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Recycled pulp and paper sludge, potential source of cellulose: feasibility assessment and characterization

E. K. Suter[a], H. L. Rutto[a], T. S. Seodigeng[a], S. L. Kiambi[a], and W. N. Omwoyo[b]

[a]Department of Chemical Engineering and Metallurgy, Clean Technology and Applied Materials Research Group, Vaal University of Technology, Vanderbijlpark, South Africa; [b]Biotechnology and Chemistry Department, Vaal University of Technology, Vanderbijlpark, South Africa

ABSTRACT

The pulp and paper industry stands out as an example of a technology based on a renewable resource, cellulose. The sludge, however, poses major environmental and public health problems. To effectively manage the sludge wastes, it is critical to fully evaluate its composition, possible environmental impacts, and the total amount of exploitable renewable resources. The study established the pH of the sludge to be 7.32 ± 0.98, an electrical conductivity (1.84 mS/cm), nitrogen concentration (2.65 ± 0.21%), and total organic matter (41.23 ± 3.11%). The cellulosic content was established to be 74.07 ± 2.71% which contributes to 53.07 ± 1.23% water holding capacity (WHC). The most abundant elements were C and O, followed by Cl, Si, Al, and Mg, with lower concentrations of S, Si, K, and iron. The polycyclic aromatic compounds (PAHs) levels ranged from 0.29 to 322.56 ng.g-1 with 1-methyl pyrene posting the highest concentration (322.56 ng.g-1. XRD peaks at 17.10°, 23.86°, 30.14°, and 36.57°, which imply the existence of CaCO3. SEM indicated that the sludge was majorly comprised of fibers materials with average particle sizes of 280 micrometers. TGA/DTG analysis showed that the sludge had the greatest cellulose and hemicellulose (64.7 wt. %).

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KEYWORDS
Recycled paper sludge; cellulosic content; PAH; disposal; pollution

Introduction

The pulp and paper industry stands out as an example of a technology based on a renewable resource, cellulose, with the ever-increasing depletion of readily exploitable material resources in the globe.[1–3] Sludge from recycled paper mills is an intricate blend of fiber from recycled paper, inorganic particles, and chemical additives used in the production of paper, such as inks, glues, clay, residues, and chemicals employed in the recovery process.[4–8] The pulp and paper industries create this sludge as the final processed waste after several steps in the production of paper, including sorting, pulping, screening, cleaning, de-inking, refining, color stripping, and bleaching.[8–10] Depending on the type of paper mill and recycling activities, up to 50 kg of primary paper sludge can be generated for every tonne of paper produced.[11] The percentage of waste generated can range from 20% in newsprint mills to 40% in tissue paper mills.[7] Sludge production is increasing, and the subsequent treatments are highly sensitive to environmental concerns.[12] The amount of mill solid waste produced daily in South Africa grew from 16,200 tons per day in 2001 to 19,100 tons per day in 2005, averaging 0.8 kilos per inhabitant per day.[13]

Primarily, paper sludge has a high-water retention capacity, meaning it can hold a lot of water because water is either trapped or linked with fiber in the sludge. Studies have shown that primary paper sludge can hold between 4.8 to 12.6 liters of water per gram of sludge.[14] This high-water retention capacity makes primary paper sludge an attractive resource for various applications, including as a soil amendment for improving soil moisture retention and fertility, as well as for use in industrial processes such as cement production.[15–17] Direct applications on land are the best and most cost-effective approach to use paper mill waste to provide nutrients for crops, however, paper mill sludge has the potential to pose major environmental and public health problems. There are also limits on putting paper mill sludge on agricultural land such as introduction of heavy metals, alteration of soil pH and composition, pathway of pathogens and contaminants etc.[12,18]

Disposing of RPMS is an inherent challenge in the paper and pulp industries.[10,15,19] It has been argued that transportation and tipping fees are the primary sources of growing rubbish disposal costs. The method endangers the environment, particularly as a result of odor and leachate.[6,12,20,21] Landfilling is the current method of disposal, however as costs rise and land becomes more expensive, this method may not be economically feasible in the long term.[22,23] Many companies adopt reckless waste disposal methods to reduce disposal costs. The air, water, and land are all negatively impacted by this situation.[24–26]

CONTACT E. K. Suter esuter78@gmail.com Department of Chemical Engineering and Metallurgy, Clean Technology and Applied Materials Research Group, Vaal University of Technology, Vanderbijlpark, South Africa.

The sludge also contain new organic material, usually cellulose from recycled paper or wood, which is combined with inorganic minerals including calcium carbonate, kaolinite, and talc.[27–29] Although primary paper sludge may be a significant resource, it must be managed and disposed of appropriately to prevent any unfavorable effects on the environment. Waste management can aid in resolving conflicts between competing demands on the planet's finite natural resources.[30,31]

To effectively manage the pulp and paper sludge wastes, it is critical to fully identify their chemical and physical properties, as well as their possible impacts on soil fertility, site quality, and the total amount of exploitable renewable resources like cellulosic materials. As a result, this research was carried out to determine the physicochemical properties of the recycled paper mill sludge (elemental composition, chemical composition, and organic contaminants), environmental pollutants possibly present like PAHs, as well as to assess the possibility of properly recycling this waste to generate cellulose for water purification membranes and other industrial applications.

