ORIGINAL RESEARCH ARTICLE

Potentials of microcrystalline cellulose prepared from wood dusts wastes of *Ficus Platyphyla, Planatus Occidentalis* and *Gmelina Aborea*

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Abstract

This research aims to prepare microcrystalline cellulose (MCCs) from native cellulosic wastes in order to find potential applications for each of the tree species. The physico-chemical characteristics of the MCCs were studied using physical and spectroscopic techniques. Acid hydrolysis in 2M HCl was used for the preparation of microcrystalline cellulose (MCC) obtained from agricultural waste sample, *Gmelina aborea (GA) Ficus platyphyla* (FP) and *Planatus occidentalis* (PO) wood dust. The MCCs obtained were off-white and powdery in appearance. The yield of MCCs were 67.55% for *Ficus platyphyla*, 77.00% *Planatus occidentalis* and 80% for *Gmelina Aborea* (GA). The functional groups in the MCC samples were confirmed by the Fourier transform infrared (FTIR) spectroscopic method with characteristic absorption bands of ;–OH stretching at 3416 cm^{-1}; C-H stretching at 2918 cm^{-1}; -OH bending at1377 cm^{-1};, 1159 cm^{-1}; and C-O-C pyranose ring skeletal vibrations at 1026-1033 cm^{-1}, with crystallinity absorption bands showing up at 1432 and 850 cm^{-1} respectively. The thermal stabilities were determined from Thermogravimetric Analysis (TGA) and showed that the MCC samples are thermally stable (50% weight loss at **450^{O}C** or PO, 50% weight loss **470^{O}C** for FP and 50% weight loss at **590^{O}C** for GA). The characteristic morphological features were established by scanning electron micrograph (SEM) and the crystallinity of the microcrystalline cellulose were further confirmed using the X-Ray Diffraction (X-RD) technique which showed three main reflections at 2θ=14.70°, 22.09° and 34.24°, this therefore indicates microcrystalline cellulose were cellulose I type and that acid pretreatment did not affect the structure of the MCC. The crystallinity index values were 69.4, 68.7 and 79.6 for FP, PO and GM MCCs and respectively. The Samples were tested for pH, Moisture content, Hydration and swelling capacities as well. These results showed that the wood dusts form the tree species are good potential sources of high-grade cellulose which can serve as useful starting materials for further processing and applications.

Keywords: *Cellulose, Microcrystalline cellulose, Physico-Chemical and Spectroscopic Characterizations*

Introduction

One of the major sources of pollution in the environment especially in the developing countries is the agricultural wastes. In order to minimize or control the negative impact of these wastes on human and animals, turning them into finished product that would serve as a source of income to man which in turn would reduce the rate at which the wastes liters the environment, is very crucial.(1). Several attempts such as their usage as sources of activated carbon for high

performance capacitors(2), composting for multiple agricultural sources(3), Enzymes (4), applications in bio medicals(5), Pharmaceuticals(6), Cosmetics and food industries (7) have been made into their conversion to useful materials. Most of these agricultural wastes are either by-product of forestry, agricultural crops, animals, microorganisms, as well as domestic and industrial practices such as food processing, wood and rice milling.(8). Examples of these agricultural wastes include, rice husk, corncobs, wood dusts, cow and chicken dungs, kitchen wastes(9), the numbers are numerous. It is worthy of note, that most of these wastes especially the plant and micro-organism-based ones are composed mainly of cellulose (9).

Cellulose is one of the most abundant and most useful polymers on earth owing to their availability in reasonable percentages in plant and **some** micro-organisms such as the oocyte bacteria. It's been estimated that cotton which contains the purest and largest percentage of cellulose has close to 99% cellulose composition. Hard wood contains about 50%, soft wood about 45% while other plant parts can contain as much as 30%. Usually, celluloses are found in combination with other components such as the Lignin and Hemicelluloses (9).

