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Structural and Thermal Evaluations of Cellulose and Lignin from Bambusa vulgaris

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Abstract: *Lignocellulose, the cheapest biomass in the world, contains cellulose and lignin as its main constituents. Earlier investigations have shown that cellulose and lignin can be extracted from maize stalk, sugarcane bagasse, tea leaf waste fibers, rice straw and wood. This study targets the extraction of lignin from bamboo tree of Nigerian origin. Bamboo sticks were sourced from Ebonyi State (6.2649°N, 8.0137°E.), ground and sieved to 600µm particle sizes. The bamboo particles (BP) were treated with 2M NaOH for 6hours. The residue was washed with distilled water to obtain cellulose particles (CP) while the filtrate was neutralized with 1M HCl at pH 5.5 with the addition of 95% ethanol at room temperature. The filtrate was later conditioned to pH (1.5) and heated to 90°C to evaporate ethanol and precipitate lignin particles (LP). Fourier Transform Infrared Spectroscopy (FTIR) revealed the functional groups present in BP, CP and LP. Results from Differential Scanning Calorimetry (DSC) showed that 481.5J/g will be required for the decomposition of LP which is higher than that for CP (134.9J/g). Aromatic rings with various branches present in LP's structure may be responsible for its highest magnitude of its temperature at the onset of decomposition (Tonset) of 284.6°C during thermal decomposition as observed via Thermogravimetric Analysis (TGA). Characterization via X-Ray Diffraction (XRD) informs that the crystallinity index of LP and CP are 48.9 and 75% respectively. It can be established from this study that bamboo tree is a sustainable material for the processing of cellulose and lignin.*

Keywords: *Controller, Differential Drive, Kinematics, Mobile Robots, Modelling*

INTRODUCTION

Lignocellulose, a renewable material, comprises cellulose and lignin that are the first two most abundant biopolymers in the world. These materials have found applications in the areas of energy, textile, and medicine. Cellulose and lignin have been chemically extracted from maize stalk, sugarcane bagasse, tea leaf waste fibers, rice straw and wood. Lignocellulose, a biomass is the cheapest and most ubiquitous renewable material in the world containing cellulose, hemicellulose, and lignin as its main constituents (Fig. 1). Cellulose is the most abundant biopolymer found in plants (Salas *et al.,* 2014). It is a biodegradable structural polysaccharide that has found its use in areas including energy, textiles and medicine. Cellulose strengthens cell walls of plants and has been confirmed to possess low density with good mechanical properties (Wulandari *et al.,* 2016). In particulate or fiber form, cellulose has been used in reinforcing other polymers (either natural or synthetic) to form composites (Wang *et al.,* 2013). The properties of cellulose are greatly influenced by the OH functional groups in its structure, which also plays a major role in the inter and intra molecular hydrogen bond interactions (Klemm et al., 2005). On other hand, lignin is second to cellulose in terms of natural abundance. It is a complex three-dimensional network polymer that makes up a continuous matrix phase in plant cell walls. This provides mechanical strength and structural support to the plant. Lignin has been used in making dyes, fuels, carbon fibers and emulsifiers (Magnus and Hakan, 2014; Podko´scielna *et al.,* 2017).

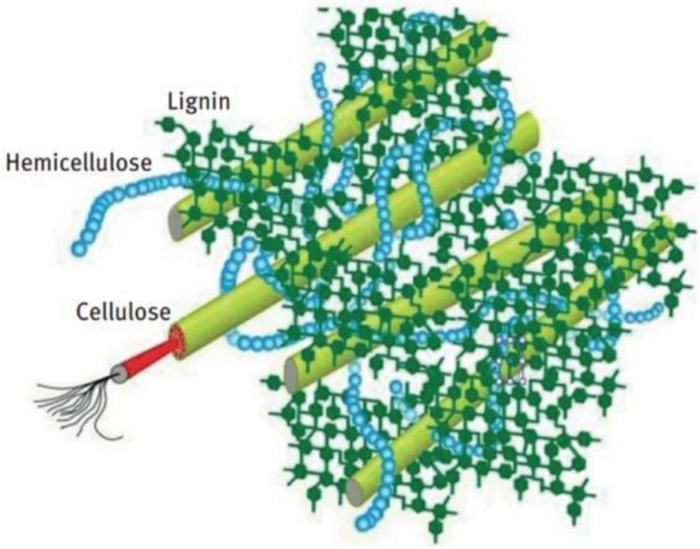

Fig. 1. Cellulose, hemicellulose and lignin in plant cell wall (**Wang *et al.,* 2017**)

