Original Research Article

Isolation, Physicochemical,and BET Aanalysisof Cellulose from *Pentaclethra Macrophylla Benth*(Oil bean) Pod biomass wastes

Abstract

Biomass energy systems are based on a wide range of feedstock, like food and agricultural waste. The research reports the isolation and physicochemical properties of cellulose obtained from *Pentaclethra Macrophylla Benth* Pod (PMBP) biomass waste. The powdered PMBP was dewaxed with toluene and ethanol (2:1) and, thereafter, treated with aqueous sodium hypochlorite (7.5% at 70 ^{o}C) and sodium hydroxide (17.5%w/v at 80 ^{o}C) to obtain isolated cellulose. Proximate, ultimate, and chemical composition analysis reveals the presence of cellulose (42.7%), hemicelluloses (7.8%), lignin (38.5%), silicon (40.72%), and aluminium (17.10%) and elements. The FTIR and Raman spectroscopic analysis revealed some of the functional groups associated with isolated cellulose and raw PMBP. The presence of hemicelluloses and lignin in the isolated cellulose implies that the isolation process did not remove all the impurities. The BET analysis reveals a better specific surface area, pore volume, and average pore diameter or size of the isolated cellulose, and could serve as a better adsorbent than the raw. As a result of the increased surface area anda high percentage of isolated cellulose in PMBP, it can be used as a sustainable energy source as well as for the environmental remediation of heavy metals.

Keywords: Biomass wastes, Cellulose, Isolation, Physicochemical Properties, *Pentaclethra macrophylla Benth* pod,

1.0 Introduction

The need to protect the environment and conserve natural resources (crude oil, coal, natural gas, etc.) has prompted study and development of renewable and eco-friendly energy alternatives. Although petroleum, the most popular energy source in Nigeria, has four times the energy density of wood, the prices of petroleum-based energy products are currently unstable, rendering them unavailable to low-income rural and urban residents [1]. Biomass energy systems use a wider range of feedstock, such as food and garden wastes [2, 3], solid wastes and sewage sludge [4], animal biomass, and agricultural byproducts[5].

The quantity of agricultural trash produced annually on the African continent demonstrates the incapacity of many African nations to follow the global trend of recycling [6,] Nigeria underutilizes a substantial portion of its annual rubbishgarbage production[1]. The burning of agro-waste and improper trash disposal (such as open dumping) are widespread waste disposal and management practises in Nigeria [7]. Previous research has demonstrated that burning agro-waste decreases agricultural output, contributes to climate change, and causes health problems, primarily respiratory illnesses such as bronchitis, eye irritation, asthma, etc. In addition to contributing to an unattractive and unappealing environment, the uncontrolled disposal of such waste also contributes to this condition [8,9,10].

The tropical tree Pentaclethra macrophylla benth (PMB) can reach a height of 21 metres. It is

densely branched, with branches extending in a canopy-like manner. The PMB seed is found in the fruit of the PMB tree, which has a 35–36 cm long and 5–10 cm broad, hard, woody pod[11] .Figure 1 depicts both the seed and the pod.

Fig I: *Pentaclethra Macrophylla Benth* Pod (PMBP)

The fermented PMB seed, known as ugba or ukpaka by the Igbo tribe of Nigeria, is a popular southern and southern-eastern condiment. The PMB seed husk/pod is a biomass that is carelessly dumped in the eastern region of Nigeria, thereby contributing to pollution.

Madukasi et al. [5] investigated the Pentaclethra macrophylla benth pod (PMBP) as an energy source and determined that the PMB seed husk and coated sludge both contained 3,456 kcal per kilogramme. According to analyses of elements, metals, and metals, PMB seed husk is an environmentally friendly biomass. Coffee husks had a higher heating value (18.34 MJ/kg) than rice husks (13.24 MJ/kg). Mhilu[12] conducted multiple studies comparing rice husk and coffee husk as energy sources and concluded that coffee husks had a higher energy content.

Cellulose is the most abundant biopolymer in nature, as it is a fundamental component of the cell walls of the vast majority of plant species [13]. ~~The fibre component of all higher plant cells, cellulose, has a distinct chemical structure, as discovered by researchers.~~As discovered by researchers, the cellulose fibre component of all higher plant cells, cellulose, has a distinct chemical structure.Some algae, bacteria, fungus, protozoans, and animal tunicates also make cellulose. It has been demonstrated that cellulose is a polymer of glucose units and that cellulose dominates the biosphere [10, 14] (See Fig. 2).

