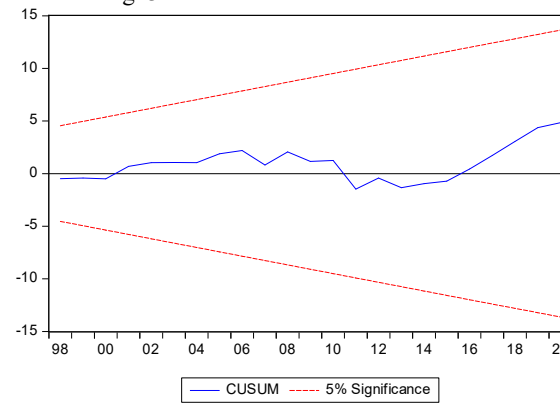

Fig. 3c: Plot of CUSUM test for Vietnam.

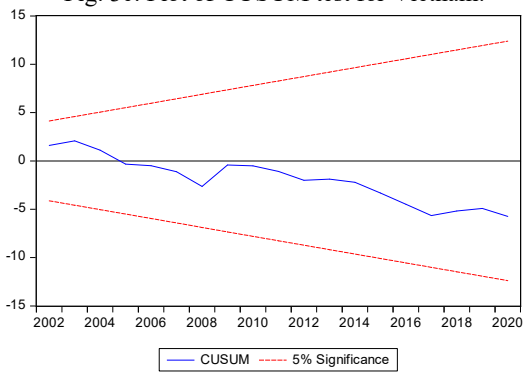

Fig. 3d: Plot of CUSUM test for Bangladesh.

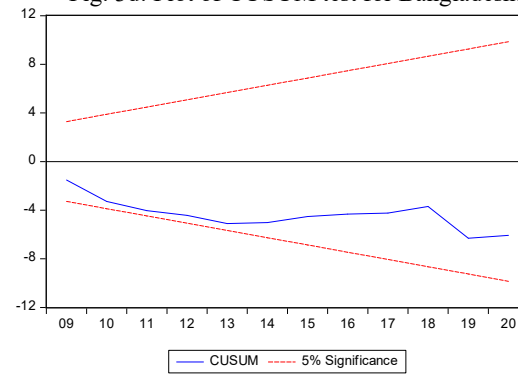

Fig. 3: Plot of the CUSUM test for different countries.

0.87 ($p < 0.001$), while precipitation also showed a significant positive impact, with a coefficient of 1.39 ($p = 0.001$) (Table 8). Temperature variables showed mixed results, with the minimum temperature showing a negative effect at -158.96 ($p = 0.083$), indicating marginal significance, whereas the mean and maximum temperatures did not exhibit a statistically significant impact on total fish production. In the short run, precipitation continues to play a critical role, with its immediate effect showing a significant positive coefficient of 0.14 ($p < 0.001$). However, the lagged effect of precipitation was negative and significant, with a coefficient of -0.12 ($p = 0.001$), indicating that an increase in precipitation may have a delayed adverse impact on total fish production. Furthermore, the first lags of the minimum and maximum temperatures were also significant, with coefficients of 27.14 ($p = 0.001$) and 35.26 ($p = 0.002$), respectively, suggesting that past temperature variations influence current production levels. Meanwhile, the immediate effect of CO_2 emissions was not statistically significant (0.03, $p = 0.576$), and the lagged effect was significant and negative, with a coefficient of -0.23 ($p = 0.002$), indicating that previous increases in CO_2 emissions may lead to a reduction in fish production over time.

In addition, the ECM coefficient is -0.31 and is significant at the 1% level. This suggests that deviations from equilibrium in the short run were adjusted at a rate of approximately 31% annually, progressively aligning towards the long-run equilibrium.

Diagnostic Inspection

The diagnostic tests for the model, as shown in Table 9, revealed no significant issues of serial correlation, heteroskedasticity, or non-normality of residuals across the different countries under study. The Breusch-Godfrey Serial correlation LM test indicates no significant evidence of serial correlation in the residuals, with F-statistics of 0.23 ($p = 0.79$) for China, 1.32 ($p = 0.36$) for India, 2.62 ($p = 0.12$) for Bangladesh, and 1.63 ($p = 0.22$) for Vietnam. The Breusch-Pagan-Godfrey Heteroskedasticity Test further supports the model's validity by showing no significant presence of heteroskedasticity, as evidenced by the p-values for all countries, which are greater than 0.05. Additionally, the Jarque-Bera Normality test confirms that the residuals are normally distributed, with χ^2 values of 0.25 ($p = 0.87$) for

China, 0.78 (p = 0.67) for India, 0.55 (p = 0.75) for Bangladesh, and 0.31 (p = 0.85) for Vietnam. Furthermore, the stability of the model was assessed using the cumulative sum of recursive residuals (CUSUM) test. As illustrated in Figs. 3 (a, b, c, d), the trajectories of total fish production remain within the 5% significance level throughout the period, thereby validating the stability of the ARDL model in all the countries studied.

DISCUSSION

Globally, climate change affects marine and freshwater fish species by shifting their distribution and altering their habitats, which in turn reduces their productivity (Allan et al. 2005, FAO 2024). Tropical ecosystems, particularly in Asia, are particularly vulnerable to these changes (Pörtner et al. 2014). This study examined the impact of climate change on fisheries in the top fish-producing countries.

China

Based on this study, precipitation has a long-term impact on the total fish production in China. We observed that a

Table 1: Variable description and data sources. Source: Author's collection from various databases.

	Variable	Code	Measurement Unit	Source
Dependent Variable	Total Fish Production	TFP	Million tons	FishStatJ, FAO
Independent Variables	Annual Precipitation	PREC	Millimeter	CCKP
	Annual Minimum Temperature	MINTEM	Degree Celcius	CCKP
	Annual Mean Temperature	MEANTEM	Degree Celcius	CCKP
	Annual Maximum Temperature	MAXTEM	Degree Celcius	CCKP
	CO_2 emission	CO_2	Metric Tons per capita	WDI

Table 2: Descriptive Statistics.

Country	Variables	Mean	Median	Maximum	Minimum	Std. Dev.	Skewness	Kurtosis
China	Total Fish production	53.19	53.79	83.93	15.11	20.14	-0.20	2.08
	Annual Precipitation	610.70	614.41	676.47	531.98	32.53	-0.14	2.84
	Annual Minimum Temperature	1.65	1.73	2.18	0.81	0.38	-0.59	2.47
	Annual Mean Temperature	7.59	7.62	8.18	6.82	0.37	-0.49	2.32
	Annual Maximum Temperature	13.54	13.63	14.21	12.84	0.39	-0.41	2.24
	CO_2 emission	4.66	4.47	7.76	1.91	2.18	0.15	1.34
India	Total Fish production	7.48	6.70	13.41	3.88	2.77	0.76	2.51
	Annual Precipitation	1114.37	1120.49	1322.44	908.97	93.05	0.12	2.74
	Annual Minimum Temperature	18.99	18.98	19.59	18.46	0.27	0.28	2.84
	Annual Mean Temperature	24.92	24.92	25.59	24.30	0.29	0.16	3.04
	Annual Maximum Temperature	30.91	30.93	31.63	30.19	0.32	0.03	3.20
	CO_2 emission	1.13	0.98	1.80	0.65	0.38	0.40	1.64
Vietnam	Total Fish production	3.83	3.44	8.19	0.94	2.32	0.37	1.80
	Annual Precipitation	1769.98	1762.83	1945.05	1621.31	104.95	0.07	1.67
	Annual Minimum Temperature	21.03	21.01	21.58	20.48	0.32	0.04	2.23
	Annual Mean Temperature	24.80	24.80	25.43	24.14	0.31	0.08	2.94
	Annual Maximum Temperature	28.62	28.59	29.38	27.85	0.34	0.09	3.58
	CO_2 emission	1.32	1.11	3.68	0.29	0.96	0.95	3.09
Bangladesh	Total Fish production	2.40	2.22	4.50	0.85	1.17	0.32	1.82
	Annual Precipitation	2185.55	2156.07	2674.16	1710.40	275.91	0.01	1.92
	Annual Minimum Temperature	21.01	21.11	21.73	20.37	0.37	0.04	2.11
	Annual Mean Temperature	25.71	25.67	26.60	25.09	0.39	0.43	2.62
	Annual Maximum Temperature	30.45	30.45	31.57	29.81	0.44	0.66	3.04
	CO_2 emission	0.28	0.23	0.59	0.10	0.15	0.59	2.03

Table 3a: Unit Root Tests.

		PP			ADF		
		At level	1st Diff	Implied order of integration	At level	1st Diff	Implied order of integration
China	LN_TFP	3.07	-1.68*	I (1)	1.41	-1.67*	I (1)
	LN_PREC	0.06	-30.69***	I (1)	0.20	-12.66***	I (1)
	LN_MIN_TEMP	-0.81	-7.30***	I (1)	0.39	-8.59***	I (1)
	LN_MEAN_TEMP	0.69	-9.60***	I (1)	0.14	-6.55***	I (1)
	LN_MAX_TEMP	0.69	-12.61***	I (1)	0.13	-7.44***	I (1)
	LN_CO2	2.46	-1.84*	I (1)	1.20	-1.90*	I (1)
India	LN_TFP	11.25	-4.50***	I (1)	6.82	-9.79***	I (1)
	LN_PREC	-0.02	-9.82***	I (1)	0.40	-9.19***	I (1)
	LN_MIN_TEMP	0.40	-10.69***	I (1)	0.22	-7.13***	I (1)
	LN_MEAN_TEMP	0.39	-9.81***	I (1)	0.26	-7.48***	I (1)
	LN_MAX_TEMP	0.28	-8.70***	I (1)	0.28	-7.83***	I (1)
	LN_CO2	-0.62	-2.03**	I (1)	-0.98	-2.03**	I (1)

Table 3b: Unit Root Tests.

		PP			ADF		
		At level	1st Diff	Implied order of integration	At level	1st Diff	Implied order of integration
Vietnam	LN_TFP	-0.22	-4.44***	I (1)	-0.77	-4.37***	I (1)
	LN_PREC	-6.81***	-21.29***	I (0), I (1)	-4.69***	-5.38***	I (0), I (1)
	LN_MIN_TEMP	-4.92***	-22.32***	I (0), I (1)	-4.92***	-5.93***	I (0), I (1)
	LN_MEAN_TEMP	-4.99***	-18.75***	I (0), I (1)	-5.00***	-8.03***	I (0), I (1)
	LN_MAX_TEMP	-5.04***	-17.90***	I (0), I (1)	-5.19***	-7.88***	I (0), I (1)
	LN_CO_2	-2.30	-5.32***	I (1)	-2.30	-4.92***	I (1)
Bangladesh	LN_TFP	0.04	-4.28***	I (1)	0.24	-4.35***	I (1)
	LN_PREC	-7.19***	-19.28***	I (0), I (1)	-7.19***	-6.91***	I (0), I (1)
	LN_MIN_TEMP	-3.24*	-14.81***	I (0), I (1)	-3.24*	-6.50***	I (0), I (1)
	LN_MEAN_TEMP	-2.87	-16.82***	I (1)	-2.94	-6.46***	I (1)
	LN_MAX_TEMP	-2.94	-16.06***	I (1)	-3.00	-6.54***	I (1)
	LN_CO_2	-3.05	-5.37***	I (1)	-3.15	-5.30***	I (1)

Note: (*) Significant at the 10%, (**) Significant at the 5%, (***) Significant at the 1% level

Table 4: Bounds Cointegration Test.

Country	F-statistic Value	Significance level	Critical value	
			Lower bound	Upper bound
China	6.99	5%	2.62	3.79
		1%	3.41	4.68
India	5.46	5%	2.39	3.38
		1%	3.06	4.15
Vietnam	1.26	5%	2.62	3.79
		1%	3.41	4.68
Bangladesh	6.50	5%	2.62	3.79
		1%	3.41	4.68

Table 5: Results of long and short-run coefficients using the ARDL model for China.

Long run				
	Coefficient	Std. Error	t-Statistic	Prob.
LN_PREC	2.44	0.53	4.57	0.000
LN_MIN_TEMP	1.38	0.68	2.01	0.063
LN_MEAN_TEMP	-13.12	8.40	-1.56	0.139
LN_MAX_TEMP	11.36	9.30	1.22	0.241
LN_CO_2	0.45	0.04	10.35	0.000
Short run				
	Coefficient	Std. Error	t-Statistic	Prob.
C	-0.21	0.01	-21.34	0.000
D(LN_PREC)	0.26	0.05	5.46	0.000
D(LN_PREC(-1))	-0.09	0.05	-2.03	0.061
D(LN_MIN_TEMP)	0.10	0.08	1.29	0.216
D(LN_MEAN_TEMP)	-0.80	0.82	-0.98	0.343
D(LN_MAX_TEMP)	0.61	0.83	0.74	0.469
D(LN_CO_2)	0.06	0.06	1.09	0.294
D(LN_CO_2 (-1))	0.16	0.06	2.97	0.010
ECM(-1)*	-0.24	0.01	-23.65	0.000
ARDL (1, 2, 1, 1, 1, 2)				

Table 6: Results of long and short-run coefficients using the ARDL model for India.

Long Run				
	Coefficient	Std. Error	t-Statistic	Prob.
LN_PREC	0.09	0.43	0.21	0.833
LN_MIN_TEMP	26.30	91.88	0.29	0.777
LN_MEAN_TEMP	-42.97	234.62	-0.18	0.856
LN_MAX_TEMP	10.35	144.33	0.07	0.944
LN_CO_2	1.08	0.13	8.07	0.000
C	17.56	11.28	1.56	0.133
Short run				
	Coefficient	Std. Error	t-Statistic	Prob.
CointEq(-1)*	-0.24884	0.035857	-6.93995	0
ARDL (1, 0, 0, 0, 0, 0)				

Table 7: Results of Short run ARDL model for Vietnam.

Short run				
	Coefficient	Std. Error	t-Statistic	Prob.*
LN_TFP(-1)	1.05	0.12	8.40	0.000
LN_PREC	0.30	0.23	1.34	0.197
LN_MIN_TEMP	-3.63	22.29	-0.16	0.872
LN_MIN_TEMP(-1)	-2.19	1.21	-1.80	0.088
LN_MEAN_TEMP	6.01	54.62	0.11	0.914
LN_MAX_TEMP	-1.83	32.68	-0.06	0.956
LN_MAX_TEMP(-1)	3.03	1.55	1.96	0.065
LN_MAX_TEMP(-2)	1.34	0.84	1.59	0.128
LN_CO_2	-0.04	0.11	-0.35	0.732
C	-5.62	4.39	-1.28	0.216
ARDL (1, 0, 1, 0, 2, 0)				

Table 8: Results of long and short-run coefficients using the ARDL model for Bangladesh.

Long run				
	Coefficient	Std. Error	t-Statistic	Prob.
LN_PREC	1.39	0.33	4.15	0.001
LN_MIN_TEMP	-158.96	84.01	-1.89	0.083
LN_MEAN_TEMP	351.15	201.62	1.74	0.107
LN_MAX_TEMP	-183.29	117.26	-1.56	0.144
LN_CO_2	0.87	0.02	40.16	0.000
Short run				
	Coefficient	Std. Error	t-Statistic	Prob.
C	-3.34	0.45	-7.39	0.000
D(LN_PREC)	0.14	0.03	5.17	0.000
D(LN_PREC(-1))	-0.12	0.03	-4.57	0.001
D(LN_MIN_TEMP)	-8.26	6.14	-1.35	0.203
D(LN_MIN_TEMP(-1))	27.14	6.07	4.48	0.001
D(LN_MEAN_TEMP)	16.60	14.75	1.13	0.282
D(LN_MEAN_TEMP(-1))	-62.85	14.70	-4.27	0.001
D(LN_MAX_TEMP)	-6.60	8.57	-0.77	0.456
D(LN_MAX_TEMP(-1))	35.26	8.61	4.09	0.002
D(LN_CO_2)	0.03	0.05	0.57	0.576
D(LN_CO_2 (-1))	-0.23	0.06	-3.99	0.002
CointEq(-1)*	-0.31	0.04	-7.43	0.000
ARDL (1, 2, 2, 2, 2, 2)				

Table 9: Diagnostic tests of the model.

	China		India				Bangladesh	
	F - Statistics	P - value	F - Statistics	P - value	Vietnam	P - value	F - Statistics	P - value
Breusch-Godfrey Serial Correlation LM Test	0.23	0.79	1.32	0.36	1.63	0.22	2.62	0.12
Heteroskedasticity Test: Breusch-Pagan-Godfrey	1.01	0.47	1.33	0.28	1.78	0.13	0.67	0.77
Normality test: Jarque-Bera	0.25	0.87	0.78	0.67	0.31	0.85	0.55	0.75

1% increase in annual precipitation could increase total fish production by 2.44%. Several studies have provided insights into these relationships. For instance, Holst and Yu (2010) found that precipitation positively impacts aquaculture outputs, whereas a 1°C increase in annual average temperature was associated with a rise in national mean output of 1.47 million tons. Similarly, Meynecke et al. (2006) identified a significant positive correlation between annual rainfall and total fish production in Australia, highlighting the importance of precipitation for fishery yields. The study indicated seasonality with a trend in annual temperature over time, ranging from 1.6°C to 13.5°C. China's seas have generally warmed over the past few decades, with the East China Sea experiencing the most significant rise and the South China Sea the least, leading to varying impacts on fisheries (Belkin 2009, Liang et al. 2018). Consistent with these findings, our analysis demonstrated that mean temperature negatively impacts fish production, suggesting that rising temperatures may pose challenges to sustaining fisheries in the region. China's national climate commitment, which aims for carbon neutrality by 2060 and an emissions peak by 2030, highlights the significance of understanding the impact of climatic factors on fishery outputs. Chandio et al. (2020) revealed that CO_2 emissions have a significant positive effect on agricultural output in China in both long-run and short-run analyses. However, they also found that temperature exerts a negative effect on agricultural output in the long run, suggesting that the benefits of CO2 fertilization

may be offset by the adverse impacts of rising temperatures. This complexity is echoed in Janjua et al. (2014), who reported that CO_2 and precipitation positively influenced wheat production in Pakistan over the long run, reinforcing the notion that different climatic variables can have varied effects on agricultural productivity depending on the context and timescale.

India

India is increasingly grappling with the impacts of climate change, which have intensified in both frequency and severity, affecting its natural environment, economy, and society (Mall et al. 2011, Kushawaha et al. 2021, Picciariello et al. 2021). The country is facing a range of extreme climate-related challenges, including heatwaves, floods, unpredictable monsoons, and declining groundwater reserves (Misra 2013, Dhara & Kolls 2021, Charak et al. 2024). Ranked as the 7th most affected nation by climate change according to the Global Climate Risk Index 2021 (Eckstein et al. 2021). India has committed to achieving net-zero emissions by 2070 and has made notable progress in decoupling its economic growth from emissions. According to the (IPCC 2022) report, India maintains a relatively low level of emissions per capita compared to other major global economies, demonstrating its commitment to sustainable development. Our study revealed a significant positive impact of CO_2 emissions on long-term fish production. CO_2 may enhance primary productivity by promoting the growth of aquatic vegetation and phytoplankton, which are key components of the food web, and the short-term effects appear less pronounced (Geider et al. 2001, Tremblay et al. 2015). These findings are consistent with the trends observed in the agricultural sector, where CO_2 emissions have also been found to positively influence long-term productivity. Ahmed & Saha (2023) reported a positive association between per capita CO_2 emissions and agricultural GDP in India over the long term, although no significant short-term effect was detected. This parallel between fisheries and agriculture highlights the complex nature of CO_2's role in enhancing productivity over extended periods. Moreover, climate change-induced physical changes, such as increased water temperature and altered dissolved oxygen levels, have been linked to a higher risk of disease outbreaks in aquatic systems (Harvell et al. 2002, Vilchis et al. 2005). These changes, driven by warming waters, could further exacerbate the challenges faced by India's fisheries and aquaculture sectors, which are critical to food security and livelihoods.

Vietnam

The results from the ARDL model for Vietnam reveal that no long-run equilibrium exists between climatic variables and total fish production, suggesting that only short-run relationships are significant. This aligns with the findings of Pham (2012), who similarly reported no significant relationship between temperature and shrimp productivity across multiple ecological regions in Vietnam. However, regional differences were noted, with temperature affecting shrimp production in the North-Central Coastal region, whereas rainfall had no notable impact. Cao et al. (2013) identified an inverse correlation between temperature and shrimp production, further supporting the complex relationship between climate and fisheries in Vietnam. The absence of long-run effects could be attributed to Vietnam's vulnerability to extreme climate events, which disrupt the consistency of the relationships between climatic variables and fish production. Vietnam is among the nations most severely impacted by climate change, as reported by the Ministry of Natural Resources and Environment (MoNRE, 2016). Frequent and extreme climate occurrences, such as typhoons, floods, and rising sea levels, have a profound effect on the fisheries sector, disrupting long-term trends and making it difficult to establish stable and long-run relationships between climate factors and production outputs. This unpredictable nature of extreme climate events may obscure long-run trends, as fish production systems adapt to short-term fluctuations rather than establishing long-term equilibrium. In response to these challenges, the Vietnamese government has implemented meticulously designed policies aimed at mitigating the impact of climate change. Vietnam's comprehensive strategies, such as the National Target Program to Respond to Climate Change (NTP-RCC), Vietnam Green Growth Strategy (VGGS), Law on Environmental Protection (2020), and National Action Plan on Climate Change (NAPCC), have been instrumental in mitigating the impacts of climate change on various sectors, including fisheries. These policies align with Vietnam's commitment to the Sustainable Development Goals (SDGs), which emphasize responsible production practices, protection of coastal and marine ecosystems, and climate resilience (Ministry of Planning and Investment 2018). Wilbanks (2003) highlights that climate change serves as both a challenge and a catalyst for advancing sustainable development, and this duality is evident in Vietnam's proactive approach. Through carefully designed government interventions, the country is managing the short-term impacts of climate change and helping to maintain the resilience of its fisheries sector.

Bangladesh

The ARDL model results for this country highlight a relationship between climatic variables and total fish production, with significant impacts observed in both the

short and long runs. Bangladesh ranked 7th in long-term climate vulnerability from 2000 to 2019 (Global Carbon Atlas, 2022), reflecting its susceptibility to climate change, which appears to influence the fisheries sector in both time frames. The long-run analysis showed that CO_2 emissions have a strong positive influence on fish production, which is explained by the rise in per capita carbon emissions from 0.107 metric tons in 1990 to 0.510 metric tons in 2020. Although Bangladesh ranks 39th in per capita CO_2 emissions globally (Global Carbon Atlas 2022), its relatively low industrial greenhouse gas (GHG) emissions (Islam et al. 2020) suggest that this positive long-run association could be linked to the ecological dynamics of CO_2, enhancing primary productivity. This is consistent with the findings of Begum et al. (2022), who also reported a positive relationship between CO_2 emissions and marine fish production in Bangladesh over the long term. Precipitation also had a significant positive impact on fish production in both the long and short runs, reinforcing the notion that rainfall plays a vital role in Bangladesh's fisheries sector. Begum et al. (2022) found that a 1% increase in average rainfall could increase marine fish production by 1.65%, a result consistent with our study where precipitation had a sustained positive influence on fish output. This relationship aligns with research showing that increased rainfall often coincides with upwelling events that bring nutrient-rich waters to the surface, significantly boosting fish catch (Atindana et al. 2019). Similar positive associations between rainfall and fish productivity have been observed in other regions, such as Malaysia (Madihah Jafar-Sidik et al. 2010) and Pakistan (Ayub 2010), further substantiating the critical role of precipitation in driving marine fish production. The short-run results reveal a more nuanced picture. While precipitation continues to be a significant factor, its lagged effect turns negative, suggesting that excessive rainfall may initially benefit fish production but may have adverse delayed effects. This phenomenon may be attributed to ecological disruptions, such as the increased risk of undesirable phytoplankton blooms during periods of high atmospheric CO_2, which can negatively affect marine ecosystems (Schippers et al. 2004). Temperature variables also had significant short-run effects on fish production. Ho et al. (2013) observed that fish landings increase with rising temperatures. However, as temperatures continue to rise, increased stratification is expected to restrict the flow of nutrients to the surface, potentially reducing productivity (Kay et al. 2018). Bangladesh's proactive climate policies, including the Bangladesh Climate Change Strategy and Action Plan (BCCSAP 2009), the National Adaptation Program of Action (NAPA 2005), and the National Fisheries Policy (NFP), reflect the country's commitment to addressing the multifaceted challenges posed by climate change. These frameworks, alongside the Bangladesh Delta Plan 2100, aim to mitigate the long-term impacts of climate change on the fisheries sector, ensuring sustainability amidst increasing climate variability.

CONCLUSIONS

This study provides a fresh perspective on the impact of climate change on fisheries in four leading fish-producing countries: China, India, Vietnam, and Bangladesh. The results reveal long-run associations for all countries except Vietnam, where only short-run relationships are identified. In China and Bangladesh, precipitation and CO_2 emissions exhibited significant positive long-run impacts on fish production, highlighting the role of favorable weather patterns and carbon availability in supporting the aquatic ecosystem. The results for India, however, showed that only CO_2 emissions have a significant long-run effect, whereas temperature and precipitation do not display any significant impacts. In Vietnam, the absence of a long-run equilibrium suggests that fish production is influenced only by short-term climatic changes, particularly the lagged effects of the minimum and maximum temperatures. Diagnostic tests confirmed the robustness of the model, with no evidence of serial correlation, heteroskedasticity, or non-normality in the residuals. The policy implications of this study are significant. In China, promoting green aquaculture and reducing emissions through innovative technologies can enhance resilience to climate change effects. India should focus on low-carbon fishing practices and adaptive aquaculture systems to mitigate the adverse effects of rising temperatures, while improving water resource management to benefit from precipitation changes. Vietnam requires enhanced flood management strategies and the development of climate-resilient aquaculture to cope with precipitation variability. Given its vulnerability to climate-induced floods, Bangladesh must prioritize sustainable water practices and species diversification to safeguard fish production. Each country should integrate climate resilience into fisheries policies to ensure sustainable development and food security and mitigate the risks posed by rising temperatures and shifting precipitation patterns.

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Enhancing S.I. Engine Performance with Metal-Doped Zeolite X Derived from Rice Husk

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ABSTRACT

The increasing prevalence of harmful NOx emissions from gasoline engines necessitates the development of alternatives to traditional three-way catalytic converters. This study investigates the potential of low-cost zeolites derived from rice husk ash as a promising alternative. Na-X zeolites derived from rice husk ash were characterized using X-ray fluorescence (XRF), Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and field emission scanning electron microscopy (FESEM) to determine their structural properties and morphology. These zeolites were then modified with copper (Cu) and iron (Fe) ions to create Cu-X and Fe-X zeolite catalysts. The catalysts were wash-coated onto cordierite honeycomb monoliths and integrated and properly housed in a steel casing catalytic converter and fitted near the exhaust manifold of a twin-cylinder nano-engine. Engine performance and emissions were evaluated under various operating loads (4, 7, 10, 13, and 16 kW). NOx, CO, HC, CO_2, and O_2 emissions were measured using an AVL DI gas analyzer. The results demonstrated that the developed Cu-X and Fe-X zeolite converters significantly outperformed the conventional catalytic converter in reducing NOx emissions, while maintaining comparable performance for other pollutants. The enhanced NOx reduction capabilities of the zeolite catalysts can be attributed to their unique structural properties and the synergistic effect of the copper and iron ions.

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INTRODUCTION

Zeolites, synthetic crystalline aluminosilicates, have become indispensable across various industries due to their unique structural properties and remarkable performance. Zeolite X, in particular, features a three-dimensional framework of interconnected tetrahedral units composed primarily of silicon (Si) and aluminum (Al) atoms. These tetrahedra are linked by oxygen atoms (O), forming a network of channels and cavities that define the material's high surface area and ion-exchange capabilities.

The discovery of zeolites dates back to 1756 when Cronstedt identified stilbite, the first known zeolite, after observing its rapid water loss upon heating, resembling boiling. The term "zeolite" originates from the Greek words zeo (to boil) and lithos (stone) (Edanol et al. 2018). Zeolites typically follow the empirical formula $M_2/Al_2O_3{\cdot}xSiO_2{\cdot}yH_2O$, where M represents an exchangeable cation with valence n. Commonly, M includes Group I or II ions, though other metal, non-metal, and organic cations may also be used to balance the negative charge introduced by aluminum in the structure (Saceda et al. 2011).

Rice husk, an abundant agricultural byproduct, consists mainly of the husk along with rice straw, plant matter, and other organic components. Significantly, rice husk contains over 90% silica, making it a cost-effective and plentiful source of high-quality amorphous silica. Extensive research has been devoted to extracting silica from rice husk for various applications, including the production of nano-

silica (Ma et al. 2014). Silica's versatility enables its use in a wide range of industries, such as ceramics, chromatography, adhesives, detergents, electronics, pharmaceuticals, and vegetable oil processing. Rice husk ash (RHA), produced by burning rice husk, has gained attention as a low-cost raw material for synthesizing silica powders and gels (Das et al. 2022). However, silica extracted from RHA may contain impurities like CaO, Al_2O_3, and MgO in addition to SiO_2. These impurities must be considered and, if necessary, removed depending on the intended application (Hassan & Jaaron 2021).

Several studies have focused on synthesizing zeolites from RHA-derived silica. Dalai et al. (2010) synthesized zeolites such as NaX, PC, analcime, and hydroxysodalite using standard procedures and reported evidence of new phase formation at elevated temperatures through XRD analysis. Yusof et al. (2010) successfully synthesized high-purity Zeolite NaY at a controlled temperature of 600˚C, utilizing seeding and aging processes to eliminate undesired phases. Saceda et al. (2011) compared silica extraction from both rice husk (RH) and RHA, concluding that RH is a more efficient source for Zeolite NaY synthesis due to simpler extraction and higher recovery. Silica from RH consisted solely of SiO_2, while that from RHA contained 97.56% SiO_2 along with 2.14% K_2O and 0.31% Al_2O_3. The resulting Zeolite NaY exhibited a high BET surface area of 882 $m^2.g^{-1}$ and a micropore volume of 0.38 $cm^3.g^{-1}$, typical of Y-type zeolite structures. Setthaya et al. (2013) synthesized faujasite and analcime zeolites using RHA and metakaolin through a dissolution-precipitation method followed by hydrothermal treatment. They found that faujasite formed at lower hydrothermal temperatures, whereas analcime developed under higher temperatures and prolonged synthesis durations. They concluded that time and temperature affect phase transitions and crystal development, while stirring duration impacts specific surface area but not zeolite type. Santasnachok et al. (2014) synthesized pure Na-A and Na-X zeolites using alkali fusion followed by extraction, residue removal, and hydrothermal treatment. Zeolite A was synthesized at 90˚C for 10 to 24 h using Si/Al molar ratios of 0.5 to 1.0. NaX zeolite was produced under the same conditions with Si/Al ratios ranging from 1.5 to 2.0. Both zeolites were tested for cadmium ion removal, with Na-A proving more effective, reducing Cd^{2+} concentrations below the WHO limit of 0.01 $mg.L^{-1}$. Pandiangan et al. (2017) reported successful synthesis of zeolite X from rice husk silica and aluminum, confirmed by Fourier-transform infrared (FTIR), X-ray diffraction (XRD), and Field emission Scanning Electron Microscopy (FESEM) analysis. Furthermore, transesterification experiments verified by GC-MS demonstrated the catalytic activity of zeolite X in converting palm oil into biodiesel.

Substantial research has explored the influence of varying hydrothermal conditions on zeolite synthesis (Saleh et al. 2018). The literature consistently supports alkali fusion combined with hydrothermal treatment as an efficient strategy for producing various zeolite types, such as X, Y, NaX, NaY, NaP, PC, analcime, and hydroxysodalite—from rice husk ash with different compositions. The central problem addressed in this study is the substantial emission of nitrogen oxides (NOx) from gasoline engines, which significantly contributes to air pollution and poses serious environmental and health risks. Traditional catalytic converters often show limited effectiveness under the oxygen-rich conditions typical of gasoline engine exhaust. Hence, there is a pressing need for more efficient and sustainable catalysts. The primary objective of this research is to develop novel zeolite-based catalysts synthesized from rice husk ash, an eco-friendly and low-cost silica source. These zeolites are further modified with copper and iron ions to improve their catalytic performance for NOx reduction. The study aims to assess the effectiveness of Cu-X and Fe-X catalysts under different engine operating conditions and compare their performance with conventional catalytic converters. Ultimately, this research aspires to contribute to cleaner emission control technologies for gasoline engines, supporting the advancement of more environmentally friendly transportation systems.

MATERIALS AND METHODS

The best raw material of rice husk (RH) used for the production of zeolites-x. Initially, the formation of zeolites-X from silica, therefore silica is produced by the acid treatment of RH during incineration. Here, the main precursor for the synthesis of the silica source is dissolved rice husk. Raw rice husks undergo a multi-step purification process to yield a refined ash product. Initially, the husks are sieved to remove impurities like clay and dust. Following this, they are steeped in a 10M concentrated hydrochloric acid solution. The treated husks are then thoroughly washed, filtered, and air-dried. The final step involves calcination at 850°C for 5 hrs. Initially, 50 g of silica and 20 of sodium hydroxide were dissolved in 500 mL of distilled water. Then the mixture was agitated with a magnetic stirrer. After a little while, the required amount of 10 g of sodium hydroxide and 10 g of aluminum oxide were both dissolved in separate beaker, in addition, the solution formed into sodium aluminate, and the pH value is 11, occur seems to have a bluish color. The mixture of sodium aluminate dissolved in silicate solution. Stir well until 4hrs around 250 rpm until the silica dissolves clearly.

Table 1: The Elemental Composition of Zeolite X.

METAL OXIDES	RRH %	IN-HOUSE MADE ZEOLITE X%
SiO_2	59.57	67.612
Al_2O_3	3.58	15.675
Na_2O	0.58	8.789
P_2O_5	18.85	1.789
K_2O	6.85	0.9
CaO	1.95	0.971
TiO_2	0.24	0.303
Fe_2O_3	0.14	4.476
MgO	4.58	0.00
Cl	0.58	0.00
SO_3	3.08	0.00
MoO_3	0.02	0.00

At last, the sample is obtained. Take it out and wash it several times until the impurities are removed. The obtained place in the oven at 100°C for 24 h. At last, the obtained zeolite X-like material.

XRF

The elemental composition of the raw rice husk (RRH) and the synthesized zeolite X was determined using X-ray fluorescence (XRF) spectroscopy. Table 1 revealed a significant increase in the silica (SiO_2) content from 59.57 wt.% in RRH to 67.612 wt.% in the final zeolite X product, indicating the successful enrichment of silica during the synthesis process. Silica (SiO_2) forms the primary framework of zeolite X, consisting of tetrahedrally coordinated silicon atoms linked by oxygen atoms to create a three-dimensional network. Alongside SiO_2, sodium oxide (Na_2O) and aluminium oxide (Al_2O_3) constitute the fundamental components of the zeolitic structure. Notably, Na_2O was present at 8.789 wt.% in the synthesized material, balancing the negative charge introduced by Al atoms within the framework. These sodium ions are located within the pores of the zeolite and are exchangeable with other cations such as K^+, Ca^{2+}, and Mg^{2+}, providing versatility for ion exchange and catalytic applications. The presence of Al_2O_3 further enhances the structural stability and acidic properties of the material, which are crucial for catalytic performance. Additionally, trace quantities of K_2O, CaO, TiO_2, and Fe_2O_3 were detected in the final product. These minor oxides may contribute to the modulation of the zeolite's physicochemical and catalytic behavior

FTIR

FTIR spectroscopy was employed to analyze the functional groups and structural features of the synthesized zeolite X. Fig.1 revealed characteristic absorption bands that are consistent with the framework vibrations of zeolitic materials. A strong and broad peak near 1000 cm^{-1} corresponds to the asymmetric stretching vibrations of Si–O–Si, confirming the formation of the zeolite's tetrahedral silica framework. A distinct signal observed at 880 cm^{-1} is attributed to internal ring vibrations, which are typically associated with specific pore structures and crystalline arrangements within zeolite frameworks. A broad band around 3000 cm^{-1} indicates the presence of hydroxyl (–OH) groups, likely arising from adsorbed silanol species or water molecules on the surface. The presence of water is further supported by a peak near 1800 cm^{-1}, which is attributed to bending vibrations of molecularly adsorbed H_2O. An additional feature at approximately 2350 cm^{-1} is ascribed to adsorbed atmospheric CO_2, commonly seen in FTIR spectra of porous materials exposed to ambient air. Moreover, C–H stretching bands overlapping around 3000 cm^{-1} suggest the presence of residual organic species from synthesis or precursor materials. The high transmittance

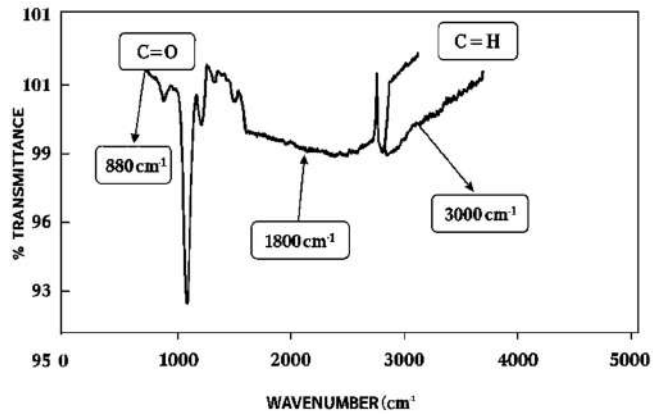


Fig. 1: FTIR Diffraction of in-house-made Zeolite X.

observed in the 4000–5000 cm^{-1} region indicates the absence of significant absorption bands in this range, confirming the structural purity of the sample. Overall, the FTIR data validate the successful synthesis of zeolite X and reflect its compositional and structural fidelity.

