Microcrystalline cellulose of Oil Bean Pod: Extraction, Physico-chemical, Brunauer–Emmett–Teller (BET), and Flow-ability Analysis

Abstract

Oil bean pods (OBPs) are a biomass that are indiscriminately dumped in eastern Nigeria, thus causes a lot of pollution. In this study, microcrystalline cellulose (MCC) was isolated from oil bean pods using acid hydrolysis, and several characterizations were performed using a variety of sophisticated techniques. Fourier transform infrared (FTIR) spectroscopy analysis has indicated the removal of lignin and hemicellulose from MCC extracted from oil bean pods. Scanning Electron Microscopy and Energy Dispersive X-ray (SEM-EDX) revealed a rough surface and minor agglomeration of the MCC. Furthermore, the isolated MCC has slightly higher inorganic minerals than the raw oil bean pod on the basis of SEM-EDX and the ash content. The Brunauer–Emmett–Teller (BET) analysis reveals that the specific surface area of MCC is 331.94 m^2/g, which is greater than that of OBP, with 164.728 m^2/g. Other characteristics like pore volume, and average pore diameter or size demonstrate that MCC has a superior property than the raw OBP. This implies that MCC could serve as a better adsorbent than the raw OBP. As a result of the increased surface area and high percentage of MCC, which is associated to chemical treatment of raw OBP, MCC can be used in environmental remediation of heavy metals. The bulk density of MCC was recorded at 0.447, which is slightly above the United States Pharmacopeia (USP) specification of 0.32, and the tapped density was recorded at 0.532. The flow ability of MCC powder determines its suitability as a direct compression binder. Thus, the isolated MCC might be used as a reinforcing element for the production of green composites, binder, adsorbents, and plastic polymers.

Keywords: BET, Cellulose, Flowability, Microcrystalline cellulose and Oil bean pod

1.0 Introduction

The current rapid industrial development has resulted in an increased need for various nano/micro functional and structural materials, which are employed for applied research and development in a variety of industries. Modern industrial societies are utilizing more expensive materials for a wide range of cutting-edge applications due to the application of microtechnology. This is due to the fact that they frequently have higher qualities than currently available conventional materials; advanced materials could potentially outperform them in terms of properties and applications [1]. Many high-tech companies, including those in the medical, aerospace, automotive, and power industries, have made extensive use of sophisticated materials. These materials are often expensive and rare due to a lack of upscaling infrastructure and high production costs [2, 3]. It has been aggressively pursued to convert currently accessible raw materials into functionally and structurally active materials. Numerous supplementary efforts have also been made to make use of the raw resources that are accessible for the creation of these smart materials. Rapid industrial expansion is hastening the deterioration of our environment, which is directly related to the use of numerous resources. The non-biodegradability of items disposed of in the soil continues to harm soil fertility and increase pollution in the ecosystem. Non-biodegradable soil components reduce soil fertility by changing soil properties. Deteriorating soil conditions are

a red flag for all living organisms [4, 5, 6]. As a result, there is a noticeable increase in demand for green materials that can replace materials derived from fossil fuels while benefiting human society. “Numerous research projects have focused on environmentally friendly materials that could be used for a wide range of applications without sacrificing production costs, scalability, or final properties. There is a lot of pressure on the future materials industry to focus on using renewable bio-resources. The steps taken in this direction will result in environmentally friendly and green science and technology. Using natural redeemable advanced materials to make polymer composites is a critical step in this direction” [7,8].

“Cellulose is an important polymer that can be tailored to meet specific requirements and can also be used as a functional and structural material in the creation of valuable composites. It is the most common type of organic compound on the planet and has been around for a very long time” [1, 9]. “Furthermore, it is naturally occurring, inexpensive, biodegradable, a low-density compound, and perfectly suited to the topic of renewability. Cellulose is regarded as a virtually limitless supply of naturally available raw materials, accounting for approximately 1.51012 tones of total yearly biomass output. It has an unusual structure, consisting of a linear carbohydrate polymer and long chains of -d-glucopyranose units connected by a 1,4-glycosidic linkage”[10]. “It also has some very important characteristics, such as renewability, biocompatibility, and biodegradability. It also has extensive chemical modification capabilities”[11]. “Cellulose can be manufactured from a variety of natural resources such as wood, plants, bacteria, and algae. The hydrolysis process can convert cellulose into either a microcrystalline form called microcrystalline cellulose (MCC) or a nanocrystalline form called nanocrystalline cellulose (NCC)” [9].