Cellulose and its numerous derivatives have been used for several applications due to the special qualities which includes, biocompatibility, good mechanical properties, high thermal properties, low density as well very good tunable properties (10). The amorphous part of cellulose can be broken down by hydrolysis to obtain Micro or Nano crystalline form of cellulose depending on the duration of hydrolysis to obtain cellulose materials with higher thermal properties due to their well-ordered crystalline nature. The importance of cellulose in its various forms cannot be overemphasized as it has found its way into all areas of life and it is being projected to replace most of the non-biodegradable petroleum feed stocks in the future.(11). The practice of processing cellulose from **agricultural** or industrial wastes into useful raw materials can be summarized as " **Taking abandoned materials from nature, processing it, and giving it back to nature in a better form for the benefit of man**". This work therefore seeks to contribute to the reduction of wastes in the environment by focusing on the treatment of cellulosic wastes and converting them into useful raw materials for better applications.

Methods

Materials, Reagents And Solvents

Materials used in this research is the Native cellulosic wastes. Solvents and Reagents were purchased from Central Drug House (CDH) Mumbai, India are were of analytical grade and used without further purification. They include: 98% Ethanol, distilled water, potassium bromide (KBr), Sublimated Iodine (I_2), Potassium Hydroxide (KOH), Sodium chlorite (NaOCl), Hydrochloric acid (HCl) and Acetic acid (CH_3COOH).

Sample Collection

The Cellulosic wastes were collected at a wood milling factory in Kwara state, North central region of Nigeria. The tree species were identified by the factory workers and documented immediately at the site of collection in their local names. Their botanical names were subsequently sourced from the internet and confirmed at the **herbarium** of the University of Ilorin, Ilorin, Nigeria.

Extraction and Purification of Alpha Cellulose

The method employed by Arowona *et al.*, 2018 **(12)** was adopted in the isolation of cellulose as well as the preparation of MCCs with slight modifications. In this method;

A 100 g of the sample was weighed and transferred into a 2L quick fit flask containing a 1L solution of 200 g sodium chlorite adjusted to a pH 4.0 using 10% acetic acid and stirred under

reflux at 75^{O}C for 3 *h*. The resulting solution was filtered, the residue was washed with distilled water and ethanol **(98%)** until its pH became neutral, and then dried in an oven at 60^{O}C for 5 *h*. Afterwards, the dried residue was treated with 10% potassium hydroxide for 6 *h* to complete the delignification process. The cellulose obtained was washed with distilled water and ethanol and dried for 3 *h* at 60^{O}C. Thereafter, it was treated with Sodium hypochlorite, washed with distilled water and ethanol and dried in an oven at 80^{O}C for 6 *h* to get rid of excess water that might be trapped in the cellulose backbone. The cellulose was blended to obtain a smooth powdery texture, weighed and stored for further treatment.

Preparation of Microcrystalline Cellulose

Known quantity of alpha cellulose obtained above was transferred into a quick fit flask and 500 mL of 2 M HCl was added and then refluxed at 60^{O}C for 3 *h*. The microcrystalline cellulose obtained was then washed with distilled water and ethanol, dried in the oven, blended, weighed and then stored for further characterization.

Starch Test

A 0.1g amount of each **sample** were placed in 50mLs beaker, about 5mLs of iodine solution was added to each sample. For comparison**,** 0.1g of starch was also placed in separate 50mLs beaker and the same quantity of iodine solution was added. Absence of blue-black coloration means positive result **for cellulose**.

ph Determination

Exactly **0.2g** each of the samples **was** placed in a 10ml clean measuring cylinder. Few mLs of distilled water was added to the samples, shaken together and the volume of the sample and water were made up to 10 **mLs.** The mixture was left to settle and the pH of the supernatant liquid were taken with a digital pH meter.

Moisture Content

About 1g each of the samples were placed independently in a white porcelain crucible with cover. The samples were dried in an oven for 3hrs at 105ºC. During drying, the weight of the samples was taken at 30 min intervals until constant weight was obtained. The moisture content was calculated using the equation proposed by Kharismi *et. al*, 2018:(7)

$$\frac{\{(\boldsymbol{A}-\boldsymbol{B})\}}{\boldsymbol{A}} * \mathbf{100} - - - - - - - - - - (\mathbf{1})$$

A= initial weight of MCC, B= final weight of MCC after drying

Swelling Capacity (Sc)

