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Physicochemical Characterization of Pulp and Black Liquor Derived from Corn Husk and Coir Blends

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ABSTRACT

This study investigates the suitability of an agrowaste biomass blend, specifically corn husk and coconut coir, for producing sustainable pulp through kraft pulping and elemental chlorine-free (ECF) bleaching. An equal weight ratio (1:1 w/w) of oven-dried corn husk and coir was pretreated with steam at 120°C for one hour, then subjected to kraft pulping in a batch reactor at 80°C for two hours using white liquor (NaOH and Na_2S) at a solid-to-liquid ratio of 1:18. The unbleached pulp, with a yield of 38.7%, was bleached with hydrogen peroxide (1:10 w/v pulp-to-peroxide ratio) at 60°C for one hour. The properties of both unbleached and bleached pulps were analyzed using Fourier transform infrared (FTIR) spectroscopy, water retention value (WRV), cellulose content measurement, scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), and thermogravimetric analysis (TGA). Black liquor from the pulping process was examined via UV–visible spectroscopy. SEM images revealed a fibril-bound network in unbleached pulp, which became more porous and fibrillated after bleaching. FTIR spectra confirmed lignin removal by decreased aromatic bands in the 1600–1400 cm^{-1} range. The cellulose content increased from 60.2% to 85.4% post-bleaching. Elevated WRV indicated enhanced fiber swelling and porosity. TGA showed thermal degradation peaks between 200–350°C in bleached pulp, signifying improved thermal stability. UV-Vis analysis of black liquor revealed significant lignin peaks (an absorbance of 0.8 at 200–205 nm), indicating effective lignin removal. Overall, these results demonstrate that the pulping and bleaching procedures are viable for producing high-quality, cellulose-based pulp suitable for environmentally friendly applications.

1. INTRODUCTION

Paper is a nonwoven sheet composed of pulp fibers measuring 0.25–4.5 mm in length and 10–50 µm in width (Pydimalla & Adusumalli 2020). These fibers are interwoven to form a three-dimensional matrix. The use of paper products often serves as an indicator of a country's educational development. Paper plays a crucial role in many aspects of modern life, including communication and the dissemination of knowledge. Currently, there are approximately 759 pulp and paper manufacturing plants worldwide, with India accounting for about 4% of global paper production, ranking 15th (Pydimalla & Adusumalli 2020). Projections estimate that India's paper consumption will reach 23.51 million tonnes annually by 2030 (Van Ewijk & Stegemann 2016). Most of India's paper mills are concentrated in Andhra Pradesh, Telangana, Tamil Nadu, Maharashtra, Punjab, Madhya Pradesh, and Gujarat. Raw materials used for paper production vary regionally; for example, subabul wood is utilized in Andhra Pradesh mills, bagasse from seasonal crops

in Karnataka, and wheat straw in Punjab. Recycled paper is also widely used to produce various grades of paper. The paper industry is generally categorized into four main segments: printing and writing (P&W), packaging paper and board, specialty papers, and newsprint (Dutt 2013). Within these segments, the industry manufactures a diverse array of products, including printing and writing sheets, packaging boards, corrugated materials, newspapers, and tissue paper. Writing and printing papers constitute approximately 26% of the world's total paper output. Packaging products, such as corrugated board and paperboard, represent the largest share, making up around 51% of the market (Furszyfer Del Rio et al. 2022). Newsprint and tissue paper account for roughly 7% and 8%, respectively, of global production.

Fibers, which are the primary raw material for paper manufacturing, are elongated, hollow, non-living plant cells composed mainly of lignocellulosic biomass (Smook 2002). These plant-based fibers are vital in producing paper, a material extensively utilized in various human activities. Chemically, a typical fiber contains approximately 40–50% cellulose, 25–30% hemicellulose, 25–35% lignin, and a minor fraction (1–5% extractives) (Rowell 2012). Cellulose, the structural backbone of fibers, significantly dictates the mechanical strength and bonding potential of paper. It is a linear polymer made up of -D-glucose units, with hydrogen bonds forming between cellulose chains across neighboring fibers, which is crucial for fiber-to-fiber adhesion within the paper matrix (Wohlert et al. 2022). Hemicellulose, the second most prevalent carbohydrate in wood fibers, differs from cellulose as it comprises branched chains of various sugars, including glucose, xylose, and mannose. These shorter chains notably improve papermaking by increasing fibers' water retention and swelling, thereby facilitating bonding during sheet formation (Pere et al. 2019). Lignin, the third major component, is a complex aromatic polymer that covalently links with cellulose and hemicellulose. It imparts rigidity and structural stability to the plant cell wall, particularly in secondary wall layers and the middle lamella (Peracchi et al. 2024). Acting as a natural adhesive, lignin binds cellulose microfibrils together and provides a protective barrier against environmental damage. However, its presence can weaken paper strength by impeding effective fiber bonding. Therefore, an important goal in pulp and paper manufacturing, as well as viscose rayon production, is to reduce lignin content while maintaining cellulose levels.

The modern cellulose industry employs a wide variety of fibrous raw materials derived from hardwoods such as eucalyptus and subabul, softwoods, and non-wood sources including bagasse, wheat straw, and rice straw. Both hardwood and softwood fibers are essential raw materials in the production of paper and board products (Karlsson 2006). In India, fast-growing hardwood species with short rotation cycles are often utilized. Fiber lengths generally range from 0.2–4 mm, with cell wall thicknesses of 3–4 μm (Liu et al. 2024). A key difference between hardwood and softwood lies in their chemical makeup: hardwoods typically contain higher levels of cellulose and hemicellulose and lower lignin content than softwoods. This composition makes hardwoods more suitable for pulping processes that aim for higher yields and improved fiber bonding (Tarrés et al. 2023). Despite these benefits, the Indian paper industry faces several challenges, including high energy costs, limited technological advancement, environmental regulations, and, most importantly, a scarcity of quality raw materials. The primary issue is restricted access to government-managed forest resources. To mitigate raw material shortages, many paper producers are promoting agroforestry practices, especially in regions favorable to rapid tree growth. Agricultural residues are regarded by industry experts as a more environmentally sustainable alternative to wood-based fibers. Their porous cell wall structure facilitates faster pulping at similar cooking temperatures, yielding pulp of comparable quality (Worku et al. 2023). As a result, processing agricultural residues typically consumes less water, energy, and chemicals (Gavrilescu 2008). Currently, the Indian pulp and paper industry sources roughly 30–35% of its fibrous materials from wood, 20–22% from agricultural waste, and 45–50% from recycled fibers, reflecting a balanced approach aligned with environmental and economic goals.