Materials and methods

Experimental section

Kimberly Clark's spring mill, South Africa (26°12'26.7" S, 28°26'26.7" E), provided sufficient volumes of recycled paper mill sludge for analysis. The Kimberly mill recycled pulp and paper sludge (KMRPPS) obtained were in a wet solid state, sticky and grayish during the collection. They were transported to the lab, air-dried, and crushed with a blender for further analysis (Fig. 1a–c).

Methanol (≥99.9%), hexane, acetone (≥99.9%), anhydrous sodium sulfate (Na_2SO_4), (≥99.5%), sulfuric acid (H_2SO_4) (≥99.9%), potassium sulfate, K_2SO_4, (≥99.0%), dichloromethane, and silica gel were used. All chemicals were purchased from Sigma–Aldrich, Merck-South Africa, and used as received.

Physio-chemical properties

To understand the effect of different sludge fiber compositions, KMRPPS was analyzed for moisture content (MC), pH, electrical conductivity (EC), elemental and chemical composition, functional group identification, particle size distribution (PSD), water holding capacity (WHC), surface morphology, crystal properties, and thermal stability.

Moisture content

The TAPPI T 210 standard method was used to measure the moisture content of the dried KMRPPS sample.[32] The process involved weighing approximately 2.0 grams of the sample on a tared weighing glass beaker and then heating in an oven at 105 °C for 2 h. The sample was then placed back into the oven for an additional hour after cooling in a desiccator. The weight was then maintained by repeating this cycle three times. Equation 1 below was used to calculate the sample's moisture content.

$$Moisture\ content\ (\%) = \frac{(Weight\ wet\ KMRPS - Weight\ dry\ KMRPS)}{Weight\ wet\ KMRPS} * 100\% \quad (1)$$

pH

The pH of the KMRPPS was determined using a SANXIN (IP67) Digital pH5 meter. A 1:2.5 ratio of distilled water was added to the sludge sample and stirred vigorously to form a slurry.[33] The pH was then determined using the resulting supernatant mixture.

Electric conductivity

The EC of the saturated paste solution of the KMRPPS that was prepared to record pH was measured using an electrical conductivity meter ASTM 1152.

Total carbon

The total carbon present in KMRPPS was evaluated using the combustion method.[34] and a LECO Corporation CR-412 carbon analyzer.[35]

Total organic matter (TOM)

The KMRPPS TOM content was calculated from the loss on ignition with a slight modification using Equation 2 with a correction factor of 1.78.[36]

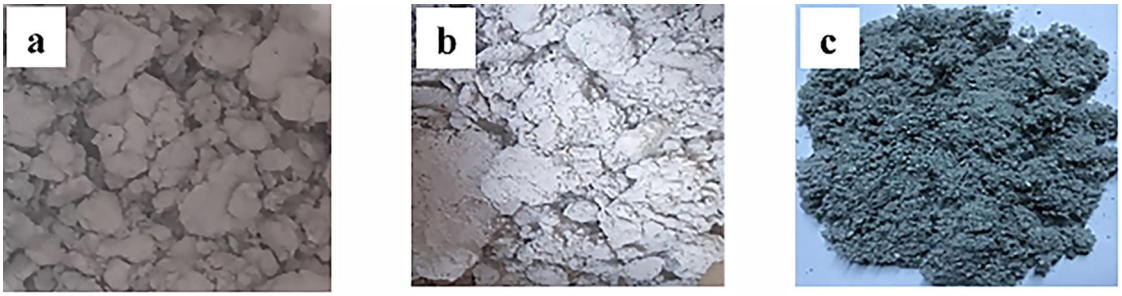

Figure 1. KMRPPS as received (a), dried (b), and crushed (c).

$$TOM = \frac{(mass\ of\ oven\ dry\ KMRPS - mass\ of\ ignited\ KMRPS)}{mass\ of\ oven\ dry\ KMRPS} * 100\% \quad (2)$$

Total nitrogen

The modified Kjeldahl technique,[37] was used to calculate the total nitrogen present in KMRPPS. The total nitrogen laboratory tests were performed in duplicate. The process involved heating a sample of KMRPPS to 360–410 °C with concentrated sulfuric acid (H_2SO_4), which oxidatively decomposes ("digests" or "destructs") the organic sample to liberate the reduced nitrogen as ammonium sulfate.

Water holding capacity (WHC)

Equation 3 was used to calculate the % WHC of the KMRPPS. Before the analysis, the dry crushed powder of KMRPPS was weighed and then submerged in water for 48 h. It was then removed from the container, and the wet weight of the KMRPPS was obtained using the vacuum technique.

$$WHC = \frac{(Wet\ weight\ of\ the\ KMRPS - weight\ of\ the\ dried\ KMRPS)}{weight\ of\ the\ dried\ KMRPS} * 100\% \quad (3)$$

Cellulose content

The cellulose content was determined using the Kürschner-Hanack technique.[38,39] This approach is based on cellulose's insolubility in water and resistance to the action of dilute acids and bases. The KMRPPS was degraded using a nitric acid-acetic acid combination and cooked in a Liebig's condenser-equipped reactor. After that, the solution was filtered *via* a Büchner funnel. The filter paper containing an insoluble residue was then dried and measured in an oven.

$$\%\ Cellulose = \frac{(W1 - W2)}{W1} * 100\% \quad (4)$$

Where W1 is the weight of dried KMRPPS (g), and W2 is the weight of dried insoluble residue (g).