A desired amount of the samples were placed in a measuring cylinder of known volume. The measuring cylinder was tapped until there was no further increase in volume. This volume was noted as V**t** . A dispersion of the powder was made by adding water up to the highest mark on the cylinder. The measuring cylinder was left undisturbed for 24hrs and the volume of the sample sediment was noted as V**s**. The swelling capacity, Sc. was calculated using the equation:

$$\boldsymbol{Sc} = \frac{\boldsymbol{Vs}-\boldsymbol{Vt}}{\boldsymbol{Vt}} - - - - - - - - - (\mathbf{2})$$

Sc. = Swelling Capacity; Vs = Volume after Swelling; Vt = Tapped volume

Fourier Transform Infrared Spectroscopy (Ftir)

The FT-IR spectroscopy was used to identify the chemical composition of each sample by defining the functional groups. To achieve this, **a 25mg** sample of dried cellulose and microcrystalline

cellulose (MCC) samples were mixed with potassium bromide (1:90) and compressed into transparent tablets using a hydraulic press (M-15, Technosearch) to enable electromagnetic radiation to pass through easily. Then, in the range of 4000–400 cm^{-1}, the FT-IR machine, (ALPHA-II, Bruker, Germany) was used to analyze the transparent tablets.

Scanning Electron Microscopy (Sem)

Surface morphology of the cellulose fibers and metal oxides composites was investigated using Phenom ProX Scanning Electron Microscopy, USA. Before the analysis, the composites were sputtered with thin gold layer to avoid electrostatic charging during examination. The micrographs with a magnification of 500 times were obtained by back scattered electron detector (BSE) in order to register both topography and compositional contrast.

Thermogravimetric Analysis/ Differential Thermal Analysis (Tga/Dta)

The TGA machine, STA449 F3, Netzsch, Germany was used to determine the degradation temperature and weight loss (TGA and DTA) of the samples under a nitrogen atmosphere (40 mL/min), while heating at 10 °C/min from 25 °C to 800 °C. The weight loss (%) was evaluated by measuring the residual weight at 800 °C.

X-Ray Diffraction (Xrd) Spectroscopy

The degree of crystallinity of the MCC samples were determined using X-Ray Diffractometer machine **(PANalytical, X'pert PRO, Netherlands)** powered by a 40 kilovolt X-Ray generator at an input of 30Ma with Cu K alpha radiation.

Results and Discussion

Appearance

The cellulose obtained were white to off white in color with smooth to rough appearance while the microcrystalline cellulose obtained were smooth white to off white fine powder. The appearances of the cellulose samples are due to the presence of both the amorphous and crystalline region while MCC samples appeared smooth due to the breakdown of the amorphous region of cellulose leaving behind fine powdery crystalline region.

Starch

There was an immediate change of colour of the starch sample to deep black while the colours of the MCC samples remained the same (brown) in iodine solution. Iodine coloration disappeared from the cellulose samples after about 20 min while the blue-black coloration of the starch sample persisted. This result show that starch was completely absent in the samples.

Figure 1a: Appearance of cellulose and starch in iodine solution

Figure 1b: Disappearance of Iodine from cellulose solution after about 30mins

pH, Moisture Content and Swelling Capacity

The pH is the measure of neutrality, acidity or basicity of samples. The pH of pure MCCs should fall within the standard values of 6-7.5 (13). The moisture content indicates the amount of water the MCCs are able to absorb from the atmosphere. The lesser the moisture content, the better is the suitability of the MCC to be used as excipient. The swelling capacities of a sample is the increase in volume of water taken up by sample after absorption. The values obtained in this research falls within the standard range of less than 7% as reported by Kharismi and Suryadi (7). The results of the pH for the samples showed that they are neutral and falls within the 6-7.5 standard acceptable limit values for pH of neutral MCCs (13). Comparing the results of the three different samples, *Gmelina aborea* MCC had the highest pH value and this is connected to its highest water holding capability which was evident in its moisture content and swelling capacity values as compared to others. Therefore, pH of MCC samples can be said to be affected by the amount of water held in the sample provided there are no anionic or cationic materials embedded in the molecule backbone.

