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Enhanced Microplastics Removal from Paper Recycling Industry Wastewater Using Membrane Bioreactor Technology

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ABSTRACT

Urbanization and industrialization have caused a ubiquity of microplastics in the environmental system. An effective elimination technique is required for microplastics from industrial effluent and other wastewater systems due to its growing threats to the ecosystem and human health. The present study endeavors to evaluate the potential of the membrane bioreactor (MBR) technique in the removal of microplastics from paper recycling industry wastewater effluent. The effectiveness of the MBR system was evaluated relative to the conventional method used in industry for wastewater treatment. The paper recycling industrial effluent consists of 148 pieces/L of microplastics. The conventional treatment plant's effluent is used as an MBR system influent, and MBR removes 64.9% of the microplastic present after the conventional treatment plant, which is ascribed to the complementary actions of membrane filtration. MBR technology offers a reliable and workable plan to decrease the quantity of microplastics in industrial wastewater. It also offers a scalable solution that is consistent with sustainable environment management.

INTRODUCTION

Global plastic production was 370 million tonnes in 2019, with European production accounting for nearly 58 million tons. By 2020, the largest consumers of plastics in Europe were the packaging industry (39.6%) and the building and construction sector (20.4%). Despite advancements in recycling technologies, only 32.5% of plastics are recycled in Europe, while 42.6% are used for the production of energy, and 24.9% end up in landfills (Mishra et al. 2021, Yadav et al. 2024a).

Although recycling rates have been increasing, it is significant to note that around 50% of plastics are designed for single-use application, contributing significantly to environmental accumulation. Single-use plastics are widely used in disposable consumer goods, packaging, and agricultural films. By comparison, just twenty to twenty-five percent of plastics are used in long-term items such as structural materials, cable coatings, and pipelines. The remaining polymers are utilized in furniture, automobiles, and electronic devices that have intermediate lifespans. The significant increase in global plastic production has resulted in a massive amount of plastic waste on land, much of which eventually enters aquatic environments, causing growing concerns (Yadav et al. 2022, Yadav et al. 2023).

Smaller than 5 mm plastic particles are known as microplastics (MPs), and they constitute a major ecological hazard for the Earth's biosphere. MPs are produced when larger plastic garbage breaks down (Ahmed et al. 2023). With an estimated 51 trillion plastic particles floating in surface waterways globally, the microplastic problem is a direct result of global plastic pollution (Edo et al. 2020). Numerous freshwater bodies, estuaries, and oceans have been found to contain these microscopic particles. Microplastics can build up in aquatic food webs and the biota because of their widespread and incredibly slow rate of biodegradation (about 100 years) (Andrady 2017).

Numerous studies indicate that a substantial portion of microplastic fibers in aquatic environments originates from the washing of synthetic clothes. Ingestion of microplastics can obstruct the digestive tracts of aquatic organisms and facilitate the transfer of adsorbed contaminants, with uncertain consequences for the health of both aquatic life and humans.

Microplastics are widely present, as evidenced by samples taken from surface water, beaches, marine sediment, and marine creatures (Bellasi et al. 2020). MPs in waterbodies cannot be efficiently collected for recycling or management, in contrast to bigger plastic waste. MPs have been discovered in wastewater treatment plant effluents in addition to being present in oceans and other bodies of water (Sun et al. 2019). According to Li et al. (2018), MPs can also find their way into ecosystems through food, clothing, cosmetics, and other industrial emissions. These dangerous materials eventually find their way into the environment if they are not adequately recycled or treated, endangering both human health and aquatic species. According to research by Li et al. (2020), a lot of MPs come from wastewater treatment operations because of their tiny size and restricted treatability.

The capacity of microplastics to adsorb different common environmental pollutants, such as metals, medications, personal care items, and others, is a significant problem. Therefore, diseases like cancer, abnormalities in humans and animals, decreased immunological response, and impaired reproductive function can all be brought on by microplastics.