FESEM

FESEM was employed to investigate the surface morphology and microstructure of the synthesized zeolite X. As shown in Fig. 2, the sample consists of relatively small particles with an average size in the ~1 µm range, which aligns well with typical zeolite particle size distributions. The particles exhibit an irregular and porous morphology, featuring a honey comb like framework with interconnected pores, characteristic of zeolite X. This microstructure is beneficial for catalytic applications due to enhanced surface area and accessibility to active sites. Slight aggregation of particles was observed, Fig. 3 shows potentially caused by electrostatic interaction during the synthesis and drying processes. Moreover, the particle surfaces appear rough and textured, suggesting the presence of surface defects or structural irregularities, which can significantly influence the material's adsorption and catalytic performance. These morphological features collectively confirm the successful formation of zeolite X with a structure suitable for advanced catalytic applications.

Preparation of Cu-X and Fe-X Zeolite Catalysts

Cu-X and Fe-X zeolite catalysts were synthesized via ion-exchange of zeolite X, followed by washcoating onto cordierite monoliths using a silica-based slurry to achieve 15 wt.% coating and thermal activation.

To prepare the metal-exchanged zeolite catalysts, 10 g of zeolite X powder synthesized from rice husk ash was subjected to an ion-exchange process. The powder was dispersed in 1000 mL of 0.05 M cupric chloride dihydrate ($CuCl_2 \cdot 2H_2O$, Fisher Scientific) solution and stirred vigorously using a magnetic stirrer at ambient temperature for 24 h. The suspension was vacuum-filtered and rinsed thoroughly with deionized water to remove excess ions. For the Fe-exchanged zeolite, ferrous chloride tetrahydrate ($FeCl_2 \cdot 4H_2O$, Fisher Scientific) was used instead of cupric chloride. The resulting solids were dried in an oven and calcined at 550°C for 6 h to activate the catalysts.

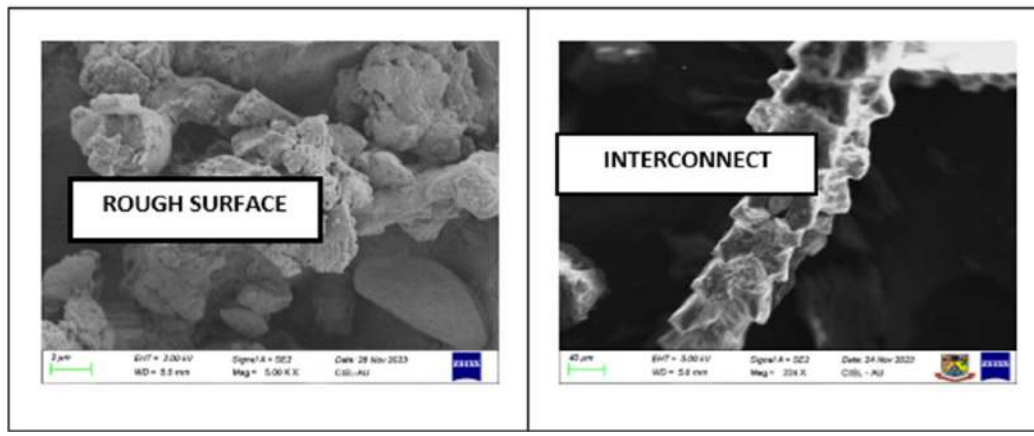
Cordierite monoliths (supplied by Bocent Advanced Ceramics Co., Ltd.) were used as substrates. These monoliths had a cell density of 400 CPSI, a wall thickness of 0.17 mm, and were 90 mm in both diameter and length. One monolith was platinum-coated, and the other was uncoated, serving as a control.

The washcoating slurry consisted of 20 wt. % Cu-X zeolite, 4 wt. % silica gel, and distilled water as the solvent. Each monolith was dipped in the slurry for one minute, and excess slurry was blown out with compressed air through both ends. The monoliths were dried in a muffle furnace at 120°C for 2 h, and the process was repeated until the washcoat loading reached 15 wt.% of the monolith's weight. Finally, the coated monoliths were calcined at 500°C for 5 h. The same washcoating and thermal treatment protocol was applied to prepare Pt-Fe-zeolite and Fe-X zeolite-coated monoliths.

Fabrication of Catalytic Converter-Catalytic Activity Tests on a Twin-Cylinder Petrol Engine Were Conducted Using Two Fabricated Catalytic Converter Types

Type 1

Two catalytic converters were fabricated with dimensions matching the original equipment manufacturer's catalytic converter. A Pt-Cu-X zeolite-coated monolith was tightly wrapped with resilient matting (to dampen vibrations) and then mounted in a steel casing. Inlet and outlet cones were



Figs. 2 and 3: Morphology of Raw rice husk and the morphology of in-house-made zeolite X.

welded to both ends of the casing. The catalytic converter was firmly attached to the engine's exhaust pipe. A stainless steel cover acted as a heat shield. Pt-Fe- coated monolith catalytic converters were fabricated using the same procedure. Fig 4 shows the in-house and commercial catalytic converters, respectively.

Type 2:

For the different configuration, a commercially sourced platinum (Pt)- coated monolith and a conventionally prepared copper-exchanged zeolite X (Cu-X) coated monolith were meticulously assembled within a welded steel casing, equipped with integrated inlet and outlet cones. This design strategically combined the catalytic activity of the Pt- coated monolith with the NOx reduction capabilities of the (Cu-X) zeolite coating. To ensure secure installation within the engine's exhaust system, appropriate mounting provisions were incorporated. A similar fabrication process was employed to create a catalytic converter utilizing a Pt-coated monolith and an iron-exchanged zeolite X (Fe-X) coated monolith. Fig. 5 depicts the deNOx catalytic converter, which was manufactured in-house. The second configuration involved the assembly of a commercially obtained platinum coated monolith and a custom-prepared copper-exchanged zeolite X (Cu-X) coated monolith within a steel casing. This casing was then proficiently welded and fitted with inlet and outlet cones to facilitate gas flow. The resulting catalytic converter was securely integrated into the engine's exhaust pipe. A parallel fabrication process was used to create a separate catalytic converter, this time combining a platinum-coated monolith with an iron-exchanged zeolite X (Fe-X) coated monolith. It shows a visual representation of the deNOx catalytic converter, which was produced within our own facilities.

Experimental Setup

The test engine for this research was a 624 cc, four-stroke, twin-cylinder, water-cooled spark-ignition type (Table 2). Fig. 6 describes the test facility, which comprised the engine fixed on a test bed and directly linked to an eddy current dynamometer. This dynamometer provided precise engine loading control and switching capabilities. The experiment used an AVL-Di gas analyzer utilized for the accurate measurement of CO, HC, NOx, and O_2 emissions. Exhaust

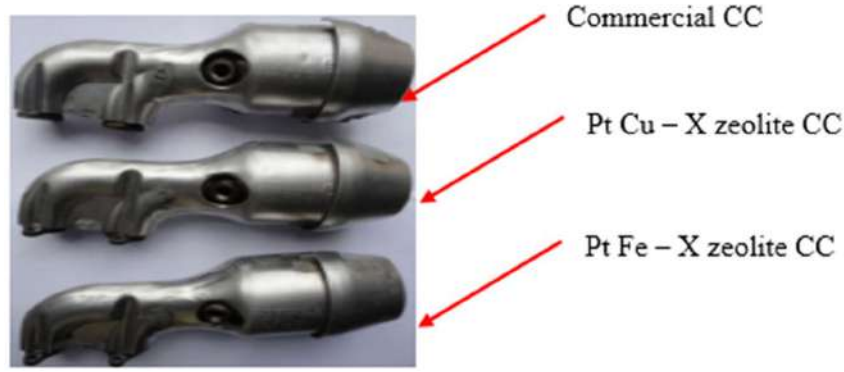


Fig. 4: Photographic view of an in-house-made and commercial catalytic converter.

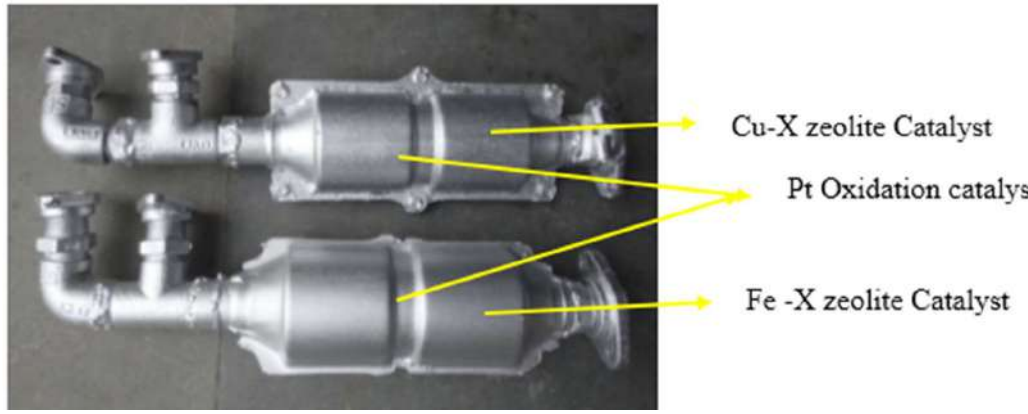


Fig. 5: Depiction of the in-house built deNOx catalytic converter.

Table 2: Specification of Petrol Engine.

Type	Vertical In-line Engine with MPFI
Number of cylinders	2
Displacement	624 cc
Bore	73.5 mm
Stroke	73.5 mm
Compression Ratio	9.5:1
Fuel	Petrol
Cycle	4-Stroke
Max. Engine output	25.74 kW @ 5250 rpm
Max. Torque	48 Nm @ 3000 rpm
Speed	2500 rpm
Orifice Diameter	20 mm
Cooling System	Water
Loading Device	Eddy current dynamometer

gas temperatures were monitored using strategically placed Chromel-Alumel thermocouples at three distinct locations on the converter.

Initially, standard engine performance was established by operating the engine without a catalytic converter across a range of loads (4, 7, 10, 13, and 16 kW) while sustaining a constant speed of 2500 rpm. Emissions of HC, CO, CO_2, O_2, and NOx were methodically recorded for each load condition. Thereafter, in Fig. 7, a commercially available catalytic converter was affixed to the engine exhaust manifold, ensuring axial entry of the exhaust gases. The engine was then operated under the Equivalent load and speed conditions previously used for the baseline measurements, and emissions of HC, CO, CO_2, O_2 and NOx were again recorded. This comparative testing procedure was replicated using both the Fe-zeolite and Cu-zeolite converters. Emissions measurements were conducted under the same operating

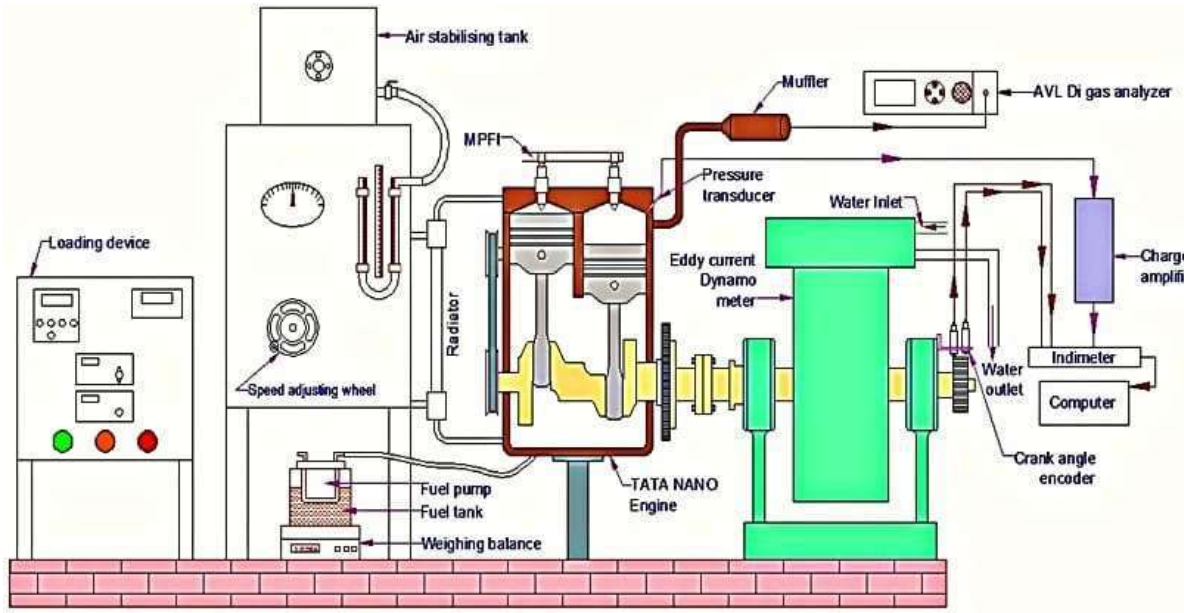
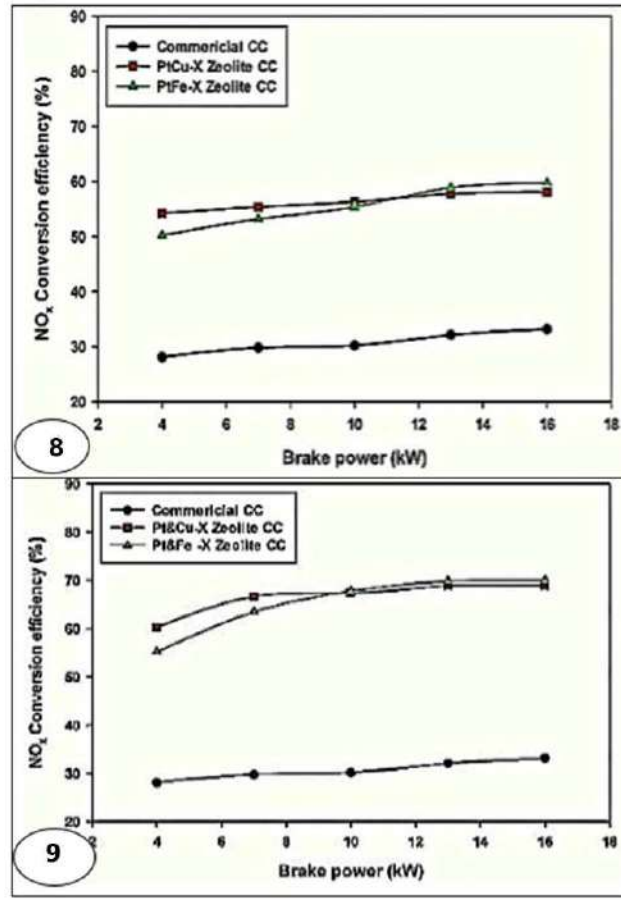


Fig. 6: Experimental setup of petrol engine.

Fig. 7: Photographic view of a catalytic converter fitted in the engine.



Figs. 8 & 9: Percentage reduction of NOx Vs. load (dual layer) and Percentage reduction of NOx Vs. load (deNO*x*).

conditions to rigorously evaluate the performance of these in-house fabricated converters against the commercially available unit.

RESULTS AND DISCUSSION

A twin-cylinder, MPFI petrol engine, connected to an eddy current dynamometer, was used for the experiments. The conversion efficiencies were then determined using the following calculations:

- NOx Conversion Efficiency = (Inlet NOx - Outlet NOx) /Inlet NOx * 100
- HC Conversion Efficiency = (Inlet HC - Outlet HC)/ Inlet HC * 100
- CO Conversion Efficiency = (Inlet CO - Outlet CO)/ Inlet CO * 100

Nitrogen Oxides Emission with Load

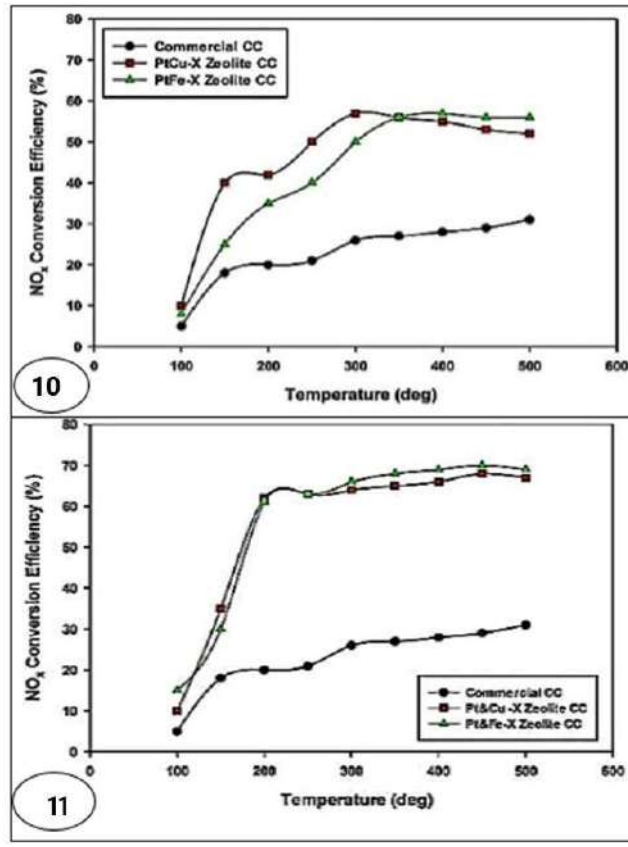
Figs. 8 and 9 demonstrate the NOx reconfiguration efficiency at various engine loads. The data clearly demonstrate that all in-house developed catalytic converters achieve significantly higher NOx conversion than the commercial converter. This effectiveness arises because commercial catalytic converters are designed for stoichiometric air–fuel ratios. However, the engine used is a lean-burn Multi Point Fuel Injection (MPFI) engine, resulting in oxygen-rich exhaust, which negatively affects the efficiency of conventional converters. To resolve this, a more effective catalytic system utilizing zeolite X was developed to diminish NOx emissions. Zeolite active sites exhibit a high affinity for NO, facilitating its separation into nitrogen (N) and oxygen (O). As shown in the results, enhanced conversion efficiency is observed with the introduced system. While in Figs. 8 and 9, it is seen that the double-layer monolith catalytic converters (Pt-Cu-X zeolite and Pt-Fe-X zeolite) exhibit lower performance compared to the combined monolith catalytic converters (Pt & Cu-X zeolite and Pt & Fe-X zeolite). Notably, the Pt-Fe-X zeolite converter achieves approximately 50% NOx conversion at a 4 kW load, gradually increasing to about 59% at 16 kW. In contrast, the Pt & Fe-X zeolite converter reduces NOx by roughly 55% at 4 kW and 70% at 16 kW.

The dual-layer catalytic converter's performance is limited because it requires simultaneous reactions of CO, HC, and NOx, restricting NOx reduction to the channel corners. Distinctly, the combined Pt and Fe-x zeolite monolith catalyst offers more active sites, enhancing NOx conversion. This occurs as exhaust gas flows through the platinum monolith, facilitating NO decomposition into adsorbed nitrogen and oxygen. The nitrogen atoms then combine to form N_2, and the oxygen reacts with CO to produce CO_2. However, at a 4 KW load, the Pt and Cu-X zeolite catalyst showed superior performance compared to the Pt-Fe-X zeolite catalyst.

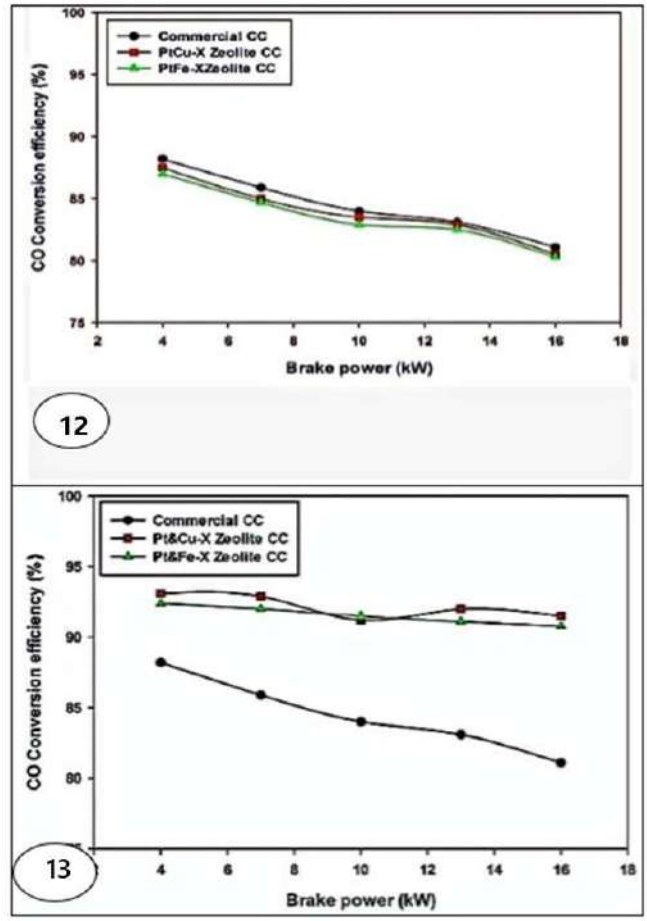
This performance difference can be attributed to the fact that three reactions (the reduction of CO, HC, and NOx to CO_2, H_2O, and N_2) occur simultaneously within the dual-layer catalytic converter, limiting the active NOx reduction sites to the corners of the monolith channels. The combined monolith catalytic converter (Pt & Fe-X) provides more active sites for NOx conversion, leading to improved performance. As exhaust gas passes through the platinum (Pt) monolith and subsequently contacts the deNOx monolith, the following reactions occur: (i) NO is adsorbed onto the active metal sites and dissociates into adsorbed nitrogen (N (ads)) and oxygen (O (ads)), (ii) two N (ads) atoms combine to form N_2, and (iii) the dissociated O (ads) reacts with the remaining CO in the exhaust gas to produce CO_2. At a 4 kW load, the Pt & Cu-X catalytic converter demonstrated better performance than the Pt and Fe-X catalytic converter.

Effects of Exhaust Gas Temperature on Nitrogen Oxide Emission Levels

The relationship between exhaust gas temperature and NOx conversion efficiency is compared in Figs. 10 and 11. The dual-layer catalytic converter demonstrates a progressive, albeit gradual, increase in efficiency, achieving a 50% NOx conversion rate at approximately 300°C. Conversely, the combined monolith catalytic converter (deNOx) exhibits a remarkably rapid surge in conversion efficiency, attaining a 60% conversion rate at approximately 190°C. This enhanced performance continues to improve steadily up to 500°C, beyond which any further significant enhancements are negligible. It is well-established that catalytic converter efficiency is profoundly influenced by temperature. It is expected that the zeolite catalyst will begin to function at approximately 180°C. Below 150°C,



Figs. 10 &11: Percentage reduction of NOx Vs. Temperature (dual layer) and percentage reduction of NOxVs. temperature (deNO*x*).



Figs. 12 & 13: Carbon monoxide against brake power (dual layer) and Carbon monoxide against brake power (deNO*x*).

its catalytic activity decreases. However, exceeding 180°C triggers a notable activation of the zeolite, resulting in a substantial improvement in NOx conversion efficiency. This temperature-dependent behavior is conspicuously absent in conventional commercial converters, which often struggle to achieve effective NOx reduction in oxygen-rich exhaust environments.

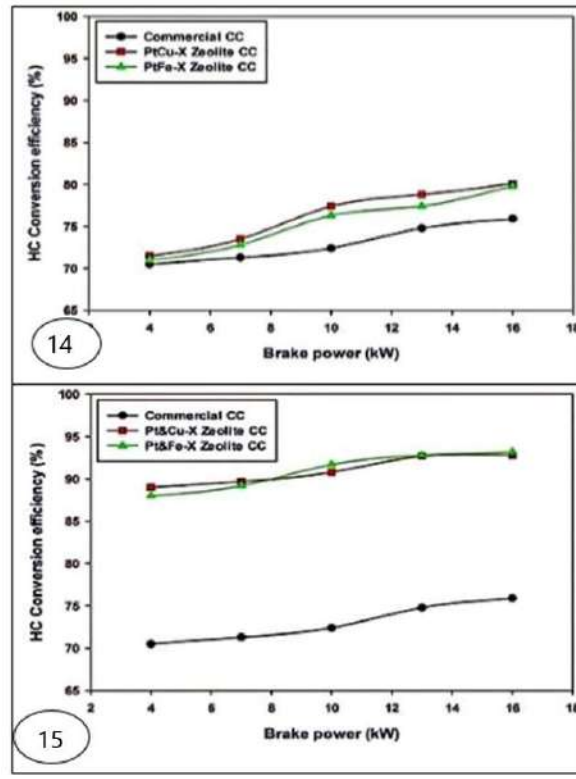
CO Conversion Efficiency

Figs. 12 and 13 show the CO conversion efficiencies of both the dual-layer monolith catalytic converter and the deNOx catalytic converter. Previous studies (Alkemade et al. 2006) have shown that commercial monoliths effectively reduce CO and HC emissions under lean exhaust conditions. Fig. 12 denotes only a slight decrease in CO changeover efficiency compared to the commercial catalytic converter. This is likely because the platinum (Pt) layer in the dual-layer monolith is partially obscured by the zeolite layer, reducing the number of active sites available for CO conversion.

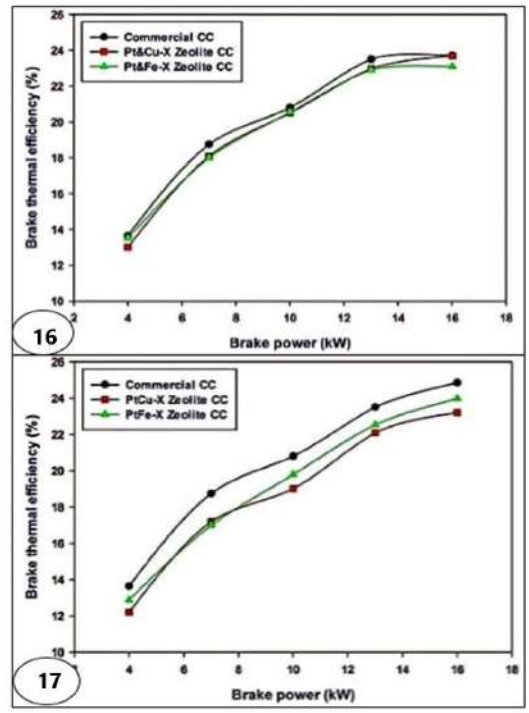
Fig. 13 illustrates that the deNOx catalyst achieves approximately 93% CO conversion efficiency at a 4 kW load, increasing to nearly 95% at 16 kW. The improved performance is due to residual CO from the Pt-coated monolith reacting with adsorbed oxygen on the deNOx catalyst, resulting in CO_2 formation. Prior studies have shown that CO oxidation aids NOx reduction in metal-exchanged zeolite catalysts.

Efficiency of Hydrocarbon Conversion

Figs. 14 and 15 show the percentage reduction in HC emissions as a function of engine brake power. The dual–layer catalytic converter achieves 72% HC conversion at a 4kW load, increasing to 79% at 16Kw. The deNOx converter system, however, achieves approximately 90% HC conversion at 4 kW, rising to 93% at 16 kW. This higher conversion is attributed to the reaction of unreacted HC emissions exiting the Pt-coated catalyst with adsorbed oxygen, producing CO_2 and H_2O. As demonstrated in earlier work (Liu et al. 2016),



Figs. 14 &15: Hydrocarbon against brake power (dual layer) and Hydrocarbon against brake power (deNO*x*).



Figs. 16 & 17: Brake thermal efficiency against brake power (dual layer) and deNOx Performance: Brake Thermal Efficiency as a Function of Brake Power.

hydrocarbons act as reducing agents by removing adsorbed oxygen generated during NOx dissociation. This action preserves the reduced states of the active sites (Cu^+ or Fe^+), which facilitates further NOx reduction.

Brake Thermal Efficiency

Figs. 16 and 17 present a comparative analysis of the engine's thermal efficiency in relation to its brake power output. A subtle yet discernible reduction in brake thermal efficiency is observed when employing the dual-layer monolith catalytic converter, as opposed to the commercially available counterpart. This diminution can be attributed to the marginally increased flow resistance encountered by the exhaust gases as they traverse the monolith walls. The introduction of an additional wash coating layer within the monolith channels effectively reduces the overall flow area, thereby impeding gas flow. Conversely, the deNOx system demonstrates a brake thermal efficiency that closely mirrors that of the commercial catalytic converter. Notably, despite the inclusion of two distinct monoliths, the deNOx system does not introduce any significant flow resistance within the exhaust gas pathway.

CONCLUSION

This study suggests a potential alternative to conventional three-way catalytic converters for reducing NOx emissions from gasoline engines. The synthesized rice husk ash-based Na-X zeolite catalysts, modified with copper and iron ions (Cu-X and Fe-X), demonstrated superior NOx reduction compared to the conventional converter while maintaining comparable performance for other pollutants. The enhanced performance can be attributed to the unique structural properties of the zeolite-based catalysts and the synergistic effect of the copper and iron ions. These findings indicate that the developed Cu-X and Fe-X zeolite catalysts show promise as a cost-effective and environmentally friendly option for reducing NOx emissions from gasoline engines. Further research and development are needed to optimize their

- The custom-made metal-doped X zeolite converter outperformed the commercially available one. Performance and scale up production for commercial applications.
- Cu- zeolite X was more effective at reducing NOx in cooler conditions, whereas Fe- zeolite X excelled at higher temperatures.
- Both carbon monoxide and hydrocarbon emissions were substantially lowered across all engine loads and during speed variation tests.
- The catalyst bed did not create excessive back pressure.
- The catalytic converter showed no noticeable loss of activity after 50 h of testing.

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Assessing the Educational Significance of Microplastic Impact on *Sardinella gibbosa*: Implications for Marine Sustainability and Public Health in Caraga Region, Philippines

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ABSTRACT

This research study aims to provide an educational assessment of microplastic contamination by examining the presence of microplastics in the gut contents of Sardinella gibbosa collected from the coastal waters of Magallanes, Agusan del Norte. Sixty fish samples were analyzed, revealing that 75% of them had ingested microplastics, with over 90% of the particles identified as fibers. These fibers are presumed to originate from environmental pollutants such as discarded fishing nets, degraded plastic debris, and other synthetic waste. The results highlight the urgent issue of microplastic pollution in local marine ecosystems and its potential impact on food safety and sustainability. Importantly, this study serves as an educational tool to raise awareness about marine pollution and its cascading effects on public health, marine biodiversity, and community livelihoods. While the research focused primarily on detecting microplastics in fish digestive systems, it underscores the need for further educational programs and research initiatives to explore the broader implications, particularly the health risks to humans consuming contaminated seafood. Integrating these findings into marine science education can foster environmental stewardship among students, policymakers, and local communities. Continued monitoring and public education on plastic waste reduction are crucial steps toward mitigating microplastic pollution and promoting sustainable marine practices in the Caraga Region.

INTRODUCTION

The Science History Institute (2022) defined plastic as a group of materials, either synthetic or naturally occurring, that may be shaped when soft and then hardened to retain the given shape. With plastic being durable and long-lasting, the British Plastics Federation (2022) stated that it is used in every sector, including to produce packaging, in building and construction, in textiles, consumer products, transportation, electrical and electronics and industrial machinery. This results in the massive production of plastic, which, according to the Natural Resources Defense Council (2022), amounts to 300 million tons of plastic each year. In the Philippines, plastic plays a major role in the national economy and supports access to low-cost consumer goods for low-income and middle-income households. This widespread dependence on small, affordable packaging has earned the country the label of a "sachet economy," as highlighted in the Market Study for the Philippines: Plastics Circularity Opportunity and Barriers (2021). However, the mass production and rapid consumption of plastic have intensified waste management challenges. According to a 2021 study by the UN Environment Programme, an estimated 75 to 199 million tonnes of plastic now pollute the world's oceans (UNEP 2021).

According to the National Oceanic and Atmospheric Association (2021), plastic is the most prevalent type of marine debris that can come in all shapes and sizes, but those that are 5 millimeters in length are called microplastics. Microplastics that are found in the sea emerged from land-based resources and ocean-based resources, including sewage and stormwater, discarded and lost fishing items (Li 2018). Microplastics are found everywhere. The study of Dris et al. (2015) stated that microplastics have been observed in both sediments and water samples. Its biodegradation-resistant properties allowed it to persist and accumulate in the marine environment, thus causing physical and chemical effects on marine organisms after ingestion (Li 2018).

A study by Ory et al (2018) stated that visually oriented planktivorous fish are susceptible to ingesting microplastics resembling or floating close to their planktonic prey. Along with that, Botterell et al. (2019) stated that high concentrations of microplastics could induce suppressed feeding activity, prolonged gut residence times, inflammation, and reduced energy reserves, impacting growth, reproduction and ultimately survival. In connection with that, You Li et al. (2021) implied that microplastics inhibit the growth and development of the Lugworm (*Arenicola marina)*, and the inhibition degree is positively correlated with the concentration of microplastics. Also, the study of Wen et al. (2018) stated that microplastics can cause blockage of the digestive tract and induce satiety, which may result in reduced growth performance and survival rate of discus fish. It has also been noted that the accumulation of microplastics significantly increased the rate of respiration and excretion while significantly decreasing feeding and absorption efficiency, leading to a reduced amount of energy available for growth of the Manila clam (Jiang et al. 2021).

The Philippine Daily Inquirer published an article written by Subingsubing (2022), whereby fishermen of Mambacayao Island observed the decreased number of fish caught and a noticeable trace of microplastics was found when they opened the fish. A total of 51 microplastics were also isolated from the various fish species based on the study of Espiritu et al. (2019). Barboza et al. (2019) concluded that the lipid oxidative damage in gills and muscle, neurotoxicity through lipid oxidative damage and acetylcholinesterase induction are directly related to the presence of microplastics in the gastrointestinal tract, gills and dorsal muscle of the fish. The study of Chen et al. (2020) concluded that exposure to microplastics significantly delayed the hatching time, altered the heartbeat and decreased the hatching rate of embryos of marine medaka and not only that, the genes involved in cardiac development were significantly upregulated, pathways involved in metabolism, immune response and genetic information processing and diseases were significantly enriched thereby concluding that microplastics have negatively affected embryogenesis and the immune response e of marine medaka.

Microplastics are found in marine residue, seabed, water fragments and manufactured substances, even inside the gastrointestinal tract, dwelling in skin, gills, liver and fish (Kazam et al. 2021). It is inevitable for microplastics to be in contact with humans. Microplastics can absorb harmful chemicals and with this being in a human system, this may cause danger to the human body as the study of Prata et al. (2020) stated that the inability of the immune system to remove synthetic particles may lead to chronic inflammation and increase the risk of neoplasia such that that article by Blackburn & Green (2021) stated that high concentration of microplastics could provoke immune and stress responses and inducing reproductive and developmental toxicity. Added to that, the review study conducted by Peixoto et al. (2019) mentioned that microplastics have the potential to bioaccumulate in secondary organs with possible impacts on the immune system and cell growth (Lusher et al. 2020) and may be responsible for negative biological responses like inflammation, genotoxicity, oxidative stress, cell apoptosis, and tissue necrosis. Its potential hazardous effect on humans has also been noted in the study of Sharma & Chatterjee (2017), and it includes alteration in chromosomes, which may lead to infertility, obesity, and cancer.