Table 1: Table showing the pH, Moisture content and Swelling Capacities of the MCCs

S/No	SAMPLE	pH values	Moisture content	Swelling capacity
1.	Gmelina	6.98	11.70	122.22
2.	Ficus	6.53	03.00	027.8
3.	Planatus	6.17	08.40	045.45

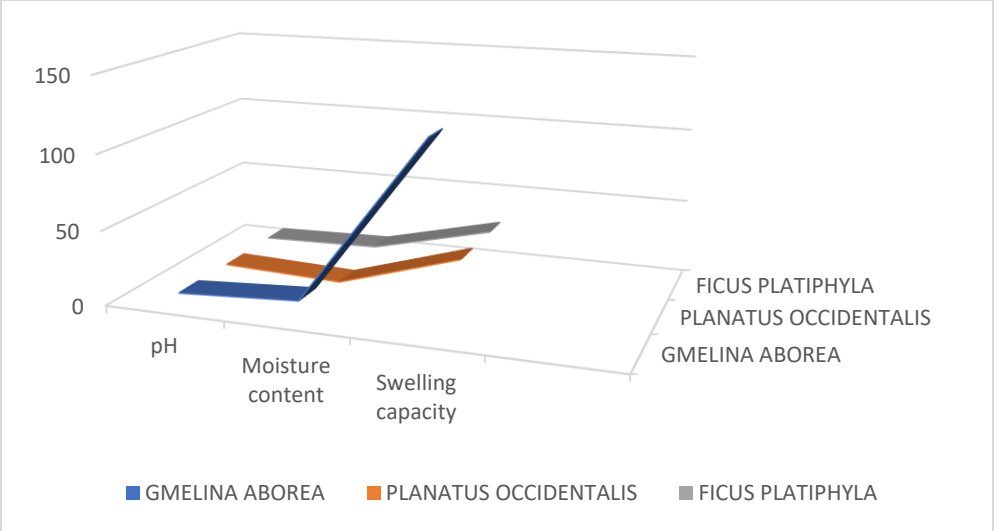


Figure 2: Relationship between pH, Moisture content and Swelling capacities of MCC samples