The removal of microplastics from aquatic environments has become an urgent challenge in the past decade due to their negative impact on aquatic animals as well as human health. The microplastics were detected in several aquatic ecosystems, including oceans, rivers, lakes, and sewage waste-water effluent. Based on their size, these plastics are classified as microplastics (MP) and nanoplastics (NP) (Poerio et al. 2018). Most existing studies have predominantly focused on municipal wastewater treatment plants (WWTPs) (Park 2020, Yuan 2022, Lee et al. 2023), focusing on a significant research gap concerning microplastics (MPs) in industrial wastewater sludge.

The production operations of the pulp and paper industry yield a substantial amount of wastewater, which in turn produces a substantial amount of sludge as a byproduct (Upadhyay & Bajpai 2023, Upendra & Kaur 2023). Historically, landfilling and incineration have been used as sludge disposal techniques. But there are also possible uses for these byproducts in land alteration and agriculture (Rissanen 2020).

The process of pulp and paper making includes wood preparation by chipping and debarking for the production of pulp, pulp bleaching, and the production of the papers. The recovered papers are separated chemically or mechanically method for the removal of ink, adhesives as well as other impurities, which are further rewetted and reduced into pulp to provide a valuable supply of fiber for the paper-making process. Pressing and drying process used for water removal.

Over the past several years, developing countries have seen an increase in demand for recycled paper of more than 7%–8% annually (Recycling Magazine 2018). The basic material for recycled paper is supplied from recovered paper. Recycling paper helps preserve natural resources like trees and water while significantly lowering production costs (Lares et al. 2018).

Its recycling material is stored and processed using plastic material, and it is the major source of microplastic generation from paper recycling plants (Yadav et al. 2024b). The wastewater or paper industry effluent involves primary treatment including neutralization, screening, and sedimentation for the removal of suspended solids. These solids are subsequently dewatered into a sludge that needs to be disposed of. Secondary and tertiary treatments are used less frequently to remove harmful organics and color from wastewater and lower its organic concentration. Consequently, it is essential to research MPs in pulp and paper wastewater sludge to monitor sludge quality and stop MPs from building up in terrestrial ecosystems (Pham 2023).

Membrane bioreactor (MBR) technology, a modern advancement in wastewater treatment, offers significant advantages over traditional activated sludge treatment. With its ability to operate at higher sludge ages and densities, MBR technology enhances the removal of pollutants, including microplastic particles. Unlike conventional methods, which struggle to eliminate microplastics effectively, MBR processes achieve more efficient elimination, preventing these particles from entering aquatic environments through final effluents. Scientific studies have shown that nearly 99% of the microplastic particles can be eliminated using activated sludge processes, particularly over the use of membrane bioreactor. The MBR technique reduces the average concentration of microplastic from the primary to final effluent by 96.2%, highlighting the crucial role of this tertiary treatment step in addressing this emerging pollutant (Mishra et al. 2021). The primary aim of the present study is to the identification of MPs and their extraction protocol specifically for effluent and waste sludges from the recycling paper industry.

MATERIALS AND METHODS

Sample Collection

The paper recycling industry wastewater samples were collected from the Yamuna Nagar Industrial area, Haryana, India. Samples were collected on October 16, 2023 to December 20, 2023. The industry set up a conventional treatment plant for wastewater treatment, which consists of a primary treatment plant that includes screening and grit chamber, then secondary conventional activated sludge with sedimentation. The samples collected for the study were influent-industrial raw wastewater (S1), effluent from the primary chamber (S2), and the final effluent sample from the conventional wastewater treatment plant (S3) were collected in 5-10L of water bottles. Sludge sample (SS) also collected 1kg packet. The final effluent wastewater sample (S3) and sludge (SS) were kept in a clean and dried container and stored in the refrigerator. Then, the collected sample S3 used as an inlet, was run in a design pilot setup of membrane bioreactor, and outlet/ effluent of MBR (S4) was also studied for microplastic.