In papermaking, pulp fibers are derived through either mechanical or chemical pulping methods utilizing wood or non-wood sources. The main purpose of pulping is to liberate individual fibers by breaking down the lignocellulosic matrix, primarily through the removal of lignin, a natural adhesive located in the intermediate lamella (Manfredi 2024). The kraft process is the most commonly used chemical pulping technique worldwide owing to its adaptability in processing diverse raw materials and its capacity to yield high-quality pulp. It also offers an efficient, proven method for reclaiming and recycling pulping chemicals. The key chemicals in the kraft process include sodium hydroxide (NaOH) and sodium sulfide (Na_2S). Together, these substances, known as white liquor, break the β-aryl ether bonds, among the most prevalent linkages in lignin, thus easing lignin removal (Madhuri et al. 2016). Consequently, lignin becomes soluble, enabling the separation of cellulose fibers. NaOH also contributes significantly by neutralizing organic acids and interacting with resinous components in the raw material, which further supports the delignification process.

The primary objective of bleaching is to remove residual lignin while preserving carbohydrate components,

particularly cellulose. This process helps retain the pulp's strength and improves its brightness. The removal of residual lignin during bleaching is regarded as an extension of the pulping process, albeit carried out more gently (Bajpai 2012). In chemically pulped fibers, the residual lignin is generally more condensed, showing an increased proportion of carbon-carbon and lignin-carbohydrate linkages and fewer β-O-4 ether bonds (Britt 1970). Bleaching, targeting lignin fractions remaining after initial delignification, is often seen as a continuation of the pulping process. Although reductive methods are occasionally used, oxidative techniques remain the predominant approach in chemical bleaching (Gellerstedt 2003). Modern bleaching methods typically employ oxidizing agents such as hydrogen peroxide (P), chlorine dioxide (C), oxygen (O), and ozone (Z). These chemicals improve the pulp's processing properties and whiteness by introducing hydrophilic functional groups and facilitating lignin depolymerization, leading to its breakdown.

In recent years, there has been a growing emphasis among researchers on utilizing non-wood fibers as sustainable alternatives for paper production (Pydimalla & Reddy 2019, El-Sayed et al. 2023, Nagaraja Ganesh et al. 2022, Das et al. 2023). Agricultural residues such as maize stalks, bagasse, rice straw, and wheat straw have been explored for their pulping potential, providing an environmentally friendly and energy-efficient option compared to traditional wood sources. Evidence indicates that non-wood fibers can be employed to produce high-quality, bleached pulp suitable for specialty papers. Advances in pulping techniques, including organic acid pulping and enzymatic treatments, have enhanced both the efficiency and sustainability of processing these materials (Singh & Bajpai 2025, Wang & Liu 2022). For instance, research on maize stalks showed that lignin could be effectively extracted using the caustic soda–anthraquinone process, yielding pulp with favorable tensile properties (Mishra & Sharma 2020). Moreover, the exploration of bast fibers such as hemp and kenaf continues, with the potential to create durable, eco-friendly paper products (Austin et al. 2024). These developments underscore the increasing significance of non-wood fibers in addressing the rising demand for environmentally sustainable paper production.

There remains a significant research gap in evaluating the synergistic potential of mixed agricultural residues. While progress has been made in utilizing non-wood biomass for pulp production, most studies focus on individual materials. Additionally, limited attention has been given to the combined processing of fibrous materials with high cellulose content, such as corn husk and coconut coir. This research aims to investigate whether a blend of coconut coir and corn husk, two commonly available agricultural wastes, can serve as an alternative fiber source for pulp production in paper manufacturing. It also seeks to systematically analyze the characteristics of pulp and black liquor following cooking and bleaching. The study assesses pulp quality and suitability for papermaking through kraft pulping and elemental chlorine-free (ECF) bleaching. This work is unique in promoting the efficient use of mixed lignocellulosic wastes, contributing to the development of sustainable raw material alternatives. Additionally, it supports circular economy principles and addresses fiber shortages in the paper industry. The pulping parameters were selected based on preliminary screening trials, serving as baseline laboratory conditions to optimize fiber separation and facilitate detailed physicochemical characterization of the pulp and black liquor.

2. MATERIALS AND METHODS

Corn husk and coconut coir were obtained from local markets in Hyderabad, India. The raw materials underwent manual washing to eliminate dust and impurities. The corn husk was then cut into small, uniform pieces, while coconut coir was cut and sieved through a 5 mm sieve to obtain appropriately sized particles for pulping. Both materials were subsequently dried in an oven at 103.5°C until reaching a constant weight, confirming moisture removal (Fig. 1). Analytical grade chemicals, including sodium hydroxide (NaOH), sodium sulfide (Na_2S), acetic acid, nitric acid, ethanol, and hydrogen peroxide (H_2O_2), were purchased from Merck Specialties Pvt. Ltd. For pulping, 50 g each of dried corn husk and coconut coir were mixed in equal proportions. The cooking parameters, including temperature, alkali charge, and cooking time, were optimized on a mild laboratory scale to minimize carbohydrate degradation and achieve controlled delignification. Preliminary experiments established conditions ensuring adequate fiber separation and acceptable pulp yield. An equal weight ratio (1:1 w/w) of corn husk to coconut coir served as the baseline formulation to explore the synergistic pulping properties of these agricultural residues. Corn husk, being richer in cellulose and lower in lignin, facilitates easier delignification, whereas coconut coir has a higher lignin content and is more fibrous. This balanced mixture was selected to assess their compatibility, uniformity in chemical saturation, and delignification performance. The 1:1 ratio acted as a screening criterion for process feasibility before further optimization with different ratios.