Researchers like Wulandari *et al.* (2016), Minu *et al.* (2012) and Xiao *et al.* (2011) had chemically isolated cellulose from sugarcane bagasse powder using sodium hypochlorite (NaOCl). This reagent was used in bleaching the dried powder to remove soluble lignin (as filtrate) while the washed residue was treated with sodium hydroxide (NaOH) to isolate cellulose (residue) from hemicellulose that remained in the filtrate. Motaung and Mtibe (2015) compared the quality of cellulose sourced chemically from pulverized maize stalk to that obtained via mechanical technique. Potassium hydroxide (KOH) was used to isolate cellulose (residue) from hemicellulose and lignin, which went into the solution as filtrate. The mechanical treatment entailed further subjecting the extracted cellulose in sulphuric acid (H_2SO_4) under vigorous stirring for 30 minutes. Results from their investigation showed that the mechanical treatment promotes the formation of cellulose nanowhiskers with diameters between 3-7nm. Thermal stability of cellulose with enhanced crystallinity was improved via chemical treatment as a result of new functional groups that were formed during reaction. Lignin and hemicellulose were eliminated from tea leaf waste by treatment with NaOH to leave cellulose residue followed by bleaching with aqueous chlorite, $NaClO_3$ (Abdul Rahman *et al.,* 2017). In the works of Dai *et al.* (2018), corn residues were added to ethanol and stirred at 60^{o}C for 1 hour. The solution was filtered after cooling and the three volumes of water were added to the filtrate. The pH of the filtrate-water solution was adjusted to 1 and a lignin precipitate was yielded.

Concentrated H_2SO_4 (14M, 20ml) was added to banana skin devoid of extractives to obtain lignin as residue while dissolved cellulose was formed in the filtrate (Ajani *et al.,* 2011). The cellulose was precipitated on addition of 8M NaOH. Earlier studies however, have shown that cellulose and lignin have been extracted from maize stalk, sugarcane bagasse, tea leaf waste fibers, rice straw and wood. This study thus, focuses on the extraction of cellulose and lignin particles from bamboo. The plant is abundant in the southern region of the country, which is characterized by the presence of water bodies. Bamboo has found various uses in materials including furniture, charcoal, paper and artistic substances owing to its abundance and cheap means of processing **(**Emamverdian *et al*., 2020**)**. Bamboo is a woody plant that finds its natural habitat in the tropical and subtropical regions in the world **(**Okokpujie *et al.,* 2020**)**. In Nigeria, the plant is mostly domiciled in the southern region (Fig. 2) which is characterized by the presence of water bodies **(**Okokpujie *et al.,* 2020). Bamboo in Nigeria has enjoyed wide applications as it is a cheap bio resource, readily available and easy to process.

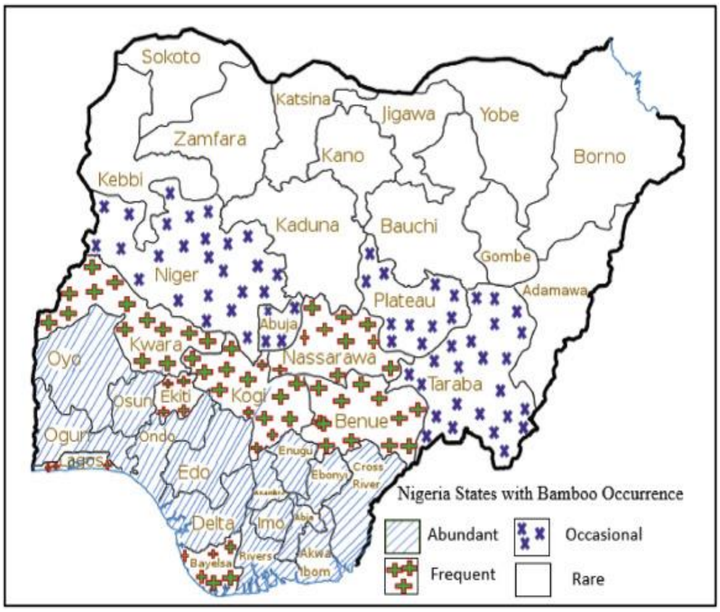

Fig. 2. Map of Nigeria displaying the states where bamboo is readily available in nature (Okokpujie *et al.,* 2020)