Sample Processing/ Analysis and Chemical Reagents

The NOAA Marine Debris Programme defines microplastics (MPs) as particles with a size range of 0.3 mm to 5.0 mm. Samples were analyzed following this technique. Samples were sieved using mesh sizes of 0.3 mm and 5 mm to achieve this size requirement. Particles falling within the designated size range were washed with deionized water and gathered in a beaker to be subjected to wet peroxide oxidation (WPO), a process that separates MPs from other particles. To oxidize organic matter for WPO, 20 mL of 0.05 moL.L^{-1} Fe(II) solution and 20 mL of 30% hydrogen peroxide solution were added to the samples. After five minutes at room temperature, the mixtures were heated to 75°C and stirred. If there was still visible biological matter, this process was repeated. All the chemical reagents were analytical grade and obtained from Merck.

The WPO solution was then transferred to a density separator. To ensure that all the remaining particles were included, the samples were rinsed with deionized water (dH_2O) and transferred to the density separator, where microplastics (MPs) were allowed to settle down overnight. The settled MPs were then drained and manually removed. These collected MPs were dried at 75°C for 24 hours and subsequently stored in a desiccator until analysis.

Using a microscope with ×40 magnification, microplastics were counted and classified into the following categories: (i) spherical shape, (ii) fiber type, (iii) fragmented pieces, (iv) thin sheets, or (v) irregular shape, according to protocols from the NOAA Marine Debris Program (2015) and Hidayaturrahman & Lee (2019), as illustrated in Fig. 1.

Infrared micro-spectroscopic analysis was conducted using a Bruker ALPHA Fourier Transform Infrared (FTIR) spectrometer. Individual MP samples were transferred to the FTIR base. IR spectra were examined at a wavenumber range of 600 cm^{-1}– 4000cm^{-1} and compared against a material database as per Qiu et al. (2016).

RESULTS AND DISCUSSION

Occurrence of Microplastic in Pulp and Paper Industrial Effluent

Microplastic production from the chosen paper recycling industry is measured and characterized. These emissions wind up in municipal sewage water. According to reports, a significant source of MPs was the effluent from the pulp and paper industries (Kay et al. 2018). With an average value of 148 pieces/L, the average number of MPs in the industrial influent was greater (Table 1). In the primary treatment plant (S2), the MP number increased as well, reaching 67 pieces/L. Sludge was held in the reactor for a considerable amount of time until biofilm grew on the MP surface (Michels et al. 2018), which would facilitate particle settling. The microplastic content of the sludge sample (SS) was 131 pieces/L.

The effluent's MP number decreased to 13 pieces/L, which explained the 91.2% total MP removal capacity. In contrast, MPs were removed in the range of 82-189 percent in China's sophisticated drinking water treatment facilities by the use of coagulation, flocculation, sedimentation, and sand and GAC filtration procedures (Wang et al. 2020). It was discovered that MPs were able to pass through the screening and grit chamber primary treatment processes by overflow and that these processes were unable to eliminate MPs.

Conversely, the traditional plant utilized in activated sludge processes significantly contributed to the increased removal of microplastics (MPs) in WWTPs (Table 2). This is because MPs have a hydrophobic characteristic, allowing them to quickly attach to organisms or sludge in the treatment plant (Crawford & Quinn 2016). In light of this, MPs are kept in the sludge, some are transferred to the drying bed for disposal or drying, and some are recycled back into the aeration tank. According to Murphy et al. (2016), these MPs may, therefore, be discharged into the environment or accumulate in the soil and food chain, where they may eventually endanger both humans and the environment.

Pham et al. (2023) studied the WWTP of Kraft Paper Factory A, which has a capacity of 24,000 m^3day^{-1}. It uses treatment facilities such as level I (Dissolved Air Flotation-DAF), level II (Up-flow Anaerobic Sludge Banket-UASB and Conventional Activated Sludge-CAS), and level III (DAF and Fenton). The findings indicate that, despite a

99.8% removal rate and 12 items per m^{-3} concentration of microplastics in treated effluent, the microplastic load of this factory was 288,000 pieces per day. The microplastics were removed most efficiently by the primary and secondary treatment methods (75.8–97.9%), with DAF having a microplastic removal effectiveness of >95%.