To improve chemical impregnation and remove residual moisture trapped within the chips, the dry raw materials underwent autoclaving at 120°C for one hour. This process also partially eliminated low-molecular-weight organic extractives, thereby enhancing the efficiency of

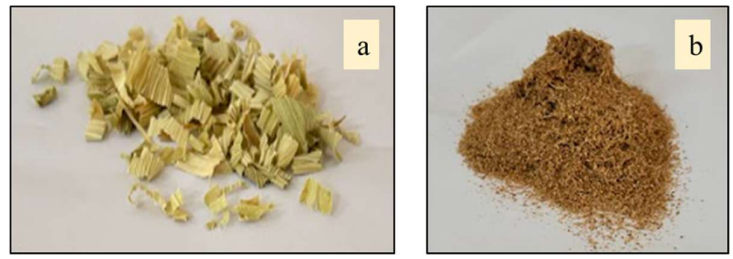

Fig. 1: (a) Corn husk; (b) coconut coir.

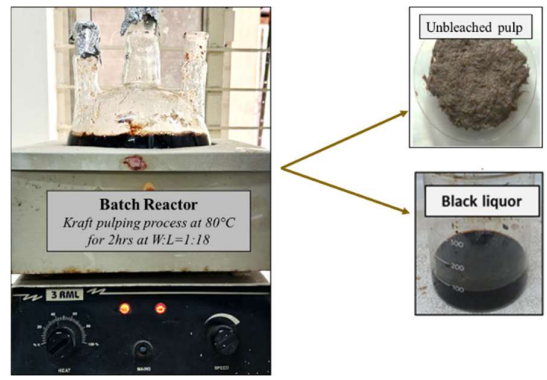

Fig. 2: Kraft pulping process in a batch reactor producing unbleached pulp and black liquor.

subsequent pulping. Laboratory-scale pulping experiments were conducted to evaluate the feasibility of using mixed agro-residues for pulp production and to analyze the physicochemical characteristics of the resulting pulp and black liquor. Biomass samples were placed into a batch reactor with white liquor, consisting of sodium hydroxide (NaOH) and sodium sulfide (Na_2S), at a solid-to-liquid ratio of 1:18 (w/v). Pulping was carried out actively for two hours at a controlled temperature of 80°C (Fig. 2). The choice of 80°C for kraft pulping in this study reflects a mild, laboratory-scale condition designed to enable controlled, gradual delignification of agro-residues while minimizing cellulose degradation and maintaining fiber integrity. Pre-steaming at 120 °C before pulping promotes fiber swelling, extractive removal, and improved chemical penetration, thus facilitating delignification at lower cooking temperatures. Such low-temperature alkaline pulping conditions (around 70–100 °C) have been used in laboratory research on non-wood lignocellulosic materials to achieve selective delignification and fiber preservation prior to bleaching or further processing (Rousu et al. 2002, Ververis et al. 2004).

Following the cooking process, the delignified pulp samples were separated from the black liquor and meticulously washed 4–5 times with deionized water to eliminate extractives and any residual black liquor on the pulp fibers. The cleaned pulp was then dried in a hot air oven at 103.5°C for 12 hours until a steady weight was attained. Subsequently, both the pulp and black liquor were characterized to determine the lignin content in the black liquor and the residual lignin remaining in the pulp.

Following the preparation and analysis of the unbleached pulp, the bleaching process was conducted using hydrogen peroxide as an elemental chlorine-free (ECF) bleaching agent. A specific quantity of pulp was placed in a beaker and subjected to bleaching on a hot plate maintained at 60°C. Hydrogen peroxide was added at a pulp-to-peroxide ratio of 1:10 (w/v), and the mixture was stirred occasionally while kept at a constant temperature for one hour (Fig. 3). Hydrogen peroxide effectively oxidizes residual lignin, thereby enhancing pulp brightness and reducing discoloration. This approach is more environmentally friendly than conventional chlorine-based bleaching, as hydrogen peroxide breaks down into non-toxic by-products, mainly water and oxygen. After bleaching, the pulp was filtered, oven-dried to remove moisture, and weighed. The bleached pulp was then further characterized and analyzed to assess improvements in fiber quality and optical properties.

The unbleached and bleached pulps were thoroughly characterized through a range of techniques, including Fourier transform infrared (FTIR) spectroscopy, water retention value (WRV), cellulose content analysis, scanning

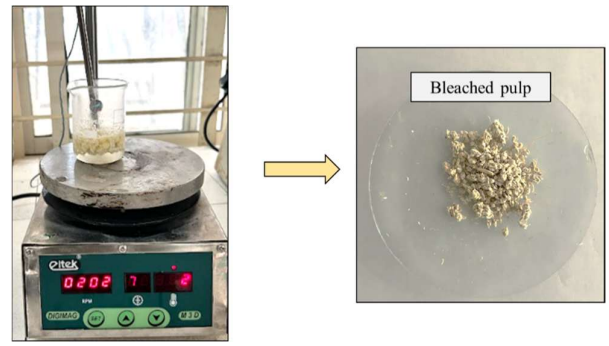

Fig. 3: Pulp bleaching process.

electron microscopy (SEM), energy-dispersive X-ray (EDX), and thermogravimetric analysis (TGA), to assess their structural, chemical, and thermal properties. Additionally, the black liquor recovered after pulping was analyzed using UV–visible spectrophotometry to measure dissolved lignin concentration and evaluate pulping efficiency.

3. RESULTS AND DISCUSSION

3.1. Pulp Yield

Fig. 4 presents digital photographs of both unbleached and bleached pulps. The ECF bleaching process markedly increases fiber brightness, as evidenced by the images. These visuals underscore the effectiveness of kraft cooking combined with ECF bleaching in producing white pulp. In Fig. 4(a), an oven-dried kraft pulp sample, obtained after cooking, is shown. The individual fibers in the mixed pulp cooked at 80°C are distinctly separated, indicating successful fibrillation. This suggests significant lignin degradation in the middle lamella and cell wall, likely facilitated by enhanced diffusion of white liquor under elevated temperatures. The breakdown of lignin, particularly within the compound middle lamella (CML), increases cell wall porosity, allowing deeper penetration of cooking chemicals into the fiber matrix (Kron et al. 2024). Fig. 4(b) demonstrates the effect of ECF bleaching on pulp brightness. The bleaching agents target chromophoric groups in lignin, such as quinones, which absorb light. The white appearance of the bleached pulp indicates substantial removal of lignin fragments and non-cellulosic components like extractives. These images effectively showcase the success of the optimized kraft pulping process followed by peroxide bleaching in producing high-brightness white pulp.

