



Physical and Spectroscopic Characterization of the Microcrystalline Cellulose Derivatives from Corn Cob and *Daniella Oliveri* Wastes

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Abstract: Cellulose was extracted from wood dust waste samples of *Daniella oliveri* and corn cobs by acetic acid and alkaline pretreatment methods, while microcrystalline cellulose (MCC) derivative was produced by acid hydrolysis in 2 M HCl. The samples were tested for pH, moisture content, swelling capacities and ash contents. The data obtained were compared with those of commercial MCCs found in the literature. The functional groups in the microcrystalline cellulose derivatives was 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 at 1377 cm⁻¹; 1159 cm⁻¹; and C-O-C pyranose ring skeletal vibrations at 1026-1033 cm⁻¹. The crystallinity absorption bands appeared at 1436 and 850 cm⁻¹. The characteristic morphological features were established by scanning electron microscopy (SEM). Furthermore, the crystallinity of the microcrystalline cellulose was further confirmed using the X-ray powder diffraction (X-RD) technique, which showed three main reflections at 2θ=14.70°, 22.09°, and 34.24°. These results supported that microcrystalline cellulose derivative as cellulose I type and the acid pretreatment did not affect the structure of the MCC. The crystallinity indices were 69.3 and 73.2%, respectively. *Daniella Oliveri* and corn cob microcrystalline cellulose are, therefore, potential materials for further processing.

Keywords: Microcrystalline cellulose derivative, *Daniella oliveri*, corn cobs, wood dust waste, FTIR, XRD, TGA/DTA, SEM, physical characteristics, potential applications

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1. INTRODUCTION

Pollution is a pronounced concern in our community/environment as a result of poor waste management and disposal. The majority of these wastes are either piled up as an eyesore or left to fester, putting human and animal life at risk. It is worth noting that the majority of these wastes are cellulose-rich agricultural byproducts, including maize cobs, rice husks, and wood dust, from a variety of tree species. The production of agricultural byproducts is projected to be approximately 12 million tons per year, with the majority of them not being converted into other materials for future use (1). Cellulose is a polymer of D-glucose linked by the -1,4-D-glucopyranose

ring (2). It is made up of hundreds to thousands of monomers of glucose that are linked together. An anhydroglucose unit of cellulose consists of two glucose units linked together by a -1,4-glycosidic bond and weighs 162.1 gmol⁻¹ (3). Cellulose and its derivatives have important applications in a variety of fields, including pharmaceuticals, biomedical, paint industries, water purification, and cosmetics (1,4-6). Microcrystalline cellulose (MCC) is a partially depolymerized cellulose in which the amorphous region of cellulose is hydrolyzed to produce crystalline cellulose with a larger surface area for better application (7-9). MCCs, like cellulose, are white, colourless, and odourless but have a larger surface area and a more ordered crystalline structure.

Corn is cultivated all over the world and consumed daily by man and animal in different forms. Knoema estimated the global corn cultivation to be 3.13 million thousand tonnes in 1971 alone, with an annual 3.06% increase, the production rise to 1.162 billion tonnes in 2020 (10). It is estimated that the ratio of corn grains to corncob is 100:18 (11). From the figures quoted earlier, this implies annual corn cob generation of corncob in 2020 alone is approximately 210 million tonnes. Corncobs find application in their uses as poultry feed formulation, polisher, bio-oil and biochar. The efficacy of corncob as alternative source of microcrystalline cellulose in pharmaceutical formulation has also been investigated (12,13). Apart from the applications stated above, several reports are available on the use of corncob as a very good source of compounds such as furfural, ethanol, xylitol, succinic and propionic acid, phenolic compounds, bio-adsorbent, biobased rigid polyurethane foam, hollow spherical carbon, and p-hydroxycinnamic esters (1,14,15).

Daniellia oliveri (DAN) is a soft wood deciduous mostly cultivated in the tropical region of Africa (16). This plant is basically cultivated for medicinal and economic benefits; the young leaves of this plants are cooked and consumed (17), the matured are used for the treatment of stomach troubles, diabetes, dysmenorrhoea, haemorrhoids due to their abortifacient, aphrodisiac, astringent, diuretic, emmenagogue and stomachic properties (18). The seeds are eaten (19), decoction from the roots are used in the treatment of gonorrhoea and skin diseases (20). The gum-resin from the stem-bark and wood is used in the treatment of gonorrhoea and laxative and also used for the manufacture of wood polish, perfumes and varnishes (21). The gum-resin obtained from the wood of this plant has also been used as binder in pharmaceutical formulation (22). An aqueous extract of the powdered bark has been shown to have effective pain-reduction properties and also to be antioxidant. The trunk are sold as timber for furniture and manufacture of equipment (23).