The sludge sample had a microplastic content of 22,772 items kg^{-1} of dry weight. Regarding morphologies, the only forms of microplastics found in the wastewater and sludge samples were fragments (55% and 91%) and fibers (44% and 9%), respectively. The proportion of blue and white microplastics in the total was 37% and 30%.

Throughout the tropic chain, exposure to MP has been linked to a wide range of toxic insults, including disturbances in eating and reproductive outcomes, as demonstrated by numerous studies (Anbumani & Kakkar 2018). Consequently, to stop MP pollution and the consequences that come with it, the management of this sludge needs to be taken seriously.

Shapes of Microplastics

The findings of the MPs' classification into the four shapes—with a fifth type known as "irregular shape"—are displayed in Table 1 and Fig. 1 & Fig. 2. The findings show that all 20 pieces/L of the sample (S1, S2, S3, S4, and SS having 15, 1, 2, 0, and 2 pieces/L, respectively) had low concentrations of spherical-shaped MP. According to Table 2, fiber was the most prevalent fraction in all samples, accounting for roughly 30.5% of all MPs in wastewater samples and 35% in sludge samples. Another significant portion was thin sheets, which ranged from 20% in WW. Plastic bags, packaging, covering, and lining materials are the main sources of thin sheet MPs (Efimova et al. 2018).

Furthermore, samples from all sites showed almost the same proportions of MPs, even though sample 1 uses wastewater from Yamuna Nagar's paper recycling industry. The MP compositions (i.e., five forms) of wastewater and sludge samples are contrasted in Table 2. Fiber, fragments, thin sheets, and irregular shapes make up the majority of MPs in WWTPs. According to a recent study (Kay et al. 2018), fiber and fragments made up more than 75% of the total MP number in WWTP samples that were collected throughout the north of England.

The present study's findings show that the mean composition of fiber MPs is higher in the water phase (30.5%) than in the sludge (32%). However, for MPs with irregular shapes, the distribution is different, with fewer in the water phase (22.6%) than in the sludge (29%). Regarding thin-sheet and fragmented MPs, slight variations were observed between the sludge and water phases. It is important to highlight that certain MPs in the sludge could not be identified based on their shape, most likely due to microbial attachment on the surface of the microplastic. As a result, the unidentified fraction was relatively high at 29%, compared to 22.6% in the water phase.

The average MP shape distribution for the samples under study is shown in Table 2. The four main portions of the sludge (solid phase) were equally divided among MP shapes: fragment, thin sheet, fiber, and unclassified. In total, fibers make up 30% of MPs.

Microplastics-Polymer Type

Fourier transform infrared spectroscopy (FTIR) is the most widely used method for examining the individual chemical bonds or surface chemical composition of plastic particles (Hidalgo-Ruz et al. 2012). By comparing the unique infrared spectra produced by the FTIR technique with known reference spectra, MPs can be identified. The FTIR technology detects changes in the dipole moment of chemical bonds. According to Doyle et al. (2011) and Harrison et al. (2012), plastics may be clearly distinguished from other organic or inorganic particles by their distinct FTIR spectra, which also allow for the determination of the MPs particle composition and particular polymer type. The physicochemical weathering of measured MP particles may be determined by utilizing FTIR spectroscopy to analyze various band patterns (Corcoran et al. 2009, Ahmed et al. 2021).

Table 1: Classification based on the shape of microplastic in different samples.

Samples	Microplastics (pieces/L)				
	Spherical	Fiber	Fragmented	Thin sheet	Irregular shape
S1	15	38	30	25	40
S2	1	26	12	18	10
S3	2	15	8	8	4
S4	0	2	3	2	6
SS	2	42	28	21	38