The pulp yield, defined as the ratio of unscreened pulp collected to the amount of steam-pretreated biomass used in the cooking process, was observed to be 38.7% for unbleached pulp and 34.8% for bleached pulp. Yield loss during kraft cooking mainly results from the dissolution of low-molecular-weight carbohydrates in an alkaline medium and peeling reactions (Knott 2021). During bleaching, treatment effectively removes chromophoric compounds and residual lignin from cell walls, resulting in higher pulp brightness, increased cellulose content, and a slight reduction in pulp yield (Bajpai 2013).

3.2. Cellulose Content in Pulp

This test measures the remaining cellulose content in the pulp after cooking. The procedure involves adding 15 mL of acetic acid and 1.5 mL of concentrated nitric acid to the samples, followed by refluxing for 20 minutes, as illustrated in Fig. 5(a). The treated samples are then washed with ethanol, filtered through a cotton cloth, and dried in a hot air oven for 12 hours to produce material A (Fig. 5(b)). Subsequently, the dried samples are incinerated in a muffle furnace at 525°C until their weight becomes constant, resulting in material B (Madhuri et al. 2016) (Fig. 5(c)).

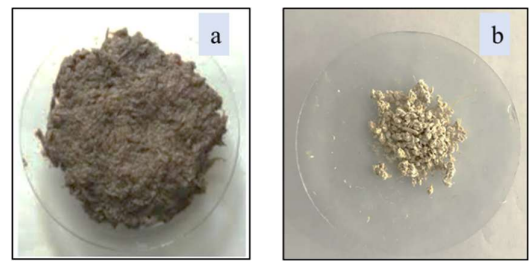

Fig. 4: (a) Unbleached pulp; (b) bleached pulp.

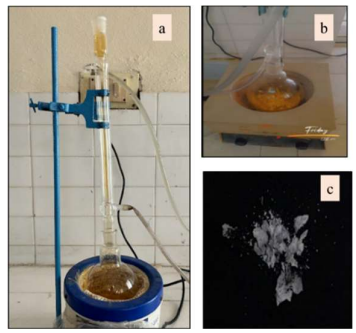

Fig. 5: (a) Cellulose isolation test; (b) pulp sample during reflux; (c) pulp after ash test.

Table 1: Cellulose content of pulp after pulping and bleaching.

Sample	Cellulose (%)
Raw material (corn husk + coir)	32.5
Unbleached pulp	60.2
Bleached pulp	85.4

The cellulose content is calculated using the following equation, and the results are presented in Table 1.

$$\%\ Cellulose = \frac{weight\ of\ material\ A - weight\ of\ material\ B}{Total\ weight\ of\ the\ sample} \times 100$$

The cellulose content (%) in both bleached and unbleached pulps was higher than that of the raw material, as shown in Table 1. This increase in total cellulose from raw material to pulp mainly results from the reduction of lignin content during pulping. Throughout the process, both lower- and higher-molecular-weight lignin fractions, particularly those linked with phenolic units in the middle lamella and secondary cell wall, are dissolved. This leads to decreased residual lignin and an increased percentage of cellulose in the pulp fibers (Brännvall 2017). The bleached pulp, containing minimal lignin, exhibited an even higher cellulose percentage, indicating successful removal of extractives, acetyl groups, and uronic acids from hemicelluloses, as well as the degradation and dissolution of α- and β-aryl ether linkages in phenolic units present in the middle lamella and cell wall regions (Santos 2013). Consequently, the relative cellulose content of the pulp fibers was further enhanced. The pulp yield was 38.7%, with the cellulose content rising to 85.4% after bleaching. These results align with reported ranges for non-wood agro-residue pulps processed under mild kraft cooking conditions (Ververis et al. 2004, Rousu et al. 2002), confirming the effectiveness of the delignification strategy employed.

3.3. Water Retention Value

Samples of 5 g each from the raw material, unbleached pulp, and bleached pulp were analyzed for WRV using the following equation:

$$WRV = [(weight\ after\ centrifuge)/(weight\ after\ drying)] - 1$$

The WRV was expressed on a g/g basis (dimensionless ratio). The wet samples were centrifuged at 2600 rpm and 23°C for 15 minutes, followed by overnight drying in a hot air oven at 103.5°C (TAPPI Standard UM 256).

The water retention value (WRV) serves as an empirical indicator of the water-holding capacity of pulp fibers, commonly used to evaluate fiber swelling. Lignin, characterized by its aromatic network structure, exhibits hydrophobic properties that hinder water absorption (Takada et al. 2020). An increase in WRV generally signifies greater lignin removal. Partial delignification in unbleached pulps leads to enhanced surface fibrillation and increased porosity at fiber interfaces, resulting in higher WRV compared to the raw material (Fig. 6). Further lignin extraction in bleached pulps induces internal fibrillation, enlarging internal pores and causing cell wall delamination, a process known as "swelling" (Bian et al. 2016). This process is also associated with the formation of external fibrils, which further improve water retention. As a result, the bleached pulp showed a

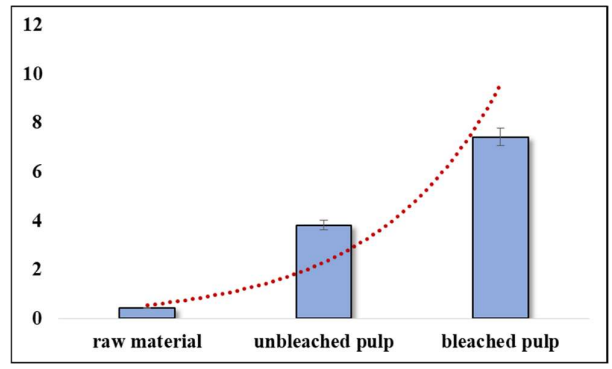

Fig. 6: Water retention value of raw material (before cooking) and pulp (after cooking and bleaching).

higher WRV than the unbleached pulp, due to its increased porosity, higher cellulose content, and lower levels of residual lignin and hydrophobic resins.