Characterization and use of the cellulosic content of the DAN. plant have not been widely studied in literature, to the best of the author's knowledge. Cellulose however, can be obtained from different part of the plant: For example, the wood dusts which is a waste or by product of milling the timber obtained from the DAN. for production of furniture and other tools are good source of cellulose for extraction and characterization. Some of the physical and spectroscopic characteristics of corncob MCC in comparison to that obtained from DAN, another plant specie is not widely investigated for its cellulosic content. This research therefore, reports some of the physical and spectroscopic properties of MCCs produced from *Daniellia oliveri*'s cellulosic waste and corncobs. This broadens the

horizon of cellulose sources and applications because, it is expected to replace toxic and non-biodegradable raw materials in the near future.

2. MATERIALS AND METHODS

2.1. Sample Collection and Preparation

Corncob (C.C) wastes lying fallow in the environment were randomly picked in the north-central part of Nigeria during the maize harvest season. This was to help clean up the litters from the environment. Wood dust of DAN tree species was obtained from a local wood milling factory environment in Kwara state, north-central, Nigeria. The local name for the *Daniella oliveri* sample was identified at the point of collection by its local name, while the botanical name was obtained through herbarium of University of Ilorin. Both samples (corn cob and *Daniella oliveri* wood dust) were sun dried to remove excess water for easy grinding and further processing.

2.2. Extraction of Cellulose

Cellulose I was extracted by the chemical pretreatment method, while Cellulose II was obtained by acid hydrolysis in 2 M HCl. The method of cellulose extraction used in our previous publication (24), we employed a slight modification. 500 g of DA or Corn cob was weighed into a quick fit flask, and 10% sodium hydroxide was added. It was then refluxed for four hours. The mixture was left to cool. It was then filtered and washed with water until neutral, washed with ethanol and dried. The dried residue was poured into a beaker, 10% acetic acid was added, and the beaker was heated for three hours. It was then filtered and washed with water and ethanol and dried. Hydrogen peroxide was added to the sample, and it was refluxed for three hours to further delignify. It was filtered and washed with water and ethanol. Hypochlorite was added and refluxed for three hours (until it turned white/off-white). The sample was thereafter washed with water and ethanol, dried, and blended into finer particles and stored for further use.

2.3. Preparation of MCC

MCC was prepared by acid hydrolysis of the cellulose samples obtained above in 2 M HCl under reflux in a 500 mL quick-fit flask. The reaction was carried out at 80 °C for 4 h with a continuous stirring speed of 700 rpm. The reaction mixture was cooled to room temperature and filtered, and the residue was repeatedly washed with distilled water until it was free from acid residue by testing the pH of the solution with a digital pH meter. The obtained MCC was dried in a hot air oven at 80 °C until a constant weight was achieved. Using the dry blender compartment, the dried MCC lumped together was ground into a fine powder.

2.4. Percentage Yield

The percentage yield of cellulose was calculated by dividing the final weight of cellulose obtained by the initial weight of the cellulosic sample. The percentage yield of both cellulose and MCC was obtained using the expression below;

$$Y_{cell} = \frac{Fw_{cell}}{Cw_{cell} * 100} \quad (1)$$

$$Y_{mcc} = \frac{Wh}{Fw_{cell} * 100} \quad (2)$$

Y_{cell} = Percentage Yield of cellulose;
Fw_{cell} = Weight of cellulose extracted;
Cw_{cell} = Weight of crude cellulose
Y_{mcc} = Percentage Yield of MCC;
Wh = Weight of Hydrolyzed cellulose;

2.5. Starch Test

The presence of starch in the sample was investigated by adding approximately 3 mL of iodine solution to 0.1 g MCC samples in a beaker. For comparison, the same amount of iodine solution (3 mL) was added to 0.1 g of reference standard starch in another beaker. Blue-black colouration indicates the presence of starch, while no change in colour indicates the absence of starch in the cellulose backbone. The absence of blue black colouration was taken as an indication of a positive result for cellulose.

2.6. Ash Content

Each of the prepared MCCs (2 g) was placed in a furnace at 600 °C for 6 h. The ash content was determined using the following equation:

$$\left[\left(\frac{W_i - W_f}{W_i} \right) \times 100 \right] - 100 \quad (3)$$

where W_i = initial weight of the sample W_f = final weight of the sample (i.e weight of ash)

2.7. Moisture Content

Each of the samples (1 g) was placed independently in a white porcelain crucible with a cover. The samples were dried in an oven for 3 h at 105 °C. During drying, the weight of the samples was taken at 30 min intervals until a constant weight was obtained. The moisture content was calculated using the equation below. (25)

$$\left(\frac{A - B}{A} \right) * 100 \quad (4)$$

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

2.8. pH Measurement

Each of the samples (1 g) was placed in a 10 ml clean measuring cylinder, a few milliliters of distilled water was added to the samples and shaken together, and the volume of the sample and water was brought to 10 mL. The mixture was left to settle, and the pH of the supernatant liquid was measured with a digital pH meter.