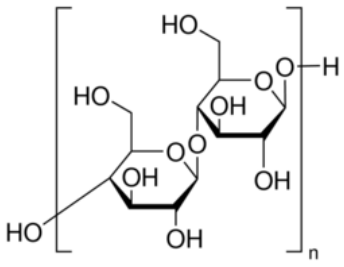

Figure. 2: The Structure of Cellulose

The chemical composition and conformation of cellulose chains, as well as their hydrogen bonding system, are responsible for their susceptibility to formingcrystalline aggregates. It is

an anhydroglucose homopolymer with the glucose residues connected in a ß-1,4 manner[15]. Cellulose is a polymer of $(C_6H_{10}O_5)n$ connected by β -(1,4) glucosidic bonds with a regular network of intra and intermolecular hydrogen bonds structured into micro fibrils [16, 17, 18]. ~~Micro fibrils~~Microfibrillarsare structured in such a way that they comprise repeating and interlaced units of crystalline and amorphous segments.Chemical treatment of cellulose fibres is known to dissolve the amorphous domain, resulting in cellulose nano-crystals [17].
Complex sugars found in cellulosic biomass, a structural component of plants, cannot be employed directly as food ingredients or fermentation substrates. It is made from leftover agricultural products like corn stover, sugarcane bagasse, discarded sugar beet pulp, and sweet sorghum, as well as agricultural feedstocks grown for energy like switchgrass, fast-growing hybrid poplar trees, and leucaena trees [19]. In order to convert cellulosic biomass into biofuels (ethanol), it is necessary to basically damage the network of plant cell walls. This releases simple sugars, which are then fermented into ethanol by bacteria or yeast [20].
The aim of the study is to isolate and characterize the physicochemical properties of cellulose made from *Pentaclethra macrophylla benth* pod (PMBP). The isolated cellulose and the raw PMBP were characterized with Fourier transform infrared (FTIR), Raman-FTIR, SEM, and BET analysis. The rationale behind BET analysis is to ascertain the adsorption potential of PMBP, which could be used as an adsorbent for the remediation of heavy metals in simulated water.The function group of isolated cellulose PMBP's was also described in the study using Fourier transform infrared (FTIR). Other analysis carried out was proximate, ultimate, and chemical compositional analysis.

2.0 Materials and Methods

2.1 ~~Sample collection and Preparation~~Collection and preparation of samples

The *pentaclethra macrophylla benth* pod (PMBP) was gathered in Aku, Igbo Etiti L.G.A, 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, then chopped with a cutter. To expand 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.

2.2 Dewaxing of *Pentaclethra Macrophylla Benth* Pod (PMBP)

The dewaxing technique used was consistent with [21]. 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.

2.4 Bleaching of the cellulose residue PMBP

~~The resulting sample residue was bleached for 30 minutes at 70 ^{o}C using a 1:1 aqueous solution of sodium hypochlorite (7.5 percent). The resulting holocellulose was extensively cleaned and filtered. The resultant holocellulose was then treated with 17.5 percent w/v sodium hydroxide at 80 ^{o}C for 30 minutes. The above-mentioned alpha-cellulose pulp was thoroughly rinsed with water. The alpha cellulose pulp was whitened further by employing a 1:1 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 ^{0}C [10, 22].~~

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2.5 Proximate Analysis of the Pod of PMB Samples

2.5.1 Determination of Natural Detergent Fibre (NDF)

At room temperature, 100ml of NDF solution, sodium sulphite, and 3 drops of N-Octanol were added to 1g of powdered PMBP. After one hour of refluxing, the mixture was filtered and washed three times with hot water and twice with cold acetone. It was then dried in an oven at 105 ^{0}C for 8 hours before being placed in desiccators to cool. The sample was weighed and recorded when it had cooled[10, 22]. The percentageof Neutral Detergent Fibre (NDF) was computed as follows:

$$\%NDF = \frac{(\text{weight of Crucible + weight of Residue}) - \text{weight of Crucible}}{\text{Weight of sample}} X100 \quad (1)$$

The % Neutral Detergent Soluble is calculated as; $\% NDS = 100 - NDF\%$ (2)