MATERIALS AND METHODS

2.1 Sample Preparation

Bamboo sticks were sourced from Ebonyi State (Eastern region of Nigeria; 6.2649°N, 8.0137°E), ground and sieved to 600μm particle sizes. The ground bamboo particles were treated with 2M NaOH and heated in a water bath for 6hours after, which the solution was filtered. The residue was washed with distilled water to neutral pH to obtain cellulose while the filtrate was neutralized with 1M HCl at pH 5.5 with the addition of 95% ethanol at room temperature (32°C). Pellets (precipitates) formed during the neutralization process were filtered from the NaOH/HCl/ethanol solution and air dried to yield hemicellulose. The filtrate (NaOH/HCl/ethanol solution) was conditioned to a pH 1.5 (this was achieved by adding 1M HCl/NaOH droplets until the pH value was attained) and heated to 90°C to evaporate ethanol and precipitate lignin.
The lignin obtained was washed and dried for characterizations.

2.2. Characterization of Bamboo, Cellulose and Lignin Particles

2.2.1. Fourier Transform Infrared spectroscopy (FTIR)

A Nicolet 6700M equipment was employed in observing the functional groups of the samples. Ten milligram of each particle was dispersed (in KBr), compressed and their absorbance was measured between 500–4000 cm^{-1} at a resolution of 4 cm^{-1}.

2.2.2. Thermogravimetric Analysis (TGA)

Two milligram of particles was heated to 750°C in a TGA Q500 instrument. Lignin and cellulose contents, their temperatures at the beginning and end of decompositions were recorded. The TGA equipment was operated at 10°C/minute heating rate.

2.2.3. Differential Scanning Calorimetry (DSC)

This was done with use of a Mettler Toledo DSC equipment as samples were heated to 150°C at a similar heating rate as done for TGA. Change in enthalpy values (ΔH) were calculated by integrating the area under consideration using the expression shown in Equation (1).

$$\Delta H = \int Cp \, dT \quad (1)$$

Where Cp is the specific heat capacity (J/g) and T, is the temperature measured in degree Celsius. The Cp was obtained from the expression shown in Equation (2).

$$Cp = \text{Heat flow/heating rate} \quad (2)$$

Where the heating rate is 10°C/minute

2.2.4. X-Ray Diffraction (XRD)

This was carried out using a PANanalytical Empyrean equipment which was operated at 40 kV and 40 mA. Crystallinity (Xc) was calculated using Equation (3) as in Juarez-de la Rosa *et al.,* (2012).

$$X_c\,(\%) = [I_c / (I_c + Ia)] \times 100 \quad (3)$$

I_c and I_a are the intensities of the crystalline and amorphous regions respectively

RESULTS AND DISCUSSION

3.1. Fourier Transform Infrared Spectroscopy (FTIR)

The functional groups present in bamboo particles (BP) between1800-500cm^{-1} via FTIR shows fingerprints of seven major peaks (Fig. 3). The OH stretching of absorbed water is indicated on 1644cm^{-1} while absorbance on 1591 and 1485cm^{-1} show the presence of C=C vibration in benzene ring and CH_2CH_3 vibrations respectively. Cellulose and hemicellulose present in the biomass are evidenced with C-O-C stretching on 1163cm^{-1} while their CH stretching is absorbed on 1341 and 905cm^{-1} respectively. Spectrum obtained in this result is typical of most lignocellulosic materials including maize stalk (Motaung and Mtibe1, 2015), tea leaf waste fibers (Rahman *et al.,* 2017), wheat straw (Adapa *et al.,* 2011), albezia pods (Gbenebor *et al.,* 2020), sugarcane bagasse (Mzimela *et al.,* 2018) and palm kernel shell (Adeosun *et al.,* 2016). The major peaks in cellulose (CP) and lignin particles (LP) extracted from the BP are also illustrated in Fig. 3 while the functional groups in these distinct peaks are presented in Table 1 for comparison.