3.4. Fourier Transform Infrared (FTIR) Spectroscopy

Fourier Transform Infrared (FTIR) spectroscopy was employed to examine differences between bleached and unbleached pulp samples, highlighting various functional groups with an emphasis on aromatic skeletal vibrations. For spectral analysis, the samples were dried, finely ground, and mixed with potassium bromide (KBr) at a ratio of 1:100 (pulp to KBr), then pressed into thin pellets. The absorbance bands were recorded for interpretation (Fig. 7). In the fingerprint region of 900–1800 cm^{-1}, bands correspond to different functional groups typically associated with lignin. Specifically, the bands between 1600–1400 cm^{-1} signify aromatic or benzene rings present in lignin (Pydimalla et al. 2019). The peaks between 1300–1000 cm^{-1} relate to C–O vibrations found in primary and secondary aliphatic alcohols within cellulose, hemicellulose, and lignin, as

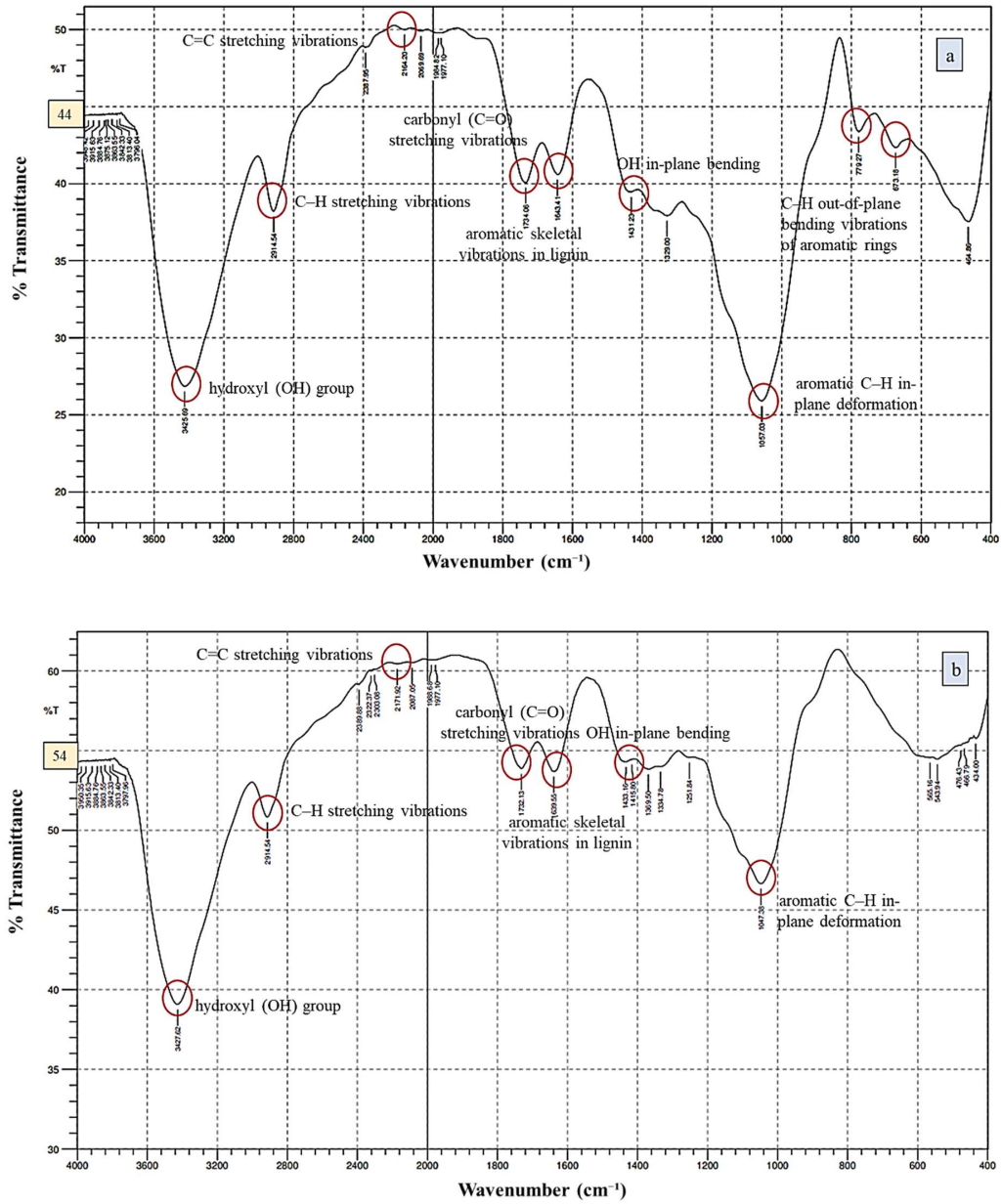

Fig. 7: FTIR analysis: (a) unbleached pulp; (b) bleached pulp.

well as aromatic alcohols in lignin. Both spectra display aromatic C–H in-plane deformations at approximately 1160, 1114, and 1055 cm^{-1} (Colom et al. 2003). Bands around 2130–2135 cm^{-1} are indicative of C=C stretching vibrations, while peaks between 1470–1455 cm^{-1} correspond to OH in-plane bending associated with cellulose. A broad absorption band spanning 3600–3200 cm^{-1} suggests hydroxyl (OH) groups are present in both bleached and unbleached pulp samples. Additionally, an increase in intensity within the 2900–2800 cm^{-1} range is attributed to C–H stretching vibrations (Wong et al. 2023). The FTIR spectra reveal that bleached pulp exhibits higher transmittance than unbleached pulp, indicating a purer cellulose content. Notably, a peak around 1430 cm^{-1} in the bleached pulp spectrum indicates crystalline regions of cellulose, while the band between 1610–1640 cm^{-1} signifies moisture absorption within the cellulose matrix (Hospodarova et al. 2018).

The FTIR spectra of the kraft unbleached pulp exhibit distinct peaks at 779 cm^{-1} and 673 cm^{-1}, corresponding to aromatic C–H out-of-plane bending vibrations, which indicate the presence of residual lignin. These peaks are clearly visible in the raw pulp spectrum but are absent in the bleached pulp spectrum, confirming the removal of lignin during bleaching. Additionally, the unbleached pulp shows a transmittance of 44%, while the bleached pulp shows 54%, suggesting a reduction in light-absorbing functional groups such as those associated with lignin.