The insoluble residue was then ashed in the Biotech muffle furnace at 550 ^{O}C for 2 hours and then cooled in a desiccator. After which the weight was recorded and ash insoluble in the Neutral Detergent fibre was calculated as;

$$\text{Ash insoluble in Neutral Detergent Fibre} = \frac{\text{Loss on ashing}}{\text{weight of sample}} \times 100 \quad (3)$$

2.5.2 Determination of Acid Detergent Fibre (ADF)

100ml of ADF solution was added at room temperature with 3 drops of N-Octanol and refluxed for 1 hour. The mixture was placed in the powdered PMBP sample (1 g) in boiling water. It was left to dry for 8 hours in the oven at 105 ^{O}C and cooled in a desiccator. After cooling, the sample weight was recorded. The % ofAcid Detergent fibre (ADF) was then calculated as thus:

$$\%ADF = \frac{(\text{weight of Crucible + weight of Residue}) - \text{weight of Crucible}}{\text{weight of sample}} X100 \quad (4)$$

The insoluble residue was then ash in the Biotech muffle furnace at 550 OC for 2 hours and then cooled in a desiccator. After that ~~which~~, the weight was recorded and the ash was insoluble. The acid detergent fibre was calculated as;

$$\text{Ash insoluble in Acid Detergent Solution} = \frac{\text{loss on ashing}}{\text{Weight of sample}} X100 \quad (5)$$

2.5.3 Determination of lignin, Hemi Cellulose, and cellulose Content in of PMBP

The method employed to evaluate the cellulose, lignin, and hemicelluloses content of PMB Pod was consistent with Van-Soest et al [23]. The process was donerepeated numerous times, and the average was utilized for extrapolation.

%Hemicelluloses content = NDF% -ADF% (6)

%Cellulose content = %ADF - %Ash insoluble in Acid Detergent solution (7)

%Lignin content = 100-%Hemicellulose + % Ash insoluble in Acid Detergent Solution + Cellulose Content) (8)

2.5.4 Determination of the Moisture Content

The moisture content of the pod sample of PMB was determined in-line with Abugu*et al.*[24]. The weighed sample was heated for 3 hours at a constant temperature of 105 ^{o}C. It was rapidly removed and put into a desiccator to prevent moisture uptake from the atmosphere, after which the sample was reweighed. The procedure was repeated several times until a constant weight was obtained. Moisture content is then calculated as thus:

$$\%\text{Moisture Content} = \frac{w_2 - w_3}{w_2 - w_1} X\ 100 \quad (9)$$

Where:

W_1 = Weight of container, W_2 = Weight of Container + Sample before drying

$W_2 - W_1$ = Weight of Sample before drying, W_3 = Weight of container + Sample after drying, W_2-W_3 = Weight of Sample after drying

2.5.5 Determination of the Ash Content

This was done by weighing out a known amount of the resin into a container and placing it in the Biotech Muffle furnace. The biomass is then heated for hours at a constant temperature of 550 ^{o}C. The sample was then rapidly removed and put into a desiccator to prevent moisture uptake from the atmosphere, after which the sample was reweighed. The procedure was repeated several times until a constant weight was obtained. Ash content is then calculated as thus:

$$\%\text{Ash Content} = \frac{w_3 - w_1}{w_2 - w_1} X100 \quad (10)$$

Where:

W_1 = Weight of container, W_2 = Weight of Container + Sample before ashing

$W_2 - W_1$ = Weight of Sample before drying, W_3 = Weight of container + Sample after ashing, W_2-W_3 = Weight of Ash in sample

2.5.5 Determination of the Volatile Matter

The volatile matter was determined in accordance with Abugu*et al.*[24]. This was done by weighing out a known amount of the biomass into a container, and placed it in the Biotech Muffle furnace. The biomass is heated for 3 hours at a constant temperature of 900 oC. The sample was then rapidly removed and put into a desiccator to prevent moisture uptake from the atmosphere, after which the sample was reweighed. The procedure was repeated several times until a constant weight was obtained. Volatile matter is calculated as follows:

$$\%\text{Volatile Matter} = \frac{W_3 - W_1}{W_2 - W1} X100 \quad (11)$$

W_1 = Weight of Container, W_2 = Weight of Container + Sample before ashing @ 900^{O}C