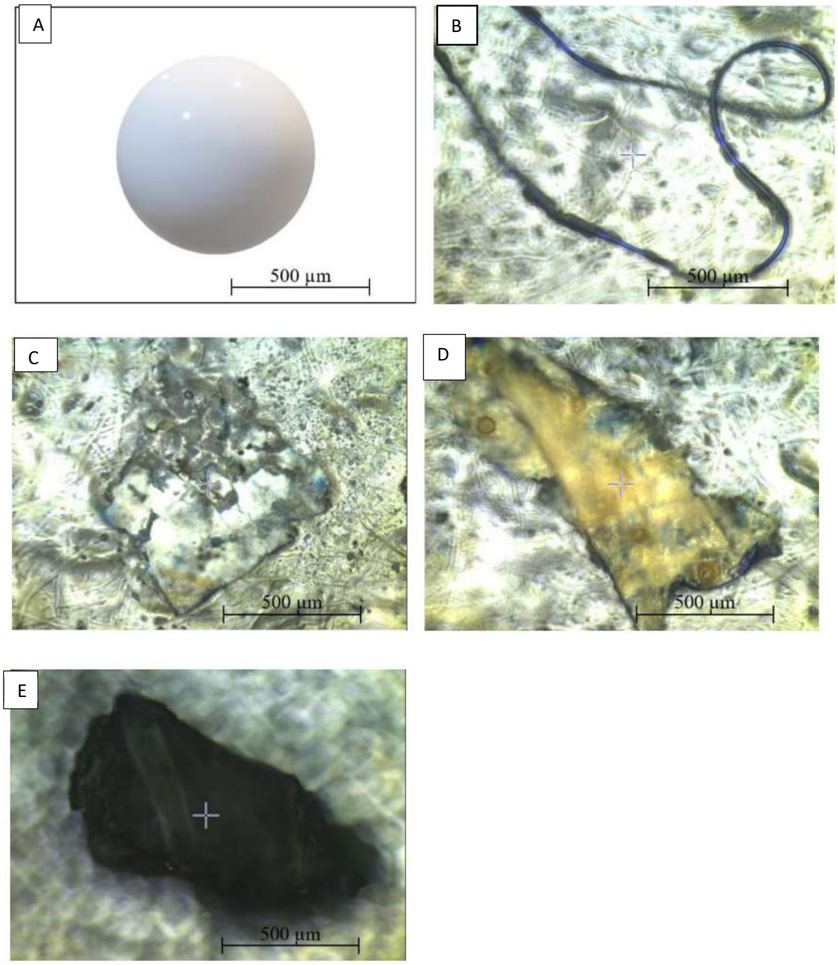

Fig 1: Shapes of Microplastic: A- Spherical, B- Fiber, C- Fragmented, D- Thin Sheet, and E- Irregular shape (Environmental and Climate Change Canada 2015, Hongprasith et al. 2020).

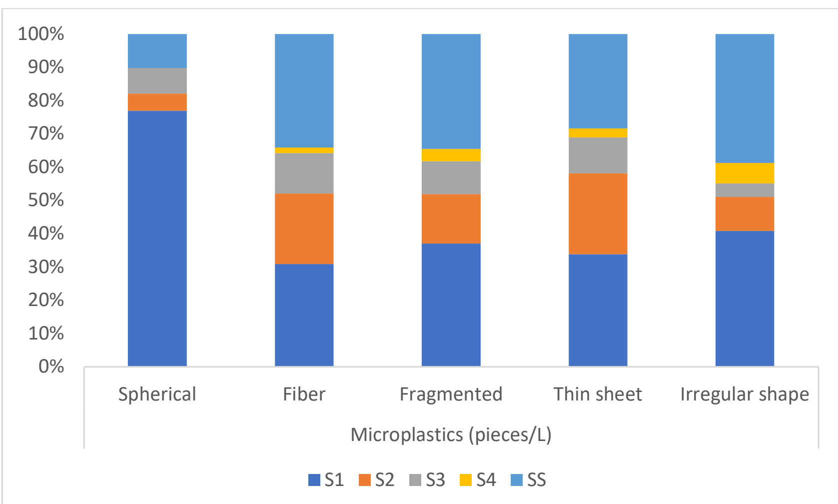

Fig. 2: Classification of microplastics (Pieces/L) in samples.

Table 2: Mean composition of total MP in wastewater sample and sludge.