3.5. Thermogravimetric Analysis

Fig. 8 illustrates the TGA curves of the pulp sample both before (unbleached pulp) and after (bleached pulp) treatment. These curves were used to evaluate the thermal degradation behavior of the pulp samples. The slight weight loss observed below 100 °C in unbleached pulp results from the volatilization of low-molecular-weight organic extractives and the evaporation of physically adsorbed moisture (Pydimalla & Reddy 2019). The peak degradation temperature (Tmax) indicates that the primary thermal degradation of bleached pulp occurs between 200°C and 350°C. In contrast, the initial degradation stage in unbleached pulp takes place between 250°C and 300°C. The pattern of weight loss in unbleached pulp can be attributed to thermal stability of composite structural units formed by lignin residues, which create complexes with polysaccharides in the cell wall matrix (Zhao et al. 2020). Conversely, the weight loss in bleached pulp results from lignin removal and the absence of those composite structures, including micron-scale pores that could serve as thermal insulators during heat exposure.

Significant weight loss occurs between 200°C and 400°C, mainly due to the degradation of hemicellulose, cellulose, and lignin. The unbleached pulp exhibits a broader degradation range because of the presence of lignin and hemicellulose, whereas the bleached pulp shows a sharper degradation peak, indicating a more cellulose-rich structure after non-cellulosic components are removed. Overall, the unbleached pulp retains more residual mass thanks to the thermally stable aromatic structure of lignin.

To enhance understanding of the degradation process, the peak decomposition temperature (Tmax) was identified from thermogravimetric trends corresponding to the maximum degradation rate. The unbleached pulp exhibited a broader degradation zone, indicating the presence of lignin and hemicellulose alongside cellulose. In contrast, the bleached pulp displayed a more distinct and sharper degradation peak within the 200–350°C range, characteristic of cellulose-rich material. The sharper Tmax observed in the bleached sample suggests improved homogeneity caused by the removal of lignin and non-cellulosic substances during bleaching.

The unbleached pulp showed a slightly higher residual mass at elevated temperatures, likely due to lignin residues, which are known for their slower degradation and wider temperature range for breakdown. Conversely, the bleached pulp lost weight more rapidly during the main degradation phase, indicating that it was richer in cellulose and contained less lignin.

The thermal degradation behavior of the bleached and unbleached pulps was analyzed over a temperature range of 30–600°C. The initial weight loss at lower temperatures was primarily associated with the evaporation of physically bound moisture and volatile components, while the major thermal degradation occurred approximately between 200 and 400°C, corresponding mainly to the decomposition of hemicellulose and cellulose, with contributions from residual lignin. Kraft pulping and subsequent bleaching remove substantial portions of lignin and hemicellulose from the lignocellulosic matrix, resulting in an increased relative cellulose content in the pulp fibers (Kron et al. 2025). Consequently, the bleached pulp exhibited a somewhat different thermal degradation profile compared with the unbleached pulp, which can be attributed to differences in lignin and hemicellulose content and the resulting changes in fiber composition. The removal of amorphous components during chemical treatment alters the thermal decomposition behavior of cellulose-rich fibers, as reported in previous studies. Overall, the TGA results indicate that kraft pulping and bleaching modify the thermal degradation characteristics of the pulp primarily through changes in lignin, hemicellulose, and relative cellulose content.