“Microcrystalline cellulose (MCC) is a naturally occurring particle that is a derivative of cellulose. It's a fine, odourless, white, crystalline powder with non-toxicity, biocompatibility, biodegradability, high mechanical strength, a large surface area, and low density” [12]. “Because of these characteristics, it has received a lot of attention in recent decades and has been used in a variety of industries. It is widely used in the culinary, cosmetic, and medical industries, among others. a binder and filler used in food, pharmaceutical pills, and other products It has also been used as a reinforcing agent in the manufacture of polymer composites. MCC has also been used as a suspension stabilizer, water retainer, viscosity regulator, and emulsifier in pastes and creams” [7, 11, 13].

The oil bean seed, also known as ugba or ukpaka by the Igbo tribe of Nigeria, is a popular condiment in the southern and southern-eastern parts of the country. The seed husk/pod is a biomass that is carelessly dumped in Nigeria's eastern region, contributing to pollution. Researchers previously estimated that the oil bean seed is edible and a good source of essential oil. Nsude et al. [11] recently discovered some essential minerals and alpha cellulose in oil bean pods. According to Madukasi et al. [2], an oil bean pod contains 3,456 kcal per kilogramme of energy and microelements that could be used as feedstock. MCC is typically made from partially depolymerized, purified cellulose. Oil palm biomass [4, 6], rice husk [10], cotton wool [14], mangosteen [15], and roselle have all been used to make MCC[16].

“BET analysis is a method for determining the specific surface areas and pore size distributions of solid materials. The method is based on the physical adsorption of an inert

gas, such as nitrogen, on the sample's solid surface. The specific surface area measured is given in m^2/ g. BET analysis is commonly used to determine the fineness of cement and concrete, the adsorption capability of activated carbon, catalyst characterization, gas purifier adsorption performance, and the study of nano-materials" [16]. "The properties of the sample determine the appropriate inert gas for the analysis. Adsorbed gas monolayers are formed when small gas molecules are drawn to the surface of the solid sample and its open porous structure. This allows the adsorbed nitrogen gas molecules on the sample's surface to be released. The released gas molecules can then be quantified, and the sample's surface area and porosity calculated" [15].

Thus, the research aims to extract and characterize microcrystalline cellulose from oil bean pods. Fourier transform infrared (FTIR), Raman-FTIR, SEM, and BET analysis were used to characterise the MCC and raw OBP. The goal of BET analysis is to determine the adsorption potential of MCC, which could be used as an adsorbent for heavy metal remediation in simulated water. In the study, the MCC function group was also described using Fourier transform infrared (FTIR), and other analyses included proximate and flow ability rates.

2. Materials and Methods

Various sophisticated techniques have been used for the characterization of intermediate materials and final product, MCC.

2.1.1 Sample Preparation:

The Oil Bean Pod (OBP) was gathered in Aku, Igbo Etiti Local Government Area, Enugu State, and transported to the department of Industrial Chemistry Laboratory, University of Science and Technology, Enugu. The sample was carefully sorted to eliminate foreign material from the sample to prepare for pulverization. The sample was rinsed with distilled water, sun-dried for 2-3 weeks, and then chopped with a cutter to increase the surface area and improve future treatment. The sun-dried chopped pod sample of OBP was crushed into fine powder and sieved to a particle size of 0.07 mm.

2.1.2 Dewaxing Technique

The dewaxing technique used was consistent with Nsude et al. [11]. The powdered OBP 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 OBP) was dried at room temperature, weighed, and stored in a sealed plastic bag for further analysis.

2.1.3 Delignification and α-Cellulose Isolation OBP

The method of Nsude et al. [11] with some modifications was used. A 100g of dust-free, defatted OBP sample was measured out using an analytical weighing balance and placed in a beaker, followed by the addition of NaOH (4.32M, 100ml) solution in a solid liquid ratio of 1:10. The OBP sample mixture in the beaker was placed in a water bath and heated at 50 ^{O}C with occasional stirring at an interval of 5 minutes for a period of 3 hours. The slurry was

then filtered and rinsed with a fresh batch of NaOH (17.52%) and washed with distilled water until neutral. The OBP cellulose residue was air dried overnight.