Fish is a staple food and a source of income for most of the people, especially the residents of Magallanes, Agusan del Norte and considering its possible danger to human health, the researchers believed that a study should be done to be aware of and to know if microplastics can be seen in the food that is constantly consumed. Therefore, this study aimed to conduct a preliminary assessment of microplastics in the common fish species of sardines (*Sardinella gibbosa*) caught in the coastal area of Magallanes, Agusan del Norte, Philippines.

MATERIALS AND METHODS

Sample Collection and Preparation

The study requires the collection of the gut of the fish *Sardinella gibbosa* (Fig. 1) to determine whether its diet is affected by plastic pollution in its natural habitat. The researchers contracted a fisherman to be able to obtain samples of *S. gibbosa* coming from the same cohort caught in the coastal area of Magallanes, Agusan del Norte and to avoid selection bias.

Fish guts are considered waste for the market buyers and are usually disregarded after purchasing the fish. A total of 60 fish samples were bought and subjected to gut

Fig. 1: *Sardinella gibbosa* caught in the coastal areas.

content analysis. All fish samples were stored in an icebox to maintain their physical integrity. It was then brought to the laboratory for examination.

The length and weight of the samples were taken by measuring the total length using a ruler and weighing them using a digital weighing scale. All measurements were recorded accordingly for data management and statistical analysis. All the procedures were documented properly.

Based on the Diet Composition by Pinkas et al. (1971), Gut samples collected and cleaned were dissected to expose their contents. It was washed in a Petri dish with 10 ml of distilled water and was gradually stirred to release all the contents into the water. The gut was then removed, ensuring all contents were released into the solution. The Petri dish was visually examined for floating plastic materials and debris (difference in density). Plastics visible to the naked eye were then collected, photographed, and noted for documentation. For microplastics (<5.0mm) determination, the solution in the Petri dish was then diluted with 10 mL of 30% hydrogen peroxide to dissolve the remaining flesh in the solution. The solution was poured over a pre-weighed filter paper to air dry at room temperature for 30 minutes. 60 pieces of filter paper with the air-dried undissolved gut content were then weighed and examined under an electron microscope for the presence of undissolved traces of plastic/ synthetic materials in the samples.

RESULTS AND DISCUSSION

A total of sixty (60) samples of the common sardine species of *S. gibbosa* were examined for gut content analysis. Table 1 shows that the average length of the samples ranges from 9.9 cm to 12.6 cm, with a standard deviation of 11.328 cm. The average weight of the samples ranges from 5.896g to 17.690 g, with a standard deviation of 10.936 g. The data show that the samples may not be truly representative of the same cohort or biological batch, with a low positive correlation (r^2) value of 0.287 (Fig. 2). This could be due to differences in ages, genders, or other characteristics of the individuals from which the samples were taken. Despite the low correlation, the high probability of the samples being in the same cohort and of the same biological batch suggests that there may still be some underlying similarities between them.

The results of the gut content analysis demonstrate a significant presence of microplastics in the gut of the samples examined. The fact that 75% of the samples had ingested microplastics highlights the widespread nature of this issue and the potential harm it poses to the affected organisms.

Table 2 also shows that the ingestion of microplastics varies among the samples, with some having no observations while others have up to 5 observations. This suggests that there may be factors that influence the ingestion of microplastics, such as the location or behavior of the organism.

Table 1: Descriptive statistics of the size, weight, and gut content of *S. gibbosa* caught in the coastal areas.

	Length [cm]	Weight [g]	Gut Content Weight [g]
Mean	11.328	10.936	0.0629
Standard Error	0.081	0.358	0.006
Median	11.5	10.884	0.049
Mode	10.8	8.618	0.09
Standard Deviation	0.630	2.773	0.052
Sample Variance	0.397	7.692	0.002
Range	2.7	11.793	0.324
Minimum	9.9	5.896	0.009
Maximum	12.6	17.690	0.333
Count	60	60	60
Confidence Level (95.0%)	0.162	0.716	0.013

Table 2: Descriptive Statistics of the observations of microplastics ingestion of *S. gibbosa*.

Microplastics	
Mean	1.533
Standard Error	0.165
Median	1
Mode	1
Standard Deviation	1.281
Sample Variance	1.642
Range	5
Minimum	0
Maximum	5
Sum	92
Count	60
Confidence Level (95.0%)	0.331

The average of ±1 observation per sample indicates that, on average, each sample had ingested one piece of microplastic. This may not seem like a high number, but it is important to consider the cumulative effects of microplastic ingestion over time, like the study of Koelmans (2019).

Fig. 3 shows some of the microplastics documented during the gut content analysis. Most of the observed microplastics were fibers (a. and b.), accounting for more than 90% of the observations, followed by microplastic fragments and others. These fiber types of microplastics are probably from discarded nets, weather plastic fragments, polyester clothes, and others (Lusher et al 2017). It is important to note that the presence of microplastics in the gut content of marine organisms is a major concern for their health and well-being. These small plastic particles can cause physical damage to the digestive system, as well

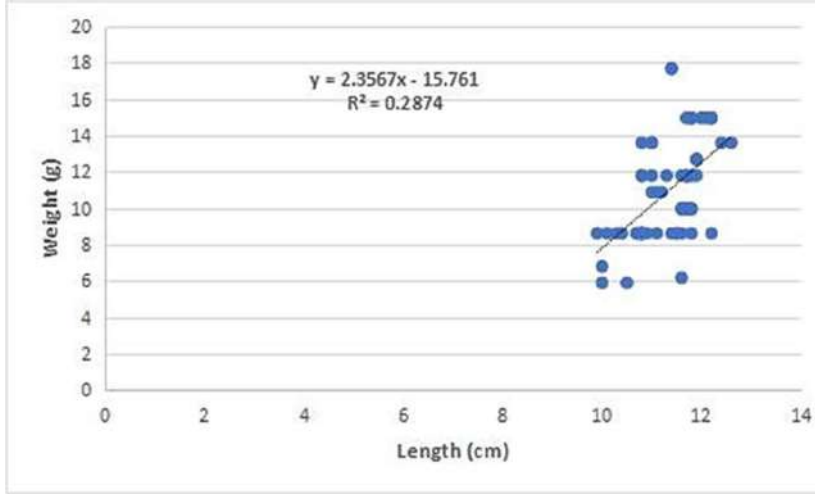

Fig. 2: Correlation between length-weight samples of *S. gibbosa*.

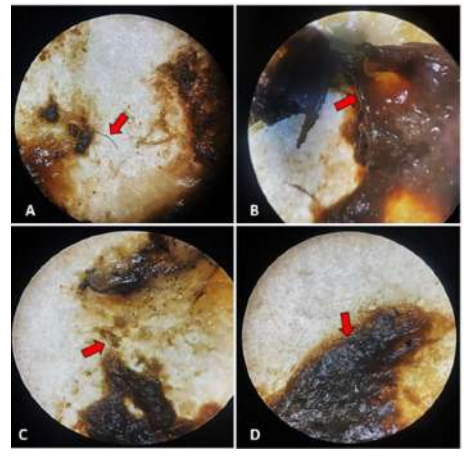

Fig. 3: Types of microplastics observed in the gut of *S. gibbosa* caught in Coastal Areas.

as potentially introduce toxic chemicals into the organism's body (Koelmans 2019). Furthermore, the fact that fibers make up the majority of the microplastics observed in this study highlights the need for better management and disposal of products such as fishing nets and clothing made from synthetic materials. These items are major sources of microplastic pollution in the ocean and can have devastating impacts on marine life (Eriksen et al. 2014).

The occurrence of microplastics in the gut of S. gibbosa was also correlated with its length and weight. Figs. 4 and 5 show that there was a very low correlation between the size and weight of the fish and their gut content, with r^2 values of 0.002 and 0.020, respectively. This means that the presence and volume of microplastics in the gut of S.gibbosa are directly proportional to their size, similar to what is reported by Pazos et al. (2017) and Filgueiras et al. (2020). This correlation does not necessarily mean that larger fish are consuming more microplastics. It could also be a result of larger fish having a larger gut capacity, which allows them to hold more microplastics. Additionally, it could also be influenced by other factors such as feeding habits and habitat. For example, fish that live in areas with higher levels of pollution may have a higher chance of consuming microplastics.

Furthermore, the presence of microplastics in the gut of S. gibbosa can also have negative impacts on their health. Microplastics can cause physical injury to the gut lining and can also disrupt the fish's metabolism and

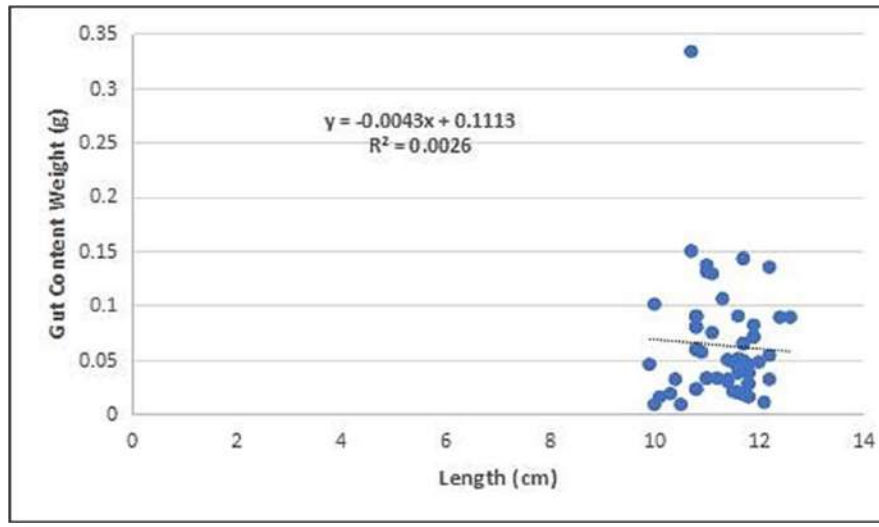

Fig. 4: Length-gut content correlation of *S. gibbosa* caught in coastal areas.

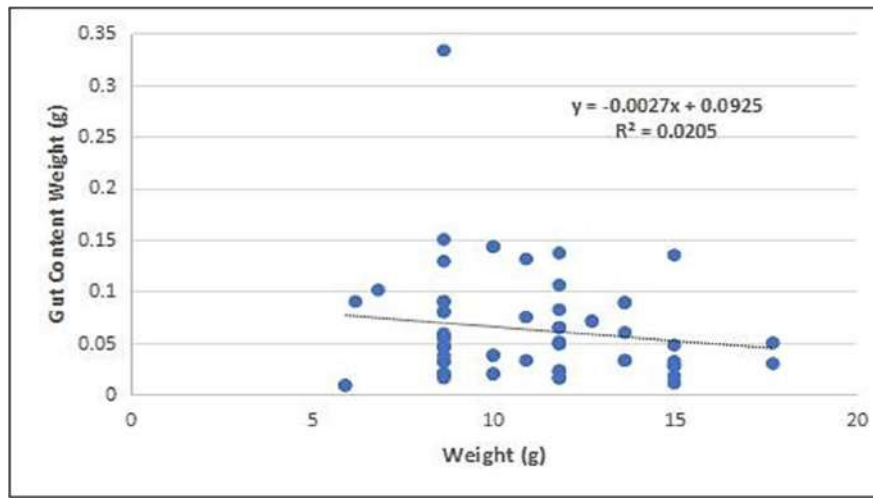

Fig. 5: Weight-gut content correlation of *S. gibbosa* caught in coastal areas.

nutrient absorption. This can lead to reduced growth and reproduction and can also make fish more susceptible to disease (Koelmans et al. 2019).

The r2 values mentioned (0.002 and 0.020) are measures of the strength of the correlation, with a value closer to 1 indicating a strong correlation and a value closer to 0 indicating a weak correlation (Fig. 5). In this case, the values of 0.002 and 0.020 indicate that there is a very weak correlation between the size and weight of the fish and the amount of microplastics found in their gut. This suggests that other factors may be more important in determining the presence and volume of microplastics in the gut of *S. gibbosa*.

CONCLUSIONS

A total of sixty (60) *Sardinella gibbosa* samples were collected from the coastal area of Magallanes, Agusan del Norte, in collaboration with local fishermen. Analysis of the gut contents revealed that 45 samples, or 75%, had ingested microplastics, predominantly in the form of fibers. These fibers likely originated from discarded fishing nets, weathered plastic fragments, synthetic fabrics, and other anthropogenic sources commonly found in the bay's vicinity. While statistical analysis showed a low correlation between the presence of microplastics and the fish's length and weight, a positive direct relationship was observed. These findings serve as an important educational resource, highlighting the pervasive nature of microplastic pollution in local marine environments. The study not only raises awareness of environmental impacts but also underscores the need for integrating marine pollution topics into educational curricula to inform students, policymakers, and the public. Although the study primarily focused on microplastic ingestion and cannot conclusively determine the bay's overall pollution status, the high percentage of contamination is a strong indication of environmental concerns. Future research and continuous educational efforts are recommended to explore the broader ecological and public health implications, fostering greater environmental stewardship and sustainable practices within coastal communities.

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Open Access Journal

Assessing Water Quality Through Remote Sensing: A Regression-Based Approach with Sentinel-2 Data

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ABSTRACT

Monitoring water quality is essential for ensuring human health and environmental sustainability. Traditional methods that rely on laboratory analysis and point-based sampling often lack sufficient spatial and temporal coverage. This study assessed the water quality along the Sabarmati Riverfront in Ahmedabad, India, using Google Earth Engine (GEE) and Sentinel-2 satellite imagery. Key parameters, including pH, turbidity (Tur), Electrical Conductivity (EC), Total Suspended Solids (TSS), Total Solids (TS), Dissolved Oxygen (DO), Biochemical Oxygen Demand (BOD), Total Phosphorus (TP), Fecal Coliform (FC), and ammonia (NH_3), were estimated using remote sensing. An empirical regression model was developed to relate in situ data to satellite-derived spectral indices. Results revealed significant seasonal and spatial variations, with some areas showing favorable levels of TSS, BOD, and FC. The model showed strong predictive accuracy for pH, TSS, and TP (R^2 = 0.80, R^2 = 0.76, R^2 = 0.75, respectively) and moderate performance for turbidity (R^2 = 0.62). Integrating remote sensing and GIS enables scalable, cost-effective, real-time water quality monitoring, providing critical insights for pollution control and water resource management. Future research should explore hyperspectral imaging and machine learning to enhance predictive accuracy and broaden the applicability of satellite-based monitoring models.

INTRODUCTION

Water is a vital resource for sustaining life on Earth and contributes significantly to the economic and social development of nations (Ingrao et al. 2023). It plays a central role in various sectors, including industrial and domestic infrastructure, agriculture, recreation, navigation, and water storage systems (Bănăduc et al. 2022). Ensuring water quality through regular and continuous monitoring is essential for informed and timely decision-making in water resource management (Kapalanga et al. 2021). However, water quality can vary substantially across geographic regions, necessitating the consideration of both quantity and quality in strategic planning processes (Kumar et al. 2024). Accurate, real-time data are therefore crucial for the effective distribution and planning of water resources at regional scales (Imiya et al. 2023).

The integration of cloud computing platforms with advanced predictive models has the potential to improve decision-making efficiency in water management systems (Sherjah et al. 2023). Traditional water quality monitoring approaches, which rely heavily on point-based sampling and laboratory analyses, are limited by their spatial and temporal coverage. These methods are time-intensive and costly and are inadequate for capturing the dynamic behavior of water bodies, particularly

in remote or large-scale areas (Adjovu et al. 2023, Essamlali et al. 2024). Consequently, there is a growing need for comprehensive real-time monitoring systems capable of supporting effective environmental governance.

Water quality is typically assessed using various chemical, physical, and biological parameters (Misman et al. 2023). Among these, certain physical and chemical attributes, such as chlorophyll-a, turbidity, and colored dissolved organic matter (CDOM), are classified as optically active parameters (Fu et al. 2022), whereas others, including dissolved oxygen (DO), total nitrogen (TN), and total phosphorus (TP), are considered non-optical parameters (Gao et al. 2024). Remote sensing (RS) platforms equipped with optical and thermal sensors mounted on boats, aircraft, or satellites provide both spatial and temporal data for environmental monitoring (Kanjir et al. 2018). These technologies have been increasingly used to monitor water quality variations and support the development of improved water management strategies (Adjovu et al. 2023).

Recent studies have advanced the development of algorithms that estimate optically active water quality parameters from RS data. For example, regional retrieval algorithms have been compared with established models to evaluate the efficacy of Sentinel-2 MultiSpectral Instrument (MSI) indices for estimating chlorophyll a concentration in optically complex aquatic environments. Similarly, Bonansea et al. (2019) demonstrated that satellite-derived spectral indices strongly correlate with in situ measurements of optically active water-quality parameters. These findings underscore the potential of satellite data for operational water quality assessment, particularly when coupled with machine learning techniques (Najafzadeh et al. 2023).

The continuous evolution of cloud computing, machine learning, and big data analytics has transformed environmental monitoring practices (Chi et al. 2016; Di et al. 2023). The authors (Chen et al. 2022) have provided a comprehensive evaluation of remote sensing big data frameworks and techniques, focusing on water extraction and quantitative water quality estimation. These advances enable the characterization of multispectral signals that reflect the hydrological, biological, and chemical attributes of water bodies and the physical properties of the surrounding environment. Spectral data in the 0.36–2.36 μm range, particularly within the visible and near-infrared bands (0.4–0.9 μm), have shown promising capabilities for detecting water contaminants using their spectral signatures (Seyhan et al. 1986). Further exploration of machine learning applications for estimating non-optically active parameters is discussed in the subsequent literature review section.

Unlike previous studies that focused on regional or global models, this study uniquely integrated Google Earth Engine (GEE) with regression analysis to estimate water quality parameters for the Sabarmati River, enabling scalable and efficient monitoring in a data-limited context.

LITERATURE REVIEW

Effective monitoring of inland water quality is essential for managing eutrophication, pollution, and ecological degradation, particularly in areas experiencing intensified anthropogenic activity and climate variability. In recent years, remote sensing (RS) has emerged as a valuable tool for observing water quality parameters (WQPs) over broad spatial and temporal scales, complementing or even replacing traditional in situ methods in certain contexts. The integration of RS with field measurements has proven effective in capturing spatially distributed information on key indicators, such as Total Suspended Solids (TSS), Total Nitrogen (TN), Total Phosphorus (TP), Chemical Oxygen Demand (COD), and chlorophyll a. For example, Muhoyi et al. (2022) demonstrated the use of Sentinel-2 imagery in conjunction with in situ sampling to map eutrophication-related contaminants in Zimbabwe's Lower Manyame Sub-catchment (LMS), identifying upstream sources of pollution and highlighting nutrient-driven degradation. Similar approaches have been applied in Egypt's Timsah Lake (Seleem et al. 2022) and India's Renuka Lake (Jally et al. 2024), revealing long-term eutrophic conditions exacerbated by anthropogenic pressure.

Despite these advances, satellite-based monitoring of inland water systems faces technical limitations such as low signal-to-noise ratios, atmospheric interference, and coarse resolution in narrow or heterogeneous water bodies. To mitigate these issues, researchers have incorporated proximal-remote-sensing techniques. For instance, Sun et al. (2022) developed empirical algorithms based on spectral reflectance and in-situ concentrations of COD, TN, and TP from multiple sites in China, achieving model accuracies exceeding 80–90%. These hybrid techniques improve precision while retaining the broader observational advantages. Parallel to sensor improvements, the adoption of statistical and machine learning methods has enhanced the retrieval accuracy and interpretability of WQPs. Studies employing multivariate analyses, such as principal component analysis (PCA) and varimax rotation, have been instrumental in identifying dominant pollution factors, including industrial discharge and ion exchange dynamics, as observed in the Daman Ganga River (Seth et al. 2025) and groundwater systems in Patna (Zafar et al. 2024). Moreover, artificial intelligence models, including recurrent neural

networks (RNNs) and ARIMA forecasting methods, have been proposed to dynamically predict coastal water quality using RS data (Bodapati 2023).

Comprehensive reviews as presented in Table 1, further illustrate the expanding role of RS in water quality monitoring. These studies categorize RS methods into empirical, semi-empirical, analytical, and machine learning-based approaches and emphasize the use of both optical and microwave sensors for retrieving parameters such as colored dissolved organic matter (CDOM), turbidity, TSM, and chlorophyll-a (Yang et al.2022; Adjovu et al.2023). The increased availability of hyperspectral sensors and unmanned aerial vehicles (UAVs) has further enhanced spatial and spectral resolution, enabling near-real-time assessments.

However, challenges in standardization remain. Variability in Water Quality Index (WQI) computation methods, such as those applied in the Aksu and Kali rivers (Şener et al. 2017, Said et al. 2021), limits the cross-regional comparisons and the operational use of RS data in policymaking. Addressing these inconsistencies requires improved model calibration, regional adaptation of algorithms, and harmonized data-reporting frameworks. Recent assessments (Tsitsi et al. 2024) advocate for the deeper integration of RS with ground observations, enhanced sensor calibration, and algorithm optimization. These steps are crucial for overcoming current limitations and realizing the full potential of RS for continuous, accurate, and scalable water quality monitoring in support of environmental sustainability.

CONTEXTUALIZING THE OBJECTIVES

The main objective of this work is to present a methodology that requires remote sensing for the evaluation of the variation in water characteristic as pH, Electrical Conductivity (EC), Turbidity (Tur), Total Suspended Solids (TSS), Total Solids (TS), Biochemical Oxygen Demand (BOD), Dissolved Oxygen (DO), Total Phosphorus (TP), Fecal Coliform (FC), and Ammonia (NH_3) via a regression model and the GEE platform. With this purpose, the intention was as follows:

- To use different indices and band ratios for water quality determination via Sentinel-2 satellite images,
- The Google Earth Engine (GEE) platform is used to obtain satellite images, and their processing is used to evaluate the water characteristics.
- Analysis of an inland water body located in the Sabarmati Riverfront, Ahmedabad, Gujarat, India,
- A regression model is used to determine the relationships among field data and remote sensing data.

MATERIALS AND METHODS

Study Area

The Sabarmati Riverfront is located at the coordinates of Latitude: 23.0341367°N and Longitude: 72.5723255°E. The Sabarmati Riverfront is a waterfront developed along the banks of the Sabarmati River in Ahmedabad, India, as shown in Fig. 1. Since 2012, the waterfront has been gradually opened to the public as facilities are constructed. The riverbed land is reclaimed on both the east and west banks to construct an around 11.25 kilometer long riverfront. The project aims to provide Ahmedabad with a meaningful waterfront environment along the banks of the Sabarmati River and to redefine the identity of Ahmedabad around the river. The average annual rainfall in Ahmedabad city is approximately 782 mm.

Table 1: Represents the comparative literature review.

Source	Approach	WQ Parameters	Accuracy Assessment	Method
Muhoyi et al. 2022	RS: Sentinel-2	TSS, TP, TN, COD	R^2 Range: 0.63 – 0.78	Empirical Models
Sun et al. 2022	Proximal Remote Sensing	TN, TP, COD	R^2 Range: 0.84–0.93	Empirical & Machine Learning Models
Seleem et al. 2022	RS: Sentinel-2 and Landsat-8	Total Suspended Matter, Chlorophyll-a	Ground truth data was not available	Empirical and Semi-Analytical Models
Maciel et al. 2023	RS: Sentinel 2	Chlorophyll-a	R^2 Range: 0.77 – 0.98	Empirical Models
Zhang et al. 2021	RS: Sentinel 2 and Sentinel 3	Water Quality Index	R^2 Range: 0.69 - 0.81	Machine Learning Models
Jally et al. 2024	RS: Landsat 8 & LISS III	Secchi Disk Transparency & Trophic State Index	R^2 = 0.94	Regression Models
Zafar et al. 2024	GIS with Conventional Approach	11 WQ Parameters	Poor WQI	PCA, HCA and Interpolation Technique
Seth et al. 2025	Conventional Approach	17 WQ Parameters	Poor WQI	PCA followed by Factor Analysis

RS: Remote Sensing

Field Data

Field data was collected from five sites along the Sabarmati Riverfront, as shown in Fig. 2, at a depth of 1 m from the water surface. The samples were collected by following the standards from collection to transportation to storage until the experiment was conducted. The physical and chemical characteristics of water, such as pH, Electrical Conductivity (EC), turbidity (Tur), Total Suspended Solids (TSS), Total Solids (TS), Biochemical Oxygen Demand (BOD), Dissolved Oxygen (DO), Total Phosphorus (TP), Fecal Coliform (FC), and ammonia (NH_3), were determined in an NABL Accredited laboratory in Ahmedabad, India. Samples were collected on three dates in February, March, and April 2024 (Table 2).

Satellite Data (Google Earth Engine)

Google Earth Engine (GEE) is used by various researchers and integrates real-world applications and visualizations of geospatial datasets through the application of algorithms to map, identify, and measure variations on Earth's surface, for real-world applications (Haifa et al. 2020, Pham-Duc et al. 2023, Velastegui-Montoya et al. 2023). In a study conducted by Pérez-Cutillas et al. (2023), the most prevalent methodological use of the GEE (22%) was for the evaluation and prediction of water resources. Imagery data collected from the Sentinel-2 satellite with a spatial resolution of 10 m for B2 (band 2 with Blue Color bandwidth), B3 (band 3 with Green Color bandwidth), B4 (band 4 with Red Color bandwidth), and B8 (band 8 with Near Infrared bandwidth)

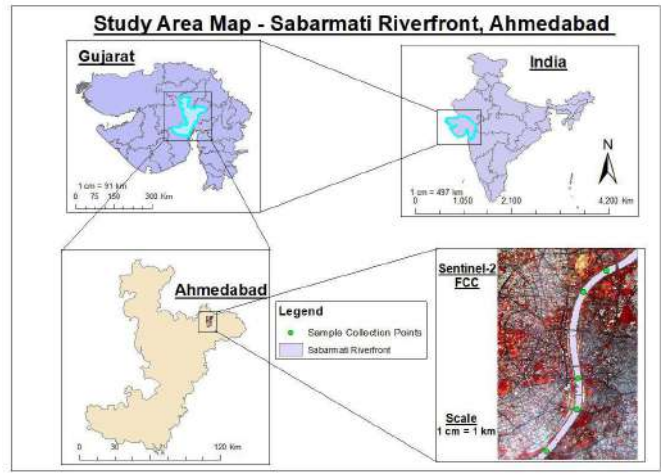

Fig. 1: Study Area Map of Sabarmati Riverfront showing with Sentinel –2 FCC satellite image of Sabarmati Riverfront.

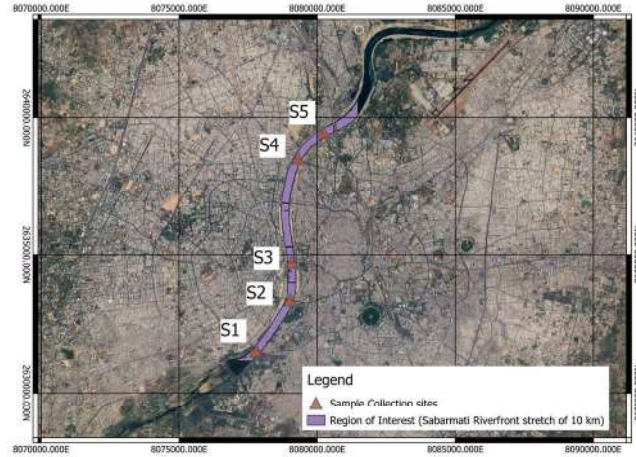

Fig. 2: Representing the sample collection sites over approximately 10 km Sabarmati Riverfront, Ahmedabad, as a region of interest.

Table 2: Represents the sample collected for the water quality parameters

S. No.	Abbreviation	Parameters	Unit
1.	pH	pH	pH Units
2.	Tur	Turbidity	$mg.L^{-1}$
3.	EC	Electrical Conductivity	$\mu mho.cm^{-1}$
4.	TSS	Total Suspended Solids	$mg.L^{-1}$
5.	TS	Total Solids	$mg.L^{-1}$
6.	BOD_5	Biochemical Oxygen Demand of 5 days	$mg.L^{-1}$
7.	DO	Dissolved Oxygen	$mg.L^{-1}$
8.	TP	Total Phosphorous	$mg.L^{-1}$
9.	FC	Fecal Coliform	$mg.L^{-1}$
10.	NH_3	Ammonia	$mg.L^{-1}$

Table 3: Representing the dates of sample collection through Field and Satellite.

Field Sample Collection Date	08th Feb 2024	09th March 2024	10th April 2024
Satellite Image Collection Date	06th Feb 2024	07th March 2024	06th April 2024

were used to evaluate the water characteristics in line with the field collection data sample dates, as shown in Table 3.

Methodology

Approach (Relationships Between Remote-Sensing and Field-Based Water Quality Parameters)

Many studies have been conducted with combinations of various bands, either individually or as ratios of the entire visible wavelength region, to monitor the variation in the spectral response (Doxaran et al. 2005, Vakili & Jamil 2020). The present study was performed with the band range from Blue to NIR to monitor the variation in the spectral response due to a change in the various water parameters.

The methodology can be categorized into three major stages, as represented in Table 4 and Fig. 3. This study illustrated a decision-making system for water quality monitoring with Sentinel-2 satellite images using the Google Earth Engine (GEE) platform, with a particular emphasis on temporal and spatial fluctuations along Ahmedabad's Sabarmati Riverfront. The Sentinel-2 data were obtained in February, March, and April of 2024, ensuring that the dates of the imagery corresponded with the dates of the collection of water samples. This made it possible to precisely record seasonal fluctuations in the quality of the water. Five different locations along the Sabarmati Riverfront were used for the study to capture the spatial heterogeneity in the river's water properties, as shown in Fig. 2.

The primary phase of the procedure is to access and pre-process Sentinel-2 imagery for the chosen months of 2024 via the Google Earth Engine (GEE) platform. With its 13 spectral bands, Sentinel-2's Multispectral Instrument (MSI) is especially well suited for tracking important water quality metrics, with this study focusing on 10 water quality parameters as shown in Table 2.

Regression Model Using Spectral Properties

By examining the spectral reflectance characteristics of satellite images, remote sensing technology offers an effective way to estimate water quality indicators. Sentinel-2 provides useful information for developing correlations between spectral indices and indicators of water quality because of its multispectral bands. The spectral properties of the water body are extracted and analyzed using zonal statistics, which compute the statistical values as the mean, median, standard deviation, variance, minimum and maximum of the reflectance values for the various bands as B2, B3, B4, and B8 bands along with the combination of bands such as B2/B3, Normalized Differential Water Index (NDWI) and Normalized Differential Turbidity Index

Table 4: Represents the main stages for the determination of Water Quality parameters.

Stage	Description
1	Use the GEE platform to filter images from Sentinel 2 collections of February 2024, March 2024 and April 2024, sync with the in-situ sample collection dates.
2	Applying the water index NDWI to automatically identify the water surface that qualifies for processing using the GEE platform.
3	Evaluate the water quality parameters value for each valid image by applying the developed correlation expression using pixel values of various bands and their combination with field-measured data through a regression model.

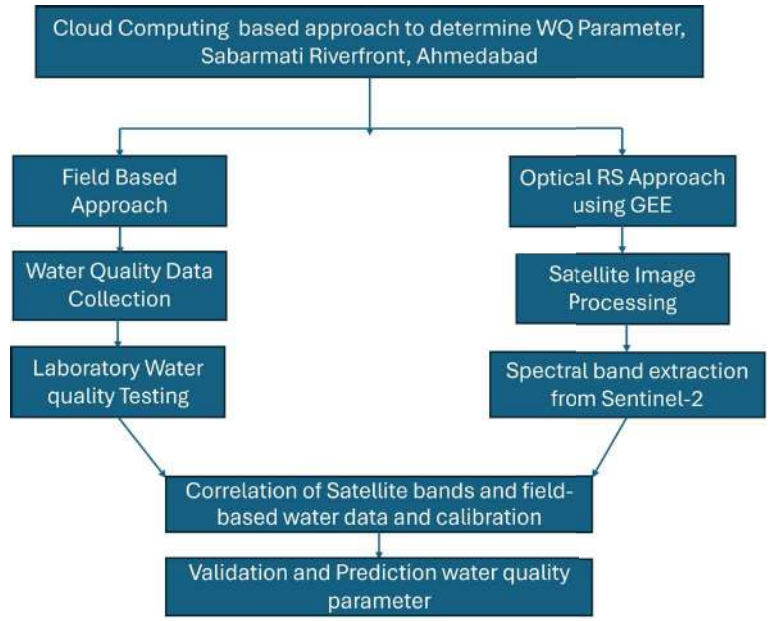

Fig. 3: A broad framework of methodology.

Table 5: Representing the area of the polygon plotted to extract zonal statistics at various stations.

Polygon Station	S1	S2	S3	S4	S5
Area [m^2]	41063.853	40519.885	28578.369	50656.604	46731.693

(NDTI) spectral band at all 5 stations. The zonal statistics were extracted based on the considered region near each station of sample collection, as shown in Table 5.

$$NDWI = (B3 - B8)/(B3 + B8) \quad \ldots(i)$$

$$NDTI = (B4 - B3)/(B4 + B3) \quad \ldots(ii)$$

These mean values of reflectance were used as data inputs to create empirical models. Furthermore, a correlation study was performed to determine the relationship between the spectral data obtained from the satellite and the 10 water characteristics measured in the laboratory. Field samples were taken at each of the five locations along the Sabarmati Riverfront throughout the designated months to obtain ground truth data for water characteristics.

The linear regression model can be created via the Data Analysis tool in Microsoft Excel after the data has been organised, with each parameter being modelled separately. The output consists of an R^2 value that indicates the goodness of fit of the model and regression coefficients that show how each spectral band contributes to the prediction of the water quality metrics. Higher R^2 values indicate a more robust correlation between the reflectance data and the parameter being modelled for water quality, as shown in Table 6. Empirical models based on this association are then used to forecast water characteristic concentrations throughout the study area by applying them to spectral data. The created expressions are utilised to estimate water characteristic concentrations within the GEE, and the results are mapped and displayed to produce spatially explicit water quality information.

By comparing the estimates obtained from satellite data with independent ground truth data of water characteristics, the precision of these predictions is validated. The models' performance is evaluated by computing the statistical metric R^2. Modifications are applied as needed to improve the models' prediction power. Using Sentinel-2 imagery through the Google Earth Engine, this system captures both spatial fluctuations along Ahmedabad's Sabarmati Riverfront and temporal fluctuations over the designated months, demonstrating the effective and scalable application of these tools for water quality monitoring. This approach offers insightful information for regional environmental management and decision-making.

RESULTS AND DISCUSSION

Relationships Between Remote-Sensing Data and Field-Based Water Quality Parameters

The geospatial images of the Sentinel-2 satellite data are utilized to depict the geographical distributions of the water

Table 6: Representing the correlation expression developed using measured ground truth data and mean spectral reflectance values.

S. No.	Parameters	Expressions
1.	pH	$31.46 - 0.0296 \times B2 + 0.0584 \times B3 - 0.0226 \times B4 - 0.0088 \times B8 - 22.446 \times \left(\frac{B3}{B2}\right) - 16.97 \times NDWI + 52.249 \times NDTI$
2.	Turbidity	$-2311.71 + 2.0317 \times B2 - 2.284 \times B3 + 0.60877 \times B4 - 0.0448 \times B8 + 1968.82 \times \left(\frac{B3}{B2}\right) - 441.042 \times NDWI - 2052.5 \times NDTI$
3.	Electrical Conductivity	$9007.85 - 7.87 \times B2 + 10.2 \times B3 - 3.86 \times B4 + 0.529 \times B8 - 7764.93 \times \left(\frac{B3}{B2}\right) + 1207.43 \times NDWI + 9502.98 \times NDTI$
4.	Total Suspended Solids	$-536.082 + 0.68294 \times B2 - 1.45326 \times B3 + 3.21347 \times B4 - 2.2606 \times B8 - 5338.93 \times NDWI - 7511.93 \times NDWI - 7511.8 \times NDTI + 392.034 \times \left(\frac{B3}{B2}\right)$
5.	Total Solids	$-791.931 - 45318.7 \times NDTI - 15.8 \times B8 - 40432 \times NDWI + 16.65 \times B4$
6.	BOD_5	$159.613 - 0.2488 \times B2 - 0.52 \times B3 + 1.577 \times B4 - 0.802 \times B8 - 177.88 \times \left(\frac{B3}{B2}\right) - 2053.87 \times NDWI - 4061.06 \times NDTI$
7.	DO	$-2681.63 + 2.33 \times B2 - 3.524 \times B3 + 1.879 \times B4 - 0.3 \times B8 + 2249.49 \times \left(\frac{B3}{B2}\right) - 945.11 \times NDWI - 4727.05 \times NDTI$
8.	Total Phosphorous	$419.5 - 0.372 \times B2 + 0.624 \times B3 + 0.0057 \times B4 - 0.31 \times B8 - 358.8 \times \left(\frac{B3}{B2}\right) - 760.23 \times NDWI - 71.325 \times NDTI$
9.	Fecal Coliform	$1090.826 - 1.14 \times B2 + 1.414 \times B3 + 1.56 \times B4 - 1.97 \times B8 - 944.97 \times \left(\frac{B3}{B2}\right) - 4997.83 \times NDWI - 4323.97 \times NDTI$
10.	Ammonia (NH_3)	$628.69 - 0.577 \times B2 + 0.655 \times B3 - 0.036 \times B4 - 0.114 \times B8 - 547.31 \times \left(\frac{B3}{B2}\right) - 303.51 \times NDWI + 36.87 \times NDTI$

quality metrics in three separate months (2024): February, March, and April. A crucial resource for evaluating the temporal and geographical changes in the river's water quality along the Sabarmati Riverfront is provided in this section. Fig. 4 for Optically Active Water Characteristics and Fig. 5 for Non-Optically Active Water Characteristics depict the distributions of these images, which show important patterns associated with both natural and human-caused processes, as well as fluctuations in each water characteristic. A deeper and thorough examination of each figure is given in Table 7 for Optically Active Water Characteristics and in Table 8 for the Non-Optically Active Water Characteristics, with particular attention given to the trends in spatial distribution and any possible ramifications for the management of water resources.

