ISOLATION AND CHARACTERIZATION OF CELLULOSE FROM *Pentaclethra macrophylla Benth* POD BIOMASS WASTES FOR POLYMER REINFORCEMENT COMPOSITE

O. P. Nsude*[1], Agboeze, Emmanuel[1], E. C. Ezeh[1], O. C Ike[1], O. C. Omuluche[2], K. J. Orie[2], O. Ogbobe[1]

[1]Department of Industrial Chemistry, Enugu State University of Science and Technology Enugu State, Nigeria
[2]Department of Pure and Industrial Chemistry, University of Port Harcourt, Rivers State Nigeria
*Corresponding author**:** okechukwu.nsude@esut.edu.ng (Okechukwu Paul Nsude), +2348036102633

ABSTRACT

The interest in natural reinforced polymer composite materials is rapidly growing both in industrial and basic research applications. This is based on its availability, renewability, low density, cheapness, biodegradability, and satisfactory mechanical properties. The research reports on the isolation and characterization of cellulose from *Pentaclethra macrophylla Benth* Pod (PMBP) biomass wastes for polymer reinforcement composites. Cellulose was successfully isolated from PMBP biomass *via* delignification and bleaching. X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier Transform Infrared Spectrometry (FTIR), thermal gravimetric analysis (TGA), and deformation gravimetric analysis (DGA) were used to characterise the raw and isolated cellulose. The isolated cellulose has better thermal stability, crystallinity, and porosity than the raw cellulose. The removal of the matrix material (most hemicelluloses and almost all the lignin) led to an increase in the crystallinity and the maintenance of the thermal stability of the cellulose. The functional group elucidation showed that both raw and isolated contained cellulose, the morphology of the cellulose revealed some essential elements that are suitable for good polymer reinforcement composites. The obtained cellulose could serve as a reinforcing material in composite products or as a raw material for other applications.

Keywords: Biomass waste, Cellulose, Composite, *Pentaclethra macrophylla* Benth pod

INTRODUCTION

The globalised commitment to stimulate research and innovation in the field of renewable Agri-Resources has contributed to the development of unique and high-value lignocellulosic biomass-based products. Cellulose is a valuable resource for the long-term growth of non-food consumer items and a variety of industrial products [1].

Cellulose is a linear polymer of glucose unit monomers and is the most abundant of all naturally occurring organic materials, with an extremely high annual production [2]. The main sources of cellulose are plant fibres (cotton, hemp, flax, and jute) and wood (with approximately 42% cellulose). It is insoluble in water and is easily separated from the other constituents of a plant. Cellulose is a polysaccharide (polymer), consisting of monosaccharide (simple sugars) called monomers joined together to form very large molecules [3, 4].

Pentaclethra macrophylla Benth is a tree that grows largely in Nigeria's Southern and Middle Belt Regions, as well as other coastal areas of West and Central Africa. It belongs to the Family leguminosae and the Mimosoideae sub-family [5]. Before the seeds can be used as a dietary supplement, they must go through arduous but thorough preparation and

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fermentation. According to the literature, the unfermented seed oil of *pentaclethra macrophylla benth* contained fatty acid [5], essential mineral elements (phosphorus and calcium) [6], vitamins (thiamin, riboflavin, and niacin) [7], sugars (stachylose, galatose, and fructose). Biomass waste of *Pentaclethra macrophylla Benth* pod/husk is dumped indiscriminately and contributes to environmental pollution in the eastern part of Nigeria. Madukasi *et al*., [8] investigated PMBP as an energy source with 3456 kcal/kg. The elemental analysis confirms that PMBP is environmentally benign and serves as a feed substrate for livestock [9].