2.1.4 Bleaching

The air dried cellulose was bleached with sodium hypochlorite (7.5%) at 70 ^{O}C for a period of 30 min with a 5 min stirring interval. The slurry was filtered and washed with distilled water until neutral. The alpha cellulose OBP pulp that was made was then dried in an oven at 60 ^{O}C until the weight stayed the same. It was then put in a sample bottle and used later.

2.1.5 Isolation of Microcrystalline Cellulose (MCC) OBP

The method adopted was in line with Rashid et al. [14] with modifications. 30 g quantity of the α-cellulose OBP obtained was placed in a glass container and hydrolysed with hydrochloric acid (2.5 M, 500 ml) at a boiling temperature of 105 ^{o}C for 15 mins. The hot acid mixture was poured into 1.5 litres of cold tap water, which was followed by vigorous stirring with a spatula, and the mixture was allowed to stand overnight. The microcrystalline cellulose OBP obtained by this process was filtered, washed with water until neutral, filtered, pressed, and dried in a hot air oven at a temperature of 60 ^{o}C for 60 minutes, followed by further milling and sieving. The fraction passing through a 650 µm sieve aperture was used for the characterization.

2.1.6 Determination of Moisture Content

This was done by weighing out a known amount of the resin into a container: and placing it in a Genilab oven. The lignocellulosic biomass is then heated for 3hours at a constant temperature of 105 ^{O}C. The sample is then rapidly removed and put into a desiccator to prevent moisture uptake from the atmosphere after which the sample was then reweighed [17]. The procedure was repeated several times until a constant weight was obtained. Mositure content is then calculated as thus:

$$\%\text{Moisture Content} = \frac{W_2 - W_3}{W_2 - W_1} X100 \quad (1)$$

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

2.1.7 Determination of 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 3hours at a constant temperature of 550 ^{O}C. The sample is then rapidly removed and put into a desiccator to prevent moisture uptake from the atmosphere after which the sample was then reweighed [11, 17]. The procedure was repeated several times until a constant weight was obtained. Ash content is then calculated as thus:

$$\%\text{Ash Content} = \frac{W_3 - W_1}{W_2 - W_1} X100 \quad (2)$$

Where:

W_1 = Weight of container, W_2 = Weight of Container + Sample before ashing
$W_2 - W_1$ = Weight of Sample before drying, W3 = Weight of container + Sample after ashing, W_2-W_3 = Weight of Ash in sample

2.1.8 Determination of Volatile Matter

This was done by weighing out a known amount of the biomass into a container and placing it in the Biotech Muffle furnace. The biomass is then heated for 3hours at a constant temperature of 900 ^{O}C. The sample is then rapidly removed and put into a desiccator to prevent moisture uptake from the atmosphere after which the sample was then reweighed Nsude et al. [1]. The procedure was repeated several times until a constant weight was obtained. Volatile Matter is then calculated as thus:

$$\%\text{Volatile Matter} = \frac{W_3 - W_1}{W_2 - W_1} X100 \quad (3)$$

Where:
W_1 = Weight of Container, W_2 = Weight of Container + Sample before ashing at 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

2.1.9 Determination of Fixed Carbon Content

Fixed Carbon Content is determined using the equation;
%Fixed Carbon Content = 100% - (Moisture Content + Ash Content + Volatile Matter)

2.1.10 Determination of Apparent Density

Method used by Abugu et al. [17] employed an empty dry graduated cylinder that was weighed. Samples of dried OBP were packed into the cylinder and were reweighed. The apparent density was calculated as;

$$\text{Density}(g/cm) = \frac{\text{Weight of dry activated carbon}}{\text{Volume of dry material}} X\ 100 \quad (4)$$

2.1.12 Determination of pH and Conductivity

The standard test technique for determining the pH of activated carbon and lignocelluloses is described by Costa et al., [18]. 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 h. 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.

2.2.1 FTIR Analysis

FTIR was carried out to understand chemical changes during the production of MCC. Perkin-Elmer 1600 Infrared spectrometer was used for the detection of various functional groups introduced during the isolation procedure. The spectra are collected by the spectrometer with 32 running scans at a resolution of 4 cm^{-1} for each sample within 650–4000 cm^{-1} range. The

"find peak tool" functionality of Nicolet software was used to determine the positions of significant transmittance peaks at a particular wave number.