Characterization

The elemental composition of samples has an impact on the synthesis, isolation, and extraction of the primary component of interest, such as cellulose and other cellulosic nanomaterials. Electron diffraction spectrum (EDS) analysis (JSM-IT500, JEOL Ltd., Tokyo, Japan) was used in this study to determine the composition of different elements contained in the KMRPPS. Fourier transform infrared (FTIR) spectroscopy, Nicolet, iS50 (Thermo Nicolet, USA) spectrophotometer was used to confirm the structural features of the KMRPPS. The FTIR spectra was collected in transmittance mode from 4000 cm^{-1} to 400 cm^{-1} at a resolution of 4 cm^{-1}. The particle size distribution of the sludge was determined using a MASTERSIZER 2000 (Hydro 2000 G (A)) analyzer. The measurements were taken from 0.020 to 3000 µm, with distilled water used as the dispersing medium. The weight loss of the KMRPPS sample was investigated using a Perkin Elmer Pyris 1 Thermogravimetric analyzer (TGA). The TGA (Pyris 1; PerkinElmer, Thermogravimetric analyzer) was connected to an inert nitrogen gas flow and heated from 30 °C to 1000 °C at a rate of 10 °C/minute. Approximately 11.000 mg of the KMRPPS sample was placed in the sample holder. The anticipated percent weight loss with temperature change for the KMRPPS was determined using TGA and DTG. For the morphological characteristics of the KMRPPS, a scanning electron microscope (SEM) (JEOL-IT 7500LA, Japan) was used to take the pictures. Before imaging, the samples were coated with gold sputtering.

Polycyclic aromatic hydrocarbon (PAH) analysis

The PAHs present in the KMRPPS sample were quantified using GC-Ms (Agilent 5977B). To purify and fractionate the PAHs present in the sample, glassware used in the experiment was washed with methanol, hexane, and acetone to remove any organic pollutants and kept in an oven at 60 °C. 20.0 g of dry powdered KMRPPS sample was homogenized with 1.5 g anhydrous Na_2SO_4 to eliminate excess water before being placed in the sample thimbles and then in a glass chamber for extraction. Dichloromethane was used as a solvent in the extraction of PAHs. The samples were extracted for 8 h in a glass room with a hot water bath using a round-bottom flask filled with 300 mL of dichloromethane. After decreasing the volume of the extract to dryness with a rotary evaporator, the extracts were transferred to a pear-shaped vial. The extracted components were purified and fractionated into aromatic fractions using silica gel column chromatography. 50 µL of the PAH surrogate internal injection standard combination (10 ppm anthracene-d10 and chrysene-d12) was added to the extract to ensure the accuracy of the PAH measurement. The extract was then loaded onto a silica gel column with 5% H_2O deactivation, and then they were washed with hexane: DCM (3:1 v/v). After that, a rotary evaporator was used to decrease the sample extracts' volume to 2.0 mL so they could be added to the column chromatography.

After the extraction, the extract was analyzed using a GC–MS (Agilent 5977B), which was set to oven conditions of the column temperature set at 80 °C and maintained at isothermal for 4 min. The temperature was then raised to 280 °C at 20 °C/minute and held isothermal for 8 min. The column was loaded with completely activated silica gel. Dichloromethane: hexane was used to elute the fractions that contained PAHs (1:3 v/v). Then, the PAH fraction was evaporated to 1 mL, transferred to a 1.5 mL amber ampule, and allowed to dry while being exposed to a moderate nitrogen stream. Next, the sample was dissolved in 50 L of isooctane with

p-terphenyl-d14 acting as an internal injection standard (IISTD) for PAH analysis.

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The injection mode was split less, with the injection temperature of 225 °C at 1 µL injection. Hydrogen gas was used as the carrier gas with 1 mL/minute at 9.5 psi pressure. Ionization detector was used, 70 eV with a transfer line temperature of 230 °C. Mass spectrometry was set to analyze the solvent for 3 min and use a total ion chromatogram (TIC) scanner with a scanning range of 40–450 amu at 1 scan/sec and set temperatures of 170 °C and 200 °C. The PAHs were monitored at specific mass-to-charge ratio (m/z) values, which were 178-(phenanthrene anthracene), 192-(2-methylphenanthrene, 3-methylphenanthrene, 2-methyl-anthracene, 1-methylphenanthrene, 9-methylphenanthrene), 202-(pyrene, fluoranthene), 216-(1-methylpyrene), 228 (benzo[a]anthracene, chrysene), 252-(benzo[a]pyrene, benzo[k]fluoranthene, benzo[e]pyrene, benzo[e]acephenanthrylene), and 278-(dibenzo[a, h]anthracene).

Results and discussion

Physio-chemical properties of the KMRPPS

Table 1 below gives an overview of the physicochemical characteristics of the KMRPPS. The pH of the sludge was 7.32 ± 0.98, and the alkalinity could be attributed to the causticizing chemicals used in the pulping process and/or the $CaCO_3$ used in the paper-finishing process. The KMRPPS exhibited an electrical conductivity of 1.84 mS/cm. Based on environmental regulations, EC levels for irrigation water and soil <4 mS/cm are regarded as safe for plants.[40,41] Most plants can survive in soils with an EC of 3–4 mS/cm.[42] This suggested that the KMRPPS was well pretreated before its disposal and would not trigger negative environmental concerns. The nitrogen concentration was 2.65 ± 0.21%. Nitrogen, which is necessary for microbial metabolism, was likely added to the mill sludge during the secondary treatment process, which may have increased the N concentrations. However, it is essential for plant growth, and a high nitrogen content might encourage the growth of pathogens that are vectors of viruses and microbes causing diseases. Thus, there is a need for alternative applications of sludge to reduce its environmental accumulation.