2.9. Water Uptake Capacity

Distilled water (4 mL) was poured into a 10 mL beaker containing MCC samples whose bulk and tapped volume had been obtained. The mixture was agitated, and the volume of the mixture reached 10 mL. The mixtures were left to stand for 72 hrs, the swelling volume V_s was obtained, and the water uptake capacity was calculated using the equation:

$$WUC(\%) = \frac{V_s - V_t}{V_t} \times 100 \quad (5)$$

WUC (%) = Percent Water uptake capacity
V_s = volume of sample after swelling
V_t = Tapped volume of the sample

2.10. Fourier Transform Infrared Spectroscopy (FTIR)

The chemical composition of each sample was determined by determining the functional groups using FTIR spectroscopy. A few milligrammes (mg) of dried cellulose and MCC derivatives were mixed with potassium bromide (1:90) and compacted into transparent tablets using a hydraulic press (M-15, Technosearch). An FTIR spectrometer (ALPHA-II, Bruker, Germany) was utilized to investigate the transparent tablets in the range of 4000–400 cm⁻¹.

2.11. X-Ray Powder Diffraction Spectroscopy (XRD)

The degree of crystallinity of the MCC derivatives was determined using an X-ray diffractometer machine (PANalytical, Model: X'pert PRO) powered by a 40 kilovolt X-ray generator at an input of 30 Ma with Cu K alpha radiation. The crystallinity indices of the samples were calculated from the spectrum obtained using the origin software by determining the area of the crystalline region and the area of the crystallinity as well as the amorphous region and then using the equation below to determine the crystallinity index of each sample.

$$C.I = \left(\frac{I_c}{I_a + I_c} \right) \times 100 \quad (6)$$

C. I = Crystallinity index; I_c = Area of Crystalline peaks, I_a + I_c = Area of Amorphous and Crystalline peaks.

2.12. Scanning Electron Microscopy (SEM)

The degree of crystallinity of the MCC derivatives was evaluated using X-ray diffractometer equipment (PANalytical, Model: X'pert PRO) using Cu K alpha radiation and a 40 kilovolt X-ray generator at an input of 30 Ma.

2.13. Thermogravimetric and Differential Thermal Analysis (TG/DTA)

Thermogravimetric analysis (TGA) was performed on the derivatives to track the thermal degradation trend, whereas DTA was used to determine how much weight was lost at a given temperature. TGA measurements were carried out with an STA449 (F3, Netzsch, Germany) under a nitrogen atmosphere (40 mL/min), and the derivatives were heated at a rate of 10 °C/min from 50 °C to 850 °C. The remaining weight loss was determined by measuring it at 850 °C.

3. RESULTS AND DISCUSSION

3.1. Appearance

The cellulose revealed white to off-white with a rough to smooth appearance due to the presence of both crystalline and amorphous regions of the cellulose molecule. On the other hand, the MCCs prepared from the initial cellulose appeared smooth with white to off-white in colour. The smooth appearance of the MCCs is related to the breakdown of the amorphous part of the cellulose compound leaving behind the powdery crystalline region.

3.2. Percentage Yield

The percentage yields of the cellulose was calculated by dividing the weight of the cellulosic biomass before extraction by the weight of the

cellulose obtained after extraction. The percent yield of the MCCs was obtained by dividing the mass of the cellulose obtained before hydrolysis by the mass of the product obtained after hydrolysis. The percentage yields of cellulose samples were 60% for C.C and 55% for DAN. The MCC yields were 80 and 75% for C.C and DAN samples, respectively. This showed that the method of preparation of the cellulose compound gave cellulose with a good degree of crystallinity, hence leaving behind a larger amount of the crystalline region than the amorphous region after extraction. This claim was proven by the small difference in the crystallinity indices of the cellulose and MCC derivatives, which was supported in the XRD analysis. The percentage yield of the cellulose is as presented in the table 1 below:



Figure 1: Cellulose extracted (left) and prepared microcrystalline cellulose (right).