3.6. Scanning Electron Microscopy

The SEM micrographs of unbleached and bleached pulp

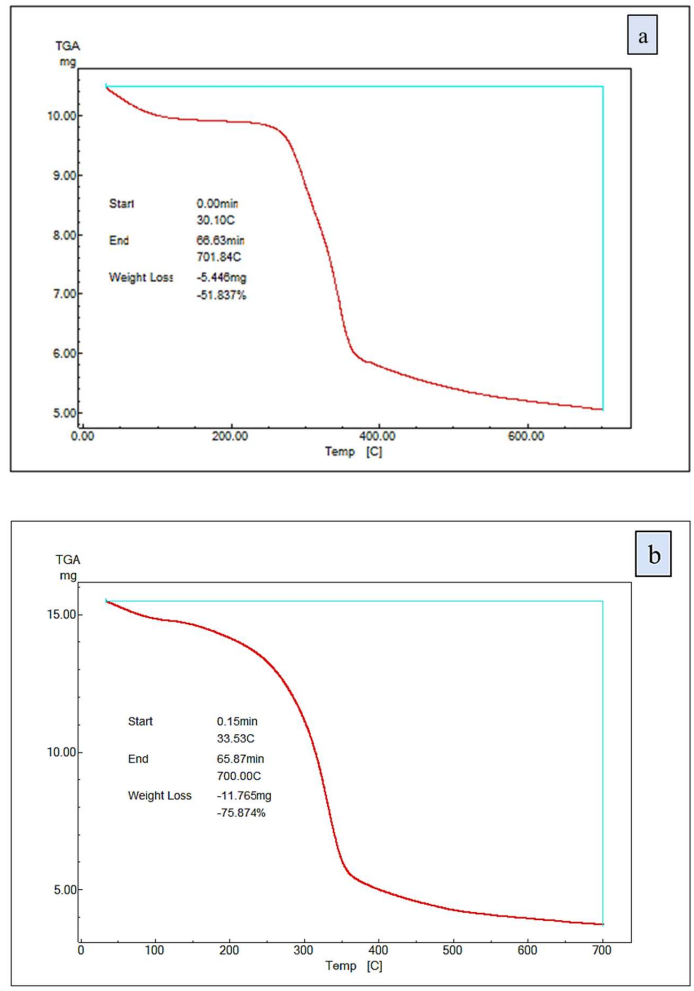

Fig. 8: TGA analysis: (a) unbleached pulp; (b) bleached pulp

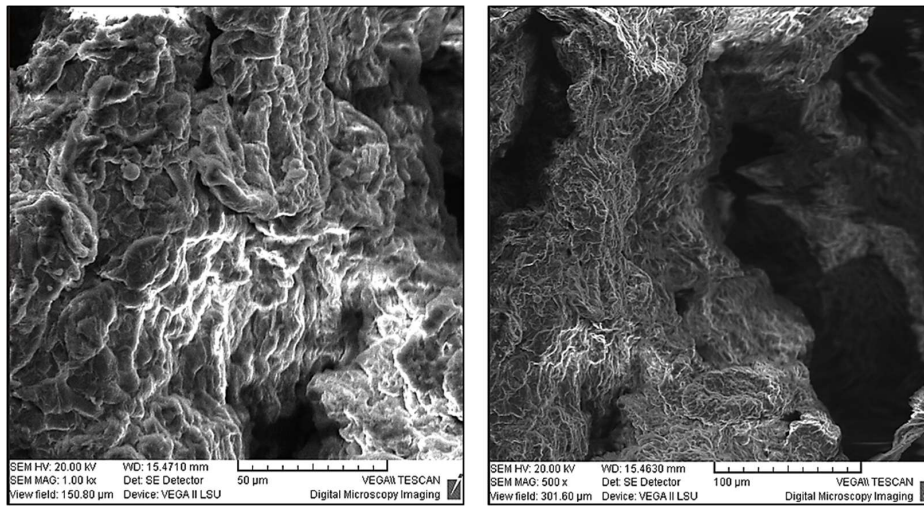

Fig. 9: SEM micrographs of unbleached pulp fibers.

fibers are depicted in Figs. 9 and 10. The micrograph of unbleached pulp shows thick, irregular fibers arranged in a complex network. Its surface appears rough, with numerous fibrils and a dense matrix. The fibers are less separated and intertwined, indicating residual lignin and hemicellulose that serve as binding agents. This compact structure results in lower porosity and less individual fiber separation.

The SEM image of bleached pulp reveals a more loosened fiber structure with more visible and distinct fibrils. The surface appears smoother, and the fibers exhibit increased individualization due to the removal of lignin and other non-cellulosic compounds during bleaching. This morphology is desirable in applications requiring high cellulose purity, such as nanocellulose production or bioplastics. Therefore, SEM characterization effectively indicates the microstructural changes in lignocellulosic fibers resulting from bleaching. The bleached pulp displays a significantly refined structure that enhances material development, whereas the unbleached pulp maintains its natural structure without fiber liberation.

3.7. Energy Dispersive X-ray (EDX) Analysis

The elemental analysis of bleached and unbleached kraft pulps derived from a mixed feedstock of coconut coir and corn husk was performed using EDX (Fig. 11), and the corresponding elemental compositions are presented in Tables 2 and 3. Both samples primarily consisted of carbon and oxygen, consistent with the lignocellulosic nature of the pulp. The unbleached pulp contained 52.57 wt% C and 46.34 wt% O, while the bleached pulp exhibited a slightly higher relative carbon content. This difference indicates a change in the surface elemental composition following hydrogen peroxide bleaching. However, EDX elemental analysis alone does not provide direct evidence of lignin removal or quantify lignin content. The observed change in elemental composition should therefore be interpreted as complementary evidence of the chemical modification of the pulp during bleaching, while the extent of delignification is better assessed from the chemical composition and other characterization results.

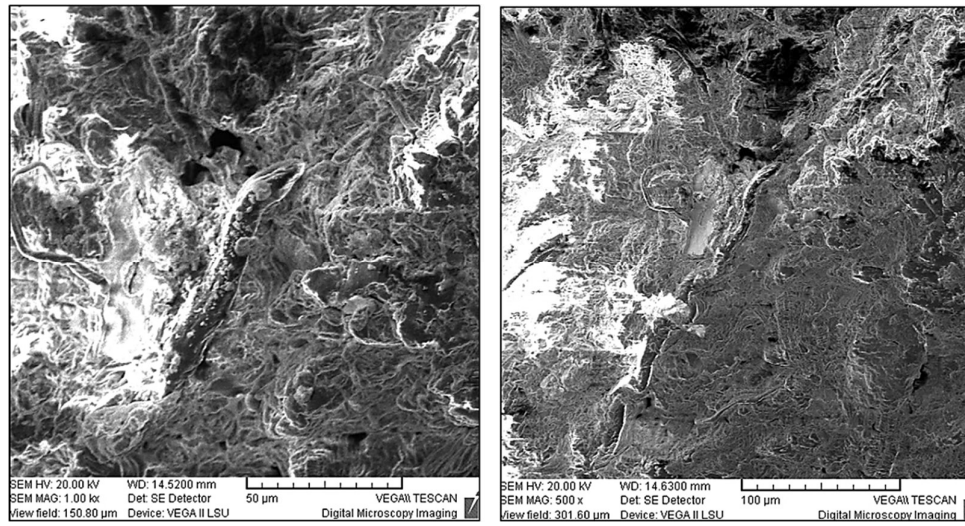

Fig. 10: SEM micrographs of bleached pulp fibers.

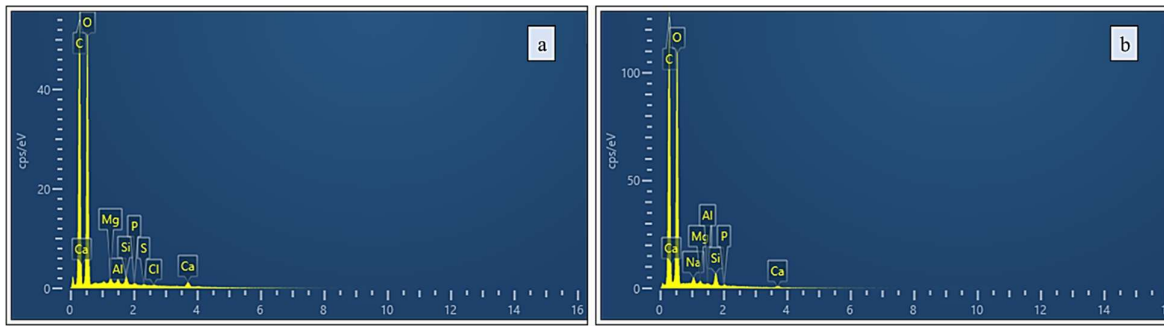

Fig. 11: EDX analysis of (a) unbleached and (b) bleached pulp.

Table 2: EDX results of unbleached pulp.

Element	Weight (%)	Weight (%) Sigma	Atomic (%)
C	52.57	0.21	59.87
O	46.34	0.21	39.62
Mg	0.23	0.02	0.13
Al	0.17	0.01	0.09
Si	0.27	0.01	0.13
P	0.06	0.01	0.02
S	0.04	0.01	0.02
Cl	0.03	0.01	0.01
Ca	0.28	0.01	0.09
Total	100.00		100.00

Table 3: EDX results of bleached pulp.