Overall Analysis

The spatial representations highlight the distribution of important water quality indicators, such as pH, turbidity, EC, Total Suspended Solids (TSS), Total Solids (TS), Total Phosphorus (TP), Fecal Coliform (FC), Biochemical Oxygen Demand (BOD), Dissolved Oxygen (DO), and Ammonia (NH_3). For tracking changes over time, each satellite image is shown for a particular date every three months. Effective management of water resources and identification of pollution sources are made possible by this temporal analysis, which provides thorough knowledge of how water quality metrics change over the course of many seasons.

- The satellite imagery of February 2024 shows that certain metrics, such as pH and EC, have rather uniform

Table 7: Represents the comparative analysis of optically active water characteristics.

Parameter	Months	Observations	Key Insights
EC	February	Increased EC readings in several river segments point to the possible presence of dissolved salts.	• Salinity and pollution from dissolved ions are well-indicated by EC.
	March	The conductivity somewhat decreased, presumably because of early rainfall dilution, although there are still some isolated hotspots.	• Management methods should concentrate on reducing salt levels, particularly during peak runoff times, as reflected by the seasonal changes in EC.
	April	Increased EC readings in several river segments point to the possible presence of dissolved salts.	
Turbidity	February	Significant increases in suspended particle matter.	• Excessive turbidity frequently signals problems with water quality, including decreased light penetration, which endangers aquatic life.
	March	The turbidity level has somewhat decreased, indicating that some sedimentation has taken place.	• Decreasing level indicates that some sedimentation had taken place.
	April	The turbidity values are relatively modest, suggesting cleaner water.	
TSS	February	TSS levels exhibit a decreasing trend from February to April, peaking in certain zones, especially in February.	• Decreases in TSS have the potential to harm aquatic ecosystems by suffocating habitats, increasing photosynthesis, and causing nutrient overload (Bilotta and Brazier 2008).
	March		
	April		
TS	February	The high TS levels are indicative of runoff and pollution sources' suspended particulates, as well as their dissolved salts	• TS distribution also exhibits a pattern similar to that of the TSS
	March	The chart for March shows some respite in the dissolved loads.	• High TS can deteriorate the quality of water for ecological and drinking reasons (Saalidong et al. 2022).
	April	Hotspot zones, especially in April, might be related to runoff or human activity upstream of the riverfront.	

distributions, but other parameters, including TSS and turbidity, show more localised variability. The maps show places that need more research as possible hotspots for pollution, where specific characteristics differ from typical values.

- There appears to be a shift in the distribution of water quality in the images from March 2024, which might be attributed to fluctuations in seasonal patterns, human activities, or rainfall. The influence of runoff and biomass, for example, appears to be reflected in the fluctuations in turbidity and TSS concentrations, particularly in populated areas along the riverside.
- The geographical patterns of water quality metrics continue to evolve by April. Desirable levels of BOD, FC, and NH_3 in particular regions might indicate stable or lower pollution levels. On the other hand, certain regions have relatively high DO levels, suggesting the potential for effective interventions or natural healing processes.

An extensive dataset for comprehending the spatial and temporal variations in the water quality of the Sabarmati Riverfront is created on the basis of geospatial images of its characteristics. Anthropogenic activities, including biomass and urban runoff, may be the main causes of the notable seasonal fluctuations that are observed across numerous parameters, notably between the months of February, March, and April. These deep insights identify regions that need targeted management measures and highlight key times when water quality is most in danger. The status of the river system can be considered healthy and may be maintained by water resource management by addressing the causes of pollution and putting targeted measures into place in the future.

Accuracy Assessment

It is crucial to test the model's performance with a fresh dataset after it has been calibrated. As a result, the model revealed positive forecast parameters related to water quality in various scenarios, such as pH with $R^2 = 0.8$, TP with $R^2 = 0.75$, and TSS with $R^2 = 0.76$. The model offers an economical and effective way to continuously monitor water quality by applying it to Sentinel-2 images taken at different times after it has been validated.

The estimations of various parameters produced from satellite data are compared with experimentally observed values to validate the water quality prediction model. Graphics are used, as shown in Fig. 6, to visualise the comparison and offer insights into the model's performance across various parameters, including pH, Turbidity, Electrical Conductivity (EC), Total Suspended Solids (TSS), Total Solids (TS), Dissolved Oxygen (DO), Biochemical Oxygen Demand (BOD), Total Phosphorus (TP), Fecal Coliform (FC), and Ammonia (NH_3). When assessing the degree to which the model's predictions and the actual measurements agree, the R^2 values are essential. The general connection between the expected and actual values is shown by the trend line in each graph, which provides information about the linearity and dependability of the model.

Table 8: Represents the comparative analysis of Non-Optically Active Water Characterstics.

Parameter	Months	Observations	Key Insights
pH	February	The pH distribution seems very regular in February, indicating a steady chemical composition on the other side of the river.	• pH levels fluctuate, ranging from almost neutral to slightly alkaline.
	March	Shows some geographical variation, especially in places where urban runoff could affect the quality of the water	• Small pH variations indicate chemical contamination or increased runoff in the early stages, thus places exhibiting these changes should be regularly watched to prevent additional damage.
	April	Slight alkalinity increases in temperature as April approaches may be a sign of increased biological activity	
BOD	February	Rising BOD levels point to organic material entering the river	• The observed BOD levels are low, and, in a few cases, they are below the detection level according to the data collected from the field.
	March	In the summer time, organic pollution is suggested by the low March and April readings.	• Decreased BOD levels do not endanger aquatic life because they maintain the amount of oxygen in the water (Chapra et al. 2021).
	April		
DO	February	A lower BOD level can correlate with higher DO levels as lower organic matter breaks down and less oxygen is consumed, resulting in the spatially defined oxygen zones observed in these months.	• Across the three months, higher DO levels correlated with lower BOD levels.
	March		• Nugraha et al. (2020) suggests lower organic input treatments are necessary, particularly in warmer months when oxygen depletion might be more severe
	April		
TP	February	Comparatively, more levels of TP dispersion are observed in February.	• Elevated total phosphorus levels are a major cause of algal blooms, which can severely deteriorate the quality of water (Li et al. 2022).
	March		
	April		
FC	February	A considerable increase in fecal coliform levels was observed, especially in upstream areas, which suggests that rainwater runoff or raw sewage are the main possible causes.	• These results highlight how crucial it is to enhance water treatment strategies and management, where intake structures are planned to be placed to avoid overloading with pollution.
	March	March's comparatively low FC readings indicate that contamination may have been lower during the month's dry spell.	• Seasonal increases in FC point to possible threats to human health, particularly in locations where the river is used for leisure (Guangzhi et al. 2022).
	April	A considerable increase in fecal coliform levels was observed.	
NH_3	February	The elevated levels in February might indicate increasing nutrient contamination.	• High ammonia levels may poison aquatic life (Edwards et al. 2024).
	March	Low spatial distribution of ammonia in March & April.	
	April		

A strong match is shown by the R^2 value of 0.802, which shows that the model explains 80.2% of the variation in pH. Past studies have shown a positive relationship and high accuracy in evaluating the remote sensing–based derived pH (Pereira et al. 2020, Jiang et al. 2022). With an R^2 of 0.38, the EC graph indicates that 38% of the variation in EC can be explained by the model, demonstrating a weak connection between the observed and predicted values. The turbidity graph, with an R^2 of 0.58, indicates a moderate correlation between the observed and expected values. The model accounts for 58% of the turbidity variability, suggesting that it accurately describes the overall trend but has difficulty explaining very murky waters. The model accounts for 76.5% of the variability in the TSS, according to the TSS validation graph, which has a strong R^2 of 0.765.

The Total Solids (TS) graph's R^2 value is 0.621. Although the trend line is almost straight, it exhibits a slightly underestimated slope at higher TS levels, much like the TSS. The model's inability to fully capture the extent of organic pollution is indicated by the BOD graph, which has an R^2 of 0.362 and indicates lower predictive accuracy. The trend line in the validation graph for DO underestimates the concentration of Dissolved Oxygen in low-oxygen areas but fits rather well at higher oxygen levels, with a modest R^2 value of 0.614. An R^2 of 0.747 indicates a strong association in the TP validation graph. The model accounts for 45.1% of the variability in FC levels, according to the Fecal Coliform graph, which has an R^2 of 0.451. The validation graph for Ammonia (NH_3) has an R^2 value of 0.31, again suggesting a lower level of model performance.

The validation findings demonstrate, as shown in Table 9, that the model performs well for a variety of parameters, including pH, TSS, and TP, with R^2 values often over 0.7, suggesting a robust match. Nevertheless, the

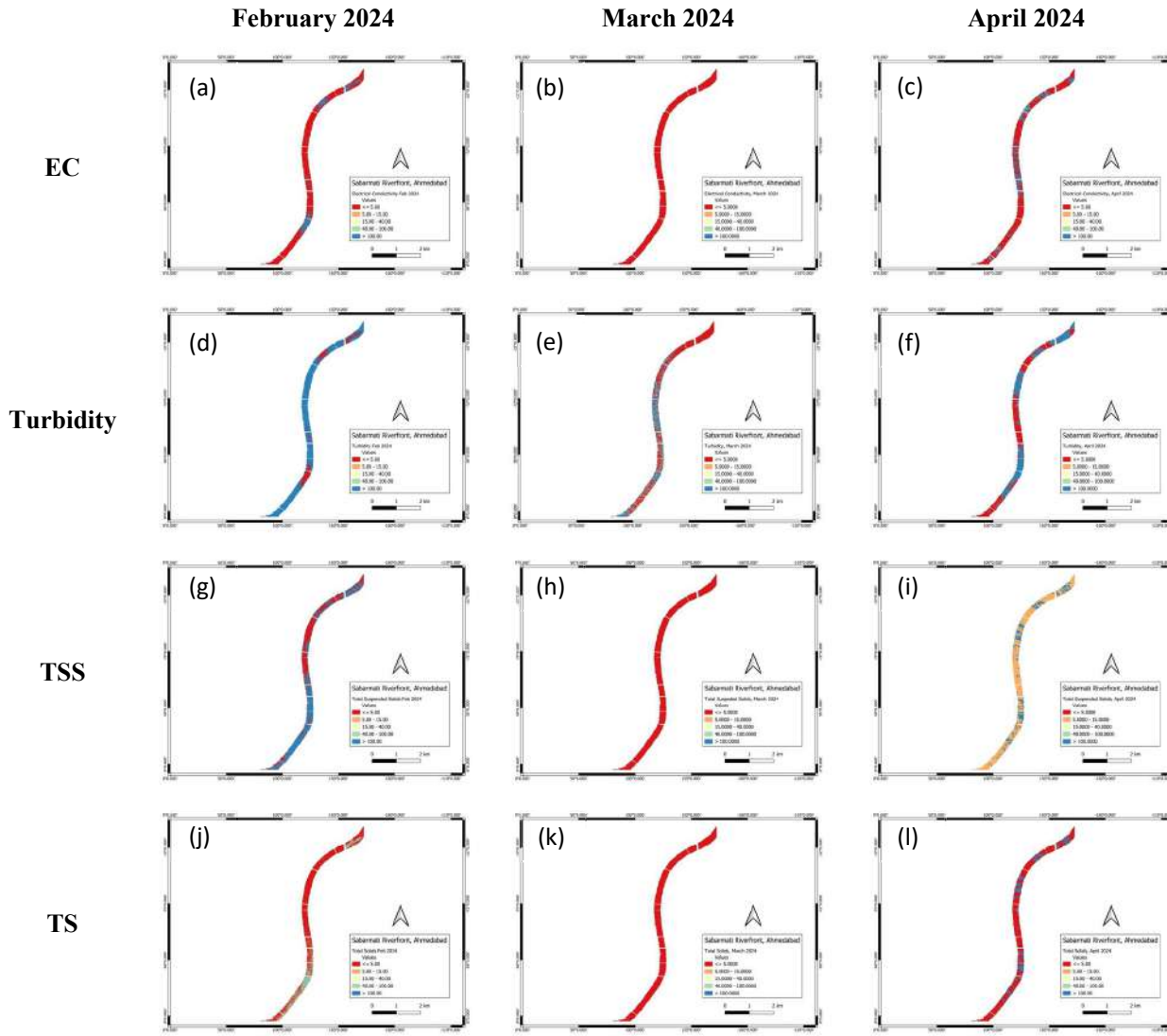

Fig. 4: Represents the spatial distribution of optically active water characterstics.

model displays more notable departures from the trend line for parameters such as NH_3, BOD, FC, and DO, with R^2 values ranging from 0.31 to 0.62, indicating the need for greater refinement. The trend line analysis shows that although the model performs well in most situations when there is a high concentration of physical contaminants, it finds it difficult to represent linear connections in other instances. As a result, even if the model works well for assessing water quality generally, further calibration or the use of nonlinear modelling approaches would be required to enhance forecasts in regions with more severe environmental circumstances. The model's prediction accuracy over a wider variety of scenarios might be greatly improved by including more advanced regression techniques or machine learning algorithms.

CONCLUSIONS

This study validated the efficacy of an integrated strategy to assess water quality at Ahmedabad's Sabarmati Riverfront, utilising both in situ measurements and remote sensing–derived data. By using multitemporal remote sensing data, natural resource managers can benefit from the integrated approach, which is an affordable technology that has been demonstrated to be a valuable source of data for defining the water quality status of Ahmedabad's Sabarmati Riverfront. Additionally, changes in water quality can be analysed through cloud-based accessibility of the data, which can quickly aid in the assessment of environmental issues and possible health hazards. Municipal authorities may face challenges in selecting locations to develop water intake

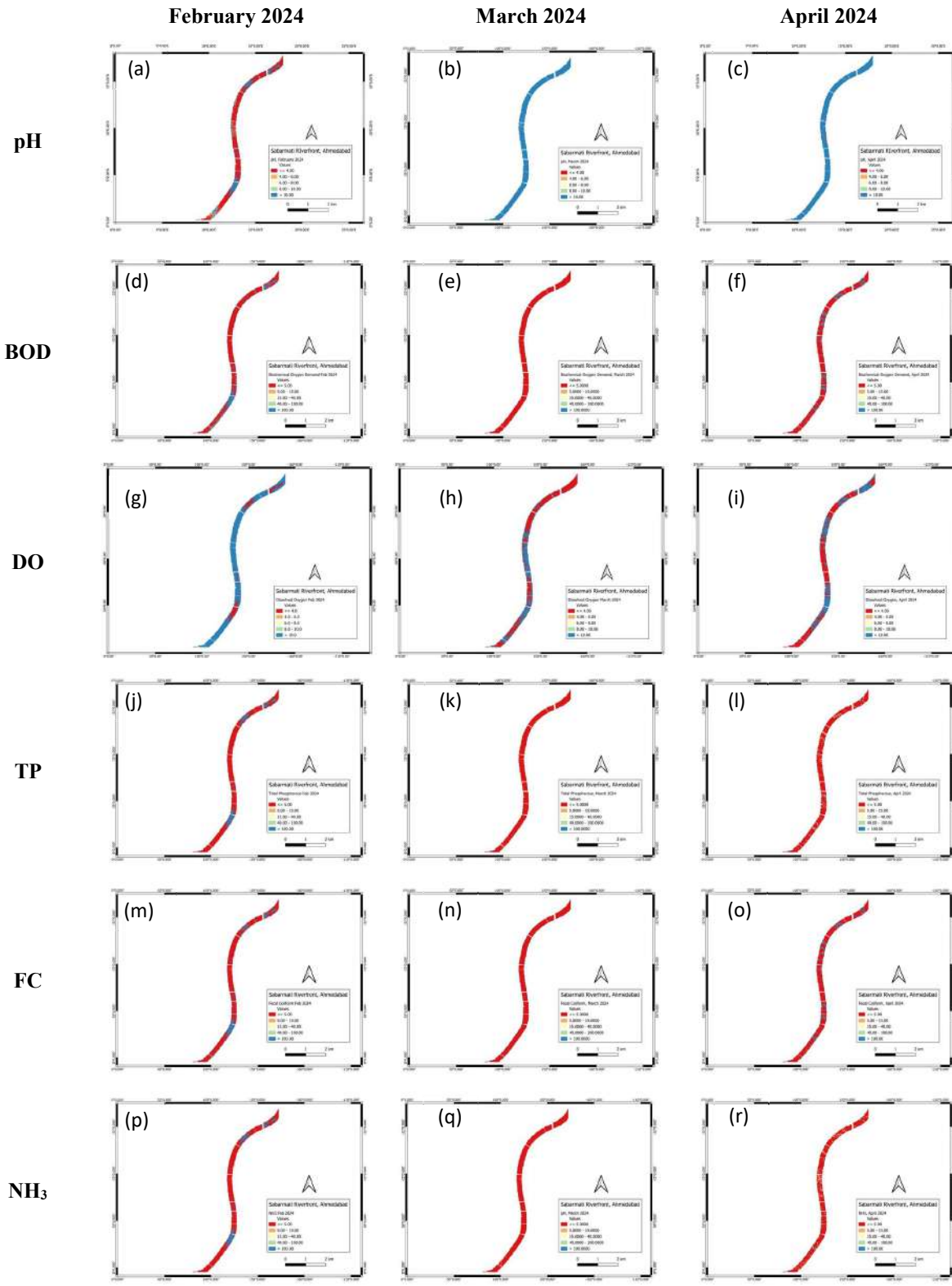

Fig. 5: Represents the spatial distribution of non-optically active water characterstics.

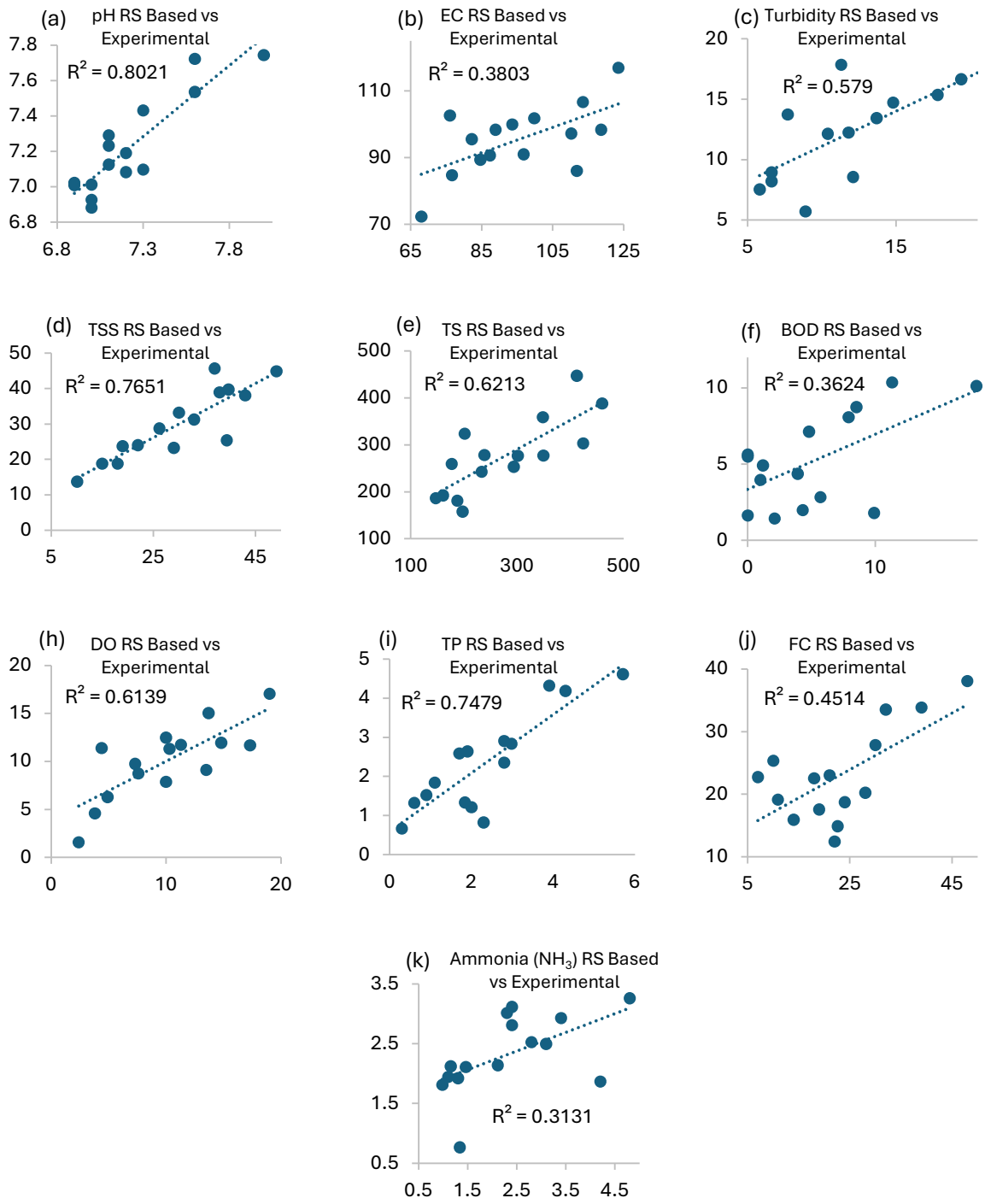

Fig. 6: R^2 values for the accuracy assessment of remote-sensing derived v/s experimental water quality concentrations.

structures that supply water to treatment plants. The chosen location should ideally be on a river stretch with lower levels of pollution. Through this approach, using GIS visualisations, authorities can identify spatial water quality indicators and select sites with the lowest pollution levels, ensuring a more effective and safer water supply.

The following future scope can be considered as a possibility: To develop a time series of water quality parameter monitoring data integrated with precipitation data and change detection in land-use–land-cover patterns for better decision-making regarding variation in water characteristics. Also, to enhance prediction accuracy, future

Table 9: Summarizing R² for all parameters

S. No.	Parameters	R^2 Range	Classification
1.	pH, TSS, TP,	>0.7	Good
2.	Turbidity, TS, DO,	0.5 – 0.7	Moderate
3.	EC, BOD, FC, NH_3	<0.5	Weak

studies can integrate machine learning algorithms such as Random Forest (RF) and Support Vector Regression (SVR) for handling high-dimensional spectral data with nonlinear relationship modelling between spectral indices and water quality parameters. Using remote sensing techniques, sediment transport and erosion patterns can also be studied to understand their effects on turbidity and total suspended solids. On the other hand, the limitations faced during the study are that the measured spectral reflectance data should be obtained after thorough spectroscopic experimental comparisons. However, the spectral range was considered to be multispectral Sentinel-2, whereas the correlation can be developed via hyperspectral remote sensing images. Real-time monitoring and analysis of water quality can help prevent pollution, safeguard ecosystems, and protect public health by enabling early detection of issues. This approach not only aids in water quality assessment but also supports decision-making related to site suitability, offering a valuable tool for regional planning and management.

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available upon reasonable request.

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What Remains When Territory Disappears? On the Possibility of Climate Sovereignty

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ABSTRACT

As rising sea levels threaten to render low-lying island states uninhabitable, international law faces an urgent dilemma - whether statehood persists without territory or not. The traditional idea of statehood is called into question if increasing sea levels submerge an entire nation's landmass. Traditional legal frameworks, anchored in the territorial criteria outlined by the Montevideo Convention, provide no definitive guidance on this unprecedented scenario. This article proposes "climate sovereignty," a novel theoretical framework designed to address the challenges of climate-induced territorial loss. Climate sovereignty redefines statehood beyond fixed territory, emphasizing the continuity of a people, their governing institutions, and collective identity, even when physical territory is submerged or uninhabitable. By shifting international legal recognition from a land-centric approach to a community-based framework rooted in self-determination, climate sovereignty offers a legal pathway responsive to the evolving realities of vulnerable states. Through illustrative cases of Tuvalu and Kiribati, this article demonstrates the normative justification and legal viability of recognizing deterritorialized statehood within contemporary international law. Ultimately, this article seeks to advance international law's response to an unprecedented existential threat, advocating for proactive recognition mechanisms and urging a fundamental reconsideration of sovereignty itself. It argues that, in a climate-altered world, nationhood must not disappear alongside territory.

INTRODUCTION

Climate-induced sea-level rise is compelling international law to address previously overlooked questions. In low-lying island states such as Kiribati and Tuvalu, the rising ocean is not only eroding coastlines but also undermining the foundations of legal identity (Yamamoto & Esteban 2013, IPCC 2021). What happens to a state when the land that defines its territory is gradually submerged? This is no longer an abstract thought experiment. An unfolding reality calls into question the basic assumptions behind the legal concept of statehood. At the center of this dilemma lies the Montevideo Convention's definition of a state, which requires a permanent population, a defined territory, a government, and the capacity to engage in international relations (Montevideo Convention 1933, Crawford 2006). Among these, territory has traditionally been viewed as indispensable. Yet this assumption, rarely scrutinized until recently, now appears fragile. The Montevideo model begins to falter when applied to communities whose land is becoming physically uninhabitable. The Convention does not account for what happens when the loss of territory is not due to war, annexation, or legal extinction, but the slow advance of the sea. The legal consequences are potentially severe: the loss of international personality, the forfeiture of maritime rights, and the dissolution of treaty capacity (Stoutenburg 2015). While the international legal order has responded to many forms of crisis over the last century, it has not yet developed a consistent or coherent response to the possibility that a state might physically disappear, even while its people, government, and political identity remain intact. Various proposals have emerged in

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response to this looming legal vacuum (Docherty & Giannini 2009, Carlarne 2014). Some suggest preserving maritime baselines regardless of physical land loss, while others propose maintaining state recognition even after a state's population relocates. There are also discussions around governments in exile, the creation of digital nations, and fixed zones for legal continuity. These ideas reflect growing awareness, but they often stop short of addressing the core problem: most of these approaches rely on exceptional recognition, political goodwill, or provisional workarounds. Few offer a principled account of how sovereignty itself might be redefined when territory is no longer a given.

This article takes that question as its starting point. It proposes the concept of climate sovereignty—a theoretical framework for understanding how a state may retain its legal identity even in the absence of permanent, habitable territory. Rather than treating territorial loss as the end of statehood, this approach suggests a shift in legal focus from the physical dimensions of sovereignty to its functional and relational aspects (Miller 2007). Climate sovereignty emphasizes the continuity of a people and the institutional structures that allow them to act collectively, regardless of geography. It offers a structured alternative to ad hoc solutions, grounded in the idea that sovereignty, at its core, is not simply about land but about political self-determination and the ongoing capacity to represent a community under international law. The idea that sovereignty could be decoupled from territory is not entirely without precedent. International law has, on rare occasions, recognized the continued existence of states or governing entities despite the loss of effective territorial control. Historical cases, such as governments in exile during wartime or the legal personality of the Holy See, demonstrate that a stable land base is not always a precondition for recognition (Burkett 2011). Yet, these examples have generally been treated as anomalies, justified by unique political or religious circumstances rather than as signals of a broader legal possibility. More importantly, they have not offered a coherent framework for how international law might respond to slow-onset, irreversible territorial disappearance caused by environmental change.

Climate sovereignty seeks to address that gap. It draws from the intuition that sovereignty is ultimately about people—their ability to act collectively, preserve their identity, and participate in global affairs—even when their physical environment can no longer sustain them. In doing so, it reframes sovereignty not as a static condition tied to geography but as a set of relationships: between people and institutions, between governments and legal systems, and between displaced nations and the international community (Miller 2007). This approach does not reject the foundations of statehood but suggests that they can and must evolve. The idea is not to dissolve the link between law and land entirely but to recognize that in the face of climate-induced loss, that link cannot remain absolute. The legal challenges of territorial loss are inseparable from the human consequences it brings. In many Pacific societies, land is not only a political resource but a living foundation for cultural identity, ancestry, and belonging (Farbotko & Lazrus 2012). When territory disappears, what is at stake is not just sovereignty in the formal sense but the continuity of meaning that land holds for the people who inhabit it. Existing legal frameworks for displacement or statelessness offer little protection for that kind of loss. While the right to self-determination is recognized under international law, its application presumes that a people still possess a space from which to exercise it. The case of climate-threatened states complicates this assumption. It calls for legal thinking that can bridge institutional continuity and cultural survival—two aspects of sovereignty that have rarely been addressed together. This article takes climate sovereignty as a starting point for that effort. It develops the concept not as a technical fix but as a way to rethink the relationship between people, territory, and law in the context of climate disruption. The following sections explore how international law has traditionally linked statehood to land, why those links are being stretched to their limit, and how a shift in conceptual focus might offer a more just and durable response. Rather than offering a single blueprint, the article aims to contribute to an ongoing conversation about how sovereignty must adapt so that the disappearance of land does not erase the rights, identity, or recognition of the people who called it home.

WHY EXISTING LEGAL FRAMEWORKS FALL SHORT

The prospect of state disappearance due to climate change has prompted a range of legal efforts to repurpose existing frameworks in the hope of preserving sovereignty beyond territory. These proposals reflect more than institutional pragmatism; they express a normative conviction that international law, despite its limitations, must provide stability when statehood's physical foundations erode. Yet while elements of the legal system have responded in part, none fully resolve the structural question at hand. They address symptoms-maritime loss, population displacement, institutional disruption—but do not supply a cohesive account of how statehood endures when territory vanishes. What follows is a review of these partial responses and the limits that render them insufficient.

UNCLOS

Among the more widely discussed proposals is the effort to fix maritime baselines under the UN Convention on the

Law of the Sea (UNCLOS). Low-lying island states face a double threat: not only the disappearance of land, but also the collapse of maritime zones- territorial seas, exclusive economic zones (EEZs), and continental shelves, whose legal status depends on coastlines. Under current rules, baselines are ambulatory, shifting as the coastline retreats. This could result in the cascading loss of maritime entitlements triggered by environmental change. In response, several Pacific Island states, backed by legal scholars, have advocated for "freezing" existing baselines and maritime claims, regardless of future inundation (Rayfuse 2010, Pacific Islands Forum 2023).

This proposal is both practical and normatively resonant. As Mayer (2016) and Zahar (2017) note, the ambulatory baseline rule was never designed for gradual, irreversible submersion. Locking in maritime boundaries could help protect not only economic rights but also symbolic continuity, allowing threatened states to retain a presence in international law. Some island states have already declared an intention to preserve maritime zones permanently, even as land disappears. This reflects a shift toward legal stability over geographic precision-a logic grounded in equity and environmental justice (Wang 2023). Still, fixed baselines address only part of the problem. They may secure access to marine resources, but cannot by themselves sustain the legal identity of a disappearing state. Even if UNCLOS is interpreted or amended to accept frozen claims, the sovereignty question remains unresolved. Zahar (2017) cautions that treating EEZs as freestanding rights detached from a viable state risks reducing sovereignty to mere extractive entitlement. Moreover, UNCLOS is silent on questions of population, governance, or legal personality. A state may retain a maritime footprint while lacking the institutional and social grounding that underpins international subjectivity. In this light, baseline strategies are protective but partial.

Governments in Exile

Other approaches look to historical precedents of displaced governance. The example of governments-in-exile, especially during the Second World War, suggests that states may persist without territory, provided their institutional structures remain intact. Exiled regimes such as those of Poland, Norway, and France retained international recognition and operated diplomatically despite occupation. By analogy, scholars have proposed that island states like Tuvalu or Kiribati could relocate their governments abroad and continue to function as sovereign actors (McAdam 2012). This reasoning draws on a longstanding presumption in international law: that once statehood is established, it is not easily undone. Talmon (1998) and Vidmar (2012) have documented cases in which continuity was preserved despite ruptures in territory, population, or government. A government-in-exile that issues passports, signs treaties, and represents a polity may, in theory, maintain legal identity. Under this model, sovereignty becomes portable, lodged in institutions rather than land.

Yet here, too, the analogy frays under closer scrutiny. Wartime exile assumed a temporary absence and eventual return. Climate-induced disappearance allows no such assumption. When land is not occupied but lost, the legal fiction of deferred sovereignty loses plausibility. Crawford (2006) notes that while international law accommodates disruption, it offers few precedents for indefinite statehood in the absence of core attributes. Practical governance challenges compound the legal uncertainties. How would an exiled government legislate, enforce, or engage a population dispersed across multiple jurisdictions? Over time, integration into host societies may dilute collective identity and undermine political cohesion. Sovereignty may persist formally while becoming hollow in function. As the institutional links between people and government weaken, recognition may preserve a name but not a polity. In light of these difficulties, some have proposed shifting the legal focus from collective sovereignty to individual dignity. Human rights frameworks offer a different mode of continuity, grounded in the protection of persons rather than the persistence of states. These frameworks ensure that even if a state disappears, its people retain certain protections under international law (Knox 2009).

Human Rights Law

Yet these protections are incomplete. Human rights law is designed to safeguard individuals, not collectivities. It conceptualizes climate displacement as a humanitarian crisis, not a constitutional rupture. Most climate-displaced persons fall outside the scope of the 1951 Refugee Convention, which requires a showing of persecution. As McAdam (2012) observes, this leaves many without access to effective international protection. Recent developments point to limited progress. The Nansen Initiative and the Platform on Disaster Displacement promote cooperative frameworks for environmental migrants. The UN Human Rights Committee has suggested that non-refoulement obligations may apply where return would expose individuals to uninhabitable conditions (UNHRC 2020). But these are soft law instruments-fragmented, non-binding, and oriented toward individuals. They provide no clear legal basis for maintaining a people as a political community once their constitutional order is lost.

The structural limits become clearer in practice. Resettlement depends on the discretion of the receiving states. There is no right to relocate with one's legal system, language, or institutional continuity intact. As Mayer (2016) notes, human rights regimes are well-suited to protecting persons from the state but poorly equipped to protect the state itself. A displaced population may survive but may no longer function as a political subject. In this light, statelessness, both legal and symbolic, emerges not just as a risk but as the likely outcome, especially when international law fails to protect collective identity beyond territorial attachment (Anaya 1996). For some scholars, recognition offers a last resort. Sovereignty, they argue, might persist if the international community is willing to treat deterritorialized states as continuing entities. Burkett (2011) suggests that "nations ex situ" could survive through sustained recognition, even absent territory. This view finds partial support in the experience of entities like the Holy See or various governments-in-exile, which have maintained legal presence despite institutional rupture (Crawford 2006, Vidmar 2012). But these cases were either temporary or exceptional. They do not constitute a generalizable doctrine. Recognition is not a legal right-it is a political decision. No state is obligated to recognize another once it ceases to meet conventional criteria for statehood. Even where granted, recognition does not restore full political function. It may preserve formal visibility-a seat at the UN, participation in treaties, but cannot guarantee effective governance or cultural continuity. The sovereign may remain visible, but its institutional reality may dissolve. Most critically, recognition provides no legal security. It can be extended, withdrawn, fragmented, or ignored. As Wang (2023) cautions, symbolic solidarity can fade into procedural silence. If recognition becomes the sole support for state survival, its fragility becomes a liability.