Fourier Transform Infrared Spectroscopy

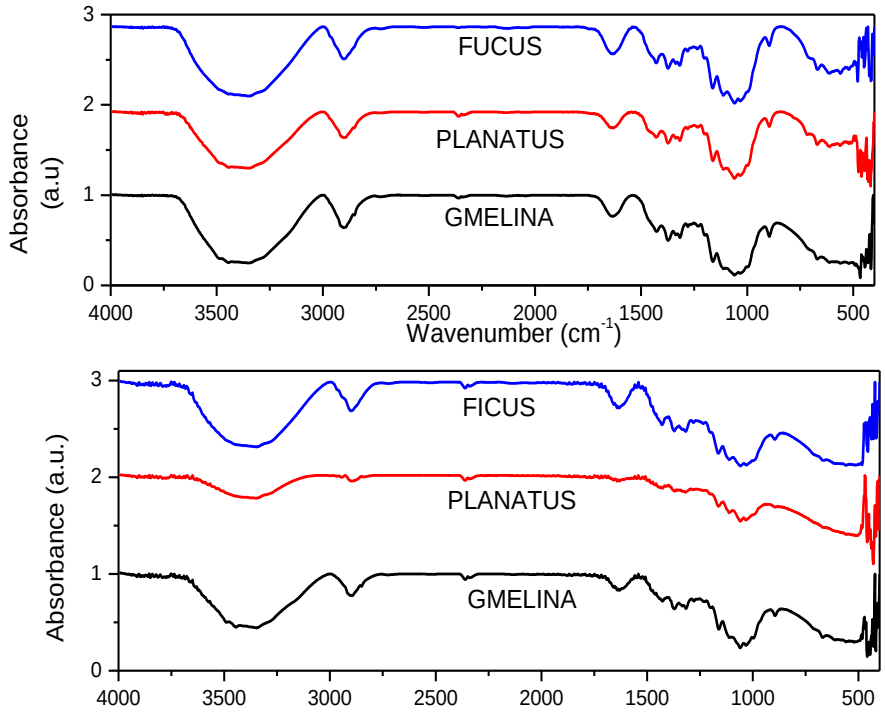
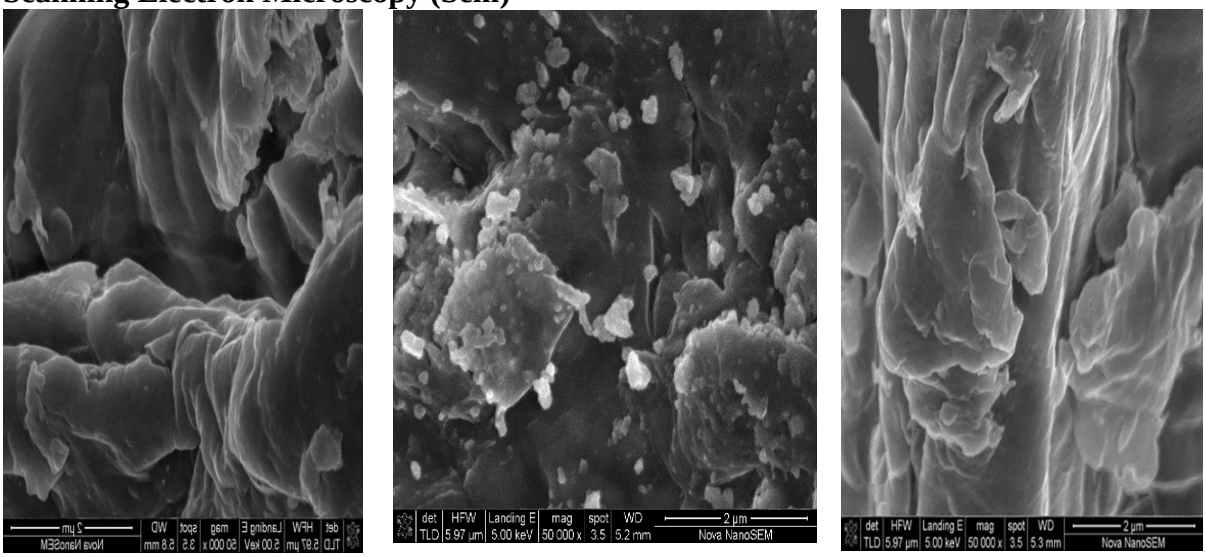


Figure 3: FTIR Spectra of Cellulose and Microcrystalline Cellulose from Different Wood Dusts

In the spectra above, the FT-IR spectra of cellulose and MCC samples are shown (A and B). The absorption bands at around 3300,2900, 1430, 1374, 1100, 1050, 890 cm^{-1} are all associated with native cellulose(14). The stretching of O-H groups and aliphatic saturated C-H are responsible for absorbance peaks in the ranges of 3450–3300 cm^{-1} and 2900–2800 cm^{-1}(14–16). The C-O-C pyranose ring skeletal vibrations are responsible for the peaks that appeared at about 1050cm^{-1}. The absorption bands at about 1374 cm^{-1} are responsible for the C-O asymmetric bridge stretching.(14). Furthermore, the spectra absorption peak at 1429 cm^{-1}, which is due to a symmetric CH_2 bending vibration and is known as the "crystallinity band" (17,18). The crystallinity of the sample is further proven with the absorption bands at 897 cm^{-1} (19,20). All these regions found in all the spectra, suggested that the cellulosic compositions are identical. In MCC, there was an improvement in strength at the 897 cm^{-1} absorbance peak, suggesting improved crystallinity, this was also observed by Zhao *et al.* 2018(15). The absorption peak at 1624 $cm^{-1}$is responsible for water absorption due to the hydrophilic nature of cellulosic material (5). The absence of peaks at 1512 cm^{-1} and 1735 cm^{-1} which are assigned to the C-C vibration as well as the aromatic C-O stretching in hemicelluloses and lignin respectively, suggested that the pretreatment method adopted suitably eliminated non-cellulosic component of the raw materials (18,20–22).