Table 1: Percent Yield of Cellulose and Microcrystalline cellulose.

| No. | Sample | Cellulose(%) | MCC(%) |
|-----|------------------|--------------|----------|
| 1. | CORN COB | 60.0±0.5 | 80.0±0.5 |
| 2. | DANIELLA OLIVERI | 56.5±0.29 | 75.0±1.0 |

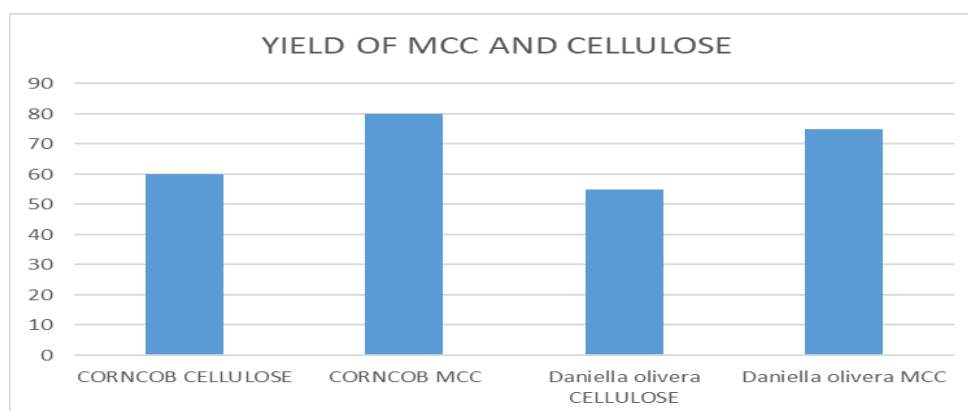


Figure 2: Bar chart representation of yields.

3.3. Starch Test

A starch test was conducted on the MCC derivatives to ascertain the absence of other carbohydrate such as starch, in the cellulose molecule. The MCC derivatives gave no colour change upon the addition of Iodine, as the reddish brown iodine remained, while starch gave blue-black colouration. The blue-black colouration of the starch was persistent, while the reddish brown colour of the iodine sublimed from the MCC solution after being kept at room temperature for 2 h. This is because, starch being a branched α -polymer of glucose, forms helical secondary structures which can hold iodine, which leads to a reaction and consequent colour change. But cellulose a linear β -polymer of glucose, does not contain helices, and so cannot hold iodine which prevents interaction, hence, no color change.

3.4. Ash Content

The percentage ash content values obtained are shown in the table 2 below, which was taken as evidence that the MCCs derivatives had very little non-cellulosic residues (25). The results were corroborated with those in the literature, with a percentage ash concentration of 1.95 for Avicel-102, a commercial excipient. This finding demonstrated that MCCs derivatives derived from these two agricultural wastes samples can compete rather well with commercially available MCCs (26,27).

Table 2: Percent Ash content of MCCs.

| No. | Samples | %Ash content (%) |
|-----|-------------------------|------------------|
| 1. | Corn Cob | 0.80±0.10 |
| 2. | <i>Daniella oliveri</i> | 1.30±0.05 |

3.5. Moisture Content

The moisture content of MCCs reveals how much water they can absorb from the environment. The lower the moisture content of the material, the better it can be utilized as a material for direct compression excipient in pharmaceutical application while the higher the moisture content (28), the better is it to be used in other applications such as hydrogel for water storage for plant (29). According to the results from this study, the values obtained are within the usual range of less than 7% reported in the literature (27). The results of this study are presented in the table 3 below:

Table 3: Percent Moisture content of the MCCs.

| No. | Samples | Moisture Content (%) |
|-----|-------------------------|----------------------|
| 1. | Corn cob | 1.80±0.12 |
| 2. | <i>Daniella oliveri</i> | 10.10±1.0 |

3.6. pH Determination

The pH of the derivatives was found to be neutral, falling within the standard acceptable limit values

for neutral MCCs of pH 6-7.5 (30). Table 4 below summarizes the findings:

Table 4: pH Values of the MCCs.

| No. | Samples | pH |
|-----|-------------------------|-----------|
| 1. | Corn Cob | 6.56±0.02 |
| 2. | <i>Daniella oliveri</i> | 6.3±0.01 |

3.7. Water Uptake Capacity

The increase in the volume of water taken up by the MCC derivatives following absorption was measured by the water uptake capacity, which measures the ability of the sample to swell. The derivatives have a moderate to high swelling ability, according to the results. The values for the swelling ability of the MCC derivatives are shown in the table 6 below, and these values favourably agreed with those reported in the literature (26). The values obtained for the two samples showed the corn has a better ability to take up more water into its cellulosic backbone than the *DAN samples* but the moisture contents value suggests the DAN sample takes up moisture easily from the atmosphere than the corncob. This phenomenon might be due to difference in their cellulosic origin which might make cellulosic sample to absorb moisture easily but releases it easily as well due smaller surface area. This can be explained by the SEM appearance of the MCC samples showing *DAN* with longer strands than the C.C with shorter agglomerates due to larger surface area and higher crystallinity as evidenced in the crystallinity indices values of the materials.