Table 1. KMRPPS physiochemical parameters.

Parameters	Mean Concentration	S. Dev
Moisture content	49.89%	2.23
pH	7.32	0.98
Electrical Conductivity	1.84 mS/cm	0.46 mS/cm
Total Carbon	32.39%	1.23%
Total Nitrogen	2.65%	0.21%
Organic Matter	41.23%	3.11%
Cellulosic Content	63.07%	2.71%
Water Holding capacity	53.07%	1.23%

Furthermore, KMRPPS exhibited a total organic matter content of 41.23 ± 3.11%, which is significant because it plays a crucial role in enhancing soil fertility and improving the physical properties of the soil. The carbon content was found to be 32.39 ± 1.23%, and the presence of such high levels of carbon and organic matter offers an additional advantage for soils, as they contribute to the overall improvement of soil quality. Based on industrial applications, the abundance of carbon is particularly advantageous due to its pivotal role as a fundamental constituent of cellulose and hemicellulose, which are key building blocks found in sludge. Consequently, a higher concentration of carbon directly corresponds to a greater proportion of cellulosic content in the paper sludge, which was determined to be 63.07 ± 2.71%. This high concentration of cellulosic materials indicates that the sludge has the potential for reuse through isolation of noble polymeric materials such as cellulose (cellulose nanocrystals and cellulose nano-fibrils).

The sludge as received from the company had a moisture content of 49.89 ± 2.23%, which was attributed to the vast volumes of water needed for processing and treatment. This proportion is comparable to those reported in the literature, where the average moisture content was found to be 51%.[43] Additionally, the majority of this water is retained in the sludge due to its tubular form and narrow interior pores from the cellulose and hemicellulose components.[44] The sludge water holding capacity (WHC) was 53.07 ± 1.23%, which is close to its moisture content. This implied that when KMRPPS is used in water purification composites/membranes without proper modification of the fibers, it can result to poor resistance to moisture-induced deformation. Thus, modification is crucial to help prevent moisture from affecting the structural features and performance of the composites/membranes, such as wettability, porosity, and microfiltration performance (flux and rejection). The modification will further guarantee their durability and functionality in various applications.[45]

Polycyclic aromatic hydrocarbons present in KMRPPS

The PAHs investigated in the study are shown in Table 2 and Fig. 2a and b. The total concentrations of the tested PAHs in the KMRPPS ranged from 0.29 to 322.56 $ng.g^{-1}$. 1-Methylpyrene had the greatest concentration (322.56 $ng.g^{-1}$), followed by Pyrene (178.35 ng.g-1), Benzo[a]anthracene (145.67 $ng.g^{-1}$), and a number of other PAHs substances with Benzo[a]pyrene (0.29 $ng.g^{-1}$) having the lowest concentration. According to the study, the overall PAH levels in the KMRPPS were less than 6 $\mu g \cdot g^{-1}$ when compared to European Union regulatory standards.[46] This amount is significant because it matched the amount suggested by the draft directive for the disposal of sludge on land from the

Table 2. Total PAH concentrations present in the KMRPPS.

PAHs	Concentrations (ng·g^{-1})	S. Dev (ng·g^{-1})	Class 1 (µg·g^{-1})	Class 2 (µg·g^{-1})	Class 3 (µg·g^{-1})
Phenanthrene	88.23	2.14	0.1	50	50
Anthracene	67.09	1.67	0.1	10	10
3-Methylphenanthrene	136.45	3.21	0.1	10	10
2-Methylphenanthrene	121.34	1.08	0.1	10	10
2-Methylanthracene	32.34	0.98	0.1	10	10
9-Methylphenanthrene	133.45	2.11	0.1	10	10
1-Methylphenanthrene	34.67	0.56	0.1	10	10
Fluoranthene	56.67	1.22	0.1	10	10
Pyrene	178.35	1.97	0.1	100	100
1-Methylpyrene	322.56	2.86	0.1	10	10
Benzo[e]acephenanthrylene	6.25	0.27	0.1	10	10
Chrysene	88.40	0.77	0.1	10	10
Benzo[a]antharene	145.67	2.02	0.1	10	10
Benzo[k]fluoranthene	1.04	0.17	0.1	10	10
Benzo[e]pyrene	33.07	0.89	0.1	10	10
Benzo[a]pyrene	0.29	0.22	0.1	10	10
Dibenzo[a,h]antharacene	2.54	0.54	0.1	10	10

*Conversion; 1 ug/g = 1000 ng/g, 1 ppm = 1000 ng/g, 1 ng/g = 1 ppb, 1 mg/kg = 1000 ng/g.