MP Shapes	Wastewater samples [%]	Sludge sample [%]
Spherical	6.7	1.5
Fiber	30.5	32
Fragmented	20	21.3
Thin sheet	20	16.03
Irregular Shape	22.6	29

Following the recovery of microplastic particles from the samples, several polymers were found (Fig.3). Polyester (PES), polyethylene (PE), polyamide (PA), and polypropylene (PP) are on the list. FTIR microscopy was widely used to identify and validate these polymers.

The proportion of different polymers remained relatively consistent across different sampling dates when considering all sampling points. Throughout the sampling campaign industrial effluent (S1) represents polyester constituted 80% of the MP and 85% polyester in the sludge sample collected. Most of the remaining MPFs were polyamide, accounting for 3.1% of all MPFs. Microplastic fibers typically exhibit a uniform thickness with three-dimensional bending, distinguishing them from cellulose-based fibers, which have a ribbon-like appearance (Noren 2007, Murphy et al., 2016). This study also found polyester fibers with a flat, cotton-like appearance. For the MP, polyethylene was the prevalent polymer, representing 30% of MP in S2 and 25% in S3 of the total MPs. The polyester represents 67% of all the wastewater samples (Fig. 4) and 85% of sludge samples (Fig. 5).

Throughout the sampling campaign, there were significant variations in the quantity of microplastics in the sludge and wastewater samples (Fig. 6). Consequently, the number of MPs in WWTPs reported for individual sample events does not provide a consistent set of data that may be used to appropriately assess and address the microplastics pollution issue. Automatic composite sampling could be used to gather more representative samples and account for diurnal variation in the estimation of microplastic concentrations in WWTP (Talvitie et al. 2017).

The majority of research investigations carried out to date took place over a few days. However, only a few numbers of additional research (Talvitie et al. 2017) noted the significant change in MP concentrations in wastewater for weeks and seasons. To determine the prevalence of MPs in wastewaters during the fall and winter seasons in a Nordic setting, a sampling program was carried out between the third week of October and the first week of December.

Municipal wastewater treatment plant in Hungary, the microplastic study was performed, and it found seventh and sixteenth weeks of 2023 saw variations in the fiber content, ranging from 1.88–2.84 and 4.25–6.79 pieces/L, respectively. The percentage of microfibers in the solid particles was 94.7% in April and 78.3% in February. Microfibers based on cellulose predominated in the effluent (53–91%), whereas polyester predominated among those based on petroleum. In April, the median length of cellulose-based fibers grew significantly from February to April (650 vs. 1250 μm), while at the same time, the median diameter increased

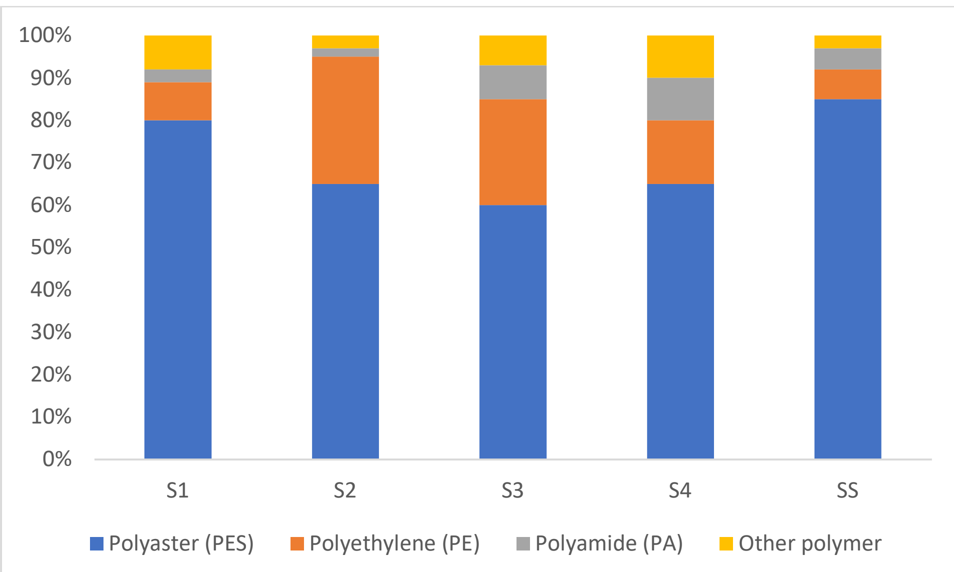

Fig. 3: Type of polymers in microplastics.