2.2.2 Raman Spectroscopy Analysis

Raman spectral fingerprint was used to identify the electrically symmetrical bond. Point and line Raman spectra of the powder particles were acquired using a DXR2 Raman microscope. The 532-nm-wavelength laser line was used as the excitation wavelength, and the laser power used was 2.5 mW. The spectra were acquired at 25 °C between 200 and 3500 cm^{-1}

2.2.3 BET Analysis for Surface Area

The OBP sample were properly weigh and placed into the sample cell, then the filled sample cell bulb were put into the heating mantle, the clamp were placed around the mantle so that the sample cell is held firm, the out gassing temperature were set at 250 ^{0}C and the system were instructed to start degassing for 3 hours and switch on the heater, when the sample has out gassed for 3 hours the heating mantle were put off and allowed allow the OBP sample cell to cool, once the heating mantle has cooled, the out gas station were unload and the sample were removed and reweighed to determine the post out gas sample weight.

2.2.4 Morphological and Elemental Analysis

To determine morphology of MCC samples, scanning electron microscope (SEM) (Hitachi Model S-3400N) was used. SEM model comes laced with energy dispersive X-ray (EDX) equipment, which has a 15kV accelerating voltage. To minimize the charging effect, the MCC samples were gold sputtered and subsequently the samples were observed. EDX diffraction was used for identification of elemental composition of the MCC samples. The analysis of the particle size of MCC samples was performed using Mastersizer 2000 particle size analyser.

3.0 Results and Discussion

3.1 Proximate analysis of raw oil bean pod and microcrystalline cellulose

The proximate analysis of oil bean pod and microcrystalline cellulose of oil beans pod is shown Table 1. The table contains the values of moisture content, ash content, volatile matter, fixed carbon content and density;

Table 1: Proximate Analysis of Raw Oil Bean Pod and Microcrystalline Cellulose of oil Bean Pod

Materials	MC	AC	VM	FCC	D	PH
Raw OBP	9.16	0.75	11.37	78.72	0.64	5.14
MCC	6.99	1.98	6.70	65.61	0.55	6.42

MC= Moisture Content, AC= Ash content, VM= Volatile Matter, FCC= Fixed Carbon Content, D= **Density**

Both the oil bean pod and the microcrystalline cellulose had low moisture content. The MCC, on the other hand, has a lower moisture content of 6.99%, whereas the raw oil bean pod has the highest moisture content of 9.16%. This is attributed to the series of chemical treatment given to the OBP, and it implies that the raw oil bean pod is more susceptible to decomposition and bacterial attack than the MCC. Because of the low moisture content,

MCC from oil bean pods could be a better biofuel than OBP. This is consistent with Grandesso et al. [19], with the conclusion that high moisture content has a significantly lower carbon burn rate and promotes bacterial activity.

The ash content is the inorganic residue that remains after the volatile matter has been removed. The OBP has a lower ash content of 0.75%, whereas the MCC has a relatively high ash content of 1.98%, as shown in Table 1. This implies that MCC may be a better source of inorganic minerals than OBP. The volatile matter of OBP in Table 1 was estimated to be 11.37%, while the MCC was 6.90%. This is an indication that raw OBP contains more volatile organic components and could be a good source of feedstock for microorganisms [20].

The raw OBP has a higher fixed carbon content of 78.72%, whereas the MCC has a fixed carbon content of 65.51%. The general variation in the properties presented in Table 1 was associated with the series of chemical treatments applied to raw OBP in order to obtain MCC. The high carbon content of OBP could be associated to the presence of lignin, hemicelluloses and other impurities, which could have been removed during MCC production. The observations were in line with Brandt et al. [21] and Nsude et al. [1], who investigated the physicochemical characteristics of selected lignocelluloses and celluloses of different biomass.

3.2 Powder Flow Properties of Microcrystalline Cellulose

The flow properties of the MCC powder are shown in Table 2. The properties considered were Bulky density, Tap density, Hasner's Index and Carr's Index

Table 2: Powder flow properties

Properties	Results
Bulky Density (g/ml)	0.447
Tap Density (g/ml)	0.532
Hasner's Index	1.19
Carr's Index	16

The bulk density of MCC of oil bean pod was recorded at 0.447, which is slightly above the United States Pharmacopeia (USP) specification of 0.32 and the tapped density was recorded at 0.532. The flow ability of MCC powder determines its suitability as a direct compression binder. Powder flow ability is measured by the Hausner index and Carr's index, the higher the values of these parameters, the lower (poorer) the flow properties of the powder [22].