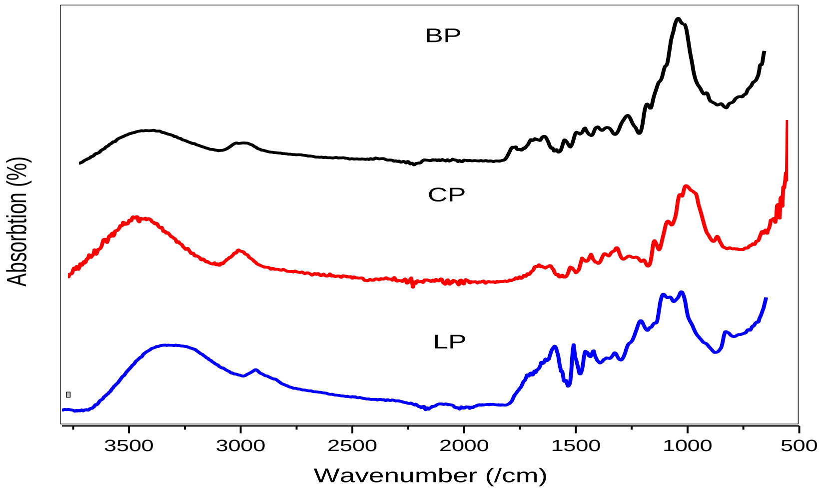

Fig. 3 FTIR spectra of bamboo, cellulose and lignin particles (BP, CP and LP)

Table 1. **Functional groups in bamboo, cellulose and lignin particles (BP, CP and LP)**

Functional groups	BP	CP	LP
		Wavenumber (/cm)	
OH stretching	3364,3273	3361	3339
C=H stretching	2919	2919	2910
Benzene ring stretching (C=C vibration)	1591	-	1432, 1510
OH stretching of absorbed water	1644	1639	1600
CH_2CH_3	1485	1449	1456
C–H stretching in cellulose, and hemicellulose	1341	1369, 1318	-
Syringyl ring (OH phenolic)	-	1213	1323
Ether	-	-	1213
Vibration of C-O-C in cellulose and hemicellulose	1163	1152	-
Secondary alcohol and aliphatic ether	-	1056	1089
C-O and C-C vibrations in glycosydic linkage	-	1029	
Aromatic ring and primary alcohol	1029	-	1023
Aromatic ring	-	-	827
C-H deformation of cellulose and hemicellulose	905	892	-

3.2. Differential Scanning Calorimetry (DSC)

The DSC thermogram of BP in Fig. 4 shows the first endothermic peak at 89.7°C. This endotherm represents the temperature at which moisture content in the particles is eliminated. Lignocellulosics are generally being reported to be hydrophilic in nature, which could be attributed to the presence of OH groups in its structure (Gbenebor *et al*., 2020). The energy required for moisture evolution from the particles is calculated to be 17.12 J/g and it is equivalent to the water holding strength of BP used in this study. Decompositions at 139.3°C (ΔH =110.7J/g), 152.5°C ((ΔH =0.39J/g) and 164.4°C ((ΔH =0.2J/g) represent release of chemically combined moisture and other volatile constituents.

Evolution of cellulose, hemicellulose and lignin present in the BP occur at low energy values, which must have occurred as a result of inorganic components interactions present in the biomass. The endotherms are at 230.5, 249.6 and 263.2°C for hemicellulose, cellulose and lignin respectively. Enthalpy value for embedded lignin dissociation is 0.79J/g while that of cellulose and hemicellulose are 0.62 and 0.58J/g respectively. Complete isolation of CP increases the stability (in terms of energy) as the enthalpy required for its dissociation is 134.9J/g. Enthalpy associated with the commencement of lignin bond dissociation at 181.2°C is 481.5J/g, which is the greatest in magnitude of the three organic molecules. This implies that LP has the highest thermal stability followed by cellulose.