Table 5: Water uptake Capacity of the MCCs.

| No. | Samples | WUC (%) |
|-----|-------------------------|-------------|
| 1. | Corn Cob | 115.00±0.58 |
| 2. | <i>Daniella oliveri</i> | 78.60±2.00 |

3.8. Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR spectra of cellulose and MCC derivatives are presented in the figure 3. Native cellulose affords absorption bands at approximately 3300, 2900, 1430, 1374, 1100, 1050, and 890 cm^{-1} Liu, 2007. Absorbance peaks in the regions of 3450–3300 cm^{-1} and 2900–2800 cm^{-1} are caused by stretching of O-H groups and saturated aliphatic C-H, respectively. The skeletal vibrations of the C-O-C pyranose ring are responsible for the peaks that occurred at approximately 1050 cm^{-1} (8,31,32). The C-O asymmetric bridge stretching was caused by absorption bands at approximately 1374 cm^{-1} . Furthermore, the absorption peak at 1429 cm^{-1} , due to a symmetric CH_2 bending vibration and referred to as the "crystallinity band," due to a symmetric CH_2 bending vibration (33). Because of the hydrophilic nature of cellulosic material, the absorption peak at 1624 cm^{-1} is responsible for water absorption (34). The absence of peaks at

1512 cm^{-1} and 1735 cm^{-1} , which correspond to the C-C aromatic bending and aromatic C-O stretching in hemicelluloses and lignin, respectively, indicated that the pretreatment procedure effectively removed noncellulosic components of the raw materials (35–38).

3.9. X-Ray Powder Diffraction (XRD)

The crystallinity indices of the derivatives indicated that the amorphous cellulose region was broken down, leaving the crystalline cellulose material behind, generating microcrystalline cellulose. *DAN*. and C.C had crystallinity index values of 69.3 and 73.2% respectively, which is a good value for microcrystalline cellulose. The increase in value for the MCCs over the cellulose samples indicated that the amorphous region of the cellulose samples was broken down during hydrolysis. The samples' XRD Spectra are shown below. The diffraction patterns of all the MCC samples showed sharp peaks at 2 angles of approximately 14.5°, 17°, 22.7°, and 35.5° for all the compounds, which were assigned to the usual cellulose I reflection planes. The results were identical to those found in the literature for carbon nanocellulose and microcrystalline cellulose (39–42).

3.10. Scanning Electron Microscopy (SEM)

From the overall view, the SEM micrographs of the cellulose samples showed the fibre strands with long and short tubes connected to one another, and the presence of both long and short tubes was due to the amorphous and crystalline structure in the cellulose backbone. The microcrystalline cellulose derivatives showed non-uniformly distributed and shorter microcrystalline particles, creating microcrystals due to hydrolysis and displaying a large sample surface area. This was caused by the amorphous region breaking down during hydrolysis, resulting in a microcrystalline structure with a huge surface area. The surface area patterns from the analyzed varieties were similar but those of corncobs formed agglomerates better than those of the *DAN sample* which makes it (C.C MCC) more crystalline as evident in the crystallinity indices of the materials calculated from the XRD results. Overall, the microcrystalline cellulose from each of these samples may be used as raw starting materials for further processing either as direct compression excipient in pharmaceutical drug formulation, biomedical and paint industry due to their crystallinity and appearance. Their water holding capacities suggests the samples as good candidates in hydrogel formation for water storage agent for plants' roots..

3.11. Energy Dispersive X-Ray Spectroscopic (EDS) Analysis

The EDS analysis of the samples showed the presence of Carbon (C) and oxygen (O) as the predominant element, which revealed materials prepared are purely cellulose. As expected for pure

cellulose products, elements like sodium (Na) and sulfur (S), which might be present as contaminants during pretreatment and Microcrystalline cellulose preparation, were absent. Conclusively, good grade cellulose successfully, were prepared. It is worthy of note that the presence of Au and Nb elements is due to gold element used in coating the samples before analysis.