$W_2 - W_1$ = Weight of Sample, W_3 = Weight of container + Sample after ashing
W_2-W_3 = Weight of Volatile Matter in sample

2.5.5 Determination of the Fixed Carbon Content

The fixed carbon content of the PMBP was estimated with the equation (12), which corroborates the idea of Krisztina and Andras [25]. The percentage fixed carbon content is shown as;

%Fixed Carbon Content = 100% - (Moisture Content + Ash Content + Volatile Matter) (12)

2.6.0 Ultimate Analysis

2.6.1 Determination of pH and Conductivity

The standard test technique for determining the pH of activated carbon and lignocelluloses is described by Krisztina and Andras [25] and Abugu*et al.* [24]. The 1.0g of biomass sample was weighed and transferred to a beaker. A known volume (100ml) of distilled water was measured into the beaker and swirled for 1 hour. The pH of the samples was tested after they had stabilized. The pH was measured electrochemically using a Hansa pH-meter and a conductivity metre. Analyses were performed in duplicate.

2.6.2 Solubility of the of PMBP Sample in Water, NaOH and HCl

For water solubility (S), 0.5g Biomass samples were mixed with 100ml of distilled water in a 250ml flask and shaken for 2 hours at room temperature at 200rpm in a shaker. Pre-weighed Whatman No. 1 size 15 filter papers were used to filter the mixtures. The filter papers containing residual carbons were dried in an oven at 105 degrees Celsius for 12 hours. The filter papers, along with residual carbons, were weighed in a dessicator after cooling to room temperature. The weight ratio of unrecovered carbon to original sample was used to calculate the percentage solubility in water. The sample's solubility in PMB HCl (0.2M) and NaOH (1M) was determined using the same method.

$$S(\%) = \frac{\text{Loss in weight on dissolution}}{\text{weight of Original Carbon}} X\ 100 \quad (13)$$

2.6.3 Determination of Apparent Density

The method used for apparent density determination of the sample of PMB was consistent with Benerjee and Bhattacharyya [26]. The samples of each dry Biomass of PBM were packed in a weighed empty dry graduated cylinder. Thereafter, it was also reweighed to determine the new when pack when pack with sample. The apparent density was calculated as;

$$\text{Density}(g/cm) = \frac{\text{Weight of dry activated carbon}}{\text{Volume of dry material}} X\ 100 \quad (14)$$

2.6.3 Determination of Iodine Value

This is measured according to the procedure established by the American society for Testing and Materials (ASTM, 2006). A stored cool dry Biomass material was used at room temperature to determination the iodine number requires for the estimation of three carbon dosages. Equation 15 was used to extrapolate the values in Table 3.

E = [A-(DF)(C)(126.93)(50)]/M (15)

M = carbon, g, A = (N2) (12693.0), DF = dilution factor, C = residual iodine

2.7. 0 Equipment Used in This Study

2.7.1 FTIR analysis

Fourier transform infrared spectroscopy (FTIR) can provide accurate predictions of the chemical composition of biomass. An FTIR spectrum contains information on the fundamental vibrations and allows for greater separation of similar signals, such as hydroxyls for carbohydrate isomers. The dried (50 ^{o}C for 24 h) powdered samples (2 mg) of biomass fibres were dispersed on IR grade ATR for FTIR analysis of the subjected biomass samples. The FTIR spectra were obtained with a resolution of 1 cm^{-1} using an Excalibur Bio-Rad spectrophotometer (Model FTS 3500 GX).

2.7.2 Raman spectroscopy analysis

Raman spectrum is one kind of spectral fingerprint to identify the electrically symmetrical bond. The intensity of IR absorption is affected by changes in the dipole moment of the bond, whereas the intensity of Raman absorption is affected by changes in the polarizability of the bond. The emphasis on Raman spectroscopy of the submitted biomass samples was undertaken to identify electrically symmetrical bonds (i.e. having no dipole moment) present in lignin and other structural carbohydrates.

2.7.3 Scanning Electron Microscopy (SEM)

The microscopic characteristics of the samples were tested using a Hitachi S2700 scanning electron microscope (SEM). A portion of the dry sample was placed on the SEM sample lens and then mounted in a vacuum chamber to prevent obstruction and other particles contamination. The imprint on the electron was converted into a three-dimensional image. The power of magnification used for the samples was 800 to1000x.