Scanning Electron Microscopy (Sem)



a **b** **c**

Figure 4: SEM Micrographs for MCCs of (a) Gmelina (b) Ficus (c) Planatus

The SEM images of the microcrystalline cellulose samples show non- uniformly dense microcrystalline particles thereby forming microcrystals from the overall view. They are densely packed and sponge-like revealing large surface area of the samples. This appearance is due to the breakdown of the amorphous region during hydrolysis affording the microcrystalline structure with large surface area. All three samples displayed similar surface area pattern showing cellulose from each of these samples could be employed as raw starting materials for further processing.

TGA/DTA

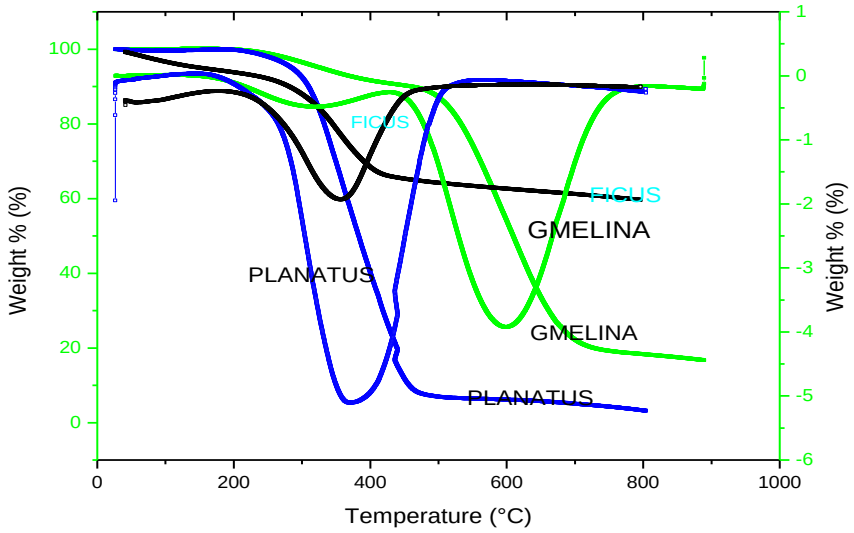


Figure 5: TGA/DTA thermogram of the MCCs

The thermogravimetric curve revealed a single step thermal degradation pattern **(Figure 5)** of the samples from 430 to 550 ^{o}C . Gmelina MCC however showed higher thermal stability with degradation temperature of about 700 ^{o}C. which might be attributed to its high swelling capacity. Overall, the thermal degradation pattern exhibited by the samples confirmed the absence of hemicelluloses, lignin as well as little or no traces of impurities in its core structure. Therefore, the MCCs are thermally stable ones. The Differential thermal analysis (DTA) revealed that the Tmax (temperature at which maximum weight loss occurs) was 440 ^{o}C as an average for the samples except for Gmelina with Tmax at about 610^{o}C. It can be said that these samples are thermally stable and can serve as good raw materials for future materials functionalization and applications.

X-Ray Powder Diffraction Specrtroscopy (Xrd)

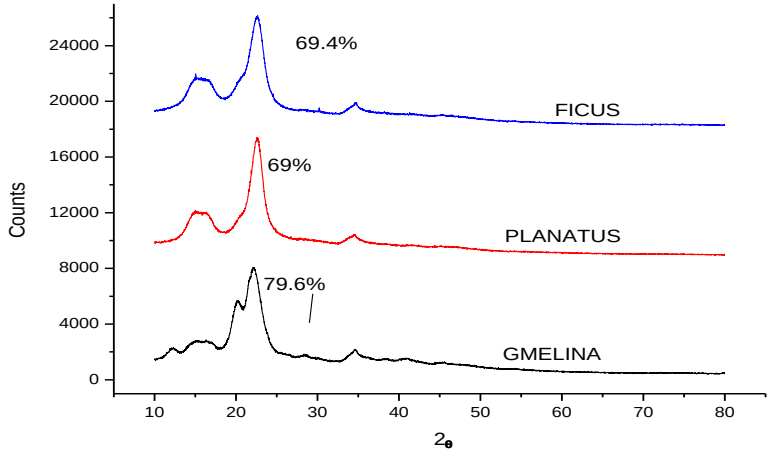


Figure 6: XRD Pattern of MCCs

The X-ray diffraction pattern of the MCC samples **(Figure 6)** revealed that *Gmelina aborea* sample had the highest value of degree of crystallinity. The crystallinity index of the samples