3.12. Thermogravimetric/Differential Thermal Analysis (TGA/DTA)

The thermogravimetric curve demonstrated that the microcrystalline cellulose derivatives degraded in a single step from 400 to 500 °C. The first degradation step is as a result of loss of water or moisture due to the hydrophilic nature of the samples while the other step involves the breakdown of the microcrystalline cellulose derivatives into ashes. From the thermogram, C.C MCC had better thermal stability than *DAN* MCC at a degradation temperature of 500 °C, which could be due to its high swelling capacity. Overall, the samples' thermal breakdown patterns verified the absence of hemicelluloses, lignin, and little or no residues of contaminants in their core structure. As a result, the MCCs are thermally stable. Similar results are widely reported in literature for microcrystalline cellulose derivatives from other plant species such as date seeds, tea wastes, rice husk, pomelo peels, wheat straw and cotton. (2,8,24,28,34,43) The T_{max} (temperature at which maximum weight loss occurs) for the samples was 450 °C. These samples, which are thermally stable, serve as raw materials for future material functionalization and applications.

4. CONCLUSION

Microcrystalline cellulose compounds were prepared and characterized from cellulose isolated through alkaline hydrolysis of cellulosic wastes. This work serve as a workable model to clean up the environment by removing some of the cellulosic wastes from the environment, therefore reducing possible environmental pollution by their presence. On the other hand, the wastes were converted into good-grade microcrystalline cellulose that will be useful in various areas such as in direct compression excipient in pharmaceutical formulation or agriculture as water storage excipient. From the properties displayed by these two MCCs, the C.C sample gave cellulose with better yield and physical as well as spectroscopic properties. Due to their (C.C) high water uptake ability, they can be employed in agriculture as water retaining hydrogels that can be prepared by grafting the MCC compounds onto other natural or synthetic polymers. This is owing to their high water uptake ability. The derivatives from *Daniella oliveri*, an underutilized plant in the area of cellulosic application on the other hand, might serve as a good or even better candidate in the

pharmaceutical, biomedical or paint industry than the corncob derivatives. Overall, good grade MCCs were prepared from wastes obtained from nature. The wastes were processed into useful raw materials that will now be sent back to nature in a useful form as raw materials for several applications.