Recently, a few researches have been carried out in attempt to get pure cellulose from certain parts of the date palm. Alothman *et al.,* [10] used alkali treatment, sodium hypochlorite bleaching, and acid hydrolysis to extract microcrystalline cellulose from date palm seeds. The cellulose crystallinity index was determined to be 62% before acid hydrolysis and improved to 72% after acid hydrolysis. In a related study, microcrystalline cellulose was recovered from sugarcane bagasse using a bleaching agent, alkali, and acid hydrolysis. It was revealed that cellulose fibres with diameters ranging from 21 to 96 m and lengths surpassing 200 m could be manufactured. Furthermore, the cellulose generated had a crystallinity index of 79.49% [11]. Another research recovered cellulose from discarded bamboo front, leaflets, and fibres using dilute acid, alkali, and bleaching with acetic acid, hydrogen peroxide, and sulfuric acid. With an average crystallinity index of 52.27 %, a yield of more than 70% alpha-cellulose was obtained [4].

Composite materials have been widely employed in the domains of material science and engineering and are now commonly used globally. This is because of its superior stiffness-to-weight ratio, low electromagnetic reflectance, and ability to combine sensors and actuators. Natural fibre reinforced polymer composites are produced and used in a variety of applications to replace metal components in corrosive conditions [12]. A composite is a structural substance composed of two or more combined parts that are macroscopically linked and are not soluble in each other. It consists of a reinforcing phase and a matrix phase. The reinforcing phase material could be fibres, particles, or flakes, whereas the matrix phase materials are generally continuous [13, 14]. In composite materials, the matrix serves to shape the composite part, shield the reinforcements from the environment, transfer loads to reinforcements, and enhance the material's toughness in connection with reinforcements. The goals of composite reinforcements are to obtain toughness, rigidity, and other mechanical characteristics that outperform other properties such as coefficient of thermal extension, conductivity, and thermal transport [15. 16].

The environmental degradation associated with the extensive usage of petroleum-based resources has prompted efforts to produce biodegradable polymers. This is based on renewable bio-based plant and agricultural products that can compete in the markets currently dominated by petroleum-based

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products [17]. Biomass-based composite materials have gained attention due to both economic and ecological concerns in the last decades. The environmental effects of pollution have pushed the scientific and industrial communities to research biodegradable biomass materials as alternatives to traditional petroleum-based non-biodegradable products. In recent years, an unexpected breakthrough has occurred in which polymers are combined with organic substances rather than chemicals [18].

The purpose of this study is to isolate and characterise cellulose from PMBP biomass wastes for use in polymer reinforcement composites. FTIR, SEM, XRD, and TG/DTG were used to describe isolated cellulose in this study. This is done to determine the structure and components of cellulose derived from Pentaclethra macrophylla Benth pod biomass that is suitable for polymer reinforcement composites.

MATERIAL AND METHODS

Materials

The natural *pentaclethra macrophylla benth* pod (PMBP), Potassium hydroxide (KOH), hydrochloric acid (HCl), ethanol, toluene, acetic acid, sodium chlorite, NaOH and other chemicals were analytical grade. All the chemicals were used as received. Deionized water was used in all experiments

Sample collection and Preparation

The *pentaclethra macrophylla benth* pod (PMBP) was gathered in Aku, Igbo Etiti Local Government Area, Enugu State, and transported to the department of Industrial chemistry laboratory, University of Science and Technology, Enugu. It was carefully sorted to eliminate foreign material from the sample. To prepare for pulverisation, the sample was rinsed with distilled water, sun-dried for 2-3 weeks, and then chopped with a cutter. To increase the surface area and improve future treatment, the sun-dried chopped pod sample of PMB was crushed into fine powder and sieved to particle sizes of 0.07 mm.

Dewaxing of *Pentaclethra Macrophylla Benth* Pod (PMBP)

The dewaxing technique used was consistent with Agboeze [19]. The powdered PMBP sample (100g) was extracted for 6 hours with 375 ml of toluene and ethanol (2:1) using a soxhlet extractor to remove chlorophyll pigments and waxes. After removing the boiling chips, the filtrate (toluene-ethanol combination) was discarded. The residue (dewaxed PMS) was dried at room temperature, weighed, and stored in a sealed plastic bag for further analysis.