The Territorial Assumption in International Law

International law has never formally declared that a state must have territory to exist. But in practice and doctrine, that assumption runs deep. It is present in the way maps organize political space, in how treaties define borders, and in the legal architecture of sovereignty that emerged after Westphalia (Shaw 2017). Territory, more than any other element, has anchored the idea of statehood in both symbolic and functional terms. The Montevideo Convention of 1933 made that anchor explicit. Alongside population, government, and the capacity to enter into relations with other states, it named "a defined territory" as a requirement for statehood (Montevideo Convention 1933, Art. 1). Though the treaty was regional in origin, its criteria came to serve as a shorthand for the customary legal understanding of what a state is—or must be (Shaw 2017). Behind that codification lay a long lineage of legal thought. Max Huber, in the Island of Palmas arbitration, had defined sovereignty in terms of independence exercised over "a portion of the globe," making control over land a core indicator of legal personality. The logic was clear: if sovereignty is authority, and authority requires jurisdiction, then jurisdiction needs a place to take effect. A state without land would seem to be a contradiction in terms.

There are reasons why this assumption became so durable. Territory does more than outline the physical extent of a state—it frames the legal and political space in which authority takes shape. First, territory defines jurisdiction. It sets the boundaries within which a state can apply its laws, enforce decisions, and exercise monopoly over violence. A government needs somewhere to govern. Without a defined space, the distinction between internal and external authority begins to dissolve, making it difficult to trace the limits of legal obligation or sovereignty.

Second, territory has long been tied to the identity and continuity of the state itself. A state, in the classical view, is not just a set of institutions but a community rooted in place. Territorial permanence was seen as a proxy for political permanence. This view helped stabilize the post-colonial international order: states inherited their borders, however arbitrary, and those borders became the vessel through which political legitimacy flowed. Recognition, membership in the United Nations, and treaty-making capacity all presupposed territorial existence—even if the population was scattered or the government in flux (Stoutenburg 2015).

Third, territory contributed to the practical stability of the international system. It served as a reference point for mapping rights and responsibilities, resolving disputes, and allocating access to resources. Whether in border arbitration, maritime delimitation, or international humanitarian law, the idea that states have "somewhere" remained fundamental (Kelsen 1945). Even in cases where states lost control of parts of their land, through war, occupation, or collapse-the assumption was that the territory still existed and that sovereignty might one day return to it. What international law has not had to confront, at least until now, is the possibility that the land itself might be lost. This implicit consensus, which regards territory as indispensable to legal statehood, has rarely been questioned. Even in moments of crisis, such as exile, occupation, or border erosion, international law has tended to treat territory as a latent constant: the legal "container" of sovereignty, waiting to be reactivated when control is restored. But climate change introduces a different kind of disruption. It does not displace governments through war or dissolve states through consent. Instead, it threatens to erase the material basis of statehood altogether.

Sea-level rise presents a scenario that existing legal frameworks were not built to absorb. For low-lying island states like Tuvalu or Kiribati, the prospect is not only of temporary loss or diminished control but of permanent uninhabitability-first through saltwater intrusion, then infrastructural collapse, and eventually physical submergence. If and when that happens, these states' territorial foundation may vanish. As Rayfuse (2010) observes, this is not a matter of occupation or failed government, but the gradual unmaking of a state's geography. With it, international law's reliance on territory as a precondition for legal personality begins to fracture. International law does contain examples of states continuing to exist without exercising control over their territory. Governments in exile, like those of Poland during World War II or Kuwait during the 1990 Iraqi occupation, retained recognition even when displaced. In such cases, sovereignty persisted de jure, even if suspended de facto. The territory, though inaccessible, still existed-and crucially, still belonged to the state. That distinction matters. These cases presumed that land remained legally assignable, recoverable, and central to the state's future. The legal personality of the government-in-exile hinged on the assumption that one day it would return. Similarly, entities like the Holy See or the Sovereign Order of Malta complicate the territorial model, but they do not displace it. Their recognition rests on religious, historical, or institutional particularities, not on a broader rethinking of how sovereignty might function without land (Crawford 2006).

In contrast, the climate crisis offers no clear path of return. The loss it introduces is not temporary or reversible, but potentially permanent and physical. As Stoutenburg (2015, 3) points out, modern legal history has no precedent for the complete disappearance of a state's land due to environmental change. If that happens, and if people are forced to relocate across borders, no doctrine tells us what happens to the state they leave behind. Recognition, in that case, becomes a legal and political choice—one for which there is no consistent guide. Still, not all scholars agree that the loss of territory would necessarily dissolve statehood. Crawford (2006) emphasized that once established, a state is not easily extinguished-even when it undergoes dramatic changes in population, government, or land. International law, he notes, tends to favor continuity. This is reflected in the treatment of fragmented or occupied states and in the reluctance to declare extinction without clear acts of succession, merger, or voluntary dissolution. However, that presumption of continuity, while powerful, is not without its limits. It presumes that the core features of the state remain recoverable or legally intact. When territory disappears not by force or choice but by natural erosion, the conditions shift. There is no roadmap for a state that loses the space within which its sovereignty has always been imagined. As Rayfuse (2010) and Burkett (2011) have argued, the Montevideo model does not accommodate this form of loss. It was never designed for it. This is not simply a technical gap. It reflects a deeper conceptual rigidity: a belief that sovereignty requires soil. Climate change forces a return to that assumption—not only to see where it came from but also to ask whether it should still hold. If the law cannot imagine a people governing themselves without a fixed space beneath them, then it risks failing the very communities whose futures it must now confront.

CLIMATE SOVEREIGNTY AS A LEGAL FRAMEWORK

Climate sovereignty denotes a state's sustained legal existence, including its international personality, sovereign rights, and institutional identity, even after the complete loss of territory caused by climate change. The concept is not proposed as a sui generis status but as a doctrinal extension of existing principles of state continuity, adapted to the unprecedented scenario of permanent and involuntary submergence. Under orthodox international law, the Montevideo Convention identifies four criteria for statehood: a permanent population, defined territory, a government, and the capacity to enter into relations with other states (Montevideo Convention 1933). These criteria were drafted to define the creation of states, not their continued existence under exceptional threats. As Crawford (2006) observes, the Montevideo standard is flexible in practice, yet no provision currently clarifies whether a state that loses its entire territory to environmental forces retains its legal status. The tension between formal criteria and the absence of extinction rules creates a doctrinal gap-a problem previously noted in exile-related jurisprudence.

The proposed framework builds on the international legal presumption of state continuity, which maintains that a state, once established, does not cease to exist merely because it no longer fulfills certain factual criteria. Marek's analysis of governments in exile underscores that legal personality can survive without territorial control, provided an institutional structure and collective identity remain in place. There is no principled basis in current international frameworks for treating involuntary territorial loss as a sufficient ground for legal extinction (ILC 2001, Draft Articles on State Responsibility). These interpretations do not challenge the traditional doctrine but expose its capacity to absorb non-territorial forms of legal presence. Climate sovereignty, in this reading, rests on a tripartite foundation for continued statehood under conditions of permanent climate-induced submergence. This framing aligns with broader trends in international law that conceptualize sovereignty as a relational and functional

structure rather than a fixed threshold (Kingsbury 1998). Second, a functioning governmental structure must exist to represent that community and maintain institutional coherence. Third, the recognition of that continuing claim by the international community, though not legally guaranteed, must be sufficiently sustained to preserve international legal personality. This formulation does not negate the importance of territory, but it reframes its loss as a condition that shifts legal analysis toward institutional persistence and collective self-definition rather than geographic presence (Talmon 1998).

While not entirely novel, this view draws partial support from scattered precedents. The Sovereign Order of Malta, for instance, has retained an international legal personality despite the absence of sovereign territory. Wartime governments-in-exile likewise sustained state functions and recognition without physical control over their homeland. These cases illustrate that legal personality, as distinct from territorial jurisdiction, can persist under exceptional circumstances. Climate sovereignty does not seek to equate these examples but rather to systematize their underlying principle: that sovereignty, once fractured from land by irreversible climate loss, may still be carried by institutional coherence and a politically self-aware community. This reframing is both conceptually plausible and normatively urgent. Yet its legal implications remain under-theorized. If climate sovereignty is to function as a doctrine of continuity under conditions of territorial loss, then its relationship to existing legal norms must be carefully examined. The aim is not to discard current frameworks but to interrogate their elasticity. What, if anything, in the current law affirms or resists the continuity of a state without land?

One starting point is the Montevideo Convention itself. While widely cited as the benchmark for statehood, it was never intended to address cases of irreversible environmental loss. Its territorial requirement is often read as axiomatic, yet neither customary law nor treaty law explicitly states that the absence of territory extinguishes legal personality. The resulting ambiguity leaves states vulnerable to an interpretive vacuum at precisely the moment when they face existential risk, a vulnerability that reflects deeper historical biases embedded in dominant sovereignty discourses (Chimni 2004). Marek's (1968) principle of state continuity offers partial support for survival without territory, but its application has been limited to cases of foreign occupation or annexation, not natural submergence. The doctrine rests on the idea that sovereignty is temporarily obstructed, not fundamentally disrupted. Climate change complicates this distinction. When territory is not seized but lost without fault and return, what counts as continuity?

This is not merely a conceptual puzzle. Without a legal mechanism for continuity, displaced nations face the prospect of losing their treaty rights, international representation, and the legal bond of nationality. As the ILC has noted (2022, para. 201), there is currently no clear protection against the risk of de jure statelessness for entire populations. While existing doctrines presume that states continue through crises, they were not built to withstand the slow erosion of coastlines. Climate sovereignty insists that the basis for continuity must expand accordingly. There are systemic implications as well. If international law permits the silent extinction of a sovereign state due to environmental collapse, it normalizes disappearance as a lawful outcome (Crawford & Baetens 2023). For many small island states already marginalized in global decision-making, this legal silence compounds structural disadvantage (Betzold 2015). That sets a dangerous precedent-not only for low-lying island states but for the coherence of the legal order. A doctrine of climate sovereignty aims to close that space of indeterminacy. It proposes not a break from international law but a necessary adaptation to preserve its most fundamental commitments: to dignity, stability, and the enduring subjectivity of peoples in international life.

If climate sovereignty is to operate within international law, it must do more than appeal to moral urgency. It must demonstrate how the foundational elements of statehood-territory, population, government, and international capacity can be interpreted in ways that accommodate continuity under conditions of permanent territorial loss. This does not require abandoning the Montevideo framework, but it does require reading its criteria as functional rather than literal. Take the question of territory. Nothing in the Montevideo Convention specifies how much territory is required or in what form. Historical practice includes states with fragmentary or contested land, and the law has tolerated considerable flexibility. What climate sovereignty proposes is an interpretive shift: that where a state's territory has become physically uninhabitable through no fault of its own, the existence of prior territorial attachment—combined with continued self-identification—can suffice to satisfy the criterion. This reading would align with the approach already suggested in efforts to preserve maritime entitlements through fixed baselines (Pacific Islands Forum 2023). In both cases, legal stability is prioritized over geographic precision.

A similar logic applies to the population. The permanent population criterion is often assumed to require physical residence within the state's territory. But this, too, is more convention than rule. Diasporic nations, governments in exile, and postcolonial transitions have all shown that the legal community does not depend on physical co-presence (Crawford & Baetens 2023). Climate sovereignty builds

on this by proposing that a national population dispersed by climate displacement can remain a "people" for statehood, provided that institutional, legal, and symbolic bonds endure. Citizenship laws, administrative capacity, and cultural continuity, rather than geographic density, become the operative markers of state identity. The requirement of the government, too, is adaptable. International law has long recognized that governments may function in exile or without territorial control. What matters is not the physical location but the capacity to represent, organize, and act. Climate sovereignty formalizes this possibility: a relocated or digitalized government, operating through transnational mechanisms, may continue to fulfill the core functions of statehood (Vidmar 2012). This idea draws strength from existing practices—such as Estonia's digital governance model or Tuvalu's initiative to preserve its institutions in virtual form—but grounds them in legal theory rather than symbolic innovation.

The fourth Montevideo element-the capacity to enter into relations with other states is, in some ways, the least problematic. A state that retains recognition, signs treaties, participates in international organizations, and represents its citizens abroad continues to act as a subject of international law. Climate sovereignty does not alter this criterion; it relies on it. The capacity for international engagement becomes not only a proof of continuity but a condition for it. In that sense, sovereignty is reinforced not by territory but by relational presence in the legal order. Taken together, these reinterpretations do not dilute the idea of statehood. They clarify its purpose. The Montevideo criteria were never meant to be metaphysical tests (Pacific Islands Forum 2023). They are tools to assess whether a community can function as an international legal person. Climate sovereignty pushes that function to the foreground. It insists that when geography fails, legal design must step in, not to invent states where none exist, but to ensure that forces beyond their control do not extinguish existing ones.

Climate sovereignty is often grouped with existing proposals for addressing climate-induced state disappearance. However, its purpose and legal structure are different. Rather than offering a workaround or technical supplement, it confronts the core legal question: what must be preserved, and how, when a state loses its land. Burkett's Nation Ex-Situ proposal anticipates the possibility of continuity but does so through the idea of new arrangements, such as international trusteeship or regional guarantees (Burkett 2011). This places the displaced nation in a dependent position, requiring external support to maintain its status. Climate sovereignty takes a different view: it asserts that a people already entitled to statehood do not lose that entitlement when their land disappears. Continuity should not depend on institutional invention or external benevolence but on the enduring rights of a political community-a normative position increasingly echoed in climate justice scholarship (Crawford & Baetens 2023).

Proposals to freeze maritime baselines, now reflected in state practice across the Pacific, focus on protecting economic entitlements under UNCLOS. Yet they implicitly assume that the state itself remains to claim those entitlements. Climate sovereignty addresses the prior question: How does legal personality endure when the land base disappears? Without answering that, maritime rights risk becoming detached from any subject to hold them. In this sense, baseline preservation is a defensive measure; climate sovereignty is a structural one. The analogy to governments in exile is also limited. Those governments are typically displaced by force and operate under the assumption that return is possible. Climate-displaced nations, by contrast, face permanent deterritorialization with no wrongful actor to invoke. Climate sovereignty does not seek to replicate the exile model. It builds a distinct legal logic: continuity without territory and the expectation of return (Vidmar 2012). Symbolic efforts, like Tuvalu's digital nation project, reflect the will to preserve national identity. However, symbolism is not legal continuity. A state's presence in the international system depends not on memory but on recognition, rights, and institutional capacity. Climate sovereignty provides the legal doctrine that digitization alone cannot supply. It does not merely commemorate statehood; it protects it.

Climate sovereignty reinterprets these scattered precedents into a principled and prospective legal doctrine: that a people's sovereign status should not be extinguished solely by the physical disappearance of their territory (Crawford & Baetens 2023). This proposition, however, remains doctrinal in character. Whether it can be translated into legal continuity within the actual structures of international law depends not only on interpretive plausibility, but on the institutional and political conditions through which recognition, participation, and personality are sustained.

APPLYING CLIMATE SOVEREIGNTY IN PRACTICE

For climate sovereignty to transition from normative aspiration to legal reality, it must integrate coherently into the existing framework of international law. The question is not one of political morality but of legal structure: Can existing doctrines be interpreted to sustain the continued statehood of a country that has irreversibly lost its territory to rising seas? (Vidmar 2012). This inquiry does not call for new institutions or formal amendments. Instead, it concerns the flexibility

of categories such as recognition, state continuity, and legal personality, whether they can stretch to meet a reality they were never designed to confront.

One possible entry point lies in how states themselves articulate continuity through their domestic legal orders. Tuvalu's 2023 constitutional amendment, particularly Article 2A, declares that the State "shall remain in perpetuity… notwithstanding the impacts of climate change or other causes resulting in loss to the physical territory." It further affirms that maritime boundaries "shall not be challenged or reduced due to any regression of the low water mark." These provisions do not directly modify international criteria for statehood, but they express a deliberate legal position, one meant to frame Tuvalu not as a disappearing object but as a persisting legal subject. The constitutional act anticipates ambiguity and attempts to fill it from within, drawing on a logic familiar from the jurisprudence of governments-in-exile, where legal identity is preserved through institutional order rather than geographic control (Talmon 1998). Here, the claim is not to restitution, but to continuity, anchored in a self-declared legal identity that seeks recognition without precondition (Pacific Islands Forum 2023).

This interpretive move gains coherence through regional alignment. In 2023, the Pacific Islands Forum adopted a declaration stating that international law "supports a presumption of continuity of statehood" in the face of climate-related sea-level rise. The document does not propose new legal categories. Rather, it affirms that those existing principles-such as self-determination, dignity, and equity-provide sufficient grounds to maintain legal personality despite territorial disappearance (Vidmar 2012). As a form of collective legal interpretation, the declaration seeks to clarify what the law already allows. In doing so, it follows a path recognized by the International Court of Justice: the formation of customary norms may begin with the consistent articulation of legal views, especially when offered by states with a direct stake in the matter (Kosovo Advisory Opinion 2010). Whether such efforts can crystallize into law depends in large part on how third-party states respond through their recognition practices or institutional behavior (Byers 1999).

This brings the problem back to recognition. International law offers no definitive rule on whether the loss of territory extinguishes statehood. Recognition remains a discretionary function-structured by legal expectation but ultimately executed through political judgment (Crawford 2006). States like Tuvalu can construct compelling legal narratives, yet their legal identity depends on whether others are willing to engage with them as sovereign equals. Here, the record of international practice is ambiguous. Entities such as Taiwan and Palestine have been variously recognized and excluded, not always by objective criteria but often in response to strategic or geopolitical concerns (Vidmar 2012). These cases underscore the fragility of legal personality when it hinges on recognition. They also remind us that persuasive claims may still falter if the institutional environment declines to absorb them.

Failure to recognize climate-threatened states has consequences beyond symbolism. The loss of territory, if followed by the loss of legal status, could result in the disappearance of treaty rights, institutional memberships, and diplomatic capacity. Individuals from such states might retain national identities under domestic law, yet face uncertain status abroad, neither citizens of a functioning state nor formally stateless. One may ask: if Tuvalu becomes physically uninhabitable, will it retain its UN seat? Will it remain a party to multilateral treaties such as the UNFCCC or UNCLOS? International law currently offers no clear process to confirm or reject such continuity (Talmon 1998). This institutional silence risks producing a legal vacuum in which the collapse of territory is quietly mirrored by the erosion of sovereign identity (Pacific Islands Forum 2023).

Some people think that climate sovereignty is a bad idea because it could hinder international collaboration and make it harder for the world to combat climate change. Some worry that governments may put their own economic interests ahead of global climate goals if national sovereignty is prioritised, which would lead to less accountability and make it harder to implement effective climate measures. On top of that, there is the argument that climate sovereignty stands in the way of the legally enforceable international agreements and protocols that would be vital to take effective action on the climate.

Problem with Global Cooperation

Prioritising national economic development and short-term benefits over long-term climate goals is a risk that can arise from an emphasis on national sovereignty. Important steps towards combating climate change, such as investing in renewable energy sources and setting strict emission reduction targets, may be impeded as a consequence. Governments' resistance to binding commitments or their pursuit of loopholes to evade their obligations, both of which stem from an emphasis on national sovereignty, can undermine the effectiveness of international climate agreements. A failure to accomplish substantial reductions in emissions and an absence of responsibility may result from this. Obstacles to fair burden-sharing and equity: Developed nations may claim they shouldn't be punished for previous emissions while developing nations are free to keep emitting at higher rates, according to the idea of climate sovereignty (Klabbers 2020).

Destroying International Agencies

State sovereignty may make states less likely to accept blame for climate change and act to mitigate it, leading to a lack of accountability. As a result, states may not be held responsible for their climate policy, and accountability may suffer. The idea of climate sovereignty raises concerns about the potential erosion of international law due to the potential erosion of adherence to international climate change norms and responsibilities. While climate change has the ability to intensify preexisting tensions and conflicts, the idea of climate sovereignty has the ability to further complicate matters by fostering fiercer rivalry over resources and territories.

Threats to Sovereignty and Statehood

Migratory patterns brought on by climate change: As a result of changing weather patterns, people may be compelled to leave their homes and seek refuge in other parts of the world. Concerning the rights of displaced people and the maintenance of statehood and sovereignty, this might lead to complicated political and legal concerns. Particularly in low-lying coastal regions, complete territory may vanish as a result of climate change. The capacity of a state to preserve its political and legal status, as well as the very concept of statehood, may be called into doubt by this. An already polarised society may see its political and social divisions deepen as a result of climate change, which might spark calls for more independence or even breakaway from current states (Klabbers 2020). Although climate sovereignty is a tempting idea in some places, it can also make international cooperation and global climate action very difficult. The efficiency of international accords, accountability procedures, and the risk of conflict and social instability can all be undermined by an emphasis on national sovereignty.

Even so, the legal system is not entirely closed to adaptation. Doctrines of continuity, the functional flexibility of treaty participation, and the procedural discretion of international organizations all offer interpretive footholds. The principle of effective participation, already embedded in treaty law and multilateral practice, could sustain a deterritorialized state's legal personality if applied purposive intent (Chinkin 1989). In this sense, climate sovereignty does not disrupt the system but tests its willingness to evolve. It asks whether the law can interpret its categories in light of existential risk without needing to rewrite them from scratch. The response need not be revolutionary. It requires, rather, a willingness to read silence as possible and treat continuity not as a geographic fact but as a legal stance that deserves engagement.

CONCLUSIONS

This article has examined how the legal notion of statehood may respond to a scenario not previously contemplated within the doctrinal tradition of international law: the complete and permanent loss of a state's territory due to climate change. Through the framework of climate sovereignty, it proposed a way to understand such loss not as the end of legal personality but as a condition that may be addressed through the reinterpretation of existing principles. The focus has remained on continuity—not as an assertion of legal exceptionalism, but as a potential outcome of applying current doctrines of recognition, personality, and self-determination to a new factual context. In assessing whether international law can accommodate this proposition, the analysis has pointed to a degree of interpretive openness. States such as Tuvalu have begun to articulate legal continuity through constitutional instruments, and regional declarations suggest an emerging normative consensus that the disappearance of territory should not be equated with the extinction of sovereignty (Pacific Islands Forum 2023). However, the legal consequences of these claims remain uncertain. International law does not provide a clear mechanism for confirming or denying the continuing statehood of deterritorialized polities, and recognition remains politically contingent. The system is procedurally silent, and this silence allows for flexibility but also creates a risk of incoherence or selective absorption.

The implications of this uncertainty are likely to become more concrete as affected states move closer to physical uninhabitability. Whether climate sovereignty will be accepted as a valid legal position may depend less on theoretical persuasiveness than on the willingness of other states and institutions to treat such claims as actionable. Participation in international organizations, treaty continuity, and diplomatic recognition will serve as the practical tests of interpretive acceptance (Vidmar 2012). At present, the conditions for such recognition remain undefined. Whether this ambiguity will be resolved through gradual accommodation or remain unaddressed will reveal much about the capacity of international law to adapt without formal transformation. Unlike questions of aggression, secession, or decolonization, climate-induced deterritorialization presents no adversarial party. The absence of a violating actor removes the legal anchor typically used to preserve continuity. What remains is the interpretive will of the system itself, its ability to acknowledge new forms of disruption without waiting for a corresponding rule to emerge. It is significant to know that how the world community responds to the threat of climate change will determine its actual effect on national sovereignty. Treaties

and decisions of international courts and arbitral tribunals establishing marine boundaries may be unaffected by these changes. In this context, climate sovereignty is less a test of doctrinal innovation than of legal self-awareness, examining whether international law can recognize its own flexibility to accommodate disappearance without dissolution.

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Integrated Flood Hazard Assessment Using AHP-GIS in the Pallikaranai Marshland, Buckingham Canal Corridor, India

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ABSTRACT

The resultant impact of climate change and urbanization has caused extensive disruption to natural hydrological processes, thus enhancing the flood risk in susceptible areas. This study evaluated flood processes in the Pallikaranai Marshland–Buckingham Canal corridor using detailed flood inundation modeling and risk assessment methodology. Important geospatial factors and variables, such as rainfall, Digital Elevation Model (DEM), slope, Land Use Land Cover (LULC), river distance, flow length, and Normalized Difference Water Index (NDWI), were weighed and ranked. These weighted parameters were assimilated to estimate the Flood Hazard Index (FHI), which was subsequently applied to create an intricately mapped flood hazard. The analysis and testing of the involved parameters by assessing flood susceptibility has been facilitated with hydrological modeling, Geographic Information System (GIS), as well as with remote sensing procedures. Deep-learning frameworks, particularly convolutional neural networks, have also shown high predictive capability for regional flood susceptibility (Kalantar et al. 2021). The findings suggest that urban growth has resulted in extensive wetland degradation, elevated surface runoff, and more frequent flooding, particularly during intense rainfall. The FHI-based flood hazard map identifies critical areas at risk of flooding, highlighting the explicit role of land cover changes in flood intensity and frequency. This study underscores the urgent need for sustainable urban planning, wetland conservation, and climate-resilient infrastructure to mitigate flood hazards and enhance long-term urban flood resilience in the region. These results help to better understand urban flood hazards and offer a scientific foundation for future flood management.

INTRODUCTION

Floods are among the most recurrent and devastating natural disasters affecting urban settlements worldwide, particularly in coastal cities with high population densities (Singha et al. 2025). Chennai, one of India's major metropolitan centers, has experienced severe flooding events, with the 2021 flood serving as a recent example of extreme urban inundation (Kartheeshwari & Elango 2022). Long-term climatic analyses indicate that temperature rise, precipitation extremes, and altered discharge patterns substantially increase flood occurrence globally (Alobid et al. 2024). Studies have attributed the increased flood risk in Chennai to a combination of excessive rainfall, unregulated urban expansion, and the degradation of natural drainage systems (Ramakrishnan et al. 2018). Chennai receives a significant portion of its annual rainfall from the Northeast Monsoon, making it vulnerable to waterlogging and infrastructure damage owing to inadequate stormwater management (National Institute of Disaster Management 2020). Comparable coastal systems show that climate-driven land cover changes can amplify runoff and exacerbate flood intensity (Song et al. 2024). One of the primary contributors to the flood vulnerability of Chennai is the rapid urbanization-induced loss of water-retaining ecosystems. Historically, the city has an extensive network of wetlands and water bodies that act as natural buffers against flooding. However,

encroachment on these ecological systems has exacerbated the severity of flood events. The Pallikaranai Marshland to Buckingham Canal corridor, in particular, has witnessed extensive anthropogenic transformations, resulting in greater vulnerability to flooding in nearby urban areas (Sudhakar et al. 2019).

The Pallikaranai Marshland, a crucial freshwater ecosystem in Chennai, helps mitigate urban flooding by absorbing excess rainfall, functioning as a natural sponge (Ramachandran et al. 2015). However, large-scale reclamation and conversion of marshland for residential, industrial, and infrastructure development have significantly reduced its water-holding capacity. Studies indicate that nearly 90% of the original marshland has been lost over the past five decades, leading to a substantial decline in ecological functions (Jayanthi et al. 2017). The Buckingham Canal, an artificial tidal waterway running parallel to the Coromandel Coast, has historically served as an inland navigation route and stormwater drainage conduit. However, pollution, encroachment, and silt accumulation have diminished its drainage efficiency (Anand et al. 2021). Restrictions on natural flow passageways between the Buckingham Canal and Pallikaranai Marshland have led to prolonged water stagnation and urban flooding during heavy rains. This study aims to map flood-prone areas and provide insights into sustainable urban planning and flood mitigation. Hybrid ensemble flood-susceptibility models have demonstrated strong accuracy in South Asian basins that exhibit comparable terrain and hydrological variability (Ahmed et al. 2022). This research will measure the effects of climate change and urbanization on flood behavior in the study region, examine land-use patterns, drainage capacity, and rainfall trends using remote sensing and GIS applications, and create a flood hazard map by applying a weighted overlay procedure based on key hydrological and topographic factors. In addition, it will identify high-risk flood-prone areas and analyze the contribution of wetland degradation to increased flood hazards.

MATERIALS AND METHODS

The study area was selected based on three main factors: flood-prone zones, land-use changes, and drainage infrastructure. Priority was given to areas such as the Pallikaranai Marshland and Buckingham Canal, which are highly susceptible to flooding owing to their low-lying nature and poor drainage systems. Regions with significant land-use changes, particularly increased built-up land cover, were emphasized because they reduce water absorption and intensify surface runoff. Additionally, areas with inadequate or poorly maintained drainage networks were considered, highlighting the role of insufficient infrastructure in exacerbating the flood risks.

The Pallikaranai Marshland, one of the last remaining freshwater marshes in Chennai, plays a vital role in flood attenuation by acting as a natural sponge that stores excess rainwater during the monsoons. However, its drainage pathway-primarily via the Okkiyam Maduvu channel into the Buckingham Canal—has become increasingly compromised due to siltation, narrowing, and loss of channel capacity. The overall workflow adopted for flood hazard assessment is shown in Fig. 1. Notably, this hydrological linkage failed during the 2015 South India floods and more recently during Cyclone Michaung (2023), causing significant inundation in

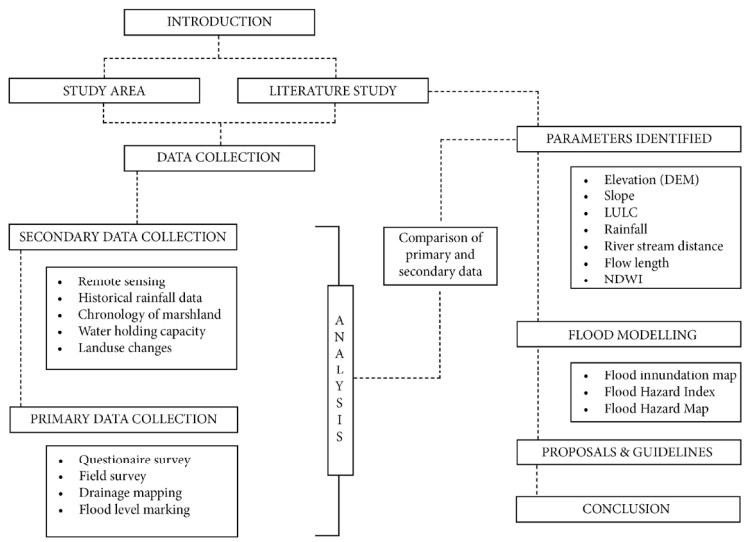

Fig. 1: Methodology.

Fig. 2: Key map.

the surrounding urban areas, such as Velachery, Perungudi, and Sholinganallur.

The Pallikaranai Marshland and Buckingham Canal, found in southern Chennai, Tamil Nadu, India, are incredibly important as they assist in flood management and are under threat from urbanization and severe weather events. The Pallikaranai Marshland is located at 12.93°N latitude and 80.21°E longitude and is a freshwater wetland of approximately 50 sq.km; however, the size of this wetland has been considerably reduced due to urban encroachment. Wetlands are natural flood buffers, biodiversity providers, and groundwater recharge areas with alluvial and clay soils that retain water.

The region has a tropical wet and dry climate, with an average annual rainfall of 1,200 mm, which mainly occurs during the Northeast Monsoon. Hydrologically connected to the Okkiyam Maduvu and Buckingham Canal, the marshland allows drainage into the Bay of Bengal. Isotopic hydrology studies in the Chennai region corroborate the complex interaction between shallow groundwater and surface-water systems (Natarajan 2023). However, rapid urbanization at a fast pace, landfilling operations, and alterations to the drainage system have disturbed its natural balance, increasing the risk of flooding and water contamination. The Buckingham Canal, particularly its Thoraipakkam–Karapakkam section, is one of the major drainage channels

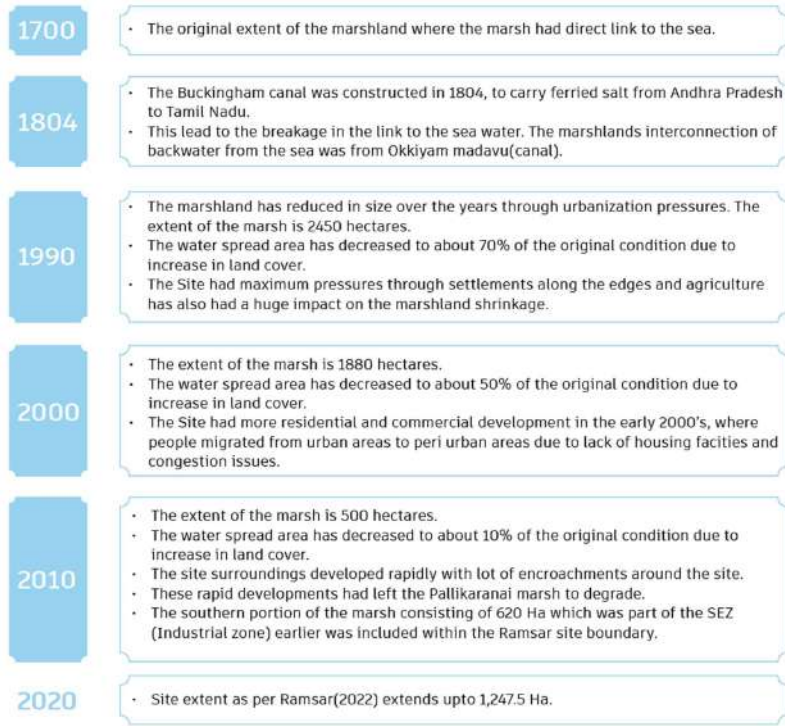

Fig. 3: Chronology of marshland timeline.

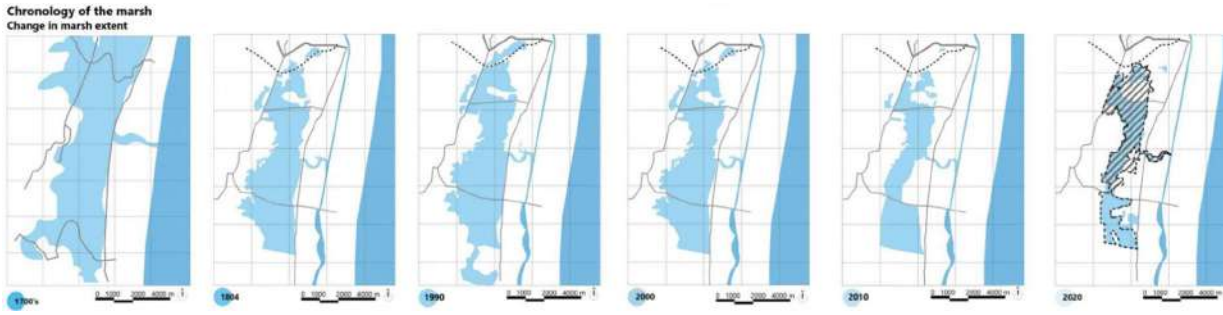

Fig. 4: Chronology of marshland.

for the city but is plagued by siltation, encroachments, and decreased flow capacity, adding to urban flooding. All these factors in unison reflect the significance of this area towards flood risk analysis and sustainable city planning (Prakriti 2025). The location and spatial extent of the study area are illustrated in Fig. 2.

Secondary Data Collection

Chronology of Marshland

The Pallikaranai Marshland initially had a direct link to the sea, with backwater inflows via the Okkiyam Maduvu (canal). The development of the Buckingham Canal in 1804 to carry salt from Andhra Pradesh to Tamil Nadu broke this natural connection, changing the hydrology of the marsh. Urbanization over the years has significantly diminished the size of the marsh. The historical evolution and timeline of marshland degradation are shown in Figs. 3 and 4.

Initially spanning an area of 2,450 hectares, the water spread area lost 70% of its land cover as settlements expanded along its boundary. By the early 2000s, when housing and commercial construction boomed with migration from overpopulated urban areas, the marsh shrank further to 990 hectares, where only 35% of its original water area remained. The development of the IT corridor in Chennai along the OMR accelerated land-use change, leading to additional reductions. The marshland now occupies an area of only 500 hectares, with scarcely 10% of its original water spread area, mainly due to rapid encroachment and illegal development. But, attempts have been made to conserve it, and 620 hectares of the south region (which was previously an SEZ industrial zone) came within the Ramsar site boundary in 2022, increasing the conserved area to 1,247.5 hectares (Prakriti 2025).

Change in Land Use Around the Region

There have been tremendous land-use changes over the years around the marshland, as illustrated in the 1990, 2000, and 2020 maps. In 1990, natural vegetation and open spaces covered most of the area around the marshland, with a few isolated built-up areas. There was a visible conversion of land by 2000, as both urban and agricultural lands started increasing in size, thereby decreasing the size of the green areas. The most drastic change took place by 2020, as significant areas of the marshland and its vicinity were taken over by urbanization and other uses. The previously wide natural habitats turned highly fragmented with serious encroachment by developed land, possibly causing the degradation of the wetland ecosystem. This trend suggests rapid urbanization, agricultural expansion, and possible environmental consequences, such as loss of biodiversity and reduced water retention capacity of the marshland. These temporal land-use changes are depicted in Fig. 5.

Fig. 5: Change in land use around the region.