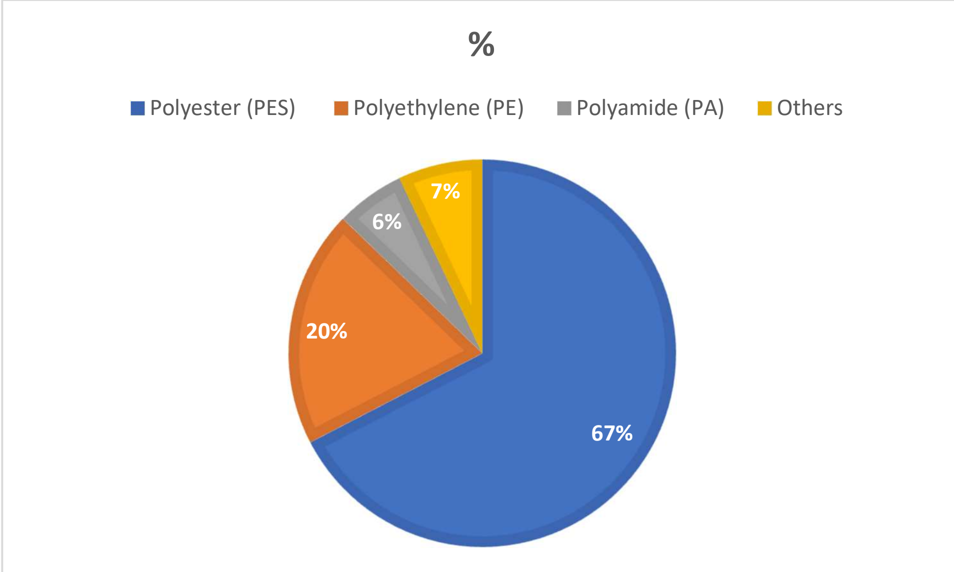

Fig. 4: Type of polymer present in wastewater samples

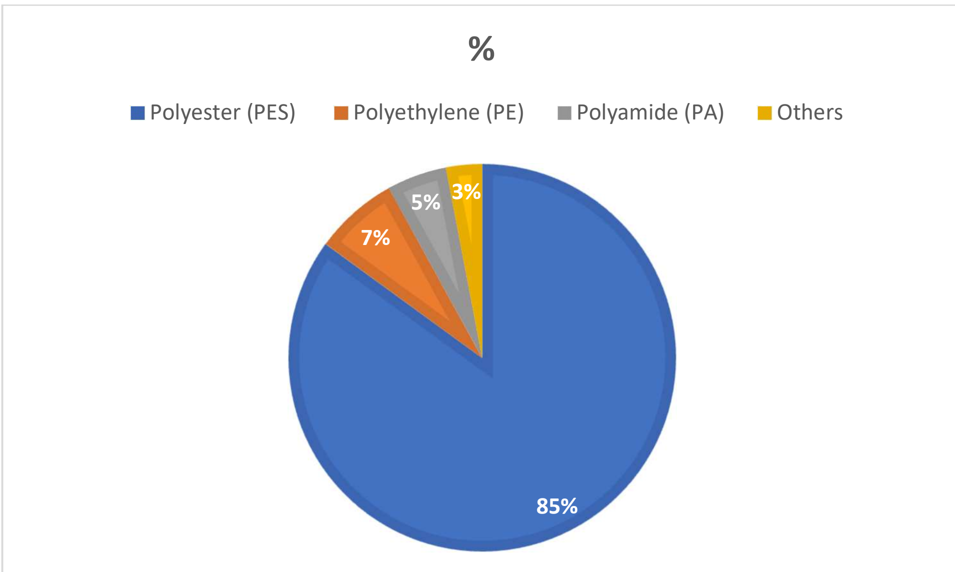

Fig. 5: Type of polymer present in sludge sample.

from 21 to 29 µm. This behavior was observed in relation to microfibers made of petroleum but to a lower degree. In February and April of 2023, the daily average transfer of treated wastewater to the Danube River varied between 0.44 – 0.69 and 0.94–1.53 billion, respectively (et al. 2024).