Bleaching of the cellulose residue PMBP

The resulting sample residue was bleached for 30 minutes at 70 ^{o}C using an aqueous solution of sodium hypochlorite (7.5%). The resulting holocellulose was extensively cleaned and filtered. The resultant holocellulose was then treated with 17.5% w/v sodium hydroxide at 80 ^{o}C for 30 minutes. The cellulose pulp was thoroughly rinsed with water. The cellulose pulp was whitened further by employing a 1:1

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aqueous solution of sodium hypochlorite (3.5 % w/v) for 5 minutes at 100 ^{0}C, followed by washing until the filtrate was clear. Excess water was manually squeezed out using a calico cloth, and the alpha-cellulose pulp was oven-dried at 50 0C [20].

Characterizations

Scanning Electron Microscope

The morphological feature of the cellulose was observed using scanning electron microscope (SEM, FEI, Quanta 200, USA), transmission electron microscope (TEM, FEI, Tecnai G20, USA) at NARICT Zaria

Fourier Transform Infrared

The IR spectra were obtained from the FTIR-8400S Fourier Transform Infrared spectrophotometer at National arbovirus research center Enugu using an ATR disc. It was used to identify the functional groups,

X-ray Diffraction

The crystalline structures of cellulose samples were determined by XRD technique. XRD analysis was carried out using a Bruker D8 ADVANCE Powder XRD instrument with CuK-α radiation of λ = 1.5404 nm and the X-ray diffractometer was operated at a voltage of 40 kV and a current of 30 mA at University of Ibadan Research Center. XRD data were collected within the range of scattering angles (2θ) of 10 to 40° at room temperature.

Crystallinity index (CrI) was calculated using the formula as stated in Equation 1:

$$\text{CrI}\ (\%) = \left(\frac{I_{200} - I_{CAM}}{I_{200}}\right) \times 100 \quad (1)$$

Where I_{200} and $I_{Cr\text{-}non}$ are the maximum peak intensities of crystalline and amorphous regions, respectively [20].

Thermogravimetric Analysis

TG/DTG curves were obtained using Seiko EXSTAR 6000 TG/DTA 6300 thermal analyzer at NARICT ZARIA. Approximately 10.2 mg of samples were placed on an aluminium pan for testing. This test was carried out from 30 to 900 °C in dynamic nitrogen atmosphere with the flow rate of 10 ml/min and heating rate of 10 °C/min

RESULTS AND DISCUSSION

X-ray Diffraction and Crystal Structure Analysis of Cellulose from PMBP

The crystallinity of cellulose is a key parameter in the determination of its mechanical and thermal properties as a reinforced component in polymer composites. In Figures 1a and 1b, XRD analysis estimated the crystallinity of the raw PMBP powder and isolated cellulose composites of PMBP. For raw powder, the intense peak around 2θ = 20° (I_{200}), represents the crystalline region of the samples, whereas, the peak around 2θ = 15.4° (I_{am}) represents the amorphous region of the powder samples. After alkaline treatment and bleaching of the raw PMBP powder, the isolated cellulose has a crystalline region of around 2θ = 34° (I_{200}), whereas the amorphous region is around 2θ = 23° (I_{am}). This finding was consistent with Yusuf *et al.*, [21], who identified the crystalline region of untreated cellulose of banana peels or eggshell to be 2θ = 30° (I_{200}) and the amorphous region as 2θ = 26° (I_{am}).

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The isolated cellulose therefore showed other peaks at 40 ^{0}C and 58 ^{O}C similar to what has been reported by Rosli *et al.*, [22] as crystallographic planes of cellulose 1 polymorph, thus indicating that isolated cellulose has typical cellulose I structure.

The raw PMBP powder and the isolated cellulose of PMBP were found to have a crystallinity index (CrI) of 23% and 32.4%, respectively. This clearly shows that the crystallinity of isolated cellulose increases with progressive chemical treatments. The removal of amorphous components and the reconfiguration of the crystalline regions into a more ordered structure result in an increase in the crystallinity index [22, 23]. The increase could be attributed to the decrease in the amorphous domain due to the cleavage of glycosidic bonds present in the disordered paracrystalline section [24]. On the other hand, the crystallinity of cellulose is determined by the amount of lignin removed and the rearrangement of cellulose molecules into a more crystalline order [25].