The Hausner's index for the MCC of OBP was recorded as 1.19. This estimation shows that MCC has inter-particle friction, since a Hausner's index value greater than 1.25 shows poor flow [6]. The Carr's index (compressibility index) shows the ability of a material to reduce in volume. Any value less than 16% indicates good flow, while values above 35% show cohesion. MCC of OBP recorded a compressibility index of 16%. Thus, indicating a fair flow ability rate [23]. The MCC of OBP has a better Hausner's and Carr's index properties than GH-MCC (1.47, 31.72) researched by Ohwoavworhua et al. [13], PP-MCC (1.379, 27.33) and CP–MCC (1.65, 39.5) researched by Ogbonna et al. [24] respectively.

3.3 FTIR Analysis of Raw Oil Bean Pod and Microcrystalline Cellulose

The FTIR investigation reveals some functional moieties of raw OBP and MCC, as shown in Table 3

Table 3: FTIR Analysis of Raw Oil Ban Pod and Microcrystalline Cellulose

	Raw OBP	MCC
1	3386.89 cm^{-1} OH	3407.00 cm^{-1} OH
2	2951.001cm^{-1} C-H stretch of aliphatic	2925.30 cm^{-1}C-H stretch of aliphatic
3	1610.303 cm^{-1} C=O at C-3 and C-C of cellulose	1602.00 cm^{-1} C=O at C-3 and C-C of cellulose
4	1465.238 cm^{-1} aromatic ring CH_2 stretch in lignin.	-----
5	1302-1379 cm^{-1} C-H deformation in cellulose and hemicelluloses	1377.0 cm^{-1} C-H deformation in cellulose
6	1224.679 cm^{-1} -Si-O-C-, stretching	1029.00 cm^{-1} O-C-, stretching
7	859.106 cm^{-1} C-H (crystalline and amorphous	812.00 C-H (crystalline & amorphous

The spectra of raw OBP and MCC contain broad bands around 3250 cm^{-1} that correspond to stretching of hydrogen bonded hydroxyl (–OH) groups, and 2871.505 cm^{-1}-3041.734 cm^{-1} that correspond to methyl and methylene groups of the cellulose and lignocellulose biomass. The peak of 1610.303-1602.00 cm^{-1} that is found across both raw OBP and MCC is attributed to C=O moieties. This is consistent to López-Malvar et al. [25] and Adapa et al. [26] that worked on FTIR screening of cellulose in maize and some biomass. The vibration frequency of 1224.679 cm^{-1} in OBP confirmed the presence of -Si-O-C-, which was absence in MCC [26]. The FTIR result reveals the presence of hemicelluloses in the isolated cellulose of the PMBP, whereas the microcrystalline cellulose was free from both lignin and hemicelluloses. This implies that acid hydrolysis is the most effective method of removing lignocellulose from the biomass of OBP. Figure 1 and 2 shows the FTIR spectral of raw oil bean pod and microcrystalline cellulose.