Element	Weight (%)	Weight (%) Sigma	Atomic (%)
C	53.22	0.18	60.48
O	45.60	0.18	38.91
Na	0.42	0.02	0.25
Mg	0.13	0.02	0.07
Al	0.04	0.01	0.02
Si	0.43	0.01	0.21
P	0.06	0.01	0.03
Ca	0.10	0.01	0.03
Total	100.00		100.00

The unbleached sample contains minor elements such as Mg (0.23%), Al (0.17%), Si (0.27%), P (0.06%), S (0.04%), Cl (0.03%), and Ca (0.28%). These may originate from natural biomass ash or residual kraft pulping chemicals. Although present in small quantities, sulfur and chlorine indicate the influence of the kraft pulping process. In contrast, the bleached pulp exhibits lower levels of inorganic impurities: Na (0.42%), Mg (0.13%), Al (0.04%), Si (0.43%), P (0.06%), and Ca (0.10%), with sulfur and chlorine undetectable. The absence of Cl and S suggests that peroxide bleaching is environmentally friendly. Overall, bleaching enhances purity and application potential, resulting in a high-quality paper product.

3.8. UV–Vis Spectroscopic Analysis

The analysis of black liquor derived from the kraft pulping process was performed using UV–visible spectroscopy to verify the presence of lignin-related compounds. The absorption spectrum spanned from 190–300 nm, with a notable peak between 200–210 nm exhibiting a high absorbance of 0.8 (Fig. 12). In addition to this peak, lignin-derived compounds generally absorb light within the 230–280 nm range, which is associated with substituted aromatic rings and conjugated phenolic structures. These spectral features support the solubilization of guaiacyl- and syringyl-type lignin units into the black liquor during alkaline pulping. The spectral characteristics align with oligomeric π–π^* electronic transitions linked to aromatic C=C bonds and n–π^* transitions from carbonyl-containing lignin fragments formed during oxidative and alkaline cleavage reactions (Hynynen et al. 2021). The peak observed at 200–205 nm indicates a high concentration of conjugated phenolic structures resulting from lignin fragmentation (Pydimalla et al. 2019). This sharp absorption suggests effective cleavage of ether and carbon–carbon bonds within the lignin macromolecule during kraft pulping, especially of β-O-4 bonds, which are highly susceptible to alkaline cleavage. Since absorbance follows Beer's law and is proportional to concentration under appropriate analytical conditions, the observed spectral features support the occurrence of lignin solubilization under the selected laboratory-scale

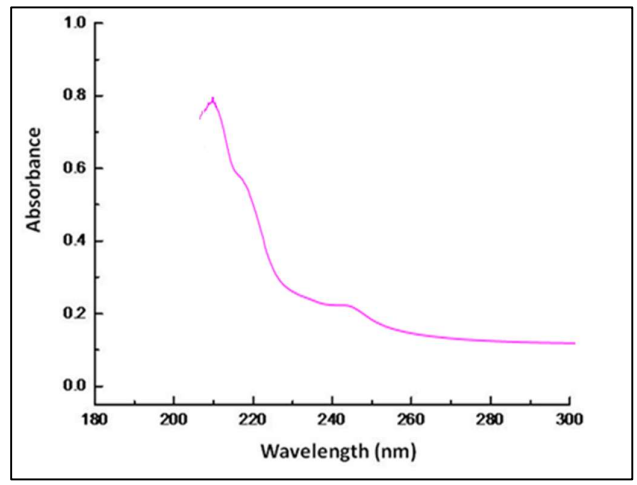

Fig. 12: UV analysis of black liquor.

pulping conditions. Furthermore, the absence of significant absorbance beyond 300 nm indicates that highly condensed chromophoric structures were rarely formed, suggesting that the pulping conditions favored lignin depolymerization over extensive condensation reactions.

This study primarily examines the feasibility of pulp production and the physicochemical characterization of fibers before conducting a comprehensive assessment of paper manufacturing performance. While standard pulping parameters such as kappa number and H-factor were not measured, the degree of delignification is inferred from qualitative analytical results. FTIR spectra indicate a notable reduction in lignin-related bands, and UV–Vis analysis of black liquor confirms the presence of dissolved lignin compounds released during pulping. These results suggest efficient lignin removal, corroborated by SEM micrographs showing fiber separation and surface modifications, demonstrating that the treatment effectively initiated lignin dissolution and fiber liberation. The findings highlight the potential for producing pulp from a mixed agro-residue system of corn husk and coconut coir. Combined physicochemical and morphological analyses verify fiber separation and lignin removal during pulping and bleaching. Although this research is conducted at the laboratory scale, future studies will systematically evaluate pulping parameters, handsheet formation, and papermaking performance to determine the pulp's suitability for industrial applications.

4. CONCLUSIONS

The current study introduces an innovative approach to utilizing mixed agrowaste biomass, specifically corn husk and coconut coir, by optimizing conditions that involve kraft pulping followed by elemental chlorine-free bleaching. This technique effectively achieved delignification, as evidenced by UV–visible spectroscopic analysis of black liquor, which exhibited a clear absorbance peak at 200–205 nm indicative of phenolic lignin derivatives. The process resulted in a pulp yield of 38.7% and significantly increased cellulose content from 60.2% in the unbleached pulp to 85.4% post-bleaching, demonstrating efficient delignification and bleaching. Additional confirmation of non-cellulosic component removal was obtained through elevated water retention value measurements and morphological changes observed in SEM images, which showed fiber loosening and fibrillation. FTIR spectra validated the degradation of aromatic lignin structures, while TGA analysis revealed improved thermal stability in the bleached fibers, attributable to lignin reduction and a more cellulose-rich matrix. Overall, this pulping and bleaching method enhanced the physicochemical and structural properties of the pulp, providing a sustainable pathway to convert lignocellulosic biomass into high-purity cellulose suitable for advanced applications such as nanocellulose, biodegradable films, and eco-friendly composites. The findings support circular bioeconomy strategies by advancing waste-to-value technologies within the pulp and paper industry. Future research will focus on systematically optimizing pulping parameters, including active alkali charge, sulfidity, and kappa number, to further improve delignification efficiency and pulp quality.

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