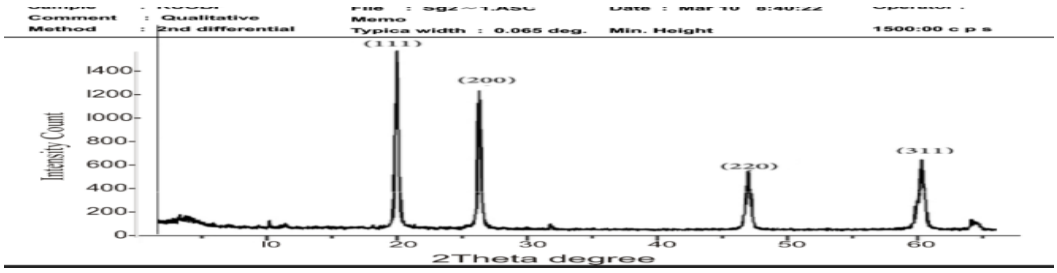

Figure 1b: XRD Diffractograms of Raw Celluloes of PMBP)

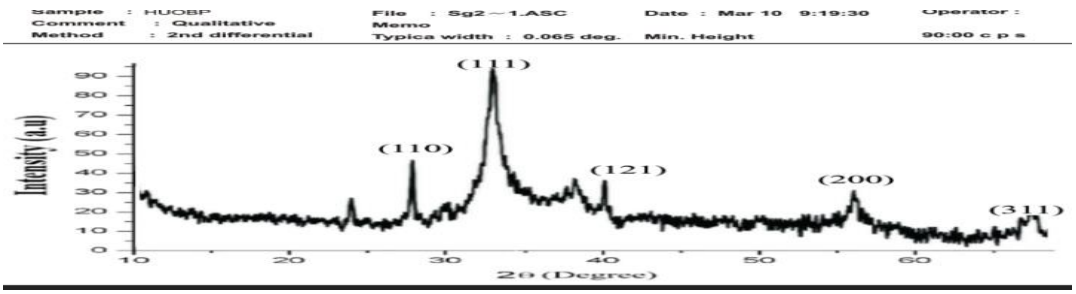

Figure 1b: XRD Diffractograms of Isolated Cellulose of PMBP

Morphological Analysis of Raw and Cellulose from PMBP Biomass

The morphology of the PMBP biomass in various phases of the chemical treatment was investigated by scanning electron microscopy (SEM). It is believed that chemical-mechanical processes have greatly influenced the morphology of cellulose. The SEM micrographs of raw PMBP powder and isolated cellulose at various stages of processing and magnifications (900x and 1000x) are shown in Figure 2. a, b, d, and e. As the image increased, the clearer the micrograph, and it was also observed that the image looked entangled and bonded together [26]. This may be that the

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lignocellulosic structure of the raw materials is not visible as they are still layered or cemented with wax, hemicelluloses, pectin, and lignin, among other impurities and other elements. Consistent with this finding is the morphology investigation of cocoa pod husk by Akinjokun *et al.*, [27], that attributed the layered or cemented to impurities due to wax, hemicellulose, pectin, and lignin.

The pore size of raw PMBP powder was 20um and that of the isolated cellulose was 50um. This indicates an increase in the porosity of the isolated cellulose. The elemental analyses shown in figures 2c and 2f confirm the presence of the same element in raw and isolated cellulose with slight percentage variation.

The presence of silicon in the cellulose will contribute to a fibre reinforcement composite and a good cross link to polymers [28].

Thermagravimetric Analysis (TGA) and Deformation Gravimetric analysis (DGA)

The results of the thermogravimetric analysis (TGA) and deformation gravimetric analysis (DTA) as a function of temperature and weight loss of raw PMBP powder and isolated cellulose of PMBP biomass are shown in Figure 3. The TGA of raw powder and isolated cellulose (Figure 3a and 3b), whereas the DGA (Figure 3c and 3d). The two samples show three major areas of weight loss. There is a slight reduction in % weight loss in Figure 3a, which is clearly seen in Figure 3c (DTG). The decrease in % weight observed in this temperature range of 20-130 ^{o}C has been attributed to vaporization of adsorbed moisture on the surfaces of the samples as well as chemisorbed and hydrogen bonded water molecules in the samples [22, 29]. Furthermore, it might be linked to the volatilization of low molecular weight organic compounds in the raw PMBP powder and residual hemicellulose in isolated cellulose [28].