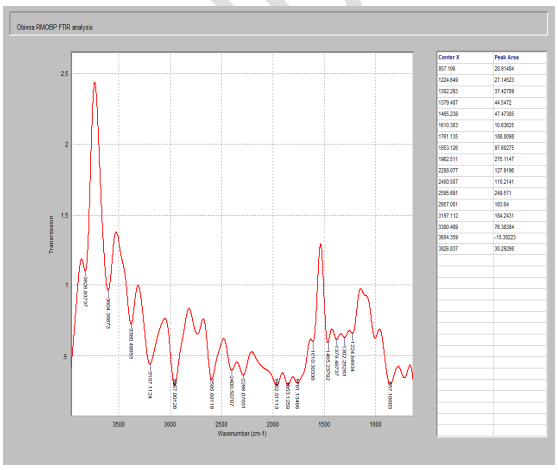

Figure 1: FTIR spectra of raw OBP

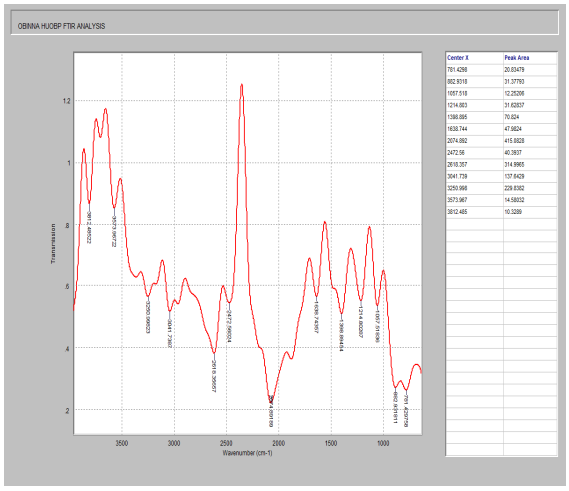

Figure 2: FTIR spectra of MCC

3.4 Raman Analysis of Raw Oil Bean Pod and Microcrystalline Cellulose

The Raman investigation classifies some functional moieties of raw OBP and MCC of OBP, as shown in Table 4

Table 4: Raman of Raw Oil Bean Pod and Microcrystalline Cellulose

	Raw OBP	MCC
1	2872 cm^{-1}, 2974 cm^{-1} Very strong CH2 stretch (alkyl)	2784 cm^{-1}, 2872 cm^{-1}strong CH2 stretch(alkyl)
2	862 cm^{-1} HCC and HCO bending at C-6 (cellulose)	786 HCC and HCO bending at C-6 (cellulose)
3	639-312.98 cm^{-1} HCC and HCO bending at C-6 (Hemicellulose)	-
4	1778 cm^{-1} Ring conjugated C=C stretch of coniferaldehyde Lignin	-

Table 4 shows the OBP contains a methylene group, cellulose, hemicelluloses, and lignin. This is revealed by the functional moieties whose vibration frequency bands are shown. The vibration frequency of 1778 cm^{-1} is associated with the presence of ring conjugated C=C-of lignin, and the frequency band of 862 cm^{-1}HCC and HCO bending of C-6 found in cellulose and hemicelluloses is linked to 394–312 cm^{-1}. This is in-line with the Zhuang et al. [27], López-Malvar et al. [25] who researched on the composition analysis of biomass via Raman-FTIR characterization. Microcrystalline cellulose is considered free from both lignin and hemicelluloses. This is attributed to the absence of vibration frequency of moieties associated with lignin and hemicelluloses. The purity of microcrystalline cellulose is supported by the investigation of Vallejo et al. [28], Costa et al. [18] and Orie et al. [20] who researched on the biomass of sugarcane bagasse, rice husk, and others synthetic compounds. Figure 3 and 4 shows the RAMAN-FTIR spectral of raw oil bean pod and microcrystalline cellulose.

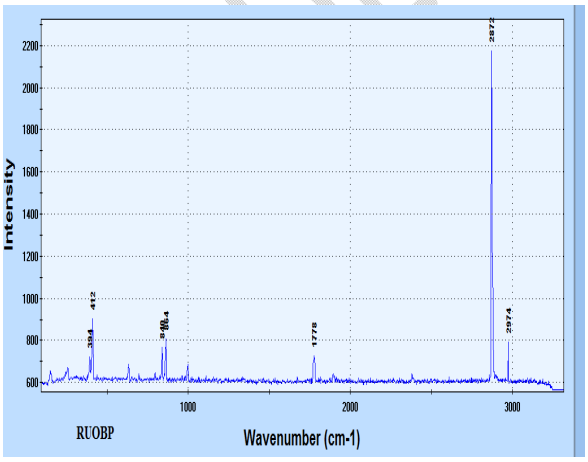

Figure 3: RAMAN spectra of raw OBP;

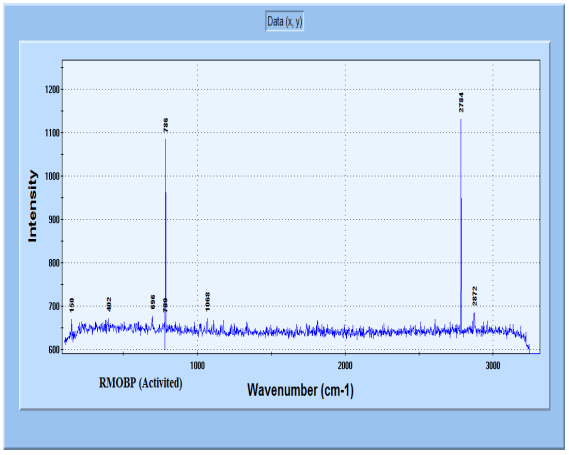

Figure 4: RAMAN spectra of MCC;