The study's authors highlight the necessity of expanding the monitoring campaign to include the spring and summer months to estimate the annual variation of MPs in wastewater and the corresponding capacity of WWTPs to manage such seasonal variation, based on the study's results. The MP concentrations in the influent may vary during the day in addition to seasonal variations. This might have increased some uncertainty in the published results because it was not taken into consideration during the sampling and computation of the MP removal efficiency in the WWTP under study.

Therefore, for more accurate evaluations, long-term sample campaigns should take the hydraulic retention time in various process components into account.

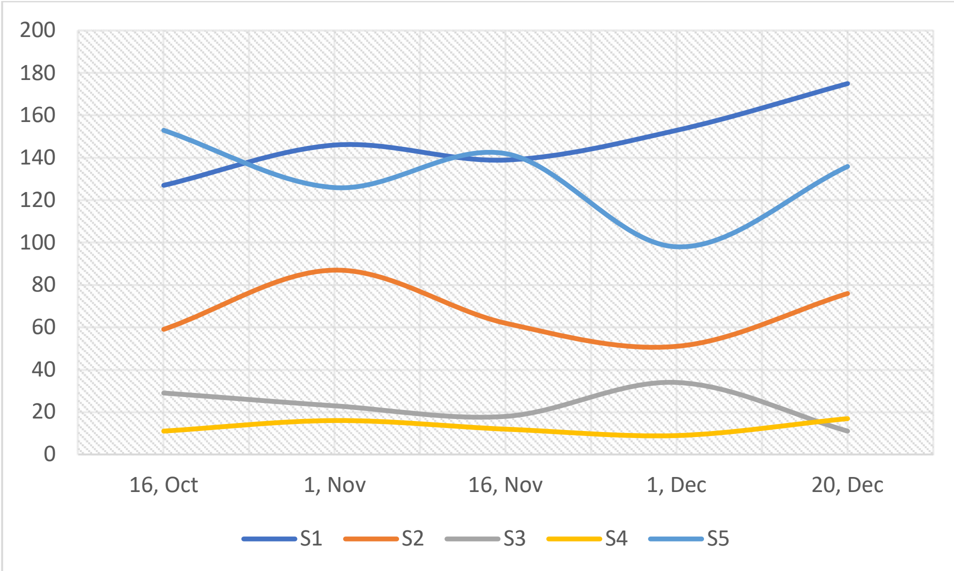

Fig. 6: Microplastic concentration with sampling dates.

CONCLUSIONS

The study's results highlight the great potential of membrane bioreactor (MBR) technology for improving the removal of microplastics from effluent from the recycled pulp and paper industry. It is clear by comparing the MBR system's performance to traditional wastewater treatment techniques that it is capable of achieving a significant 64.9% decrease in microplastics by utilizing the complementing processes of membrane filtration and biodegradation. This significant advancement emphasizes how much better the MBR system is at capturing microplastic pollutants, which are becoming more and more common as a result of industrialization and urbanization.

An effective and scalable solution that adheres to sustainable environmental management principles is provided by the MBR technology. Its efficacious treatment of industrial effluents indicates that it can be used in a wide range of industries with comparable pollution problems. Because the MBR system effectively removes microplastics, it presents a viable option for widespread implementation in industrial wastewater treatment plants, addressing a significant environmental and public health concern.

Subsequent studies ought to concentrate on refining operating parameters, examining the system's long-term stability and economic viability, and examining how well the technology works with various kinds of industrial effluents. Overall, this study offers strong evidence in favor of the use of MBR technology as a major approach to reducing the negative environmental effects of microplastics improving the quality of water bodies and ecosystems.

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