2.7.4 BET Analysis for surface area (QUANTACHROME NOVA4200e model)

The PMBP sample was properly weighed and placed into the sample cell, then the filled sample cell bulb was placed into the heating mantle, and the clamp was placed around the mantle to hold the sample cell firm. The out gassing temperature was set at 2500 c, and the system was instructed to start degassing for 3 hours and turn on the heater. Thereafter, the heating mantle was turned off and the PMBP sample cell was allowed to cool.

3.0 Results and Discussions

3.1 Physical Properties of Alpha Cellulose of PMBP

Table 1 shows the physical properties of alpha cellulose of PMBP. The cellulose of PMBP was identified with the iodated zinc chloride solution and the pH value shown in the table.

Table 1. Physicochemical Properties of Isolated Cellulose of PMBP

Identification	Turns violet-blue with iodated $ZnCl_2$
pH	6.42
Solubility test	
Distilled water	Insoluble
Dil NaOH (5%)	Slightly soluble
Dil HCL (5%)	Slightly soluble
Alcohol	Insoluble
Acetone	Insoluble

The purified PMBP residue obtained from extraction turned violet-blue, confirming the presence of cellulose. The solubility investigations revealedthat the identified cellulose was in soluble in cold water and warmed water, whereas~~,~~ it wasslightly soluble in 5% NaOH and

HCL. This soluble investigation was consistence with Mhilu[12]who investigated the cellulose solubility of rice, coffee husks, and their blends at variouspercentages.~~that worked on the solubility of cellulose of rice, coffee husks and its blends at different percentages.~~Madukasi*et al.*[5] confirmed that cellulose obtained from sugar cane bagasse is insoluble inethanol and ethyl acetate solvents.

3.2 Analysis of Holocellulose Content of *Pentaclethra Macrophylla Benth* Pod (PMBP)

Table 2 shows the holocellulose content of PMBP biomass and was estimated withthe Van-Soest[23] method. Holocellulose is a water-insoluble carbohydrate fraction of wood materials.

Table 2: Parameters of Holocellulose Content of PMBP

Parameters	Results
% neutral detergent fiber	61.5
% neutral detergent solution	38.5
% ash insoluble NDF	44.2
% Acid detergent fiber	53.7
% acid detergent solution	46.3
% ash insoluble ADF	11.0
% hemicelluloses	7.8
% cellulose	42.7
% lignin	38.5

The PMBP after being washed, dried and grinded were analyzed to determine their holocellulose content as displayed in Table 2. The analysis revealed 72.7% of cellulose, 38.5% of hemicellulose~~s~~ and 7.8% of lignin content. The estimated values were compared with resultsfromCouret*et al.*[27] that research on the characterization of cellulose nanocrystals from post-consumer wood fiberboard waste ~~are~~was comparable, and Brandt*et al.*[28] that worked on deconstruction of lignocellulosic biomass with ionic liquids. The cellulose component of PMBP was higher than the hemicelluloses and the lignin contents. These also reveal the efficiency of the purification process and were consistent with that of the literature.

3.3 Proximate analysis of Raw and Isolated Cellulose of*Pentaclethra Macrophylla Benth* Pod (PMBP)

Table 3 shows the mean values of the proximate analysis of some parameters of the raw and isolated cellulose of PMBP biomass. The experiments were carried out in triplicate and results were displayed in mean.

Table 3: Proximate Analysis of Raw PMBP and Isolated PMBP

Parameters	% Result of Raw- PMBP	% Result of PMBP-C
Density*	0.64	0.473
Moisture content	9.16	7.80
Ash content	0.17	1.19
Volatile matter	11.37	8.6
Fixed carbon content	78.72	55.17%
Iodine value*	202	124.4

The moisture content of both isolated cellulose and the raw PMBP was low. However, the isolated cellulose PMBP has the lowest moisture content ~~with~~at 7.80%, whereas the raw PMBP has the highest moisture content ~~with~~at9.16%. The variation of the moisture content is associated with the chemical treatment of the raw PMBP. The low moisture content therefore implies that PMBP can be used as a bio-fuel technology. This is in line with the view of Grandesso et al. [29] and Nuruddin et al. [30] that high moisture content has a significantly lower carbon burn rate and encourages bacterial activity.