3.5 BET Analysis for Surface Area of Raw Oil Bean Pod and Microcrystalline Cellulose

BET analysis is a method used to determine the adsorption property of a solid biomass material. Table 5 shows the BET analysis of the oil bean pod and microcrystalline cellulose

Table 5: BET Analysis for Surface Area Raw of Oil Bean Pod and Microcrystalline Cellulose

		Raw OBP	MCC
1	SpecificSurface area BJH (m^2/g)	164.728	331.94
2	pore volume (m^2/g)	0.081	0.162
3	Average pore Diameter or size BJH (nm)	2.013	2.133
4	BET summary surface area (m^2/g)	159.133	260.932
5	BET pore Diameter (nm)	2.140	3.00
6	Correlative Coefficient	0.989	0.998

Table 5 shows that the MCC has better property than OBP of all the items considered. The specific surface area of MCC is 331.94 m^2/g, which is greater than that of OBP, which is 164.728 m^2/g. Other characteristics in the demonstrated that MCC has a superior property than raw OBP. This increased property of MCC in comparison to OBP demonstrates the success of the chemical treatment applied to raw OBP. In line with Farooq et al. [29], 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 MCC can serve as a good metal remover in a polluted environment. A previous study consistent with the findings showed that modification of fungal biomass with cetyl trimethyl ammonium bromide (CTAB) increases bet surface area. The CTAB modification caused a 26% increase in BET surface area. Ghani et al. [29] has it that adsorption being a surface phenomenon, 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. The results of the correlative coefficient suggest that MCC is a better than raw OBP.

3.6 Morphological and Elemental Analysis of Raw Oil Bean Pod and Microcrystalline Cellulose

The SEM-EDX analysis was aimed at estimating the morphology and elemental composition of the raw OBP and MCC.

Table 6: Elemental Analysis of Raw Oil Bean Pod and Microcrystalline Cellulose

	Raw OBP	MCC
Oxygen (O)	14.20	20.20
Aluminium (Al)	10.30	
Silicon (Si)	40.22	2.20
Silver (Ag)	18.30	
Calcium (Ca)	4.22	0.20
Sodium (Na)	2.40	0.74
Magnesium (Mg)	1.32	
Iron (Fe)	5.70	3.54
Manganese (Mn)	0.10	
Carbon (C)	2.24	64.40
Potassium (K)		
Nickel (Ni)		3.46
Cupper (Cu)		2.22
Zinc (Zn)		0.70

Table 6 shows the elemental analyses of the OBP and MCC biomass. The analysis confirmed the presence of the same element in both the raw powder and the MCC of OBP with a slight percentage variation. This variation also implied the effectiveness of the treatment on the raw OBP sample. This result is consistent with Madukasi et al. [2], who worked on the elements present in oil bean husk and its energy composition. This also implies that the OBP material can serve as a feedstock based on the presence of the constituent minerals. Figs. 5 and 6 show SEM images of raw OBP and MCC of OBP at various processing stages and magnifications (900x and 1000x). As the image size was enlarged, the micrograph became clearer and it was also observed that the image appeared entangled and bound together. This might be attributed to the presence of wax, hemicelluloses, pectin, lignin, and other impurities that render the materials' structure invisible [11, 13, 30]. This idea is also supported by the morphology research of cocoa pod husk conducted by Rasheed et al. [31], which attributed the layered or cemented appearance to impurities spurred on by wax, hemicellulose, pectin, and lignin. The high percentage of silicon implies that the cellulose of OBP can serve as good polymer reinforcement composite and other applications.