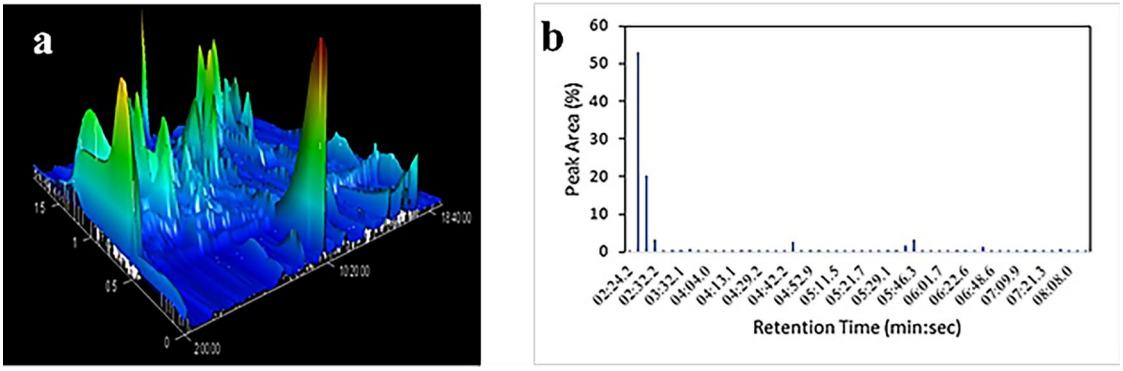

Figure 2. 3D (a) and 2D (b) GC–MS micrographs for the PAHs.

European Union.[46] This shows that the PAH levels in the KMRPPS were below what is considered acceptable for disposal by European regulations. The study also noted that the PAH levels found in the KMRPPS were below the Class 2 standard established by Environment Canada, Lands and Parks, and the British Columbia Pulp and Paper Association.[47] This suggests that there is a lesser probability of negative environmental consequences linked to PAH pollution since the PAH concentrations in KMRPPS do not exceed the regulatory limitations established by these organizations. This study implies that KMRPPS may have a reduced potential for introducing PAHs into agricultural systems, thus lessening the environmental effect associated with the use of PAH-contaminated sludge in the environment. Even though it was established that the PAH levels were below or to the recommendable levels, continuous disposal could result in bioaccumulation into the soil, water bodies, and finally into human bodies through the food chain. Thus, it is necessary to seek alternative ways of application to safeguard the environment.

Spectroscopic analysis

From the FTIR analysis shown in Fig. 3, the spectrum showed that there are no hydroxyl functional groups present in the Kimberly mill pulp sludge (KMRPPS). This indicated that all the O-H groups could have been occupied by other

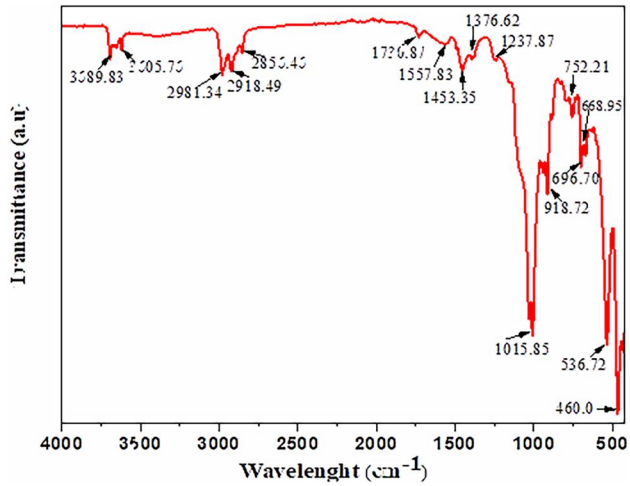

Figure 3. FTIR spectra of KMRPPS.

inorganic and organic compounds found in the sludge, which is linked to the peaks at 3689.83 and 3605.76 cm^{-1}. The triplet peak between 298.34 and 2856.46 cm^{-1} corresponds to the C-H stretching of aldehydes and ketones.[48–51] Similarly, the peak at 1730.87 cm^{-1} is associated with the C = O stretching of aliphatic and aromatic ketones. Furthermore, the peak at 1453.35 cm^{-1} is linked to C-O-H in-plane bending for carboxylic acids. The band observed at 1376.62 cm^{-1} corresponds to the bending vibrations of the C–H and C–O groups of the polysaccharides

present in the sludge. The peak at 1237.87 cm^{-1} in the sludge is attributed to the presence of hemicellulose. The peak at 1015.85 cm^{-1} is the main characteristic peak of cellulose in the polymers present in biomass. The peak at 918.72 cm^{-1} is attributed to α-D glucose and β-D glucose.[50] Stretching vibrations assigned to the C-S linkage occur in the region of 752–600 cm^{-1}, as compounds containing C–S and S–S bonds, such as sulfides and mercaptans, exhibit stretching bands in this region. Brominated compounds appear in the 600–500 cm^{-1} region of the infrared spectrum.[52–55]

From the FTIR data, there was little degradation of the cellulose and hemicellulose structures that were still present.[56] Given that the pulping process seeks to relocate or eliminate lignin while keeping the cellulose structures necessary for further processing, this was to be expected. The mechanical and chemical degradation that takes place during the production of paper and bleaching may be partially responsible for the high cellulose concentration in the paper mill sludge.[57] The bleaching process could be the cause of the lignin's absence and relatively low amount. Thus, the creation of paper mill sludge with high cellulose and low lignin concentration may be caused by the mechanical destruction of polysaccharides during the papermaking process.[58] The FTIR absorption spectra confirmed the presence of alcoholic and phenolic hydroxyl groups in cellulose and hemicellulose fibers, which are related to higher hydrophilicity.[59] The higher relative peak for KMRPPS indicated a higher water-holding capacity (WHC), which could be attributed to its hydrophilic nature. The lower hydroxyl content associated with cellulose and hemicellulose fibers also indicated higher compatibility with the inorganic hydrogel binder.