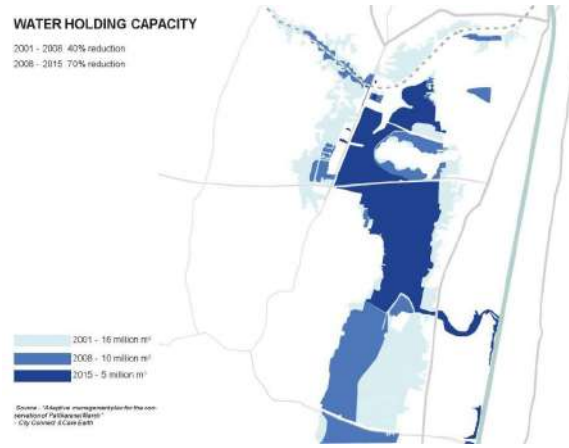

Fig. 6: Water holding capacity.

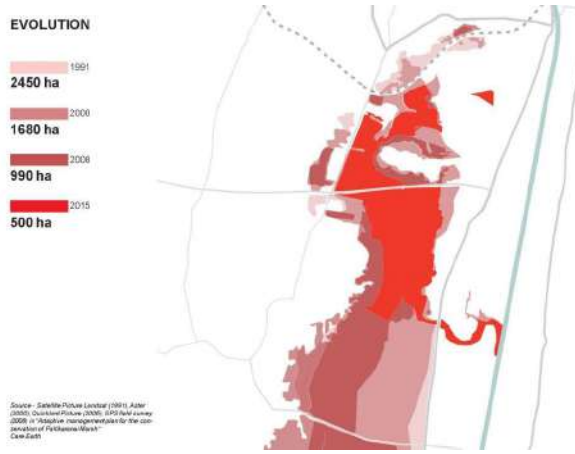

Fig. 7: Marshland degradation.

Degradation of the Marsh and Water Holding Capacity

The water-holding capacity of marshlands is influenced by the geological characteristics of the soil. Since the marshland has clayey soil that can hold water for a longer period of time and can release it during dry periods, the surrounding areas of the marsh were not much affected by the excessive rainfall during 2005. However, in 2015, some of the worst-hit areas in Chennai that suffered immensely due to flood levels of up to 1.8m were Pallikaranai, Velachery, Madipakkam, and Thoraipakkam, which are all in the vicinity of the marshland and have blocked the natural drainage network of the marshland. The encroachments include residential land use and infrastructure facilities approved by the government, namely the mass rapid transit system railway station of Velachery, which has taken up the northern part of the marsh, while the Perungudi Landfill has taken up the central part of the marsh, which takes up about 72 hectares of the marsh. In addition, the ELCOT SEZ occupies the southern part of the marsh. The spatial variations in water-holding capacity and extent of marsh degradation are presented in Figs. 6 and 7.

Historical Rainfall and Flooding in Chennai

Chennai experiences a tropical wet and dry climate, with rainfall patterns significantly influenced by the Northeast Monsoon from October to December, contributing 60-70% of the city's average annual rainfall of approximately 1,200 mm. Historical rainfall data reveal considerable variability, with some years marked by intense rainfall, leading to catastrophic flooding. Notably, the 2005 event saw over 1,000 mm of rainfall in a single day, causing widespread waterlogging and damage to infrastructure. Historical monthly and annual rainfall variability for Chennai is shown in Figs. 8 and 9. Detailed two-decadal rainfall statistics used for analysis are provided in Table 1.

Table 2 illustrates how natural rainfall extremes**,** combined with urban encroachment**,** inadequate drainage,

Table 1: Two decadal rainfall data (source: KEA weather station).

Year	Jan-May	Jun-Sep	Oct-Dec	Total
2024	87	694.2	1084.1	1865.3
2023	104	743.7	1268.8	2116.5
2022	129.8	497.7	960.3	1587.8
2021	215.9	558	1484.8	2258.7
2020	97.2	293.4	1033.5	1424.1
2019	4	492.6	605.7	1102.3
2018	5.8	432.2	390.1	828.1
2017	7.7	508.7	978.5	1494.9
2016	209.8	526.3	324.6	1060.7
2015	23.2	407.8	1663.8	2094.8
2014	23.8	518.8	752	1294.6
2013	33.6	617.4	436.8	1087.8
2012	17.8	408.2	595.2	1021.2
2011	130.8	852.4	852.4	1835.6
2010	209.4	647.6	757.6	1614.6
2009	37.8	233.2	909.8	1180.8
2008	226.8	422.6	947.6	1597
2007	7.2	677	625.6	1309.8
2006	37.4	393	892.6	1323
2005	121	337	2108	2566
2004	264.6	360	572	1196.6

and wetland degradation, have made the Pallikaranai Marshland a recurrent flood hotspot. Effective flood mitigation demands restoration of marsh connectivity, desilting of drainage channels, and long-term land use regulation.

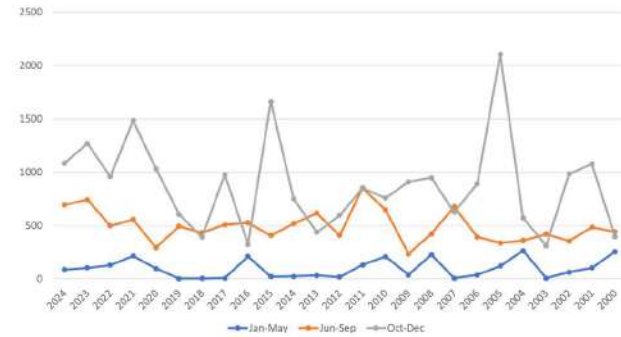

Fig. 8: Historical month-wise rainfall data.

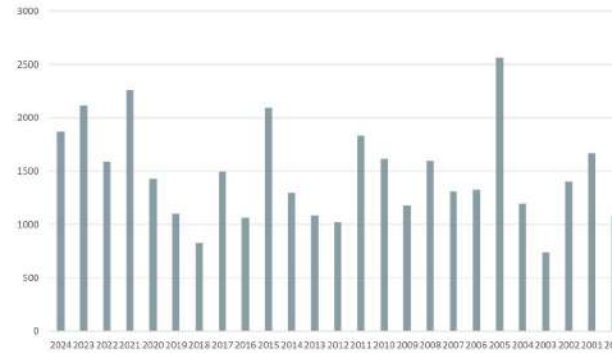

Fig. 9: Historical year-wise rainfall data.

Land Ownership Inside the Marshland

In the early 1900s, marshland occupied an area of 6000 ha (60 km^2), which is now 593 ha (Care Earth 2002). The National Institute of Ocean Technology (NIOT) and the Center for Wind Energy Technology (WET) have built institutions that have divided and minimized the marsh, and the Perungudi dump yards and effluent treatment plants have

Table 2: History of flooding in Palikarani Marshland and Buckingham Canal.

Year	Event/ Cyclone	Rainfall [mm]	Cause	Impact	Notes
2005	Heavy Monsoon Rainfall	~400 mm in 3 days	Prolonged NE monsoon, poor drainage	Major waterlogging in Velachery, Perungudi, Pallikaranai	Highlighted the lack of stormwater infrastructure
2008	Cyclone Nisha	~500 mm (Nov)	Cyclonic storm + encroachment in the marsh	Marsh overflowed; roads flooded	Marsh area reduced due to dumping & encroachments
2015	South India Floods	>1,200 mm in 30 days	Historic rainfall + blocked drains + Encroachments	Extensive flooding in South Chennai, airport closure	Pallikaranai overflowed; Okkiyam Maduvu failed to drain into the Canal
2017	Cyclone Ockhi (indirect)	~200 mm	Back-to-back rain events, poor marshland drainage	Moderate inundation; waterlogging in IT corridor	Drainage systems strained
2021	Northeast Monsoon	~1,000 mm (Oct–Nov)	Intense NE monsoon, high tide effects on Buckingham Canal	Partial flooding in marshland-adjacent areas	Canal outfall constrained by urban development
2023	Cyclone Michaung	~400 mm in 48 h	Cyclonic storm, silted Buckingham Canal, encroachments in marsh	Severe flooding in Sholinganallur, OMR, Perumbakkam	Okkiyam Maduvu unable to drain efficiently into the sea

(Source: *TN SDMA* (Tamil Nadu State Disaster Management Authority) Reports)

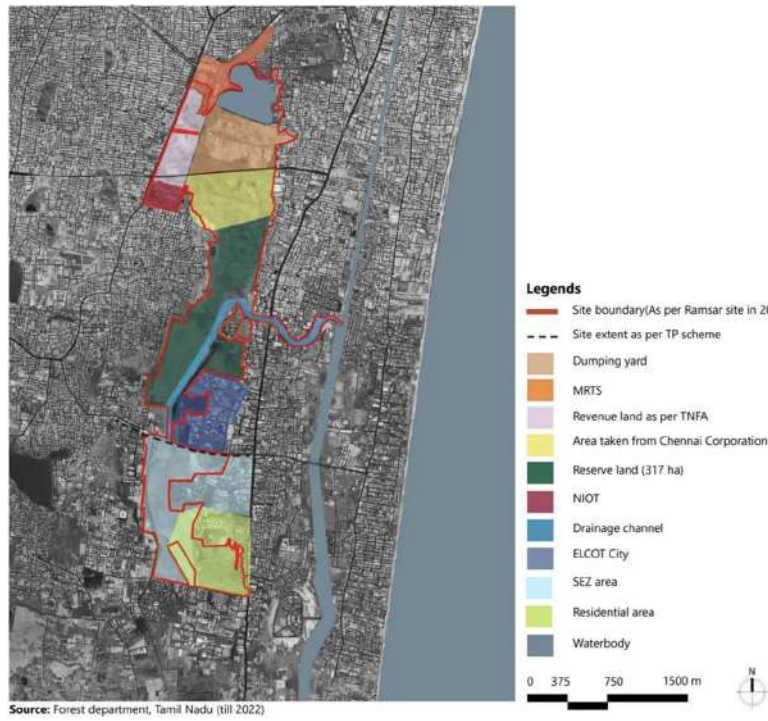

Fig. 10: Land ownership inside the marshland (source: Forest department, Tamil Nadu).

Fig. 11: Flood-affected areas.

taken over a significant area of marshland. In contrast, IT corridors and residential apartments have been developed in the area. The dumping yard comprises 173.33 Ha, land taken from Chennai Corporation is 170.405 Ha, channel of drainage is 54.21 Ha, Elcot consists of 85.43 Ha, revenue land as per TNFA is 131.55 Ha, and the SEZ comprises 445 Ha. Previously, the Palikaranai marshland was used up to the point where the Perumbakkam main road exists (Government of Tamil Nadu 2022). Subsequently, the SEZ area was also covered when it was declared a Ramsar site. The distribution of land ownership inside the marshland is mapped in Fig. 10.

Primary Data Collection

Flood-affected Areas

Fig. 11 shows the streets, residential areas, and shops in West Karapakkam, with markings indicating the water levels reached during past flood events. Specifically, the images highlight the flood levels from the 2015 Chennai floods, which were particularly devastating.

The floodwaters reached significant heights, submerging homes and businesses and causing widespread damage to infrastructure and property. The economic impact of the floods was severe, as many residents and shopkeepers lost their livelihoods and possessions due to the floods. Thoraipakkam, located below the OMR road level, has experienced recurring flooding during heavy rains due to its lower elevation. In the 2015 floods, water from the OMR flowed into the inner streets, leaving them submerged under 4 to 5 ft of water for nearly a week. A scrap shop in the area suffered severe damage, causing the owner an economic loss of approximately ₹50,000, and other residents faced similar hardships as their homes and businesses were inundated. A nearby apartment complex close to the Pallikaranai Marshland experienced severe waterlogging during both

the 2015 and 2023 floods, worsened by the overflowing marshland. Residents faced numerous challenges, including stagnant water, poor sanitation, property damage, and submerged vehicles, leading to health risks and disruption of daily life. During such events, residents struggle to access essential supplies, exacerbating their difficulties. The problem of waterlogging in Thoraipakkam persists in its inner streets because of rainwater flowing from the elevated OMR road. Stagnant water often remains for approximately a week, posing significant health and sanitation challenges and increasing the risk of waterborne diseases. These prolonged floods continue to cause economic losses and disrupt community well-being.

Questionnaire

A questionnaire survey was conducted within the study area and around the Pallikaranai Marshland to assess flood susceptibility across various environmental and infrastructural settings in Chennai. A total of 247 households comprising 1,067 individuals were selected as the survey sample. The selected locations encompassed high-, moderate-, and low-risk flood zones, enabling a comprehensive evaluation of flood hazards. The data collected serve as critical indicators for analyzing the impact of climate change, drainage infrastructure, urbanization, and hydrological changes on flood occurrences.

The findings indicate that a significant proportion of residents experienced severe disruptions due to flooding, including substantial losses of property and belongings. Many households were forced to relocate temporarily for safety as floodwaters inundated residential areas and roadways. Additionally, prolonged water stagnation and poor sanitation contributed to disease outbreaks and other health issues in the affected communities. The survey offered valuable insights into the intensity and causes of flooding in the region. The primary driver was identified as overflow of the river and marshland, exacerbated by heavy rainfall exceeding the capacity of the existing drainage infrastructure, thereby intensifying the flood impact on local populations.

Drainage Infrastructure

In several parts of Chennai, including Kannagi Nagar and West Karapakkam, the convergence of stormwater and greywater within the same drainage systems poses significant challenges to urban flood management. In Kannagi Nagar, drains discharge both stormwater and untreated greywater into the Okkiyam Maduvu canal, which ultimately connects to the Buckingham Canal system. This mixing of waste and runoff water not only degrades water quality but also reduces the drainage system efficiency by increasing the risk of clogging, sedimentation, and overflow during intense rainfall events. Similarly, in West Karapakkam, the drainage outlets exhibit similar dual usage, leading to frequent overflows during the monsoon season. The accumulation of stagnant water on streets causes considerable inconvenience to residents and poses serious sanitation risks to them. Thoraipakkam faces recurring water stagnation challenges despite stormwater drains on most streets. The primary issue lies in poor maintenance, as blockages, silt buildup, and a lack of regular cleaning prevent proper drainage during heavy rains. This causes rainwater to accumulate on the streets, thereby worsening the problem. In comparison, Karapakkam

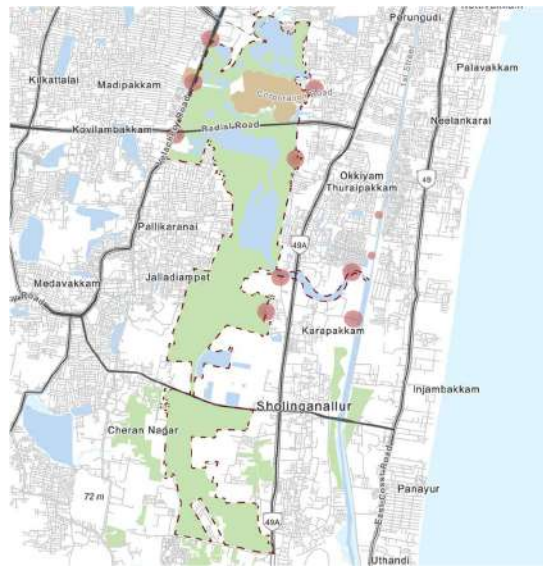

Fig. 12: Sewage outlets.

has fewer drainage facilities, intensifying the risk of flooding. Additionally, the improper use of stormwater drains for disposing of grey water in Karapakkam leads to blockages, further diminishing their effectiveness.

These cases emphasize the importance of maintaining the existing urban drainage infrastructure to ensure its continued capacity to manage water flow effectively. Integrating blue–green infrastructure into metropolitan planning has been shown to markedly improve climate adaptation and flood resilience (García Sánchez & Govindarajulu 2023). Regular maintenance, combined with the implementation of integrated drainage solutions that separate greywater from stormwater, is essential for enhancing flood resilience and promoting better environmental health.

Analysis

A detailed analysis of the existing conditions and site synthesis enabled the delineation of the marshland into three distinct zones based on environmental sensitivity and anthropogenic pressures (Fig. 12)

Environmentally Critical Zone: This parcel has undergone extensive ecological degradation due to landfill activity and infrastructural encroachments. The construction of the 200-feet radial road has disrupted natural hydrological connectivity, resulting in the stagnation and contamination of water within this segment. The proximity of the Perungudi dump yard further exacerbates the deterioration of water quality, leading to the formation of chemically polluted stagnation zones.

Sensitive Zone: The central portion of the marsh, identified as a sensitive ecological area, has been subjected to pressures from cattle grazing, debris dumping, and a lack of protective measures along its periphery. This zone is connected to the Okkiyam Maduvu drainage channel, which is approximately 15 feet above the marsh's natural elevation, creating a hydrological disconnect that affects flow dynamics.

Unprotected Zone: Previously unrecognized as part of the marsh ecosystem and falling within a Special Economic Zone (SEZ) before its RAMSAR designation, this area exhibits potential for ecological restoration. It can serve as a terrestrial habitat for diverse faunal and avifaunal species.

Furthermore, the low-lying regions within the study area (Fig. 13) have been officially designated as aquifer recharge zones by the Chennai Metropolitan Development Authority (CMDA), underscoring their hydrological significance in urban water management strategies.

Data Sets

In this research, Digital Elevation Model (DEM) and slope information were collected from the United States Geological Survey (USGS) based on the Shuttle Radar Topography Mission (SRTM) 1 Arc-Second Global dataset. This data set captures high-resolution elevations with a near 30-meter spatial resolution to ensure that precise topographic presentation is accomplished. Recent studies combining machine-learning models with geospatial datasets have further strengthened flood-risk prediction in

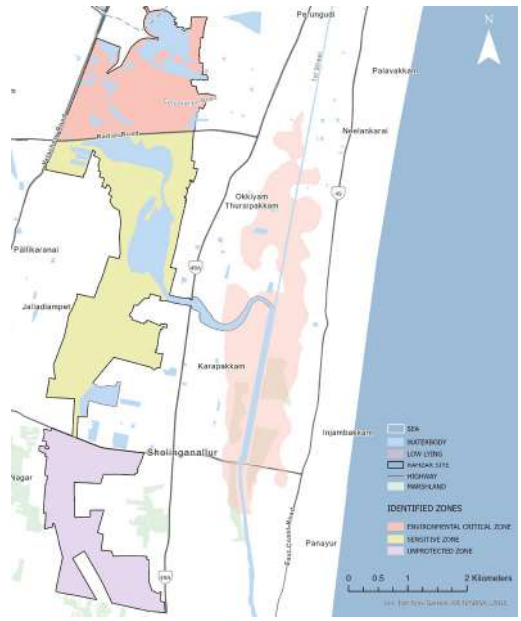

Fig. 13: Identified Zones.

urban catchments (Miranda et al. 2023). The DEM has been applied in terrain analysis and slope computed employing GIS-based image processing to provide surface gradients, as well as landscape differences assessment. Important spectral indices like the Normalized Difference Water Index (NDWI) were collected from the United States Geological Survey (USGS) based on the Landsat 8-9 OLI/TIRS C2L1 (30 Meter Spatial Resolution) dataset. Their merge enabled reliable characterization of the terrain, thereby making it viable to apply to applications in hydrology, geomorphology, as well as the environment's modelling.

Sentinel-2 (S2) multispectral imagery was used for land use classification and analysis. The high spatial and spectral resolution of Sentinel-2 (10 m resolution) data allowed precise discrimination of different land cover types, such as vegetation, water bodies, urban, and bare land.

Flood Inundation Model

Flood inundation modeling in QGIS using a Digital Elevation Model (DEM) involves a systematic methodology for identifying flood-prone areas based on topographic characteristics and hydrological analysis.

Flood Depth = Water Surface Elevation – DEM

The process begins by importing a high-resolution DEM, such as SRTM or LiDAR, into QGIS, followed by preprocessing with the Fill Sinks (Wang & Liu) tool to remove depressions and ensure accurate water flow representation. Hydrological flow analysis was then conducted using Tau DEM, which generates flow direction and accumulation maps to delineate drainage patterns and potential flood pathways. To estimate the flood extent, the Floodplain Delineation Plugin or Raster Calculator was used to subtract DEM elevations from predefined water surface levels, identifying inundated areas. Flood depth was calculated using the equation, producing a raster layer that represents varying flood depths across the terrain.

A flood inundation map was generated to assess flood depths of 1 and 2 m. As shown in Fig. 14, this resulted in severe damage, particularly in low-lying areas. Notably, regions within 500 m on either side of the Buckingham Canal experienced significant flooding, severely impacting the residents of Thoraipakkam. Additionally, the institutional and industrial zones of Karapakkam were affected, with flood impacts varying according to land use patterns. Furthermore, Velachery, Perungudi, and Medavakkam faced extensive inundation owing to inadequate drainage infrastructure and disruptions to natural water flow, exacerbating the flood severity in these regions.

Flood Hazard Index

The FHI is a multi-criteria decision analysis method that uses the Analytical Hierarchy Process (AHP) to evaluate different factors contributing to flood hazards.

Analytical Hierarchy Process

The AHP process for flood assessment and mapping involves using a multi-criteria decision-making method to evaluate various factors influencing flood risk and then assigning weights to the factors based on their relative importance. The weights were then used in a weighted overlay analysis within a GIS environment to create a flood susceptibility or hazard map. Maximum entropy modelling has also been successfully applied in

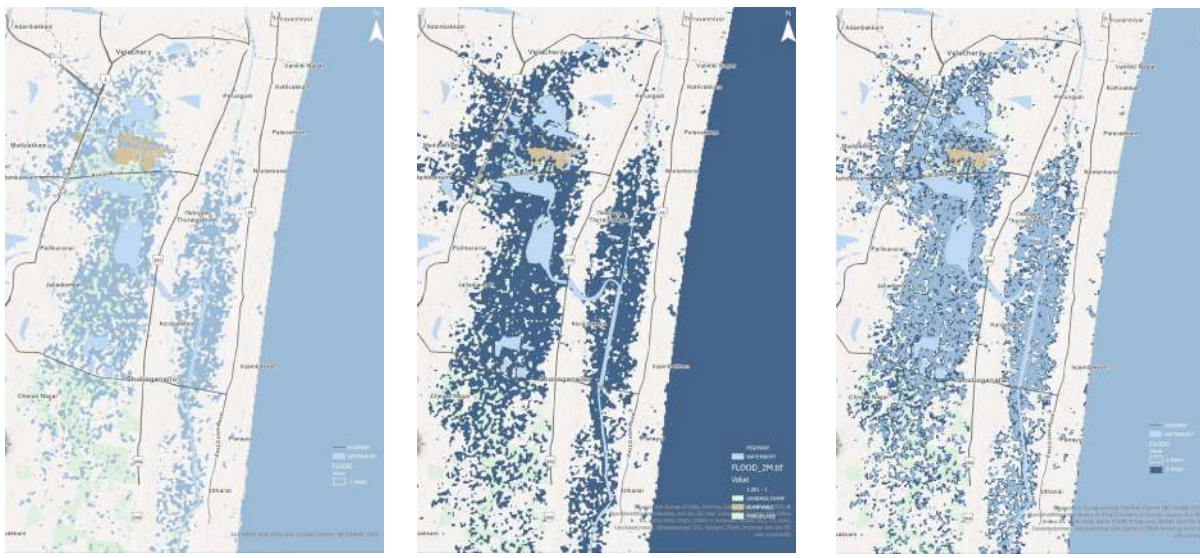

Fig. 14: a) 1 meter flood depth. b) 2 meter flood depth c) Flood inundation map.

Indian catchments for delineating flood-hazard zones (Kalita et al. 2025).

Identifying Key Factors and Criteria

The AHP focuses on identifying and structuring the factors that contribute to flooding, as shown in Table 2. Recent advancements that integrate remote sensing with machine-learning models highlight improved accuracy in identifying flood-susceptible zones (Ahmed et al. 2024). In this study, the seven most effective FCFs were selected for processing: elevation, slope, rainfall, NDWI, LULC, river distance, and flow length (Fig. 15). The metadata sources and descriptions of all FCFs are listed in Table 3.

Weight Assignment Using Pairwise Comparison

To evaluate the relative importance of various factors influencing flood hazard, a pairwise comparison matrix was constructed using the Analytic Hierarchy Process (AHP) methodology. The pairwise comparison values are calculated as:

$$PCMij = Wj/Wi$$

Where Wi and Wj are the weights of factor *i* and factor *j*, respectively.

In this approach, each factor is systematically compared with every other factor based on its contribution to flood susceptibility. The comparisons were made using the Saaty scale (1–9), which allows experts to quantify how much more important one factor is over another factor. This scale ranges from 1 (equal importance)

Fig. 15: Flood hazard index.

Table 3: Metadata of the utilized datasets for flood hazard susceptibility mapping.

Main factor	Sub Factor	Data description	Source	Data Acquired
Topographical factor	Elevation [m]	Shuttle Radar Topography Mission (SRTM) 1 Arc-Second Global dataset (30 Meter Spatial Resolution)	https://earthexplorer.usgs.gov/	2024
	Slope	Shuttle Radar Topography Mission (SRTM) 1 Arc-Second Global dataset (30 Meter Spatial Resolution)	https://earthexplorer.usgs.gov/	2024
Terrain factor	LULC	Sentinel 2 - S2, The high spatial and spectral resolution of Sentinel-2. (10 m resolution) (2024)	https://livingatlas.arcgis.com/landcoverexplorer/	2024
Hydro logical factor	Rainfall [mm]	India Meteorological Department Spatial resolution: 0.25° × 0.25° (~25 km × 25 km)	https://mausam.imd.gov.in/	2024
	River distance	Shuttle Radar Topography Mission (SRTM) 1 Arc-Second Global dataset (30 Meter Spatial Resolution)	https://earthexplorer.usgs.gov/	2024
	Flow length	Shuttle Radar Topography Mission (SRTM) 1 Arc-Second Global dataset (30 Meter Spatial Resolution)	https://earthexplorer.usgs.gov/	2024
	NDWI	USGS - Landsat 8-9 OLI/TIRS C2L1 (30 Meter Spatial Resolution)	https://earthexplorer.usgs.gov/	2024

Table 4: Pairwise Comparison Matrix.

Factors	Rainfall	Elevation	LULC	Slope	River Distance	Flow Length	NDWI
Rainfall	1	1.2	1.5	3	6	6	6
Elevation	0.83	1	1.25	2.5	5	5	5
LULC	0.67	0.8	1	2	4	4	4
Slope	0.33	0.4	0.5	1	2	2	2
River Distance	0.17	0.2	0.25	0.5	1	1	1
Flow Length	0.17	0.2	0.25	0.5	1	1	1
NDWI	0.17	0.2	0.25	0.5	1	1	1

Table 5: Derived weights.

Factors	Derived Weight	Derived Weight [%]
Rainfall	0.3	30%
Elevation	0.25	25%
LULC	0.2	20%
Slope	0.1	10%
River Distance	0.05	5%
Flow Length	0.05	5%
NDWI	0.05	5%

to 9 (extreme importance of one over the other) (Table 4).

Weights were then assigned to each factor based on expert judgment, considering the relative influence of each factor in determining flood hazards. The resulting matrix captures the preferences and priorities in a structured form, providing the basis for calculating the normalized weights and ensuring consistency in the decision-making process. The normalized weights for each factor are presented in Table 5.

Derived Weights and Consistency Check

To find the Weighted Sum Vector, multiply the Pairwise Comparison Matrix (PCM) by the weight vector (the derived weights from normalization). The results are shown in Table 6 and the consistency evaluation results are detailed in Table 7.

Let's denote:

A = Pairwise Comparison Matrix (7x7)

W = Weight Vector (7x1)

Now compute: WSV = A x W

The principal eigenvalue (λ_m) is computed by averaging the values of WSV/W:

λ_m = (7.15 + 7.06 + 7.11 + 7.05 + 7.04 + 7.04 + 7.04)/7 = 7.071

The Consistency Index (CI) is calculated as:

$CI = (\lambda_m - n) / (n - 1) = (7.071 - 7)/6 = 0.0118$

The Consistency Ratio (CR) is calculated using the Random Index (RI), which for n = 7 is 1.32:

$CR = CI/RI = 0.0118/1.32 = 0.0089$

Because the calculated Consistency Ratio (CR = 0.0089) is significantly less than 0.1, the judgments in the pairwise comparison matrix are considered consistent. This validates the reliability of the weight derivation and confirms that the matrix is suitable for further analysis in the AHP.

Flood Hazard Map

Using the collected spatial and environmental data, a Flood Hazard Map was developed in GIS through reclassification and standardization. Each input raster was first normalized and then reclassified on a scale of 1–5, where 1 represents a very low flood hazard, and 5 represents a very high flood hazard (Table 8).

Table 6: WSV Calculation.

Factor	WSV Calculation	WSV
Rainfall	(1×0.30) + 1.2×0.25 + 1.5×0.20 + 3×0.10 + 6×0.05 + 6×0.05 + 6×0.05	2.145
Elevation	0.83×0.30 + 1×0.25 + 1.25×0.20 + 2.5×0.10 + 5×0.05 + 5×0.05 + 5×0.05	1.764
LULC	0.67×0.30 + 0.8×0.25 + 1×0.20 + 2×0.10 + 4×0.05 + 4×0.05 + 4×0.05	1.421
Slope	0.33×0.30 + 0.4×0.25 + 0.5×0.20 + 1×0.10 + 2×0.05 + 2×0.05 + 2×0.05	0.705
River Distance	0.17×0.30 + 0.2×0.25 + 0.25×0.20 + 0.5×0.10 + 1×0.05 + 1×0.05 + 1×0.05	0.352
Flow Length	0.17×0.30 + 0.2×0.25 + 0.25×0.20 + 0.5×0.10 + 1×0.05 + 1×0.05 + 1×0.05	0.352
NDWI	0.17×0.30 + 0.2×0.25 + 0.25×0.20 + 0.5×0.10 + 1×0.05 + 1×0.05 + 1×0.05	0.352

Table 7: Weight Consistency.

Factor	Weight [W]	Weighted Sum	WS/W
Rainfall	0.3	2.145	7.15
Elevation	0.25	1.764	7.06
LULC	0.2	1.421	7.11
Slope	0.1	0.705	7.05
River Distance	0.05	0.352	7.04
Flow Length	0.05	0.352	7.04
NDWI	0.05	0.352	7.04

Table 8: Flood criteria ranking of the thematic layer.

Factors/criterion	Class value range	Reclassified value	Type
Elevation [m]	-7.99 – 1	5	Numerical
	1.01 – 3	4	
	3.01 – 6	3	
	6.01 – 9	2	
	9.01 – 19	1	
Slope [°]	0.01 - 0.36	5	Numerical
	0.37 - 1.29	4	
	1.3 - 3.67	3	
	3.68 - 9.8	2	
	9.81 - 25.57	1	
LULC	Bare Ground	5	Categorical
	Built area	4	
	Crops	2	
	Flooded Vegetation	3	
	Range land	5	
	Trees	2	
	Water	1	
River Distance [m]	0 – 100	5	Numerical
	100 - 223.05	4	
	223.05 - 316.31	3	
	316.31 - 576.34	2	
	576.34 - 1548.68	1	
Flow length [m]	100 - 230	5	Numerical
	230 – 510	4	
	510 – 790	3	
	790 – 1080	2	
	1080 – 1210	1	
NDWI	(-0.43) - (-0.25)	5	Numerical
	(-0.25) - (-0.18)	4	
	(-0.17) - (-0.13)	3	
	(-0.12) - (-0.03)	2	
	(-0.02) - (0.07)	1	

The individual thematic layers contributing to the FHI like DEM, slope, LULC, river distance, flow length, and NDWI are illustrated in Fig. 16. Elevation and slope are significant parameters that significantly impact flood risk. Gentle slopes and low elevations allow for water accumulation, making the area more susceptible to floods (Vojtek & Vojtekova 2019). Elevation also influences river inundation and atmospheric conditions, such as precipitation (Najibi & Devineni 2023). In this study, elevation and slope were extracted from the 30 m SRTM-DEM in GEE. In this study, elevations varied from -7.99 m to 19 m, where lower elevations were more susceptible to flooding than steeper, higher regions. A mean

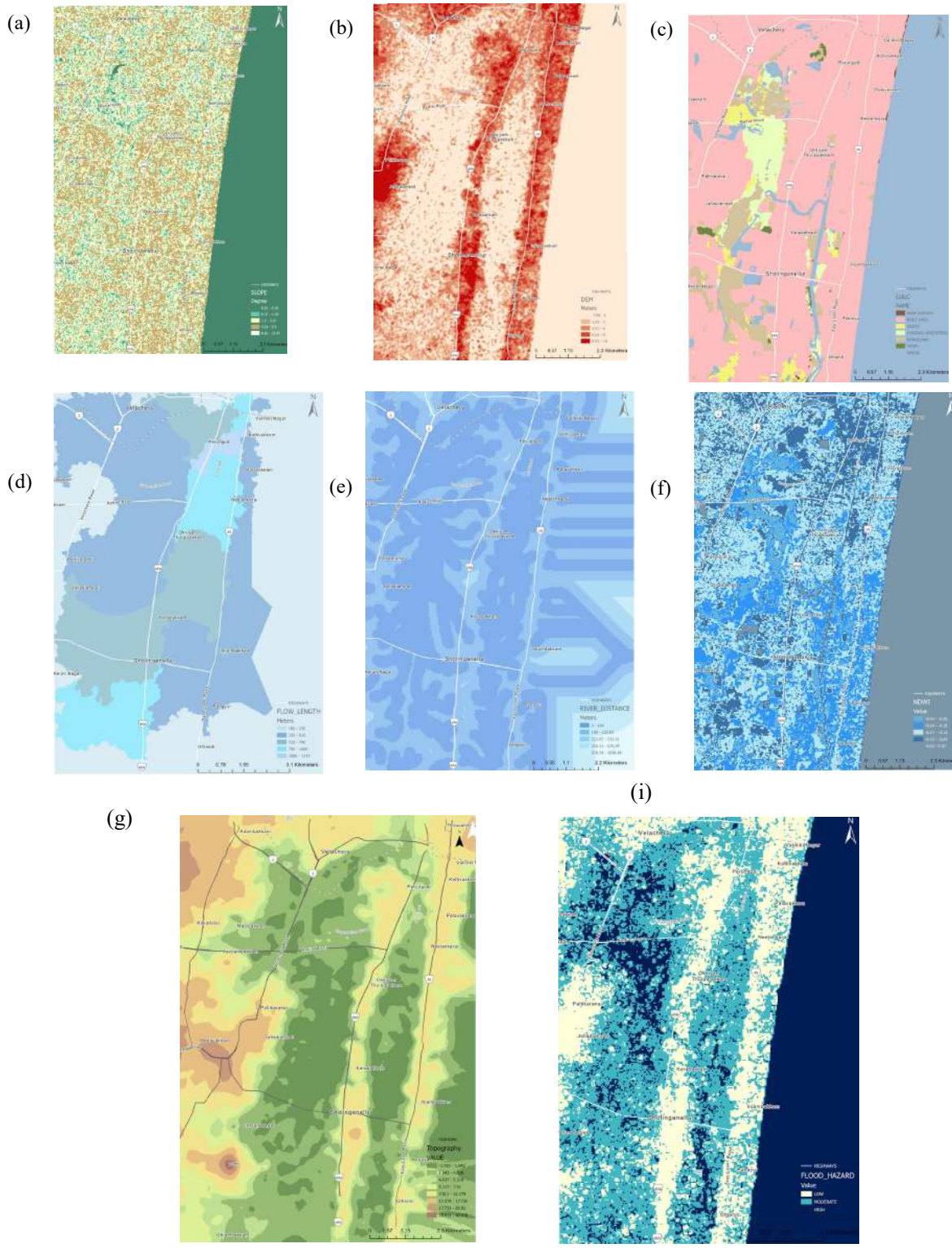

Fig. 16: a) DEM b) Slope c) LULC d) River distance e) Flow length f) NDWI g) Topography h) flood hazard map.

slope of 2.36° justifies the higher risk of flooding on flat slopes. The NDWI is a remote sensing index used to identify water bodies and moisture levels in vegetation. It is used for the important task of flood hazard mapping by delineating regions with high water content, which could be flood risk areas. It is computed using near-infrared (NIR) (Band 3 in Landsat 8) and Shortwave Infrared (SWIR) or green bands (Band 5 in Landsat 8) of satellite data. Water absorbs NIR and reflects green light; therefore, water features appear as high positive NDWI values.

$$NDWI=(Green+NIR)/(Green-NIR)$$

The NDWI values for the study area varied between -0.43 and 0.07, with a standard deviation of 0.10, showing changes in the submerged or waterlogged zones. The distance to rivers and streams controls the probability and magnitude of flood occurrences through proximity to water courses. Flow length is the path length that water moves across the land to flow to the closest stream or river. The flow length of our study area varied from 0.01 to 0.12 sq km, resulting in shorter flow lengths, which contributed to quicker runoff and increased flood risk, thereby boosting the FHI score. The 2024 LULC was obtained from ESA Sentinel-2 imagery at a 10 m resolution. Land cover differences are vital when explaining water storage and hydrological processes, as they have an immediate effect on flooding (Ma et al. 2023). The land use and land cover in this study encompassed seven prominent classes: tree cover, rangeland, flooded vegetation, cultivated crops, buildings, exposed land, and water surfaces.