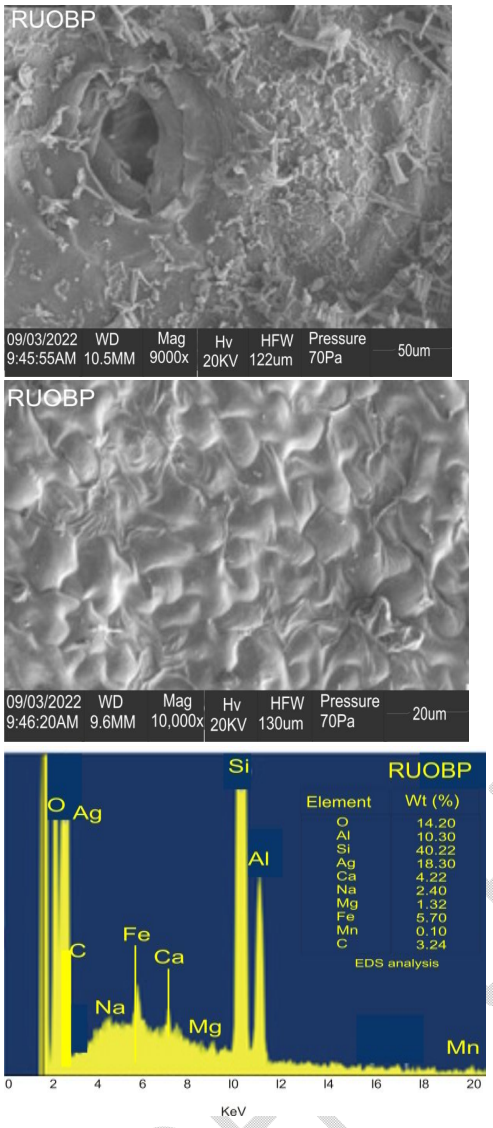

Fig. 5: SEM Micrograph and EDS Analysis for Raw OBP

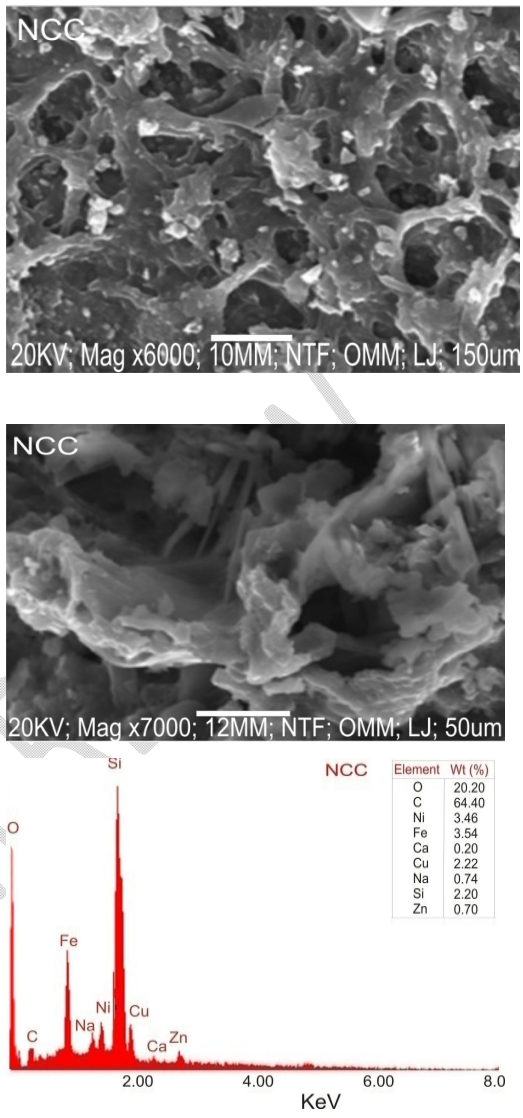

Fig.6: SEM Micrograph and EDS Analysis for MCC OBP

4.0 Conclusion

The study describes the extraction and characterization of microcrystalline cellulose from oil bean pods. The findings from the proximate analysis reveal that the reduced moisture content and ash content of MCC make it a better option than raw OBP for bio-fuel and a better source of inorganic compounds that can serve as feedstock. The high silicon and aluminium content of both OBP and MCC 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 were identified in raw OBP, whereas they were absent in MCC using FTIR and Raman spectrometers. The absence of lignin and hemicelluloses in the MCC implies that the isolation process was successful. The BET analysis shows that the MCC has a better specific surface area, pore volume, and

average pore size than the raw OBP, which implies that isolated cellulose could serve as a better adsorbent than the raw. Because of the increased surface area and high percentage of MCC in OBP, it can be used for environmental remediation. As a recommendation, MCC should be used in the adsorption of heavy metals.

References

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