Particle size distribution

The size of particles in a material can affect the way the fibers bond with them and therefore the distribution of shear stress across the material. To understand how particles in the sludge ware distributed, the particle size distribution was estimated as shown in Fig. 4. This metric represents the size of hypothetical, non-uniformly sized particles that have different volume-to-surface area ratios. The analysis revealed that the sludge particle sizes ranged from 2.52 to 125.41 µm. This great difference in particle sizes of approximately 122.89 µm might negatively impact the overall characteristics of the composite material to be prepared from the sludge. Similarly, the two sets of particle distributions of 2.52 to 7.2 µm and 10.11 to 125.41 µm implied that the particles are not well linked (this is clearly indicated by the SEM images, Fig. 8). This could have been attributed to the various inorganic components that were present in the paper sludge. Thus, to create composites that are suitable for membranes, more modification needs to be done (such as removal of unwanted materials) from the sludge to improve the bonding surfaces of the sludge particles, including the permeability and hydrophilicity.

Elemental composition

Figure 5 and Table 3 depict the results of the localized chemical analysis performed by energy dispersive spectrometry (EDS). The analysis found that C, O, Mg, Al, Si, S, Cl, K, Ca, and Fe were present in various quantities. The most abundant elements were C and O, followed by Cl, Si, Al, and Mg, with lower concentrations of S, Si, K, and iron. The presence of these elements in primary sludge indicates how the chemical composition changes over the various stages of the paper manufacturing and recycling process.[60] The elements identified in this EDS analysis are comparable to those reported in the chemical analysis conducted on pulp sludge by Koukkanen et al., who investigated the chemical and leaching properties of paper mill sludge.[61] The high carbon content suggests the viability of utilizing this waste to produce cellulosic materials such as cellulose nanocrystals and nanofibers. High carbon content in biomass aids in the development of the polymer's bulk structure and porosity for filtration membranes.[62]

Crystal properties

To assess the KMRPPS characteristics further, X-ray diffraction (XRD) was done (Fig. 6). The more crystalline (impure) phases are represented by a few different peaks in the KMRPPS. The presence of crystalline phases and the microstructure of the sludge were both impacted by the chemical

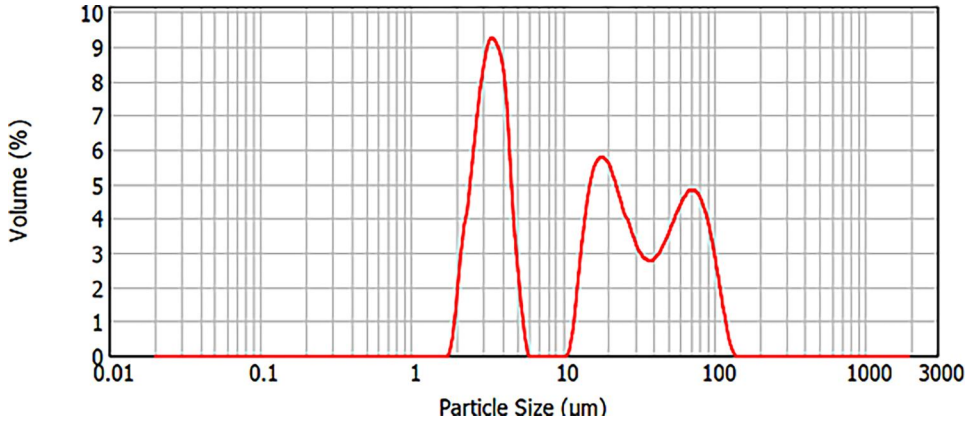

Figure 4. Particle size distribution of KMRPPS.

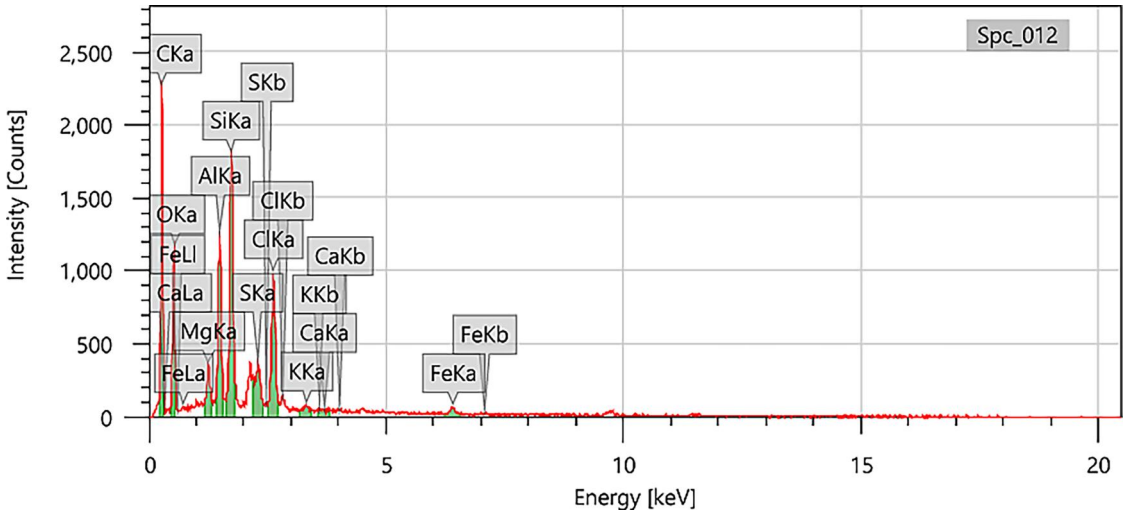

Figure 5. EDS spectra of KMRPPS.