From the TG curve in Figure 3a, the thermal decomposition of the raw PMBP powder began at about 280 ^{o}C and reached a maximum at around 435 ^{o}C, accounting for cellulose pyrolysis in the sample. Thermal disintegration began at 260 ^{o}C in the case of isolated cellulose from PMBP prepared after alkali treatment and bleaching (Figure 3d). This is lower than the values reported in the literature for cellulose isolated from the bark of mulberry trees [12] and cocoa pod husk [27], but comparable to the value reported by Sheltami *et al.*, [29] for cellulose extracted from *mengkuang* leaves.

The lower degradation initiation temperature observed in this study for isolated cellulose from PMBP might be attributed to the residual hemicellulose component left over from the chemical treatment. The isolated cellulose has a maximal peak in the DTG (Figure 3e) of about 375 ^{o}C. Decomposition at temperatures above 400 ^{o}C is related with lignin pyrolysis. Based on the 435 ^{o}C decomposition, the raw PMBP powder has more lignin, whereas the isolated cellulose contains more cellulose and less hemicelluloses. This reduction in lignin is due to the chemical treatment.

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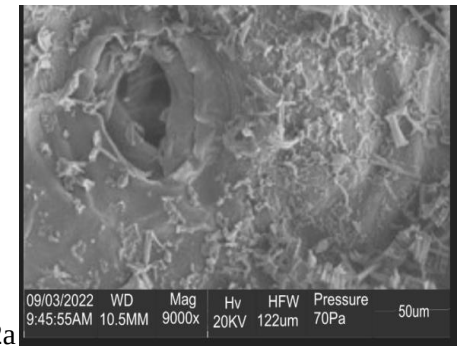

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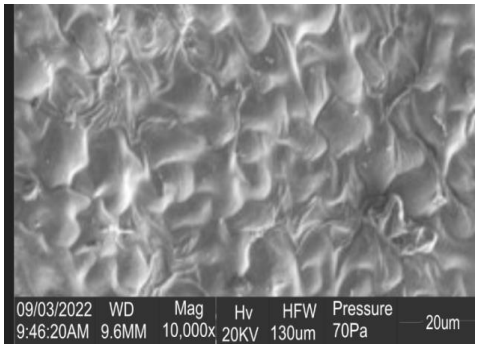

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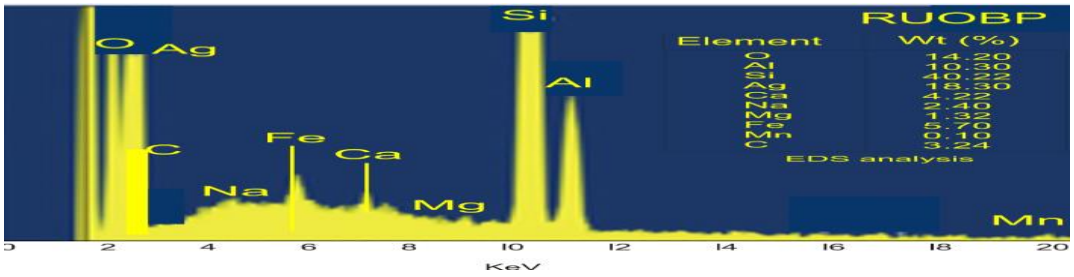

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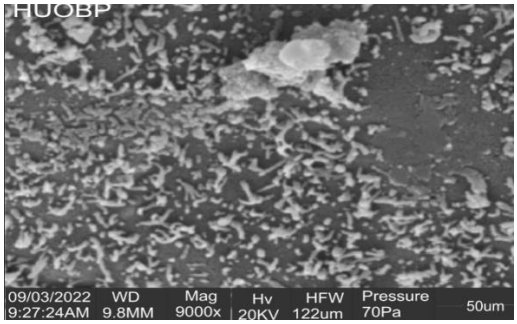