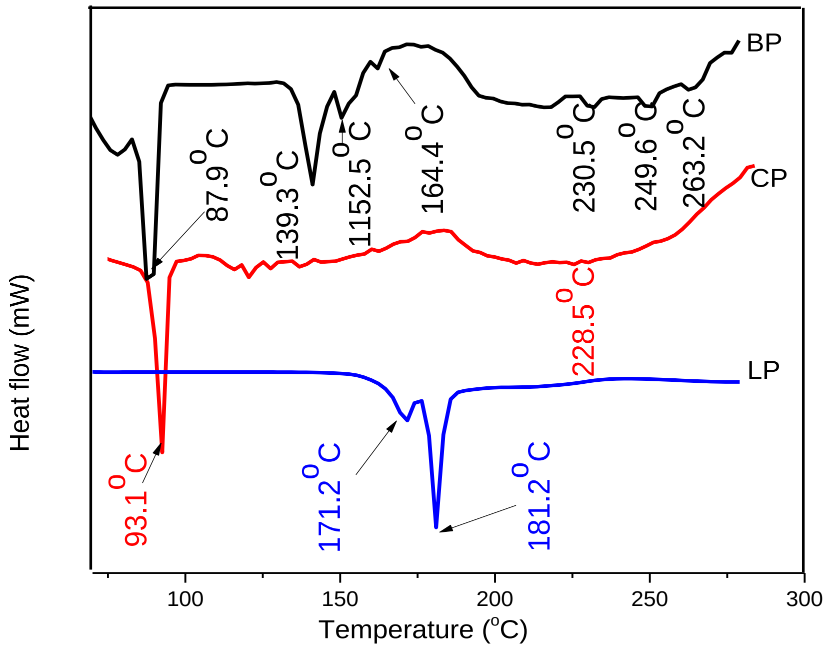

Fig. 4 DSC of bamboo, cellulose and lignin particles (BP, CP and LP)

3.3. Thermogravimetric Analysis (TGA)

Decomposition curve for BP shown in **Fig. 5** occurs in four different stages. The first occurs between 70 – 115 °C with mass loss of 0.7 % representing decomposition of water and other volatile components. Presence of hemicellulose in the particles is exemplified by the second stage of decomposition between 252 – 343°C with 29.1% content. This is comparable to the decomposition temperature of hemicellulose from corn cobs, which occurred between 200-400°C (Ma *et al.*, 2016). A second stage of decomposition was observed between 150 – 280°C in tomato and carrot wastes (Changmaia *et al.*, 2018); the investigations reported a weight loss of 7.99%, which is an indication of the hemicellulose content in the agro wastes. Result in this study also shows 0.7% weight loss of total biomass representing occurrence of cellulose and lignin in BP. This is represented by the third decomposition stage, which commences and ends at 343 and 370°C respectively. Result obtained in this study is also comparable to the observation made by Changmaia *et al.* (2018) where lignin in tomato and carrot wastes were concluded to have decomposed between 280 and 480°C. The fourth weight loss of 48.3 % is observed within the range of 370 and 474°C, which is evident by the decomposition of both lignin and cellulose. Cellulose in lignocellulosic materials decompose between 250 and 400°C (Yeng *et al.*, 2015). Thermogravimetric analysis for lignin shows two major stages of decomposition. Moisture content (11.08%) represents the first decomposition stage while lignin realization from the bamboo particle is observed within 284.- 501°C and showing 71.32% content.

This implies that a thorough chemical treatment imposed on bamboo particles will propitiate a higher yield of lignin. Comparing the mixture of cellulose with lignin contents (48.3%) in the untreated sample to the fully extracted one, there is 646% increase in content realized after extraction as 79.13% cellulose content is recorded between 271 and 381^{o}C. In this study, it is noticed that higher content of polysaccharides can be sourced from lignocellulosics after adequate chemical treatments. Aromatic rings with various branches present in lignin's structure could be responsible for its highest magnitude of Tonset (284.6 ^{o}C) during thermal decomposition. Second to lignin in terms of temperature at which decomposition commences is cellulose (271.1^{o}C). Absence of long and side chains in its structure coupled with glycosidic bond strength (which is lower compared to the strength required in breaking the aromatic rings) could be responsible for this observation.

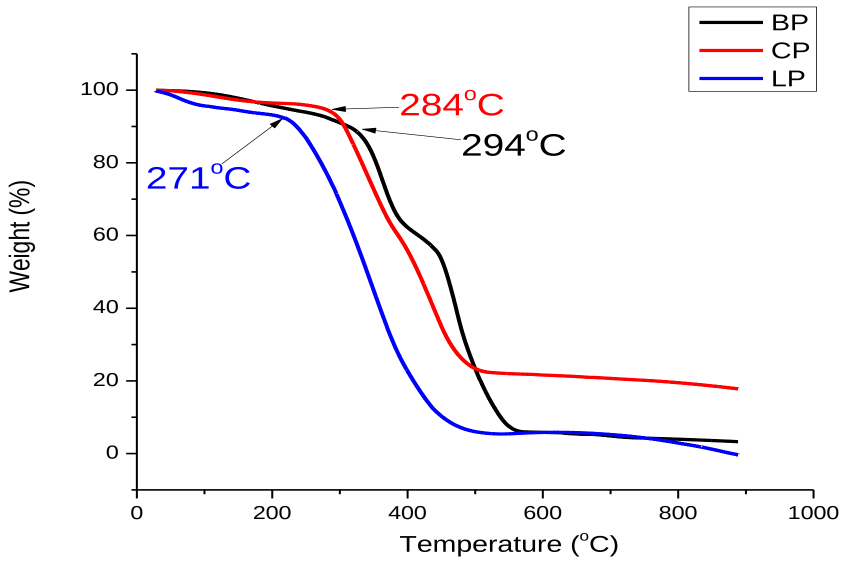

Fig. 5 TGA of bamboo, cellulose and lignin particles (BP, CP and LP)