Table 3. The elemental composition of the KMRPPS.

Element	Mass (%)	Atom (%)
C	55.04 ± 0.21	66.54 ± 0.26
O	27.85 ± 0.28	25.27 ± 0.25
Mg	1.00 ± 0.03	0.60 ± 0.02
Al	3.73 ± 0.05	2.01 ± 0.03
Si	5.87 ± 0.06	3.03 ± 0.03
S	0.93 ± 0.03	0.42 ± 0.01
Cl	4.33 ± 0.05	1.77 ± 0.02
K	0.24 ± 0.02	0.09 ± 0.01
Ca	0.09 ± 0.02	0.03 ± 0.01
Fe	0.92 ± 0.04	0.24 ± 0.01
Total	100.00	100.00

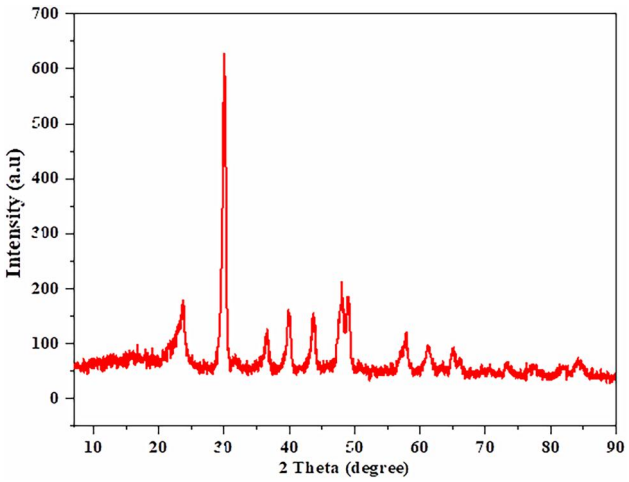

Figure 6. XRD patterns of KMRPPS.

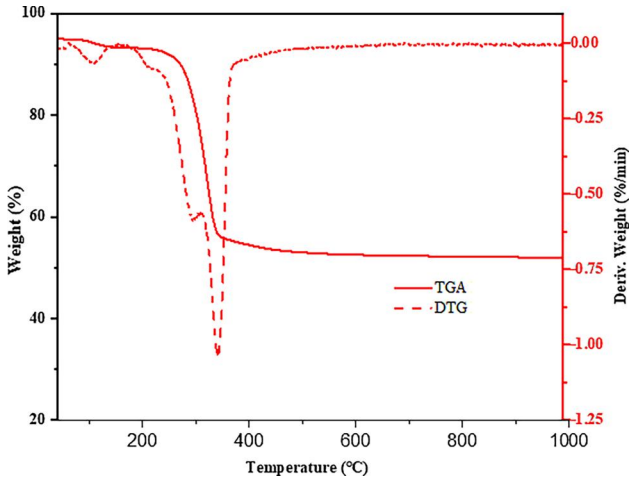

Figure 7. TGA and DTG curves for KMRPPS.

treatments, as was to be predicted. According to the XRD pattern, calcite ($CaCO_3$), which matched (JCPDS 47-1743)[63], is the primary crystal component of the paper waste. The elemental analysis that determined the presence of calcium utilized in paper manufacture is compatible with the XRD peaks at 17.10°, 23.86°, 30.14°, and 36.57°, which imply the existence of calcite ($CaCO_3$). Similar results were reported by other authors who worked on paper and pulp sludge noted that the primary crystal phase of primary sludge is calcite[29,44,64] and its predominance supports the mechanical strength of possible new materials using paper sludge in their production chain. The elemental analysis proved that the diffraction peak at 39.79° can be linked to crystalline carbon.[65] The quartz phase, such as SiO, may be responsible for the high diffraction peak at 43.82° with the JCPDS number (JCPDS card No. 5-0490).[66,67] The elemental analysis that revealed Al and Si coincides with the KMRPPS identification of kaolinite (AlSiO (OH)) at 50.07 which is supported by the JCPDS (JCPDS card No. 06-0221).[65]

Thermal properties

As shown in Fig. 7, KMRPPS, which is mostly comprised of cellulose, hemicellulose, lignin, and inert components is thermally stable over a wide temperature range and begins degrading at only around 260 °C. The early mass loss between 50 and 100 °C shown by the thermogravimetric investigation can be ascribed to moisture evaporation.[68] A maximum % weight loss was observed around 320 °C. According to Fournie *et al.* (2022), the mass loss might range from 60% to 80% depending on the inert/ash concentration of the paper sludge.[69] However, KMRPPS can still