Ash content is the inorganic residual left after the volatile matter has been removed. The findings reveal the lowest ash content was 0.17% and the highest volatile matter content was 11.37%. It has been reported that the ash content reduces the fuel quality because ash has an affiliation with fouling. However, high volatile matter ascertains easy ignition. The treatment of the raw PMBP was effective, based on the lower values of volatile content estimated for the isolated cellulose PMBP. The raw PMBP has the highest fixed carbon content of 78.72%, whereas the purified PMBP-C has a fixed carbon content of 55.17%. This implies that the carbon content of the isolated cellulose could be attributed to the removal of carbon due to lignin and hemicelluloses, which were most likely present in the raw PMBP. The observations were in line with Vallejo et al. [31], who investigated the physicochemical characteristics of selected lignocelluloses and cellulose of different biomass. Other properties of both the isolated cellulose and the raw PMBP are shown in Table 3.

3.4 Scanning Electron Microscopy (SEM) analysis of Raw and Isolated Cellulose of*Pentaclethra Macrophylla Benth Pod (PMBP)*

The SEM analysis was aimed at estimating the porosity and elemental composition of the raw powder and isolated cellulose of PMBP biomass. 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.

Table 4: Elemental Analysis of Raw PMBP and Isolated PMBP

Elements	% Raw PMBP	% IC-PMBP
Oxygen	14.20	18.50
Aluminum	10.30	17.10
Silicon	40.22	40.72
Silver	18.30	7.00
Calcium	4.22	0.20
Sodium	2.30	2.24
Magnesium	1.32	0.33
Iron	5.70	6.52
Manganese	0.10	0.10
Carbon	3.24	7.25

Table 4 shows the elemental analyses of the raw powder and isolated cellulose of PMBP biomass. The analysis confirmed the presence of the same element in both the raw powder and the isolated cellulose of PMBP with a slight percentage variation. This variation also implied the effectiveness of the treatment on the raw PMBP sample. This result is consistent with Madukasi*et al*. [5], who worked on the elements present in oil bean husk and its energy

composition. The high percentage of silicon implies that the cellulose of PMBP can serve as a good polymer reinforcement composite and other applications.

3.4 Fourier Transform Infrared Spectroscopy (FTIR) of*Pentaclethra Macrophylla Benth* Pod (PMBP)

The purpose of the FTIR spectroscopy investigation was to determine the functional groups of the cellulose of *Pentaclethra Macrophylla Benth* pod (PMBP). Table 5 shows the vibration frequency of PMBP extrapolated from the FTIR spectra.

Table 5: Summary of Selected FTIR Active Moieties of Cellulose in the PMBP

FTIR Active Moieties	Wave number (cm^{-1})
Hydroxyl (OH)	3446 - 3250
C-H stretch of aliphatic structure.	2960-2850
C-O-C vibration in cellulose and hemicelluloses.	-1158
C=O (acetyl and ester).	1735
C=O at C-3 and C-C of cellulose.	1058
Aromatic ring stretch in lignin.	1604 - 1516
C-H (Crystalline andAmorphous cellulose).	893
CH2 cellulose, lignin.	1462-1425
C-H deformation in cellulose and hemicelluloses.	1384-1346

The spectra had a broad band at 3446 to 2890 cm^{-1} that corresponded to stretching of H-bonded –OH groups and methyl, and methylene units C-H stretching absorption at 2890-2850 cm^{-1}. These findings support the work of Alemdar and Sain[32], who investigated the characterization of nano-fibres derived from agricultural residues–wheat straw and soy hulls. The C=O stretching of the acetyl group in hemicellulose was related with the absorption bands at 1736 and 1238 cm^{-1}. The aromatic C= C ring stretching and C-H deformation in lignin's methyl, methylene, and methoxyl groups are shown by the absorption bands at 1604 and 1516 cm^{-1}. The bands at 1419, 1157, 1026 and 901 cm^{-1} correspond to the $–CH_2$ scissoring, C H asymmetric, –OH bending, C O antisymmetric, C O C pyranose ring skeletal and C H rocking [33, 34]. Various studies have also found that the band at 1163 cm^{-1} and 1669 cm^{-1} corresponds to the NH, C O C α-1,4-glycosidic bond stretching [35]. In all spectra, the peak at 897 cm^{-1} represents the β-glycosidic connections of glucose ring in cellulose [35]. The overall FTIR spectra confirmed the presence of cellulose in PMBP biomass with some traces of hemicelluloses and lignin.