3.4 X-Ray Diffraction (XRD)

The diffractogram of BP in Fig. 6 shows the presence of cellulose at 2θ = 22 and 35^{o} corresponding to (200) and (004) crystallographic planes. A broad band between 2 θ = 38 and 58^{o} displays the amorphous region of hemicellulose and lignin. This is comparable to the XRD pattern of Albezia pod particles where hemicellulose and lignin were confirmed to exist on 2θ = 46^{o} (Gbenebor *et al.* 2020). The CP sample shows strong peaks at 2θ =16.8 (110) and 22.7^{0} (200) while a minor diffraction on (004) plane is observed at 2θ =34.7^{0}. This is comparable to the works of Khenblouche *et al*. (2019) where peaks at 15.14, 16.25, 22.75 and 34.39^{0} were noticed when Retama raetam stems were characterized. The crystallinity of cellulose extracted from BP in this study stood at 75%. The XRD patterns of lignin has a major peak at 2θ = 22.6^{0}. This peak is not as narrow and sharp as that of cellulose peak that also appears on (002) with 48.9% crystallinity.

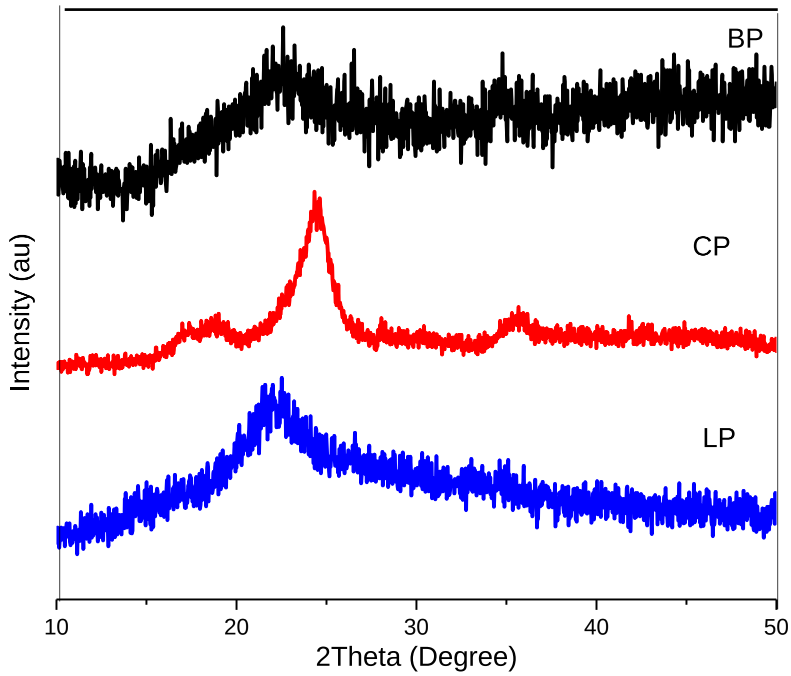

Fig. 6 XRD of bamboo, cellulose and lignin particles (BP, CP and LP)

CONTRIBUTION TO KNOWLEDGE

This study has established bamboo tree, which is abundant in the southern part of Nigeria, as a sustainable material for the processing of cellulose and lignin.

CONCLUSION

Cellulose and lignin particles (CP and LP) have been sourced chemically from bamboo in this study. The method employed informs that with the use of NaOH on the biomass, cellulose is first obtained as the residue while dissolved lignin is isolated from the filtrate (which contains hemicellulose and lignin) via precipitation with NaOH/HCl/ethanol solution. Results from the characterizations show that the biopolymers were successfully isolated as their functional groups, crystalline phases and thermal characteristics are comparable to conventional cellulose and lignin properties.

CONFLICT OF INTEREST

The research work is original and there is no conflict of interest.

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