Flood Hazard Index

The Flood Hazard Index (FHI) was derived using these standardized layers. The FHI serves as a composite indicator to assess flood risk, integrating a range of physical and environmental parameters, including rainfall, slope, elevation, land use/land cover (LULC), drainage density, Normalized Difference Water Index (NDWI), stream order, river distance, and flow length.

Each parameter was assigned a weight based on its relative importance, which was determined using the Analytic Hierarchy Process (AHP) (Table 9). The final FHI was computed using a weighted overlay technique, where the reclassified and normalized values were multiplied by their corresponding AHP-derived weights and summed across all factors.

$$\text{Normalized Value (X)} = (\text{Raw} - \text{Min})/(\text{Max} - \text{Min})$$

$$FHI = \sum (Wi \times Xi)$$

$$FHI = 0.1650+0.1750+0.1500+0.0400+0.0100+0.0275+0.0350=0.6025$$

A Flood Hazard Index (FHI) score of 0.60 signifies a moderate to high flood hazard potential within the study area. This outcome reflects the combined influence of key parameters, such as intense rainfall, low elevation, and vulnerable land use/land cover (LULC) types. A comprehensive weighted overlay analysis was conducted in a GIS environment using the Analytic Hierarchy Process (AHP), wherein each factor was assigned a weight based on its relative importance on a 1–5 scale. Seven critical factors–rainfall, slope, elevation, LULC, NDWI, river distance, and flow length–were normalized and integrated to compute the FHI. Hazard zones were classified using the Natural Breaks (Jenks) method, which effectively distinguishes areas with varying flood risks. The resulting FHI map identified the study area as being highly susceptible to flooding.

RESULTS AND DISCUSSION

The Pallikaranai Marshland and Buckingham Canal, once integral components of Chennai's natural drainage and flood mitigation systems, have been severely degraded due to rapid urbanization, encroachments, and inadequate drainage infrastructure. The marshland, historically functioning as a natural flood buffer, has experienced a significant decline in its water-holding capacity. This is primarily attributed to extensive land reclamation for residential, commercial, and industrial purposes, resulting in excess stormwater inundating

Table 9: Flood Hazard Index.

Factor	Raw Value	Min Value	Max Value	Normalized Value [X]	Weight [W]	Weighted Score [W × X]
Rainfall [mm]	891.4	264.6	1663.8	0.55	0.3	0.165
Elevation [m]	2	-8	19	0.7	0.25	0.175
LULC	5.5	1	7	0.75	0.2	0.15
Slope [°]	5	0.4	25.57	0.4	0.1	0.04
River distance [m]	300	0	1548.68	0.2	0.05	0.01
Flow Length [m]	700	100	1210	0.55	0.05	0.0275
NDWI	-0.05	-0.44	0.07	0.7	0.05	0.035

adjacent urban areas during periods of intense rainfall. Similarly, the Buckingham Canal, originally engineered as a critical stormwater drainage channel, has been compromised by siltation, encroachments, and pollution. These factors have drastically reduced its hydraulic capacity, limiting its ability to effectively convey floodwaters. As a consequence, low-lying areas such as Thoraipakkam and Karapakkam are increasingly susceptible to severe waterlogging during monsoonal events.

A comprehensive analysis of the Pallikaranai Marshland has delineated distinct ecological zones (Fig. 13) based on prevailing environmental conditions. The marshland's chronology (Figs. 3&4) shows a consistent pattern of degradation, directly increasing flood vulnerability in the region. To mitigate further deterioration and preserve the marsh's ecological and hydrological functions, a series of strategic interventions is proposed. To prevent future encroachments and monitor anthropogenic activities, geo-fencing of the marshland boundaries is recommended (Fig. 17(a)). This digital perimeter surveillance system will enable real-time monitoring of unauthorized vehicular movement and construction activities, transmitting instant alerts to enforcement authorities. Integrated with GIS-based databases, the system will also support automated land use change detection and generate actionable reports for regulatory intervention.

In parallel, ecological edge restoration (Fig. 17(b)) is proposed to rehabilitate the marsh's degraded margins. Wetland restoration has proven effective in strengthening regional flood resilience in similar urban–wetland systems (Sundaram et al. 2022). This measure aims to stabilize marsh boundaries, mitigate erosion, and reinforce habitat

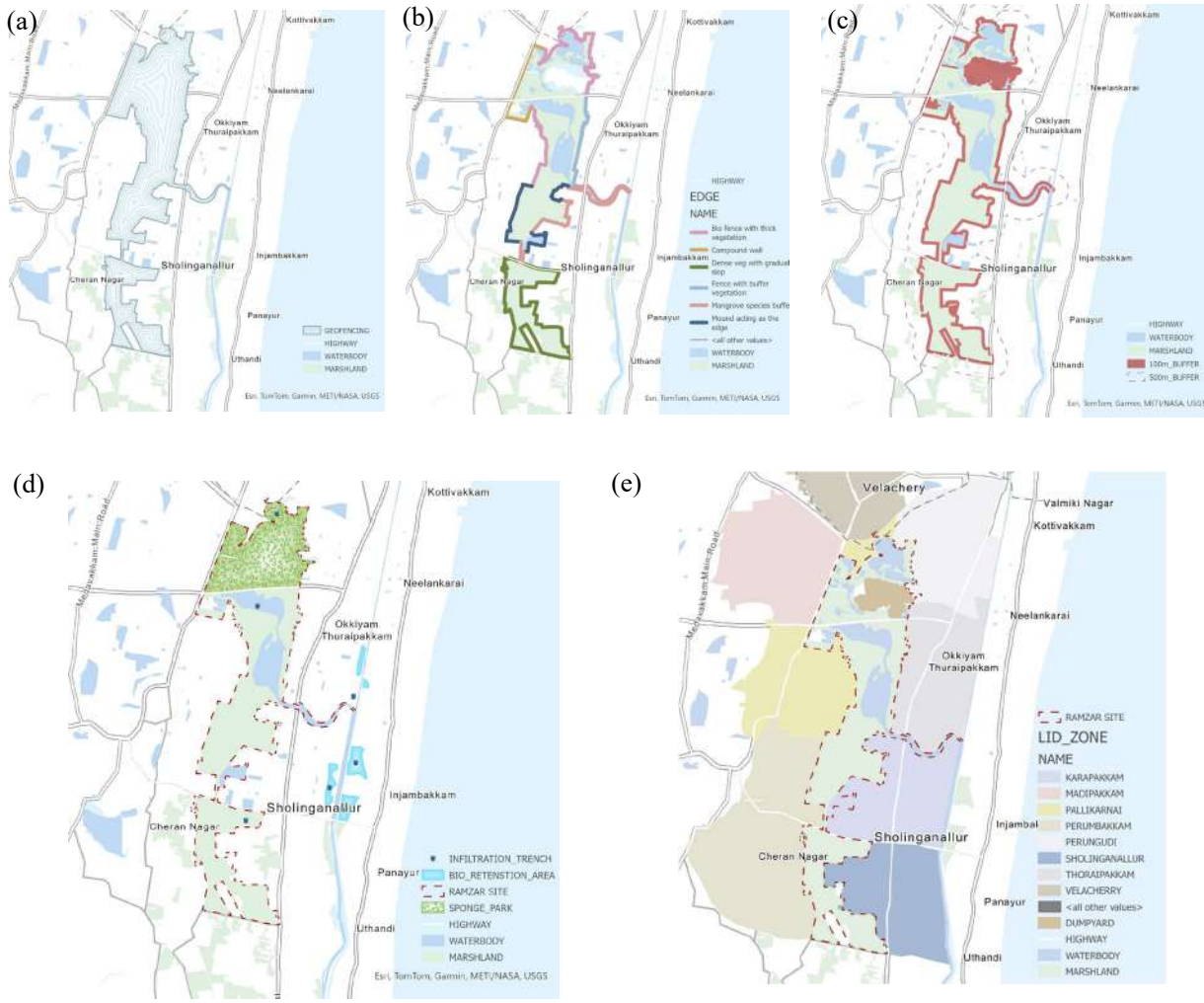

Fig. 17: a) Geo-fencing, b) Edge restoration, c) No development zone, and d) Recharge wells, bio retention ponds, bioremediation and Sponge Park, e) low-impact development zones.

conditions for native biodiversity. The restoration strategy will be tailored to the specific ecological character of each edge zone. To regulate future development, the enforcement of a 100-meter "No Development Zone" (Fig. 17(c)) around the marshland is strongly advocated, in accordance with the Wetland (Conservation and Management) Rules, 2017. This regulatory buffer will protect the marsh's core zones from further urban intrusion. The installation of recharge wells is proposed in both the designated Aquifer Recharge Area (ARA) near the Buckingham Canal and in regions of greater water depth within the marshland. These interventions will enhance groundwater recharge, mitigate seasonal water shortages, and support aquifer sustainability.

To complement these hydrological strategies, bio-retention ponds will be established in identified low-lying areas with high percolation potential. These ponds will serve as decentralized stormwater treatment systems, filtering runoff and improving water quality through natural processes (Fig.17(d)). A critical long-term intervention is the bioremediation of the Perungudi landfill (Fig. 17(d)), which currently poses severe environmental risks to the adjacent marshland. The proposal seeks to transform the degraded dump site into a sponge park, facilitating stormwater absorption, reducing surface runoff, and enabling ecological regeneration. This concept draws on a successful precedent in Indore, where a similar intervention was implemented over three years (John Snow Inc 2022).

Land-use policies should promote sustainable practices, such as green roofs, rainwater harvesting, and rain gardens, across residential, commercial, and institutional developments, with mandatory compliance with LEED or GRIHA certification standards. Low-density, non-polluting industries, such as those classified as white-category industries, may be permitted. Additionally, the installation of Decentralized Wastewater Treatment Systems (DEWATS) should be made mandatory to treat wastewater locally and prevent its discharge into ecologically sensitive areas, such as the Pallikaranai Marshland and the Buckingham Canal.

These guidelines recommend designating surrounding urban areas, Karapakkam, Madipakkam, Pallikaranai, Medavakkam, Perungudi, Perumbakkam, Sholinganallur, and Thoraipakkam as Low-Impact Development (LID) zones (Fig. 17(e)) to preserve ecological integrity. Furthermore, a 100-meter buffer zone should be established as a no-development area to curb further urban encroachment and maintain the flood mitigation capacity of the marshland. Implementing these measures is essential to enhance urban flood resilience and promote long-term environmental sustainability.

CONCLUSIONS

In conclusion, this study underscores the crucial roles of the Pallikaranai Marshland and Buckingham Canal in flood mitigation and urban water management. The application of geospatial modelling and hydrological analysis provides critical insights into flood hazard risks, facilitating the identification of vulnerable areas and the formulation of effective mitigation measures. Nature-based solutions, including wetland restoration and bioremediation, coupled with stringent land-use policies, can significantly enhance flood resilience in the region.

The compounded effects of climate change and urbanization have intensified flood risks in the Pallikaranai Marshland–Buckingham Canal corridor, highlighting the urgent need for sustainable urban planning in the region. The progressive shrinkage of marshlands due to land conversion, waste dumping, and infrastructure expansion has diminished their natural flood-buffering capacity. Similarly, the Buckingham Canal has suffered from siltation, pollution, and unregulated development, which has exacerbated urban flooding. Historical flood events, such as those in 2015 and 2021, underscore the increasing vulnerability of low-lying areas, such as Thoraipakkam and Karapakkam, owing to intensified rainfall, rising sea levels, and reduced drainage efficiency.

To mitigate these challenges, it is imperative to adopt holistic urban planning strategies that emphasize wetland conservation, the restoration of natural drainage systems, and the remediation of degraded areas. The implementation of Low-Impact Development (LID) principles, integration of green infrastructure, and reinforcement of stormwater management policies are essential steps toward reducing flood risks. A comprehensive, ecosystem-based approach that aligns climate resilience with sustainable urban expansion is critical for ensuring long-term flood mitigation and environmental sustainability in Chennai.

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A Modified Neural Network for Predicting the Solar Photovoltaic Power Generation Using Weather and Operational Parameter

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ABSTRACT

Solar PV systems often face challenges feeding power into the local grid due to weather dependence. Although solar PV generation is variable, it is predictable and can help maintain grid stability. In this study, a modified neural network is developed to forecast power generation for a 500-kW solar farm under Thailand's climatic conditions. Year-round operational data from the solar PV plant are used to train the forecasting model, and over 15% of the period is reserved for power generation prediction and validation against the actual power profile. Keras provides an effective interface, with TensorFlow as the backend engine, which is well-suited for high-computation processes. For training and testing, a batch size of 32 and 50 epochs is used as standard parameters, helping avoid overfitting and improving computational efficiency. It was found that during 75% of the sunshine period, the solar PV system generated 50% of its nominal DC capacity, indicating efficient operation. A 0.22 kW average difference between the forecasting model and the actual power profile indicates 99.86% accuracy over the testing period. The difference between actual and predicted power ranged from 2.88 kW to -4.67 kW, and the corresponding MAE, MSE, and RMSE were 0.87, 1.32, and 1.15, respectively. Furthermore, the developed ANN-based forecasting model is highly recommended for commercial use to avoid penalties from the grid authority and enhance grid stability.

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INTRODUCTION

The last three decades have been among the most significant periods of technological advancement, and electrical energy has become an integral part of daily life. In the 1990s, energy production relied heavily on fossil fuels, such as coal and petroleum. In the 2000s, following coal, natural gas became the predominant energy source in the United States and other developing countries. Limitations of fossil fuel resources, rising costs, and environmental pollution have led to a reduction in the use of conventional energy sources (Jitoko et al. 2021). Furthermore, the late 2010s saw a forced shift toward renewable energy-based power generation. Coal and other conventional energy sources have declined significantly, with a corresponding increase in renewable energy sources (Dhanraj et al. 2022). Several forms of renewable energy systems are available to meet global energy demand, particularly solar energy, which is widely recognized as an alternative to reduce fossil fuel consumption due to its simplicity of operation and ability to meet conventional loads, ranging from small electronic gadgets to spaceships. Solar Photovoltaic (PV) modules are silicon cells that convert photons into electrical energy through the photovoltaic effect (Karthikeyan et al. 2018, Velmurugan et al. 2022). A solar cell is a p-type and n-type semiconductor that favors exciting electrons and creating electron-hole pairs. The solar cell's internal electric field separates electrons and holes, driving them toward the p-type and

n-type junctions. This results in electrons flowing to the external circuit, producing current. The electron flow toward the external circuit depends on the photon energy falling on the solar cells (Techo et al. 2024, Chand et al. 2022). This is the primary concern in solar PV power, as solar irradiance is not continuous and power production follows its trend, leading to grid instability. A large-scale solar PV system often faces penalties from the grid authority division due to power feed that is lower or higher than the expected range (Gandhi et al. 2024).

To improve grid stability and maintain the lifespan of the load, the local grid authority monitors alternative energy sources on a day-ahead and hourly basis using forecasting tools (Sankari & Kumar, 2023). In recent years, solar PV power forecasting has become essential for medium- and large-scale solar PV power plants. Several forecasting methods exist, including conventional mathematical modeling, machine learning, deep learning, and Artificial Neural Networks (ANNs) (Ahmed et al. 2020, Liu & Du, 2023). Faizan Tahir et al. developed a bidirectional long- and short-term memory model to predict solar PV power generation for the 10 MW Masdar project in the UAE. The Bayesian optimization technique was adopted to maintain higher prediction accuracy. Historical data, including solar irradiance, PV module temperature, ambient temperature, angle of incidence, sun zenith angle, sun altitude, and air mass ratio, were used to train the forecasting model. Notably, hyperparameter tuning played a significant role in improving the accuracy of the power prediction, with Root Mean Square Error (RMSE), Mean Square Error (MSE), and Mean Absolute Error (MAE) of 3.2560%, 21.252%, and 1.2310%, respectively (Tahir et al. 2024). Distributed solar PV power forecasting was conducted for the Netherlands location for the day ahead using 12 different models. Ensembles and deep-learning forecasting models were found to attain lower MAE, effectively eliminate unnecessary parameters, and capture efficient information from predictor variables. Second, the physical model shows its importance for day-ahead energy trading by delivering higher profits. By comparison, random forest and LSTM models are effective for technical performance and reliable for both single and aggregated solar PV systems (Visser et al. 2022). Unlike other forecasting models, ANN is simpler to compute and requires less computational power. The faster training time makes this prediction model efficient for solar PV power forecasting and applicable to real-time applications (Tavares et al. 2022, Pazikadin et al. 2020). Ledmaoui et al. developed a hybrid system in which solar power forecasting is performed by an Artificial Neural Network (ANN), and the energy meter is connected to the Internet of Things (IoT) to monitor the forecasted power and real-time power fed into the grid. The IoT is integrated with energy meters, as the solar PV system is located in the industrial area of Benguerir, Morocco, where energy demand is high. The developed ANN model exhibits accurate power prediction, making it versatile for energy trading and increasing the power plant's income. The correlation, MAE, and RMSE were found to be 0.694, 9.913, and 13.447, respectively (Ledmaoui et al. 2023). Forecasting power production for bifacial solar PV systems is challenging, as computing the bifacial solar PV and cooling roof system for different albedos is required. The examined system features a roof cooling system, as the bifacial solar system is situated in a desert region with higher PV module temperatures than in other tropical regions. With the help of the ANN model, the microgrid power system operates efficiently and reduces power curtailment with balanced demand-side management. The albedo surface significantly impacts power production; for example, an albedo range of 0.2 to 0.5 resulted in a 7.75% power enhancement, and further increasing the albedo to 0.8 resulted in a 14.96% power enhancement. The MSE for the different albedos of 0.2, 0.5, and 0.8 was 1.19540e-1, 1.95796e-1, and 1.60751e-1, respectively, and the correlation coefficients were 9.92416e-1, 9.88912e-1, and 9.91884e-1, respectively (Ghenai et al. 2022). Furthermore, to analyze the forecasting accuracy for hot climatic conditions, a 4 kW solar PV system from Shaqra, Saudi Arabia, was used to predict the DC power and PV module operating temperature. The hybrid model combines Multilayer Feedforward Neural Networks (MFFNNs) with a Genetic Algorithm (GA) and a Multiverse Optimizer (MVO) to enhance forecasting accuracy. Second, GA and MVO with MFFNNs favor controlling local minima and optimizing the network parameters. It was found that the Normalized Root Mean Square Error (NRMSE), MSE, MAE, and coefficients of determination were 2.78e-3, 1.30e-1, 1.07e-2, and 0.997222 for MFFNNs-GA and 7.11e-4, 3.33e-2, 7.22e-3, and 0.999289 for MFFNNs-MVO, respectively (Talaat et al. 2022). Sahin et al. examined a 500 kW solar PV power plant under Turkey's environmental conditions to find the relationship between weather and power production. Principal Component Analysis (PCA) improves the forecasting accuracy by controlling the feature dimension, and ANN captures the complex relationship between features and efficiency. The developed ANN model achieved lower RMSE and MAE values of 23.89 and 25.09, respectively, compared to the Multiple Linear Regression (MLR) model, which had R^2 values of 0.9628 and 0.901 (Sahin et al. 2023). According to the literature above, it is evident that forecasting solar PV power generation has become an essential process for maintaining grid stability. Several forecasting models are in practice, among which ANN models have gained popularity due to their ease of

computation, high prediction accuracy, and the ability to train the model with fewer parameters. The main objective of the present study is given below:

- This study developed a modified neural network called SPFNet, specifically designed to perform the regression task for Thailand's geographical conditions.
- A dynamic model architecture is created based on neural layers, activation functions, and kernel initializers to forecast solar PV power generation.
- The dependent and independent variables of solar PV systems are obtained from the NREL open-source data and analyzed statistically to understand their operational behavior.
- A comparative analysis is performed for the actual and predicted power generations, and the error metrics of MAE, MSE, and RMSE are used to validate the accuracy of the forecasting model.

MATERIALS AND METHODS

Study Description

This study developed an ANN forecasting model for a 500-kW solar PV power plant in Thailand equipped with monocrystalline PV modules and fixed-array tracking systems. Given the local geography, the PV modules are mounted at a 20-degree tilt and a 180-degree azimuth, which favors higher power generation and shorter payback periods. For solar power forecasting, meteorological and plant performance data are obtained from the NREL open-source dataset. The raw data include 24-hour timestamps (hourly intervals) of beam irradiance, diffuse irradiance, POA irradiance, ambient temperature, solar cell temperature, and DC array output for one year. Furthermore, to improve forecasting accuracy, the models are trained using only daylight periods.

ANN Model Training

Importing libraries: Solar PV power forecasting uses open-licensed Anaconda Navigator to create a controlled environment. Furthermore, the Jupyter Notebook processes the data and performs the modeling. Initially, libraries are imported to build an ANN model for predicting solar power. Pandas and NumPy are used for data manipulation and to handle large datasets for numerical operations. TensorFlow and Keras are used to leverage layers for building and training the ANN model. TensorFlow is more suitable for complex computations and offers greater built-in deployment support than PyTorch. Secondly, Scikit-learn is widely used for linear regression but is less effective for deep learning models and has poor scalability, especially in PV system power forecasting. Furthermore, Matplotlib and Seaborn are used to visualize the dataset, as they are widely used for solar PV power forecasting. After importing the libraries, a dataset is loaded using the 'd.read_csv' function for preprocessing and modeling tasks.

Dataset: A one-year dataset of solar PV power plant data is imported to build and train the ANN model at an hourly frequency. The main parameters in the dataset include beam irradiance, diffuse irradiance, and plane-of-array irradiance, as well as ambient temperature, PV cell temperature, and power output. Except for power output, all parameters are independent variables, as power generation depends on the irradiance and temperature. profile. Furthermore, converting the dataframe to a numpy array facilitates splitting the dataset into X and y, where X contains the independent variables and y contains the dependent variable.

Splitting, training and test sets: For training and testing the dataset, the train_test_split function from sklearn. model is used to split X and y into X_train and y_train for training, and X_test and y_test for testing the model. In this model, 15% of the data is used for testing by setting the parameter to 0.15, which helps ensure the robustness of the developed forecasting model. The random_state parameter was set to 42 to guarantee the reproducibility of the split. To ensure data distribution across the training and testing sets, the shapes are printed as X_train.shape, y_train.shape, X_test.shape, and y_test.shape.

Feature scaling: To normalize the data, StandardScaler from the 'sklearn.preprocessing' module is used, as it is essential for determining the features and preparing the variables in a comparable state. In this case, StandardScaler is employed for both the input and output variables of the forecasting. The training data are fitted and transformed to have a mean of 0 and a standard deviation of 1; the same conditions are applied to the testing data, respectively.

Creating a neural network: The neural network forecasting model is developed using the create_spfnet function, and solar PV power plant generation is predicted using TensorFlow and Keras. The list of neuron counts for each layer, activation functions, and kernel initializers plays a significant role in initializing the model. Dense layers are added to the neuron count, and the input dimensions are initialized. As a result of the forecasting, a final dense layer is added to the neuron count and predicts the power output.

Train and test: Following the development of the neural network, data training is performed with a batch size of 32 and running 50 epochs. A batch size of 32 indicates the number of samples used at a time for computing the gradient and updating the model. The epoch 50 means the model will

learn the training data pattern 50 times. The primary reason for selecting 50 epochs is to prevent overfitting. Secondly, increasing the epochs beyond 50 could lead to higher accuracy in solar power forecasting, but the computational cost is comparatively higher. As an effective method, several studies have adopted 50 epochs as the standard setpoint. Validation occurs for each training session to evaluate the neural network's performance and continually monitors the outcome. The verbose and history functions ensure the display of the progress and error metrics for each epoch, indicating performance and convergence over time.

Error metrics: These error metrics are important parameters in solar PV power forecasting, indicating the accuracy of the forecasting model and how it differs from actual power generation. Generally, MAE, MSE, and RMSE are primary error metrics that need to be evaluated to quantify the accuracy and robustness of a forecasting model.

***Mean Absolute Error*:** MAE is directionless and does not indicate that the predicted values are higher or lower than the actual value. It simplifies the average error magnitude, and the absolute value of the error is taken to treat all errors as a positive contribution, as expressed in Eq. (1).

$$MAE = \frac{1}{n}\sum_{i=1}^{n}|y_i - \hat{y}_i| \quad \ldots(1)$$

where, y_i = actual value, $\hat{y}_i$ = predicted value, and n = total number of predictions.

***Mean Square Error (MSE)*:** MSE is one of the standard error metrics widely used for analysing the prediction error following MAE. It is the square of the errors and is often validated against the actual power generation, as expressed in Eq. (2). The MSE is sensitive to outliers, where larger errors are penalised more, and is often used to train the regression model.

$$MSE = \frac{1}{n}\sum_{i=1}^{n}(y_i - \hat{y}_i)^2 \quad \ldots(2)$$

***Root Mean Square error (RMSE)*:** RMSE is the square root of MSE as expressed in Eq. (3). RMSE is sensitive to the large errors following MSE because large errors can significantly change the outliers of the error pattern, leading to lower forecasting accuracy. It is noted that the RMSE is non-negative, and when the value is zero, the prediction and actual values are identical.

$$RMSE = \sqrt{\frac{1}{n}\sum_{i=1}^{n}(y_i - \hat{y}_i)^2} \quad \ldots(3)$$

RESULTS AND DISCUSSION

Input Parameters

This study predicts 500 kW of solar PV power production for the Thailand location. To forecast power production, one year of meteorological data, including irradiance and ambient temperature, along with plant performance data such as solar cell temperature and power production (as shown in Fig. 1), is used to train the ANN forecasting model. To improve forecasting accuracy, beam, diffuse, and Plane of Array (POA) irradiance are utilized. Fig. 1 shows raw data in a 24-hour format and is further processed into 12-hour segments for training the forecasting model. In Thailand, daylight periods naturally begin at 6:00 AM on most days of the year. The annual average diffuse irradiance at the examined site is 54.05 W/m² and 112.55 W/m², lower than the beam irradiance and POA, respectively, due to fewer clouds and other physical obstacles. On the other hand, peak POA irradiance is recorded at 1096.85 W/m², which gives solar PV farms greater potential to generate higher electrical energy. Under tropical climatic conditions, increased solar irradiance generates higher power but also raises solar cell temperature due to the high ambient temperature. Notably, peak ambient and solar cell temperatures reached 38.1°C and 75.26°C, respectively, which exceed standard test conditions and considerably deteriorate power generation. It is well known that solar PV power generation is discontinuous, and higher solar cell temperatures worsen plant operation and economic stability. In this case, a high ambient temperature significantly reduces the DC power output during the summer. This study does not focus on reducing solar cell temperature. However, solar cell temperature patterns are trained with the ANN model to achieve temperature-corrected power prediction.

Statistical Analysis

As mentioned above, the raw solar PV power plant data are processed into a 12-hour format, which contains a total of 4,408 irradiance values, ambient temperatures, cell temperatures, and DC array power outputs used to train the ANN forecasting model to predict the DC power output. The mean POA irradiance was recorded as 431.41 W/m², and the corresponding DC power was 165.07 kW. In year-round performance, for 25% of the period, the solar farm generates 56.90 kW; notably, the mean and 50% reached 165 kW. Notably, over 75% of the time, the solar farm generates more than 50% of the installed DC capacity, indicating that it is operating on a profitable scale. Diffuse irradiance is lower than beam and POA irradiance in all conditions due to the best absorption. The solar cell is a current generator; an increase in solar irradiance increases both the current profile and the power profile simultaneously. Due to the nature of the solar irradiance pattern, power generation is sinusoidal, and a wide variation is observed over the 12 hours of daytime operation, with a standard deviation (SD) of 114224.17. The difference between beam

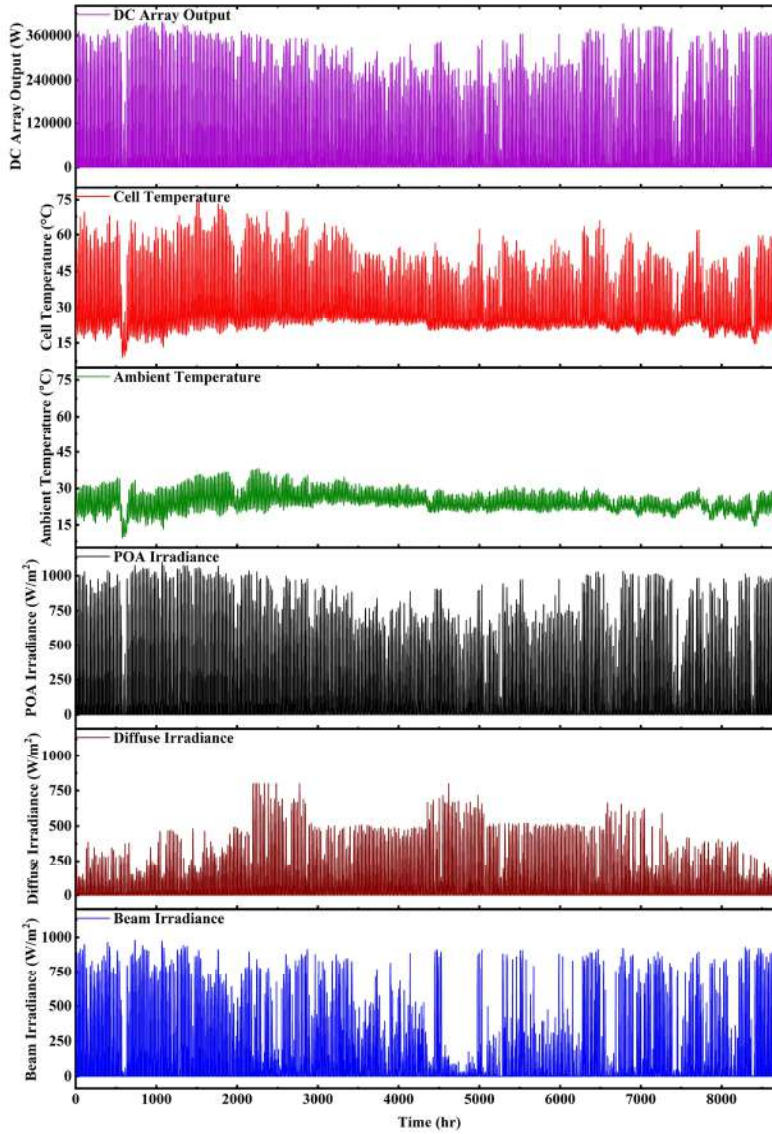

Fig. 1: Meteorological and plant performance data.

Table 1: Statistical analysis of meteorological and solar farm data.

	Beam Irradiance [W.m^{-2}]	Diffuse Irradiance [W.m^{-2}]	Ambient temperature [°C]	Plane of Array Irradiance [W.m^{2}]	Cell Temperature [°C]	DC Array Output [W]
count	4408.00	4408.00	4408.00	4408.00	4408.00	4408.00
mean	315.16	207.74	26.92	431.41	39.69	165070.88
min	0.00	1.00	9.80	0.93	10.48	0.00
25%	34.00	89.00	24.40	147.71	29.60	56909.29
50%	201.00	159.00	26.80	416.45	39.11	165067.66
75%	592.00	308.00	29.30	674.66	48.63	258488.94
max	980.00	800.00	38.10	1096.85	75.27	396421.24
SD	304.12	161.91	3.88	305.66	11.88	114224.17

and POA irradiance is 1.56, but a higher difference in SD is noted with diffuse irradiance. Comparatively, the ambient temperature attained a 3.88°C deviation, indicating that the solar farm operates under severe thermal stress, resulting in a higher solar cell temperature with a deviation of 11.88°C.

Training Model

In this ANN forecasting model, the dataset is split into training and testing subsets to ensure robust predictions. Over 365 days, 85% of the data is used to train the ANN model, and the remaining 15% is used to test and validate the model against actual data. Using 310 days of dependent and independent variables from solar farm data helps the model learn the relationships among parameters, delivering accurate predictions in the testing phase. Furthermore, the mean and SD from the training data are applied to both the training and testing sets, which helps prevent data leakage and maintain consistency.

Error and Epochs

Each layer of the developed neural network is processed iteratively, with the first layer conFig.d with a specified number of nodes, an activation function, and an initializer. The final layer is appended with a single node to produce the predicted DC array power output. In this case, a neural network with two hidden layers, containing 32 and 64 nodes, is used. Training data is processed over 50 epochs with a batch size of 32 to fit the model. Over the 50 training epochs, the training and validation RMSE values improve substantially. Initially, the training set's RMSE stood at 0.4569, indicating higher prediction errors. However, as training continued, the RMSE consistently decreased, reaching a final value of 0.0093 by epoch 50. Similarly, the validation RMSE experienced a significant reduction, starting at 0.0842 and ending at 0.0101.

Training and Testing the Model with Ambient Temperature and Beam Irradiance

As mentioned earlier, ambient temperature plays a crucial role in power generation, as an increase in ambient temperature naturally restricts heat dissipation from the PV module, thereby increasing the temperature of the solar cell. Power training and testing models are plotted against ambient temperature, reflecting their importance in power generation. It is found that higher power production periods are widely observed at ambient temperatures above 20°C, whereas temperatures below this threshold typically occur before effective sunshine hours (a typical day in winter).

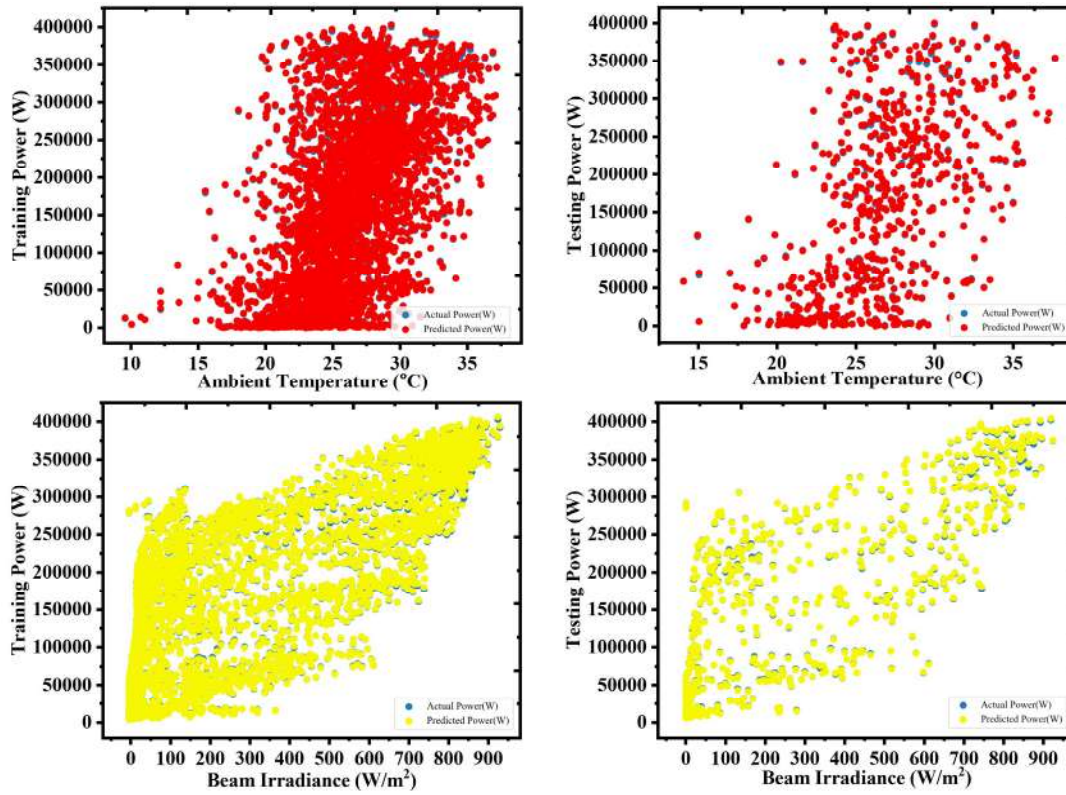

Fig. 2: Power training/testing model for ambient temperature and beam irradiance.