4.3. RAMAN Spectroscopy of*Pentaclethra Macrophylla Benth* Pod (PMBP)

Raman spectroscopy methods have good potential to determine the structural characteristics and distribution of the functional components of cellulosic biomass materials. The raw and isolated Raman spectra of cellulose obtained from *Pentaclethra Macrophylla Benth* pod (PMBP) are shown in Figure 3a and 3b.

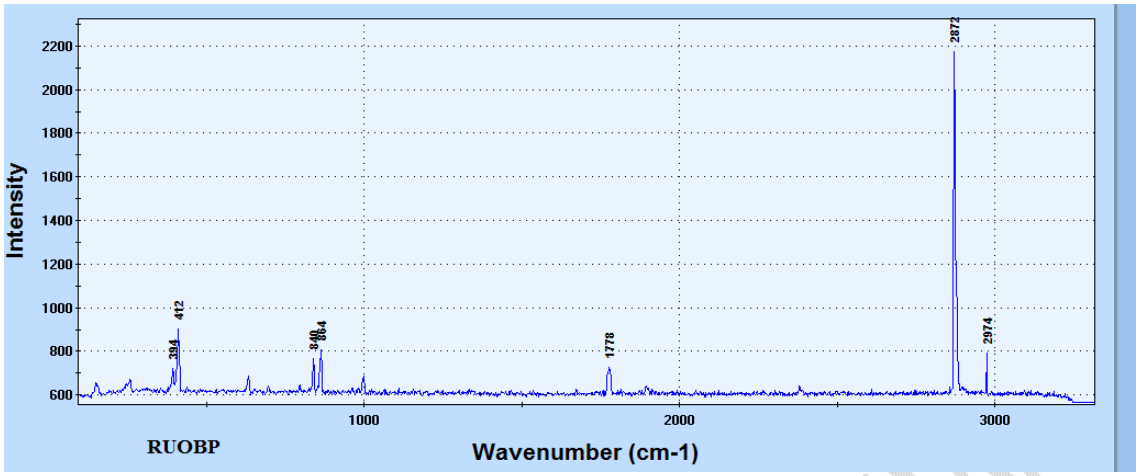

Fig 3a: Raman Spectra of Raw powder of *Pentaclethra Macrophylla Benth* Pod (PMBP)

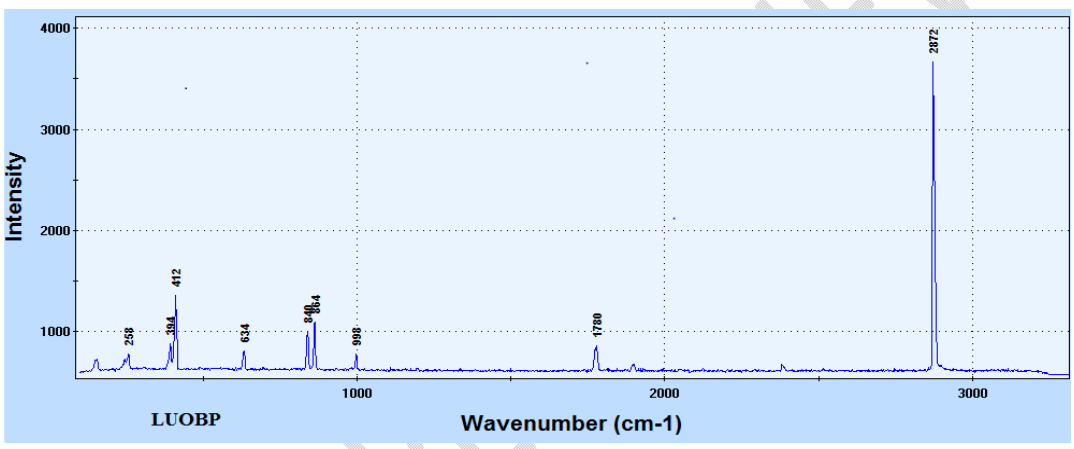

Fig 3b:Raman Spectra of Isolated Cellulose of *Pentaclethra Macrophylla Benth* Pod (PMBP)