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6. REFERENCES

- Bian B, Hu X, Zhang S, Lv C, Yang Z, Yang W, et al. Pilot-scale composting of typical multiple agricultural wastes: Parameter optimization and mechanisms. *Bioresour Technol* [Internet]. 2019 Sep 1 [cited 2021 Apr 9];287. Available from: [<URL>](#).
- Abu-Thabit NY, Judeh AA, Hakeem AS, Ul-Hamid A, Umar Y, Ahmad A. Isolation and characterization of microcrystalline cellulose from date seeds (*Phoenix dactylifera* L.). *Int J Biol Macromol* [Internet]. 2020;155:730–9. Available from: [<URL>](#).
- Granström M. *Cellulose Derivatives: Synthesis, Properties and Applications*. 2009. ISBN: 978-952-10-5485-3.
- Kumar V, Pathak P, Bhardwaj NK. Waste paper: An underutilized but promising source for nanocellulose mining. *Waste Manag* [Internet]. 2020;102:281–303. Available from: [<URL>](#).
- Czaja W, Krystynowicz A, Bielecki S, Brown RM. Microbial cellulose - The natural power to heal wounds. *Biomaterials*. 2006;27(2):145–51. Available from: [<URL>](#).
- Kostag M, Jedvert K, Ahtel C, Heinze T, El Seoud OA. Recent advances in solvents for the dissolution, shaping and derivatization of cellulose: Quaternary ammonium electrolytes and their solutions in water and molecular solvents. *Molecules*. 2018;23(3). Available from: [<URL>](#).
- Kunusa WR, Isa I, Laliyo LAR, Iyabu H. FTIR, XRD and SEM Analysis of Microcrystalline Cellulose (MCC) Fibers from Corncorbs in Alkaline Treatment. *J Phys Conf Ser*. 2018;1028(1). Available from: [<URL>](#).
- Zhao T, Chen Z, Lin X, Ren Z, Li B, Zhang Y. Preparation and characterization of microcrystalline cellulose (MCC) from tea waste. *Carbohydr Polym*. 2018 Mar 15;184:164–70. Available from: [<URL>](#).
- Garba ZN, Lawan I, Zhou W, Zhang M, Wang L, Yuan Z. Microcrystalline cellulose (MCC) based materials as emerging adsorbents for the removal of dyes and heavy metals – A review. *Sci Total Environ* [Internet]. 2020;717(Mcc):135070. Available from: [<URL>](#).
- Knoema. Search, Discover, Catalog and Access Your Data Seamlessly. October 29, 2022 ([<URL>](#). Opens in new tab).
- Cao Q, Xie KC, Bao WR, Shen SG. Pyrolytic behavior of waste corn cob. *Bioresour Technol*. 2004;94(1):83–9. [<URL>](#).
- Ravikumar C, Senthil Kumar P, Subhashni SK, Tejaswini P V., Varshini V. Microwave assisted fast pyrolysis of corn cob, corn stover, saw dust and rice straw: Experimental investigation on bio-oil yield and high heating values. *Sustain Mater Technol* [Internet]. 2017;11:19–27. Available from: [<URL>](#).
- Zhang Q, Zhang D, Xu H, Lu W, Ren X, Cai H, et al. Biochar filled high-density polyethylene composites with excellent properties: Towards maximizing the utilization of agricultural wastes [Internet]. 2020 [cited 2021 Apr 9]. Available from: [<URL>](#).
- Wang S, Gao W, Li H, Xiao LP, Sun RC, Song G. Selective fragmentation of biorefinery corncob lignin into p-hydroxycinnamic esters with a supported zinc molybdate catalyst. *ChemSusChem*. 2018;11(13):2114–23. Available from: [<URL>](#).
- Duan C, Meng X, Liu C, Lu W, Liu J, Dai L, et al. Carbohydrates-rich corncobs supported metal-organic frameworks as versatile biosorbents for dye removal and microbial inactivation. *Carbohydr Polym* [Internet]. 2019;222:115042. Available from: [<URL>](#).
- agritrop-eprint-317983. *Useful Tropical Plants*. July 20, 2022 ([<URL>](#). opens in new tab).
- Shackleton CM, Pasquini MW, Drescher AW. African indigenous vegetables in urban agriculture. *African Indigenous Vegetables in Urban Agriculture*. 2009. 1–298 p. ISBN: 9781136574993 .
- Ahmadu AA, Zezi AU, Yaro AH. Anti-diarrheal activity of the leaf extracts of *Daniellia Oliveri* hutch and Dalz (*Fabaceae*) and *ficus sycomorus* Miq (*Moraceae*). *African J Tradit Complement Altern Med*. 2007;4(4):524–8. Available from: [<URL>](#).
- Burkill HM. *The useful plants of west tropical Africa, Vols. 1-3*. 1995;(2. ed.), ISBN: 094764301x. [<URL>](#).
- Dinku W, Isaksson J, Rylandsholm FG, Bouř P, Brichtová E, Choi SU, et al. Anti-proliferative activity of a novel tricyclic triterpenoid acid from *Commiphora africana* resin against four human cancer cell lines. *Appl Biol Chem* [Internet]. 2020;63(1). Available from: [<URL>](#).
- Alagbe, J.O., Sharma, R., Eunice AbidemiOjo., Shittu, M.D and Bello KA. Proximate , Mineral and Phytochemical Analysis of *Piliostigma Thonningii* Stem Bark and Roots. *Int J Biol , Phys Chem Stud (JBPCS)*. 2020;(c):1–7. Available from: [<URL>](#).
- Adeyanju O, Olatoyinbo FA. Toxicological Studies and Utilization of DAN. Gum as Binder in Drug Formulation. *J Pharm Appl Chem*. 2018;4(3):169–74. Available from: [<URL>](#).
- Ajala, O. O., Awotedu, O.O. and Ogunbamowo PO. Comparative assessment of briquette produced from