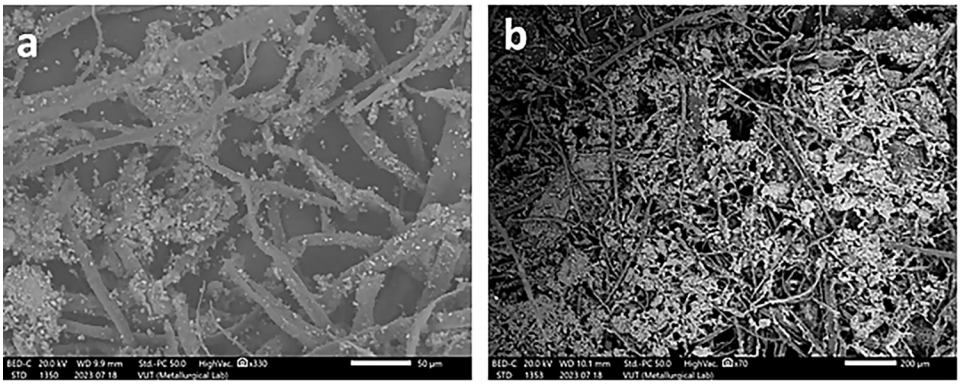

Figure 8. SEM images of KMRPPS under different magnifications.

be used in pressing processes that involve temperatures up to 250 °C without any significant degradation. This is particularly noteworthy as the composite pressing temperatures to be used in water purification membranes production range from 115 °C to 160 °C, and there is no indication that the degradation of paper sludge will impact the composite performance at these processing temperatures. As a consequence, the KMRPPS may be used without degradation in operations of water purification membranes heated to 250 °C. Paper and pulp mill sludge fibers include various amounts of cellulose, hemicellulose, lignin, inorganic components, and ash, which affects the sludge's bonding capacity.[70] From the TGA/DTG analysis, KMRPPS has the greatest cellulose and hemicellulose (66.7 wt%). The composition of the sludge concurs adequately with the compositions calculated by Poletto *et al.*, (2012).[71] Lower ash concentration (inert material) paper sludge feedstock has been found to perform better mechanically.[72]

The KMRPPS had a loss on ignition of 63.30%, while the waste analyzed by Singh *et al.* (2018) had a loss on ignition of 77.62%, which is slightly comparable.[73] Such high values are explained by the existence of a significant number of organic and inorganic compounds which might make the isolation of cellulosic materials from the KMRPPS to be inefficient.

Surface morphology

The micrographs in Fig. 8a, b were obtained using SEM under various magnifications. It reveals that the KMRPPS, an industrial waste, contains particles of various sizes and shapes that are not chemically connected (as confirmed from the PSD data in Fig. 4). Because of the link that occurs between the fibrous-natured particles, it is clear that the KMRPPS majorly comprises fibers materials. The abundance of fibers in the main sludge's composition makes it suitable for usage in membranes. Fibers' elongated form can increase interfacial adhesion in polymeric materials.[74] Additionally, the fibers have a very high energy absorption capacity and, as a result, a high mechanical strength, which might be intriguing for their ability to fit into small, constrained spaces in membranes.[75] The elongated configuration typical of the fibers also causes an increase in thermal stability. Similar properties, such as tubular elongation derived from the cellulose fibers of the KMRPPS material, may be found in the morphological structure of the paper industry wastes. However, some studies have found that the type of wood used to extract the cellulose might affect the size of the interior micro canals, which leads to additional deterioration of the fibers.[76]

Conclusions

The present study evaluated the potential environmental impacts of paper sludge disposal into the environment and its viability in the production of cellulose a renewable resource for application in water filtration membranes. The results demonstrated that the sludge could be a good soil mulch due to its excellent water-holding capacity, however, various PAHs compounds were detected which could pose environmental hazards in the long run if not well managed. High levels of cellulosic compounds (64.07 ± 2.71%) were proportional to the carbon content present in the sludge. This revealed that the KMRPPS could be a novel re-usable material in the production of cellulose for application in filtration membranes.

Abbreviations/Acronyms

PAHs	Polycyclic Aromatic Compounds
WHC	Water Holding Capacity
PSD	Particle Size Distribution
TGA/DTG	Thermogravimetric Analysis/Derivative Thermogravimetric
KMRPPS	Kimberly mill Recycled Pulp and paper Sludge
MC	Moisture Content
EC	Electrical Conductivity
PSD	Particle Size Distribution
WHC	Water Holding Capacity
TAPPI	Technical Association of the Pulp and Paper Industry
LECO	Laboratory Equipment Corporation
ASTM	American Society for Testing and Materials
TOM	Total Organic Matter
DCM	Dichloromethane
TIC	Total Ion Chromatogram

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Authors' contributions

Mr. Evans Suter contributed to the sample collection and analysis, manuscript preparation, and communication. Prof. Hilary Rutto provided the chemicals, coordinated sample characterization, and proofread the draft article. Prof. S. L Kiambi was engaged in the article development and data analysis. Prof. T.S. Seodigeng assisted in the characterization and proofreading of the draft manuscript. Dr. W. N Omwoyo contributed to the analysis and proofreading of the manuscript.

Disclosure statement

All the authors declare no competing interests.

Data availability

This article includes all of the data generated or analyzed during this research.

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