By comparison, the difference between actual and predicted power is negligible, as the developed ANN model was trained using 310 days of solar power generation data, along with other plant performance and meteorological data, providing accurate predictions, as shown in Fig. 2. A similar pattern is observed when testing the model against the correlation with ambient temperature. Furthermore, a training and testing model is performed against beam irradiance to understand energy generation, as beam irradiance represents the amount of solar irradiance received by the Earth. Although the higher count of power generation noted was less than 200 W/m², solar farms failed to meet 50% of the total installed DC capacity, as solar cells are current generators and higher solar irradiance favors higher power generation. Secondly, irradiance above 600 W/m² favors delivering 350 kW, and, beneficially, a second higher power generation count is noted between 750-850 W/m². This indicates that the solar farm effectively converts solar irradiance into electrical energy and attains higher performance efficiency. The training and testing model of the power profile shows that beam irradiance and ambient temperature follow a similar pattern. The dependent variable, DC power output, maintains a similar pattern to the independent variables of beam irradiance and ambient temperature. These findings indicate that the developed ANN forecasting model accurately predicts power generation for Thailand's location.

Actual and Predicted Power

As mentioned earlier, 85% of the dataset is used to train the ANN forecasting model, while the remaining 15% is used to test and validate the developed model. Fig. 3 shows the actual and predicted power generation for the 500-kW solar farm. The actual average power output of the testing model was 166.296 kW, and the developed ANN forecasting model predicts a power generation of 166.516 kW, which is 0.22 kW higher than the actual power generation. Based on 663 hours of power prediction testing, the model is 99.86% accurate and highly reliable for commercial solar farms to avoid penalties from grid authorities. The power prediction differences range from 2.88 kW to -4.67 kW, which is reasonably negligible. Although the power prediction differences are negligible, the high oscillation is mainly attributed to sudden fluctuations in solar irradiance, such as an unexpected cloud passing. In Fig. 3, a 36-hour insight view of actual and predicted power is plotted to visualize the error percentage in the developed ANN forecasting model, which is nearly zero or negligible. Under certain unpredictable weather conditions, forecasting errors can be observed, but these fluctuations are typically short-lived and often overlooked in real-time applications.

Error Metrics

The error metrics for the developed ANN model indicate an MAE of 0.87, with the actual and predicted power

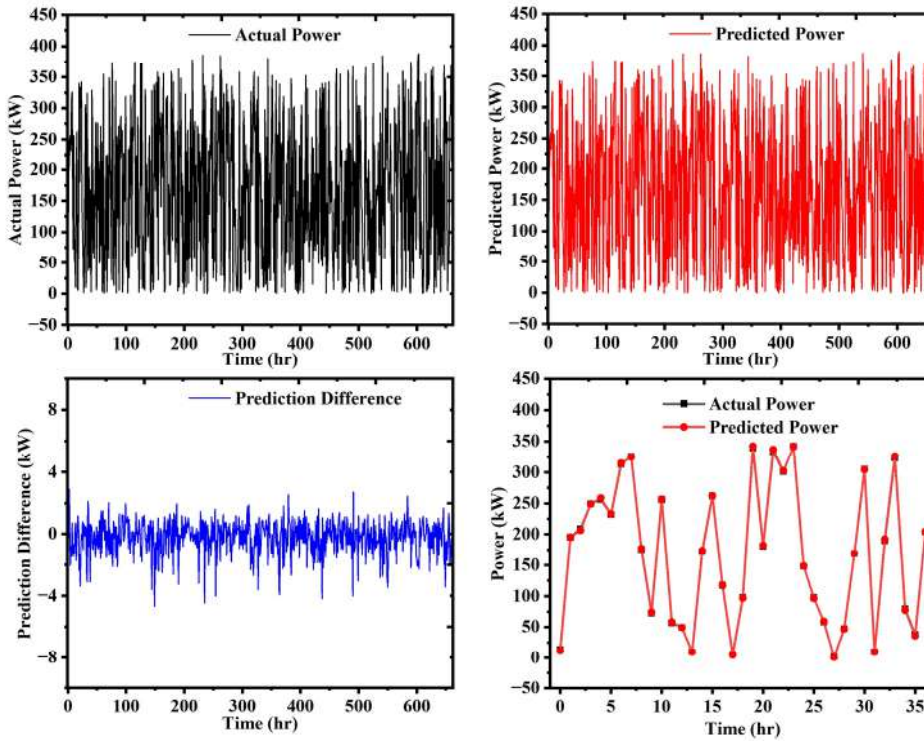

Fig. 3: Actual and predicted power profile using ANN model.

following a similar trend and minor variations in power generation. Following that, the MSE and RMSE are 1.32 and 1.15, respectively. The small difference between the MAE and RMSE indicates that the forecasting model is efficient and less affected by outliers. However, the MSE may yield a noticeable error, which is acceptable, and the forecasting model operates in a stable mode. The RMSE is critical whenever significant outliers are present; in this case, an RMSE of 1.15 is considered a minor error in forecasting accuracy. These error metrics indicate that the prediction deviated slightly by 0.87 kW from the actual power generation profile. Overall, the developed ANN forecasting model is suitable for solar farms in Thailand, and it is recommended that it be utilized for large-scale solar PV systems to prevent incurring surplus charges from the grid authority.

CONCLUSIONS

In this study, a modified neural network is developed to predict power generation for a 500-kW solar farm in Thailand. Raw data from the solar farm are processed and aggregated into 12-hour intervals to minimize prediction errors, as power generation is zero at night. The dataset is split into X (independent variables) and y (dependent variable). Both variables undergo training and testing with a 0.15 parameter setting, meaning 15% of the data is reserved for prediction. The ANN model is trained with a batch size of 32 and 50 epochs to enhance prediction accuracy through iterative optimization. The statistical analysis indicates that the maximum cell temperature reached 75.27°C, which is above the STC. During 75% of the period, the DC power output exceeded 258488.94 W, indicating that the solar farm is operating under severe thermal stress. The developed forecasting model predicts power generation with an average difference of 0.22 kW. Over 663 hours of testing, the model's actual and predicted power generation sums are 110088 kW and 110233.9 kW, respectively. The modified neural network achieves MAE, MSE, and RMSE values of 0.87, 1.32, and 1.15, respectively. According to these error metrics, the predictions are effective and reliable for large-scale and commercial implementation. Furthermore, it is recommended that future studies consider additional independent variables, such as humidity and atmospheric pressure, to enhance the accuracy of the forecasting model.

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NOMENCLATURE

ANN	Artificial Neural Network
DC	Direct Current
GA	Genetic Algorithm
IoT	Internet of Things
kW	Kilowatt
LSTM	Long Short-Term Memory
MFFNNs	Multilayer Feedforward Neural Networks
MVO	Multiverse Optimiser
MAE	Mean Absolute Error
MSE	Mean Square Error
MLR	Multiple Linear Regression
NRMSE	Normalised Root Mean Square Error
NREL	National Renewable Energy Laboratory
N	total number of predictions
PCA	Principal Component Analysis
PV	Photovoltaic
POA	Plane of Array
RMSE	Root Mean Square Error
SD	Standard deviation
UAE	United Arab Emirates
y_i	actual value
$\hat{y}_i$	predicted value

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Multivariable Analysis of Indoor VOC Dynamics in a Smart Urban Building: Advancing Evidence-Based and Health-Centered Air Quality Management

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ABSTRACT

This study investigates the dynamics of indoor volatile organic compounds (VOCs), including formaldehyde, xylene, and toluene, within a newly constructed smart urban building in Bangkok, Thailand. It aims to identify key determinants of air quality and to support data-driven strategies for healthier indoor environments. A total of 120 indoor air samples were collected from multiple zones and timeframes, considering building usage patterns, air conditioning operation, and environmental factors such as temperature, humidity, and air velocity. Statistical analyses revealed that air velocity and usage time significantly influenced VOC concentrations, while the time period was a critical factor for formaldehyde levels. Toluene concentrations exceeded the ACGIH guideline (20 ppm) in 13% of samples, and formaldehyde levels surpassed NIOSH thresholds in all samples, indicating potential exposure risks. Higher ventilation rates were linked to reduced pollutant accumulation, particularly during peak occupancy. Strong correlations between toluene and xylene also indicated shared emission sources. Although most values remained within regulatory limits, some exceeded health-based guidelines. These findings highlight the complex interactions among building operations, environmental conditions, and chemical exposure, and suggest targeted ventilation scheduling, particularly early-morning activation, to reduce VOC peaks. The study provides a practical framework for designing adaptive ventilation systems and developing guidelines to improve indoor air quality in new buildings. Insights from this research can inform future policy, urban planning, and smart building design aimed at reducing health risks and enhancing environmental resilience.

INTRODUCTION

Urbanization around the world is increasing rapidly, as the World Health Organization (World Health Organization 2022) and the United Nations (United Nations Human Settlements Programme (UN-Habitat) 2022) estimate that by 2070, the urban population, or population living in cities, will increase by as much as two times from the year 2020. Emerging studies indicate that newly constructed buildings often emit higher levels of indoor pollutants, particularly volatile organic compounds (VOCs) and formaldehyde, compared to older structures, due to emissions from new materials and limited initial ventilation (Hodgson 2004). Formaldehyde and VOCs are common pollutants found in buildings (United States Environmental Protection Agency (US.EPA.) 2021). These challenges are amplified in the context of smart urban buildings, where innovative materials and compact energy systems may exacerbate VOC release during early occupancy. Understanding these emission dynamics is essential for developing health-centered strategies in next-generation building design.

Volatile organic compounds (VOCs), notably formaldehyde, xylene, and toluene, are key contributors to indoor air pollution and are emitted from a range

of sources, including particleboard, plywood, medium-density fiberboard, insulation, textiles, wallpapers, paints, glues, cleaning agents, and electronic equipment such as photocopiers and computers(World Health Organization 2010, United States Environmental Protection Agency (USEPA) 2021, Shin & Jo 2013, Suzuki et al. 2019). Formaldehyde, a colorless, flammable, and highly reactive gas, has been classified as a human carcinogen by the United States Environmental Protection Agency due to its association with long-term cancer risks (National Institutes of Health 2010). At concentrations exceeding 0.1 ppm, formaldehyde can cause acute symptoms, including eye and throat irritation, coughing, nausea, and respiratory discomfort. (World Health Organization 2010). VOCs are semi-volatile compounds that readily evaporate at ambient conditions, often accumulating indoors at levels up to ten times higher than outdoors, particularly in recently constructed buildings with limited ventilation. (United States Environmental Protection Agency (USEPA) 2021, Hodgson 2004). Health effects associated with VOC exposure range from mucosal irritation, fatigue, and asthma to systemic damage to the liver, kidneys, and central nervous system (Mølhave 2003, Norback et al. 1995, Norback et al. 2000, Wieslander et al. 1997, Wolkoff and Nielsen 2001). Xylene and toluene, in particular, are highly flammable compounds commonly found in solvents, paints, and coatings. Xylene, a mixture of three isomers, poses significant risks through inhalation and skin contact, with documented effects including eye and respiratory irritation at 400 ppm in humans, and severe toxicological responses at 2,800 ppm in laboratory animals (Jarnstrom et al. 2006, National Research Council (US) Committee on Acute Exposure Guideline Levels 2010). Similarly, prolonged exposure to toluene concentrations up to 4,000 ppm has been shown to cause hepatic, renal, and neurological impairments in animal models (World Health Organization 2022). These findings underscore the necessity of investigating VOC dynamics under real-world conditions, particularly during the early occupancy phase of smart buildings, where emissions tend to peak, and ventilation is often suboptimal (Klepeis et al. 2001)..

Thailand is a rapidly developing country with continuous growth in many sectors, including transportation. The government has implemented policies to support the livelihoods of the Thai population, which has led to an increase in population growth(Thai Environment Institute 2013). This has led to the construction of numerous new residential and office buildings. Building upon this trajectory, Thailand's ongoing urbanization and smart infrastructure expansion provide a timely context for advancing IAQ research, with implications for health-based building regulations and sustainable urban planning in rapidly developing regions.

Consequently, indoor air quality (IAQ) analysis, particularly in newly constructed smart buildings, is vital for managing measures and developing guidelines to prevent impacts caused by chemical vapors. It will help new building occupants adjust their space usage and prevent the effects of chemical vapors inside new buildings, which can cause Sick Building Syndrome (SBS) or building syndrome(Department of Occupational Safety and Health 2022). However, the study of the relationship between physical factors that affect IAQ still involves a specific analysis of a single variable, as in the case of temperature affecting VOCs in buildings. (Jo and Sohn 2009, Zhang et al. 2020, Zhou et al. 2020), humidity affecting VOCs in buildings (Jo & Sohn 2009, Suzuki et al. 2019), and the air exchange rate influences formaldehyde concentration in the building (Huang et al. 2017). In the same direction as the study of building usage characteristics that affect IAQ, such as usage time and indoor air concentration levels (Hu et al. 2022)The time period of the building affects indoor air concentration (Guo et al. 2013, Kaunelienė et al. 2016). Despite growing awareness of indoor air quality, few studies have employed real-time, multivariable frameworks within operational smart building contexts where the dynamic interplay between occupancy patterns and environmental conditions can be most meaningfully observed and analyzed.

To address these research gaps, this study conducts a multivariable analysis of VOC dynamics in a newly constructed smart urban building. Holistic assessments that integrate building usage and environmental variables remain limited. We systematically investigated how patterns of use, air conditioning operation, and factors such as temperature, humidity, and air velocity affect indoor concentrations of VOCs and formaldehyde, based on 120 real-world air samples. These findings contribute to a mechanistic understanding of pollutant behavior, supporting evidence-based IAQ management strategies. Although conducted in a single building, this high-resolution assessment offers insights into real-time exposure dynamics and serves as a foundation for comparative research and policy development.

MATERIALS AND METHODS

Study Design and Site

This study employed a field-based multivariable observational design to investigate the dynamics of indoor air pollutants under actual building-use conditions. The assessment was conducted in a newly constructed smart office building at Navamindradhiraj University, Bangkok, Thailand. The building had been in use for less than one year at the time of data collection. A total of 120 indoor air samples were collected to analyze the concentrations of xylene,

toluene, and formaldehyde in relation to building usage and environmental conditions. Environmental parameters, including air temperature, relative humidity, and air velocity, were also recorded concurrently with each air sample to assess their potential influence on pollutant dynamics. This comprehensive monitoring aimed to generate evidence-based insights into VOC dynamics that can inform health-centered indoor air quality strategies.

Sampling and Instrumentation

Air samples were collected from six indoor zones, categorized based on building usage, over two separate monitoring periods spaced five months apart. The sampling zones reflect diverse occupancy conditions and functional areas commonly found in smart building operations, including workspaces with varying occupancy levels, shared and transitional spaces, and educational-use areas (Table 1). Zones 1 to 4 each included two sampled rooms to represent typical conditions under different workplace or classroom settings. In contrast, Zones 5 and 6 were each represented by a single sampling location due to their spatial characteristics, namely the entrance hall of a building and a learning center, respectively. This sampling strategy was designed to capture temporal and spatial variations in VOC dynamics.

Air sampling was performed using personal air pumps (Gilian GilAir Plus, Sensidyne, USA) with calibrated flow rates of 200 $mL.min^{-1}$, drawing air through solid sorbent tubes. Prior to and following each sampling session, flow rates were calibrated using a primary standard calibrator to ensure measurement accuracy. Toluene and xylene were analyzed using NIOSH Method 1501, while formaldehyde was analyzed following NIOSH Method 2541. Samples were collected at a height of approximately 1.2 to 1.5 meters to represent the typical human breathing zone. Active sampling was conducted during three distinct time points relative to air conditioning operation: before switching it on, one hour after, and eight hours after. These time points were selected to capture expected diurnal variation in VOC dynamics, including initial accumulation, short-term stabilization, and prolonged exposure under full-day usage conditions. Field blanks were included in each sampling round to assess potential contamination. All analytical procedures followed established QA/QC protocols to maintain data quality and integrity. Although duplicate sampling at identical spatiotemporal points was not conducted, the sampling strategy prioritized broader spatial-temporal coverage across distinct building zones and two operational cycles. This approach enhances the representativeness of the dataset while ensuring methodological reliability in operational settings.

Statistical Analysis

Statistical analyses were conducted using SPSS software to evaluate differences and relationships among measured variables. Data normality was assessed via skewness and kurtosis, with values of $|ZSkewness| < 3$ and $|ZKurtosis| < 10$ considered acceptable for parametric testing(Kline 2011). Group comparisons were carried out using independent-sample t-tests and one-way analysis of variance (ANOVA) (Ebrahimi et al. 2023). To examine the influence of environmental and usage-related variables on VOC and formaldehyde concentrations, multiple linear regression analysis was applied. Multicollinearity was evaluated using the variance inflation factor (VIF) and tolerance values, where VIF < 10 and tolerance > 0.2 indicated acceptable

Table 1: Classification of indoor zones by building usage and occupancy patterns.

Period	Day	Usage time	Area
Period1: (After 10 months of building use.)	Monday: (After 2 days closure))	Time 1: (Before AC turned on.)	Area1: (Area with more than 10 people working.)
Period2: (After 15 months of building use.)	Friday: (After continuous 5-day use)	Time 2: (1 hour after AC turned on.)	Area2: (Area with no more than 10 people working.)
		Time 3: (8 hour after AC turned on.)	Area3: (Classroom or meeting room.)
			Area4: (In front of the elevator (semi-open buffer zone.)
			Area5: Entrance hall of the building
			Area6: Learning center area.

Note: This classification reflects temporal and spatial variations in building use, air conditioning operations, and occupancy conditions that influence indoor VOC dynamics.

levels of collinearity. (Belsley 1991). This analytical approach supported the identification of key predictors in VOC dynamics under actual building-use conditions.

Causal Structure Analysis and System Visualization

To reveal the underlying system dynamics of indoor air pollution, relationships among key variables were synthesized into causal loop diagrams (CLDs) using Vensim PLE 9.3.0. These diagrams were developed to visually articulate feedback structures and interdependencies among building usage patterns, environmental conditions, and chemical concentrations, providing a holistic representation of the system's behavior over time. This modeling approach not only enhances understanding of VOC dynamics in smart buildings but also supports evidence-based decision-making and policy development to improve indoor air quality management.

RESULTS AND DISCUSSION

Effect of Building Usage and Environmental Characteristics on Chemical Concentrations

Indoor air sampling and analysis of 120 samples revealed significant effects of building use and environmental factors on VOC concentrations. For xylene, significant differences were observed across usage times ($p < 0.05$), with the highest average concentration recorded during Time 1 (before AC operation), followed by Time 3 and Time 2. Air conditioning system status also significantly influenced xylene levels, with higher concentrations when the system was off ($p < 0.05$).

Toluene concentrations were significantly affected by three variables: time period ($p < 0.01$), usage time ($p < 0.01$), and air conditioning system status ($p < 0.01$). Higher toluene levels were consistently observed during Time 1 and in the earlier time period (Period 1), especially when the air conditioning was off. Formaldehyde levels were significantly influenced only by time period ($p < 0.01$), with Period 1 showing higher average concentrations than Period 2 (Table 2).

All xylene concentrations were within OSHA, NIOSH, and ACGIH exposure limits (100 ppm, 100 ppm, and 20 ppm, respectively). Toluene concentrations exceeded the ACGIH threshold (20 ppm) in 16 samples, 15 of which occurred during Period 1, though they remained within OSHA and NIOSH limits. Formaldehyde concentrations exceeded OSHA limits in 17 samples and surpassed the NIOSH threshold in all samples, emphasizing the need for immediate ventilation strategies during early occupancy periods to mitigate exposure risks. Fig. 1a–c illustrates the distribution patterns of xylene, toluene, and formaldehyde across different usage times and monitoring periods, highlighting the temporal dynamics of VOC accumulation and reduction.

Table 2: VOCs concentrations by building usage and environmental conditions.

Usage Variables	Condition	Temp. [°C]	Humidity [%]	Velocity [$m.s^{-1}$]	Xylene [ppm]		Toluene [ppm]		Formaldehyde [ppm]	
		$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$	p-value	$\bar{x}$	p-value	$\bar{x}$	p-value
Time period	Round 1	25.00	66.62	0.11	0.03	0.31	13.01	0.00**	0.67	0.00**
	Round 2	25.07	65.41	0.12	0.02		7.64		0.32	
Day	Monday	24.99	63.66	0.11	0.02	0.38	10.40	0.78	0.54	0.08
	Friday	25.08	68.37	0.13	0.03		10.26		0.45	
Usage time	Time 1	27.25	70.11	0.09	0.03	0.02*	12.78	0.00**	0.48	0.96
	Time 2	24.05	64.89	0.12	0.02		5.64		0.50	
	Time 3	23.81	63.05	0.14	0.02		12.56		0.49	
Area	Area 1	25.11	66.01	0.11	0.02	0.13	10.43	0.79	0.52	0.31
	Area 2	24.55	65.24	0.15	0.02		9.44		0.41	
	Area 3	24.98	65.50	0.10	0.03		10.87		0.53	
	Area 4	25.71	69.30	0.12	0.02		9.53		0.45	
	Area 5	24.43	66.43	0.12	0.02		10.79		0.49	
	Area 6	25.24	61.60	0.11	0.03		11.93		0.62	
Air condition system	Turning off	27.25	70.11	0.09	0.03	0.01*	12.78	0.00**	0.48	0.80
	Turning on	23.93	63.97	0.13	0.02		9.10		0.50	

Note: *: p-value < 0.05, **: p-value < 0.01

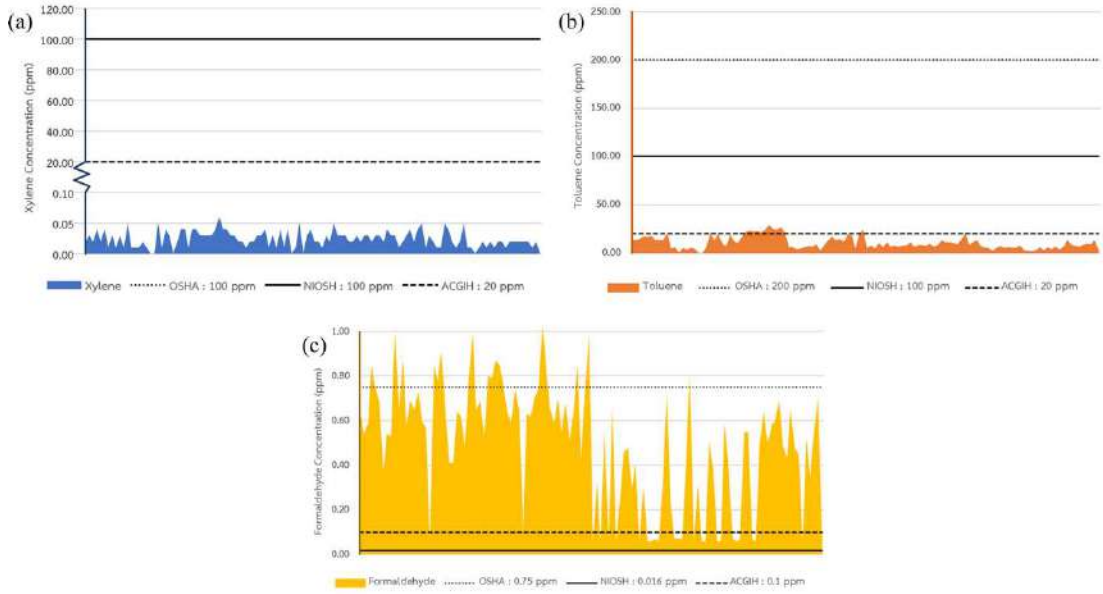

Fig. 1a-c: VOC concentration distributions across sampling times and periods.

Table 3: Key predictors of VOCs from multiple linear regression.

Independent variable	Correlation coefficient (r)	B	SE	Beta	p-value	R^2adj	Collinearity	
							Tolerance	VIF
Dependent Variable: Xylene (ZSkewness = 1.30, ZKurtosis = -1.14)								
Constant		0.02	0.00		0.00	0.37		
Toluene	0.510**	0.00	0.00	0.38	0.00		0.88	1.14
Air velocity	-0.500**	-0.05	0.01	-0.37	0.00		0.88	1.14
Dependent Variable: Toluene (ZSkewness = 2.99, ZKurtosis = -1.10)								
Constant		12.80	1.21		0.00	0.63		
Usage time 2	-0.545**	-6.16	0.73	-0.48	0.00		0.95	1.05
Time period	-0.442**	-4.95	0.68	-0.41	0.00		0.99	1.01
Xylene	0.510**	133.43	29.98	0.29	0.00		0.71	1.41
Air velocity	-0.346**	-9.68	3.88	-0.16	0.01		0.75	1.34
Dependent Variable: Formaldehyde (ZSkewness = -1.50, ZKurtosis = -2.04)								
Constant		1.27	0.15		0.00	0.71		
Time period	-0.636**	-0.34	0.03	-0.62	0.00		0.98	1.02
Air velocity	-0.548**	-1.15	0.16	-0.43	0.00		0.73	1.38
Humidity	-0.170*	-0.01	0.00	-0.19	0.00		0.96	1.04
Xylene	0.403**	2.93	1.18	0.14	0.01		0.73	1.37

Note: *: p-value < 0.05, **: p-value < 0.01

Regression Analysis and VOC Interrelationships

Multiple linear regression results revealed that the primary predictor of xylene concentration was toluene (β = 0.38), followed by air velocity (β = –0.37), with the model explaining 37% of the variance (R^2adj = 0.37). For toluene, key predictors included usage time 2 (β = –0.48), time period (β = –0.41), xylene (β = 0.29), and air velocity (β = –0.16), with an R^2adj of 0.63. Formaldehyde was most strongly influenced by time period (β = –0.62), air velocity (β = –0.43), humidity (β = –0.19), and toluene (β = 0.14), explaining 71% of the variance (R^2adj = 0.71) (Table 3).

These regression results build on the statistical findings by identifying key predictors, such as air velocity and time period, as well as interdependencies among VOCs. The notable correlation between toluene and xylene suggests common emission pathways from interior materials, offering practical insight for emission source control. High adjusted R^2 values, especially for formaldehyde, demonstrate the models' predictive strength and support the use of multivariable analysis as a foundation for designing targeted, responsive IAQ management strategies. Air velocity consistently emerged as a significant negative predictor of xylene, toluene, and formaldehyde levels, underscoring the effectiveness of active ventilation in reducing indoor VOC concentrations.

Causal Loop Analysis

The causal loop diagram (Fig. 2) visualizes interactions among VOC concentrations, environmental conditions, and building usage behaviors, highlighting feedback mechanisms and leverage points relevant to IAQ optimization in smart building environments. It illustrates the complex interplay among building usage patterns, environmental variables, and VOC concentrations. It highlights key feedback mechanisms, such as the reinforcing effect of low air velocity on pollutant accumulation and the balancing effect of increased airflow on pollutant reduction. For example, extended usage time without adequate ventilation increases VOC emissions, which in turn elevates indoor concentrations unless counteracted by enhanced air movement or environmental control.

The diagram also reflects the interdependencies among VOCs, particularly the reinforcing relationship between toluene and xylene, as well as their sensitivity to contextual factors such as time of use, humidity, and air conditioning status. This system-level view underscores that even small adjustments in operational settings can produce nonlinear impacts on air quality.

By visualizing these dynamics, the model serves as a decision-support tool for identifying leverage points to optimize IAQ management. It underscores the need for responsive, health-centered strategies in smart building design, aligning real-time building use with targeted ventilation interventions and environmental controls. In practice, the diagram can guide policymakers in identifying regulatory leverage points, such as implementing mandatory ventilation schedules during early occupancy, while also supporting building managers in refining zone-based air management strategies to reduce VOC exposure.e.

Discussion

Interpretation of Findings

This study provides evidence-based insights into how building usage characteristics and environmental factors influence indoor air quality (IAQ) in newly constructed smart buildings, reflecting the growing importance of

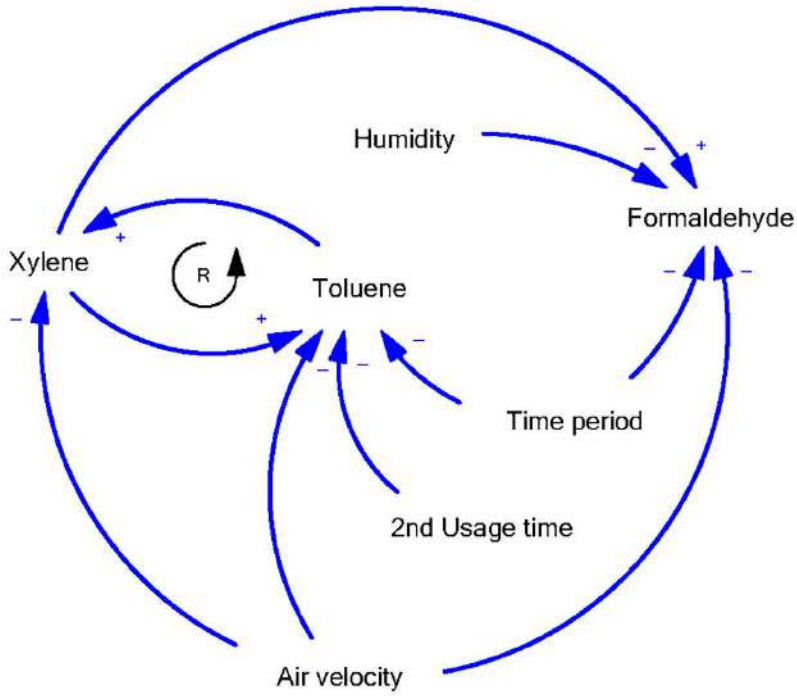

Fig. 2: Causal loop diagram illustrating VOC dynamics in a smart building context.

health-centered design in urban development. The findings emphasize that VOC dynamics are shaped not only by emissions from building materials but also by temporal occupancy patterns and operational conditions such as ventilation and air movement.

Among all tested variables, the time period of use emerged as the most statistically significant factor influencing formaldehyde concentrations. This supports prior research showing time-dependent decay of emissions due to off-gassing of construction and furnishing materials (Jo & Sohn 2009, Guo et al. 2013). The decline in formaldehyde over the 15 months underscores the need to manage exposure during early occupancy phases. Notably, air velocity was also found to be a major mitigating factor for VOCs, consistent with findings from office environments in China (Huang et al. 2017), indicating that airflow regulation, whether mechanical or passive, can substantially reduce pollutant buildup. These findings can directly inform the development of smart ventilation strategies. In particular, increasing airflow during early occupancy periods, when VOC accumulation is highest, may serve as a practical intervention to reduce short-term exposure risks. Integrating such timing-sensitive airflow control into building automation systems can significantly improve IAQ management in smart environments. These findings contribute to the growing body of evidence supporting real-time, usage-aware, and environment-responsive strategies for indoor air quality management. They also align with the principles of modern smart building design and intelligent IAQ control systems that integrate sensor-based environmental data. Specifically, the results highlight that increasing airflow during early occupancy periods, when VOC accumulation tends to peak, may serve as an effective intervention to mitigate short-term exposure. Incorporating such timing-sensitive airflow control into automated building systems can significantly enhance IAQ management in smart urban environments. Furthermore, the consistently elevated levels of formaldehyde underscore the importance of adopting additional measures, such as the use of low-emission construction materials and phased occupancy strategies, to reduce emissions during the early building use phase.

Although regulatory thresholds for xylene and toluene under OSHA and NIOSH limits were not breached, 16 air samples exceeded the ACGIH threshold for toluene, mostly during Time 1 (before air conditioning operation). This raises potential short-term exposure concerns, particularly during morning hours or upon reoccupation after periods of closure. A strong correlation between toluene and xylene concentrations also points to shared emission sources or similar dispersion behavior, consistent with studies by Ghaffari et al. (2021).

The study further examined the roles of environmental parameters. Building size and occupant density were not significant predictors of VOC or formaldehyde levels, consistent with findings from apartment complex studies in Harbin (Zhang et al. 2020). Within the observed temperature range (21–29°C), no clear association with pollutant levels was found, though past research suggests that more extreme variations could elevate emissions (Lin et al. 2021). Relative humidity was inversely associated with formaldehyde, supporting the theory that increased moisture enhances formaldehyde emissions and its interactions with materials, as observed in more recent studies (Huang et al. 2016, World Health Organization 2010). Findings from this study also align with those reported by Seng et al. (2023), who investigated indoor air quality in critical areas of Malaysian hospitals. Their study found elevated TVOC levels in poorly ventilated rooms, highlighting the importance of airflow. This conclusion is consistent with our findings at Time 1, before air conditioning was turned on, when pollutant levels reached their peak. The parallel underscores the broader relevance of ventilation control in mitigating VOC exposure across different building types.

The temporal use patterns of the building significantly shaped exposure profiles. Usage Time 1 showed the highest VOC levels due to overnight stagnation, whereas Usage Time 2, marked by high circulation and occupant movement, had the lowest concentrations. Usage Time 3, despite continuous use, showed re-accumulation in the absence of strong ventilation. These usage-sensitive findings reinforce the concept of adaptive ventilation scheduling, particularly during transitional periods of the day (Kwon et al. 2023).

Taken together, the results of this study contribute to the growing body of evidence supporting real-time, usage-aware, and environment-responsive strategies for indoor air quality management. The findings also align with smart building policy frameworks, such as those proposed by the Nusantara Capital Authority, and with modern IAQ control systems that utilize IoT technologies

These findings show that strategically managing ventilation, air velocity, and humidity in line with occupancy schedules can significantly improve health-centred building design in urban environments."

Methodological Considerations and Limitations

This study was conducted in a single smart building, which may limit generalizability. However, the focused design enabled detailed, context-specific analysis under controlled conditions. Data collection was limited to fixed weekdays and time points, which may not fully capture seasonal or behavioral variability. To address this, measurements were repeated across two periods and multiple functional zones.

While key environmental and operational parameters, including temperature, humidity, air velocity, and building usage patterns, were systematically assessed, the study did not include outdoor VOC measurements or detailed modeling of occupant behaviors, such as ventilation use, movement, or product application. These factors can influence short-term variability in indoor VOC concentrations and should be considered in future research to enhance model robustness and applicability across building types and usage scenarios.

To enhance the applicability and scalability of findings, future research should explore integrating IoT-based sensor networks with predictive, AI-driven ventilation systems. These systems can enable real-time, adaptive airflow regulation in response to pollutant levels and occupancy patterns. Additionally, extending the temporal scope to include multi-seasonal monitoring would help capture environmental variability and strengthen the evidence base for long-term IAQ management strategies.

Despite these limitations, the study offers a solid empirical foundation for health-centered air quality strategies in smart buildings, and its methodological approach provides a replicable framework for further research in similar urban contexts.

Policy Implications and Practical Applications

This study offers important guidance for enhancing indoor air quality (IAQ) policy and design practices in smart urban buildings. First, usage-sensitive ventilation strategies should be incorporated into building codes to address elevated pollutant levels observed during early occupancy. Urban building codes in Thailand should consider mandating post-construction flush-out procedures and staged occupancy requirements to mitigate VOC exposure during the initial building phase. Second, the strong influence of air velocity supports integrating adaptive, sensor-driven environmental control systems. Third, to reduce acute exposure to formaldehyde and toluene, phased ventilation protocols should be recommended during initial building use. Furthermore, aligning national IAQ policies with emerging regional frameworks, such as those proposed by the Nusantara Capital Authority, may enhance consistency and accelerate the adoption of health-centered smart building standards across Southeast Asia. Although this study focused on a single building, its methodological approach offers a replicable framework for broader applications in IAQ modeling and evidence-informed policy development across varied urban contexts.

CONCLUSIONS

This study provides comprehensive insights into indoor air pollutant dynamics in newly constructed smart buildings by integrating multivariable statistical analysis and system dynamics modeling. Key findings show that usage patterns, time of day, and air velocity significantly affect VOC concentrations, with formaldehyde particularly sensitive to early occupancy and ventilation conditions. The observed correlations between xylene and toluene highlight shared emission pathways, while the strong predictive power of the regression models underscores the relevance of targeted environmental controls.

Through real-time sampling across distinct building zones and operational periods, the study offers practical recommendations for adaptive ventilation, humidity regulation, and evidence-based air quality management. The system-level understanding generated by causal loop modeling further supports the development of smart, health-centered building strategies. These results contribute meaningfully to the ongoing discourse on sustainable indoor environments and provide a decision-support foundation for future policy development, particularly in rapidly urbanizing regions.

Beyond its immediate practical relevance, this study advances the scientific understanding of how environmental and operational variables interact to influence VOC behavior in high-density urban settings. Future research should explore longitudinal IAQ monitoring across diverse building typologies, integrate occupant behavior modeling, and evaluate the effectiveness of AI-driven ventilation systems. These directions will strengthen the foundation for sustainable, health-centered indoor environments in rapidly urbanizing cities.

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ETHICS APPROVAL

This study was ethically approved by Navamindradhiraj University on 28 September 2022 (ref: COE 002/2565).

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