In the Raman spectra of PMBP biomass reveal the presence of cellulose and other constituents that may be seen as impurities. The vibration frequency of 2872 cm^{-1} is the peak of strong C-H stretch, the peak at 998 cm^{-1} is for HCC and HCO bending at C-6, the peaks at 840 cm^{-1} and 864 cm^{-1} are the bending vibration of C-O-C and C-C. This peak confirms the presence of hemicelluloses and cellulose, and is thus consistent with the findings of Vallejo,*et al.*[31], ~~that~~who reports on the overlap of the peaks and the vibration frequency of hemicelluloses and cellulose. The peaks at 1780 cm^{-1} is the vibration frequency of conjugated C=C stretch of aromatic ring aldehyde, which indicates the presence of lignin. This also corroborates the findings of Kotaiah*et al.*,[36], ~~that~~who researched on the isolation of cellulose from sugar bagasse. The band around 380 cm^{-1} which is seen in Fig 3 is assigned as bending vibration of C-C-C and is ring deformational mode. The most significant difference in spectra in Fig 3a and 3b is the peak at 2974 cm^{-1} that is missing in the Fig. 3. This implies that the isolation process was effective on the note that it was able to remove some impurities of lignin, which is most probably the C-H methylene groupsof the lignin[37].

Table 6; Multi BET Analysis of Modified and Unmodified PMBP

S/no	Properties	PMBP-R	PMBP-C
1	Specific Surface area BJH (m^2/g)	164.728	324.120
2	pore volume (m^2/g)	0.081	0.159

3	Average pore Diameter or size BJH (nm)	2.013	2.133
4	BET summary surface area (m^2/g)	159.133	288.090
5	BET pore Diameter (nm)	2.140	2.94
6	Correlative Coefficient	0.989	0.996

Table 6 shows the multi BET analysis of isolated cellulose and unmodifiedPMBP. The specific surface area of the PMBP-R is lower than that of the isolated cellulose. This increase in specific surface area is associated with different chemical treatments given to the raw PMBP. The increase in surface area, therefore, indicates that the isolated cellulose should show enhanced biosorption over unmodified. Consistent with this finding is the research of Sieradzka et al. [38], who worked on the pyrolysis of biomass wastes. The other properties considered in Table 6 were pore volume, average pore diameter, BET summary surface area, and BET pore diameter, and they also have higher values for the isolated cellulose when compared with the raw PMBP. In line with Farooq et al. [39], the adsorption capacity is directly proportional to the BET surface area, and hence larger particles with more BET surface area should give a higher removal of metal. It therefore implies that the isolated cellulose of PMBP can serve as a good metal remover in a polluted environment. A previous study consistent with the findings showed that modification of biomass with CTAB increases bet surface area. It has been reported that modification of CTAB causes a 26% increase in BET surface area and thus is generally favoured by reduction in particle size (i.e., large surface area), and it has been reported that smaller biomass particles have higher adsorption capacity than larger particles[41, 42]. The results of the correlative coefficient suggest that the isolated cellulose is better than raw samples of PMBP, as seen in Table 6.

5.0 Conclusion

Renewable biomass sources are a viable option for addressing global energy issues and environmental remediation. The study describes the isolation and physicochemical aspects of cellulose-PMBP biomass waste. The findings from the proximate and ultimate analysis reveal that the reduced moisture content and increase in ash content of isolated cellulose make it a better option than raw PMBP for bio-fuel. The high silicon and aluminium content of both isolated cellulose and raw PMBP is an indication that they can be used as polymer reinforcement composites, among other things, as a result of their important mineral composition. Some functional groups associated with lignin, hemicelluloses, and cellulose ~~was~~were identified using FTIR and Raman spectrometers. The presence of lignin and hemicelluloses in the isolated cellulose implies that the isolation process did remove all the impurities (hemicelluloses and lignin) in the isolated cellulose of PMBP waste. The solubility studies confirmed that the isolated cellulose of PMBP was slightly soluble in 5% of NaOH and HCl, but not soluble in ethanol and acetone. The chemical compositional study reveals cellulose content of 42.7% with a lower percentage of hemicelluloses and lignin. The BET analysis shows that the isolated cellulose has a better specific surface area, pore volume, and average pore diameter or size than the raw PMBP, which implies that isolated cellulose could serve as a better adsorbent than the raw. Because of the increased surface area and high percentage of isolated cellulose in PMBP, it can be used as a sustainable energy source as

well as for environmental remediation. As a way of recommendation, further purification should be done on the isolated cellulose of PMBP using other chemical combinations.

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