suggested the amorphous region of the cellulose is broken down, leaving behind the crystalline region of the cellulose material, yielding microcrystalline cellulose. The crystallinity index values of the MCC samples ranged from 68.7% to 79.6% percent which is a good value for microcrystalline cellulose. The diffraction patterns of all the MCCs samples showed sharp peaks of the 2θ angles at about 14.5°,17°, 22.7°, and 35.5° for all the samples are assigned to the typical reflection planes of Cellulose I. The values obtained are identical to those reported by Hu *et. al* and Rahman *et. al* for carbon nanocellulose and microcrystalline cellulose respectively.(9,23–25) The crystallinity indices of the samples are shown in the bar chart below for better comparison. The origin soft ware was used to calculate the areas of crystalline regions as well as the areas of the crystalline and the amorphous region of the XRD spectra. The crystallinity index of each sample was thereafter calculated using the equation below:

$$\boldsymbol{CI = \left\{\frac{Ic}{I(c+a)}\right\} * 100 - - - - - - - - (3)}$$

CI= crystallinity index
Ic= Area of Crystalline region
I(c+a)= Area of crystalline and amorphous region.

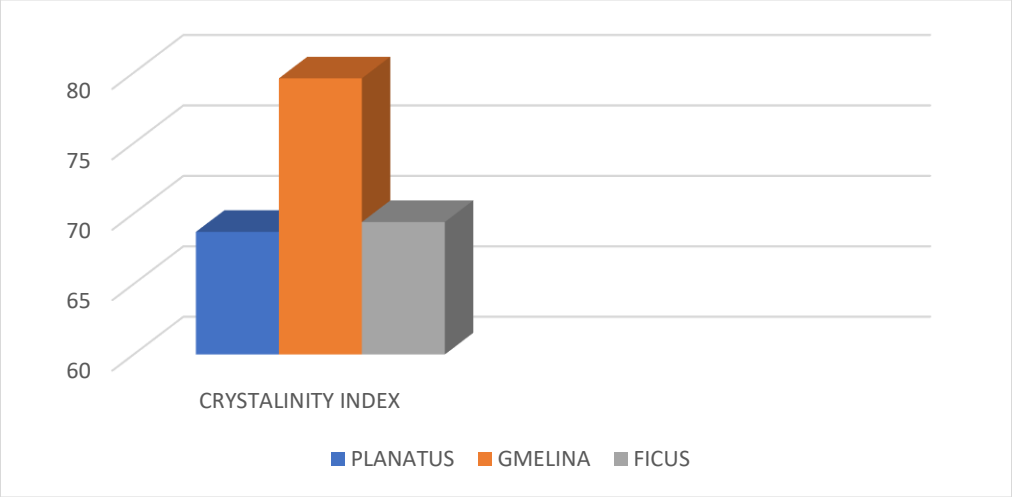


Figure 7: Bar Chart Illustration of the C.I of the three samples

Conclusion

In this study, pure, high-grade cellulose were successfully extracted from rejected wood dusts wastes from sawmills. Microcrystalline cellulose samples were subsequently prepared from the extracted cellulose and characterized. The results from this study revealed that quality raw materials that can be employed for several applications could be obtained from abandoned materials in the environment. This practice will not only help in eliminating wastes from the environment, but will also serve as source of revenue generation if practiced on a commercial scale. It can therefore, be concluded that the Gmelina microcrystalline cellulose sample due to its good swelling property, can be grafted onto other polymer samples like starch, chitin, polyvinyl pyrolidone, Polyethylene glycol, Polymethylmetacrylate and a host of others to obtain cationic or anionic hydrogels depending on the dissolution solvent for enhanced applications in pharmaceutical, biomedical as well as the agricultural fields. The Ficus and Planatus samples due to their moderate swelling ability are good candidates as direct **compression** excipient in drug formulation. Overall, 'waste materials were obtained from nature, purified and processed into

useful raw materials that are ecofriendly for several applications which will serve **beneficially** to human'.

Conflict of Interest

The authors of this research hereby declare no conflict of interest in this work

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