- selected wood species. *J Sustain Environ Manag* [Internet]. 2016;8(ISSN: 2141-0267):1-12. Available from: [<URL>](#).
24. Arowona MT, Olatunji GA, Saliu OD, Adeniyi OR, Atolani O, Adisa MJ. Thermally stable rice husk microcrystalline cellulose as adsorbent in PTLC plates. *J Turkish Chem Soc Sect A Chem*. 2018;5(3):1177-84. [<URL>](#).
25. Kharismi RRAY, Sutriyo, Suryadi H. Preparation and characterization of microcrystalline cellulose produced from betung bamboo (*dendrocalamus asper*) through acid hydrolysis. *J Young Pharm*. 2018;1 (2):s79--s83. [<URL>](#).
26. Nwachukwu N, Ofoefule SI. Effect of drying methods on the powder and compaction properties of microcrystalline cellulose derived from gossypium herbaceum. *Brazilian J Pharm Sci*. 2020;56:1-17. [<URL>](#).
27. Kharismi RRAY, Sutriyo, Suryadi H. Preparation and characterization of microcrystalline cellulose produced from betung bamboo (*dendrocalamus asper*) through acid hydrolysis. *J Young Pharm*. 2018;10(2):s79-83. [<URL>](#).
28. Krivokapić J, Ivanović J, Djuriš J, Medarević D, Potpara Z, Maksimović Z, et al. Tableting properties of microcrystalline cellulose obtained from wheat straw measured with a single punch bench top tablet press. *Saudi Pharm J*. 2020;28(6):710-8. [<URL>](#).
29. Geng H. A one-step approach to make cellulose-based hydrogels of various transparency and swelling degrees. *Carbohydr Polym* [Internet]. 2018;186:208-16. Available from: [<URL>](#).
30. Viera-Herrera C, Santamaría-Aguirre J, Vizuete K, Debut A, Whitehead DC, Alexis F. Microcrystalline cellulose extracted from native plants as an excipient for solid dosage formulations in drug delivery. *Nanomaterials*. 2020;10(5):1-12. Available from: [<URL>](#).
31. Liu C-F, Sun R-C, Zhang A-P, Qin M-H, Ren J-L, Wang X-A. Preparation and Characterization of Phthalated Cellulose Derivatives in Room-Temperature Ionic Liquid without Catalysts. 2007; Available from: [<URL>](#).
32. Beroual M, Boumaza L, Mehelli O, Trache D, Tarchoun AF, Khimeche K. Physicochemical Properties and Thermal Stability of Microcrystalline Cellulose Isolated from Esparto Grass Using Different Delignification Approaches. *J Polym Environ* [Internet]. 2021;29(1):130-42. Available from: [<URL>](#).
33. Szymanska-Chargot M, Chylinska M, Gdula K, Koziol A, Zdunek A. Isolation and characterization of cellulose from different fruit and vegetable pomaces. *Polymers (Basel)* [Internet]. 2017;9(10). Available from: [<URL>](#).
34. Hachaichi A, Kouini B, Kian LK, Asim M, Jawaid M. Extraction and Characterization of Microcrystalline Cellulose from Date Palm Fibers using Successive Chemical Treatments. *J Polym Environ* [Internet]. 2021; (0123456789). Available from: [<URL>](#).
35. Usmani Z, Sharma M, Gupta P, Karpichev Y, Gathergood N, Liang James Hawkins Michael E Ries Peter J Hine YE, et al. A study on the microstructural development of gel polymer electrolytes and different imidazolium-based ionic liquids for dye-sensitized solar cells. *Carbohydr Polym* [Internet]. 1st ed. 2017 Mar 15 [cited 2021 Mar 29];10(1):1-12. Available from: [<URL>](#).
36. Hina S, Zhang Y, Wang H. Role of ionic liquids in dissolution and regeneration of cellulose. *Rev Adv Mater Sci*. 2015;40(3):215-26. Available from: [<URL>](#).
37. Cheng W, He J, Wu Y, Song C, Xie S, Huang Y, et al. Preparation and characterization of oxidized regenerated cellulose film for hemostasis and the effect of blood on its surface. *Cellulose*. 2013;20(5):2547-58. [<URL>](#).
38. Nascimento DM d., Almeida JS, Vale M do S, Leitão RC, Muniz CR, Figueirêdo MCB d., et al. A comprehensive approach for obtaining cellulose nanocrystal from coconut fiber. Part I: Proposition of technological pathways. *Ind Crops Prod* [Internet]. 2016;93:66-75. Available from: [<URL>](#).
39. Rahman MS, H. Mondal MI, Yeasmin MS, Sayeed MA, Hossain MA, Ahmed MB. Conversion of Lignocellulosic Corn Agro-Waste into Cellulose Derivative and Its Potential Application as Pharmaceutical Excipient. *Processes* [Internet]. 2020 Jun 19 [cited 2021 Apr 9];8(6):711. Available from: [<URL>](#).
40. Hu F, Lin N, Chang PR, Huang J. Reinforcement and nucleation of acetylated cellulose nanocrystals in foamed polyester composites. *Carbohydr Polym* [Internet]. 2015;129:208-15. Available from: [<URL>](#).
41. Mahmoudian S, Wahit MU, Ismail AF, Balakrishnan H, Imran M. Bionanocomposite fibers based on cellulose and montmorillonite using ionic liquid 1-ethyl-3-methylimidazolium acetate. *J Mater Sci*. 2015;50(3):1228-36. [<URL>](#).
42. W. R. Kunusa*, I. Isa LAL& HI. FTIR, XRD and SEM Analysis of Microcrystalline Cellulose (MCC) Fibers from Corncorbs in Alkaline Treatment. *J Phys Conf Ser* 1028. 2018 Mar 15;1(1):258-66. [<URL>](#).
43. Liu Y, Liu A, Ibrahim SA, Yang H, Huang W. Isolation and characterization of microcrystalline cellulose from pomelo peel. *Int J Biol Macromol* [Internet]. 2018;111:717-21. Available from: [<URL>](#).