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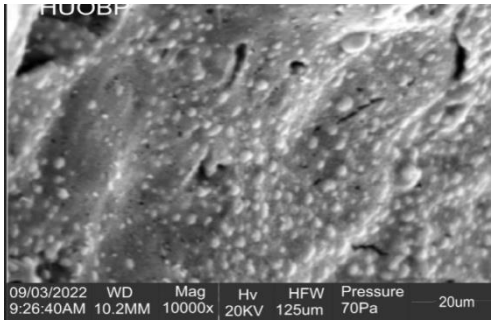

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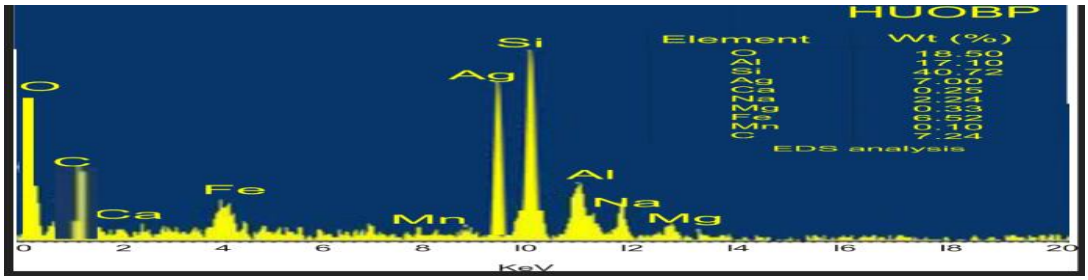

2f

Figure 2: Micrographs Analysis of Raw and Isolated Cellulose of PMBP

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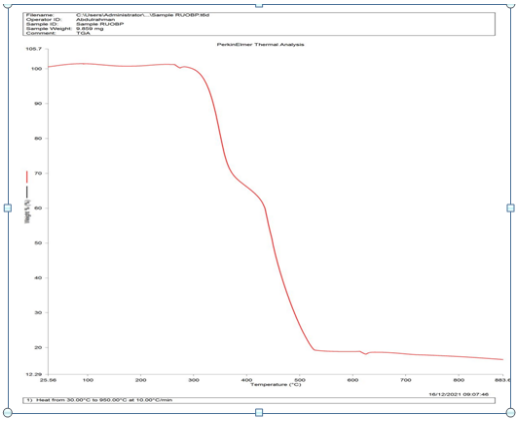

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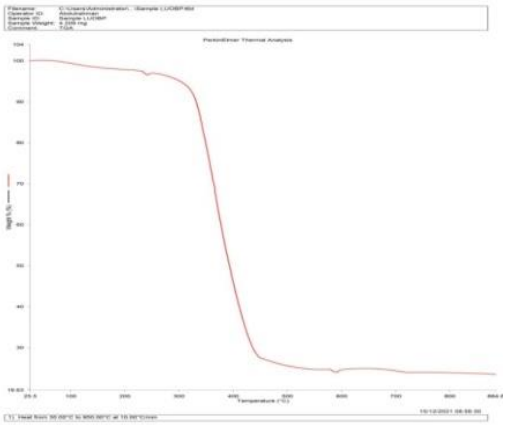

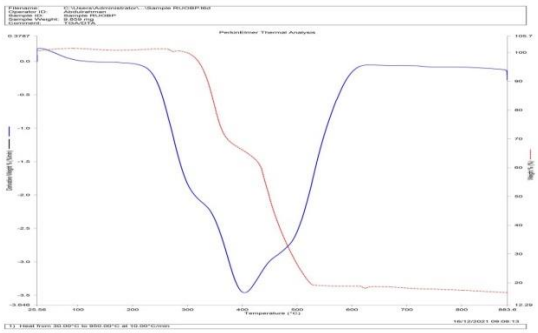

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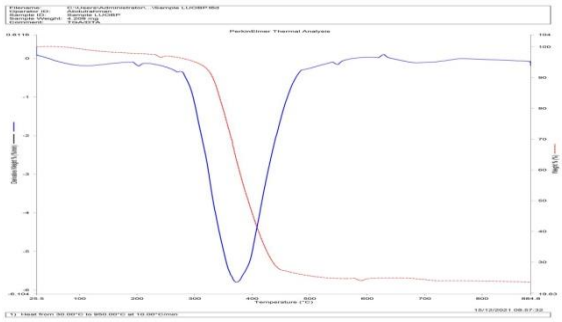

3d

Figure 3: Thermogravimetric analysis (TGA) and Deformation gravimetric (DGA) Raw and Isolated Cellulose of PMBP

ATR-FTIR Analysis of Raw and Isolated Cellulose from PMBP Biomass

Table 1 shows the functional group analysis of both raw PMBP powder and the isolated cellulose of PMBP biomass. The wide band with a maximum at 3550-3200 cm^{-1} is assigned to the O–H group. In all spectra, the peak at 1000-980 cm^{-1} represents the β-glycosidic connections of glucose ring in cellulose [30]. The band at 720-700 cm^{-1} detected in the spectra of all samples is assigned to the -Si-O-Si-, indicates the presence silicon compound both on the raw PMBP powder and isolated cellulose.

Table 1: Summary of Functional Group Analysis of PMBM Biomass

	Functional Groups	**Absorption(s) frequency (cm^{-1})**
1	OH	3446 – 3250
2	C-H stretch	2960-2850

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3	C=O stretching of carboxylic acid or ester (lignin)	1750-1735
4	C=C stretching of aromatic ring (lignin)	1470-1350
5	CH_2 symmetrical bending (lignin)	1430-1330
6	C-O stretching of acetyl (lignin)	1245-960
7	β-glycosidic linkage	1000-980
8	-Si-O-C- stretching	1250-1100
9	-Si-C- symmetric stretching	800-750
10	-Si-O-Si-	720-700

Whilst the bands at 2960-2850 cm^{-1} are due to the C–H rocking vibration of carbohydrates indicates the existence of organic molecules in the samples, and the O–H bending vibration of water [31]. Other peaks identified are show in Table 1. The overall FTIR spectra confirmed the presence of cellulose in PMBP biomass with some trace of hemicelluloses and lignin.

Composite potent of PMBP Biomass

Composites are multifunctional material systems that provide characteristics not obtainable from any discrete material. They are compact structures built by the physical combination of two or more compatible materials that differ in composition and properties. They are inexhaustible, inexpensive, fully or partially recyclable, and biodegradable [32]. Because of their availability, renewability, low density, and low cost, as well as good mechanical qualities, they are an appealing ecological alternative to glass, carbon, and man-made fibres used in composites manufacture. The characterization of raw PMBP powder and isolated cellulose of PMBP biomass reveals the presence of hemicelluloses, lignin, aluminium, silicon linked to oxygen and carbon, and other materials suitable for reinforcement. The presence of silicon and other metals adds strength and rigidity to the polymer [34]. Zhao *et al.,* [33] reported the thermal stability of poly (cyclosiloxane–carborane) at high temperatures and hash conditions. In the view of Mohamed and Hassabo, [28], silicon-based materials in cellulosic fabrics have functional applications in the industry. Rana *et al.,* [34] investigated the impact of aluminium (Al) and silicon carbide (SiC) hybridization on the mechanical and morphological characteristics of hybrid epoxy composites reinforced with Al and SiC particles. The analysis confirmed that the inclusion of aluminium (Al) and silicon carbide (SiC) provides a 50% wt of particle-reinforced composite with the optimum set of mechanical properties.

CONCLUSION

Cellulose was successfully isolated from PMBP biomass waste *via* delignification and bleaching. XRD, SEM, TGA, DGA, and FTIR measurements of the isolated cellulose revealed some level of the removal of hemicellulose and lignin from the raw PMBP powder. The

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crystallinity, pore size, stability, elemental properties, and the function group of both raw and isolated cellulose were compared. The thermogravimetric analysis demonstrated that the isolated cellulose has an enhanced thermal stability over the raw PMBP powder. The results of this study suggest that PMBP biomass can constitute an alternative raw material for cellulose polymer reinforcement composites. This is associated with the presence of some metals that have established their roles in polymerization reactions and as additives in the polymer industry. Finally, the cellulose of biomass waste exhibits structural properties and microstructure characteristics that encourage further studies on their application as a reinforcement agent in polymers as well as composites.

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