



## Isolation and Characterization of Cellulose and Microcrystal Cellulose Obtained from the Pod of African Locus Bean (*Parkia Biglobosa*)

<sup>1</sup>DIDIGWU, SO; <sup>1</sup>EZEH, EC; <sup>1</sup>NSUDE, OP; <sup>1</sup>UDEOZO, PI; <sup>\*2</sup>ORIE, KJ

<sup>1</sup>Department of Industrial Chemistry, Enugu State University of Science and Technology, Enugu State, Nigeria

<sup>\*2</sup>Department of Chemistry, Ignatius Ajuru University of Education Port-Harcourt, Rivers State, Nigeria

\*Corresponding Mail Address: [oriekingsley81@gmail.com](mailto:oriekingsley81@gmail.com)

\*ORCID: <https://orcid.org/0000-0002-5110-7161>

\*Tel: +2348106148644

Co-Authors Email: [samsondidigwu11@gmail.com](mailto:samsondidigwu11@gmail.com); [emmanual.ezeh@esut.edu.ng](mailto:emmanual.ezeh@esut.edu.ng); [okechukwu.nsude@esut.edu.ng](mailto:okechukwu.nsude@esut.edu.ng); [prisca.udeozo@esut.edu.ng](mailto:prisca.udeozo@esut.edu.ng)

**ABSTRACT:** The pod of African locus bean (*Parkia biglobosa*) is a type of biomass that is haphazardly discarded in eastern Nigeria and other parts of the country, thus leading to significant pollution. Hence, the objective of this paper was to isolate and characterization of cellulose and microcrystal cellulose obtained from the pod of African locus bean (*Parkia biglobosa*) using appropriate standard techniques. The FTIR revealed the presence of O-H, C-O-C pyranose rings, and cellulosic  $\beta$ -glycosidic linkages. The SEM revealed a rough surface and MCC agglomeration. An EDX study of the cellulose found the following elements: C (80.42%), Na (4.21%), O (15%), N (10.12%), Al (5.66%), Mg (2.73%), Si (2.11%), and Na (4.21%). The MCC also included the elements C (61.32%), Na (19.59%), O (17.43%), Cu (30.67%), Si (9.23%), Mn (6.15%), and Na (4.21%), albeit with some variation compared to cellulose. Both cellulose and MCC demonstrate a sharp peak around 400 °C, with a mass loss of approximately 66.584% and a slight variation in weight masses with the temperature range of 30-100 °C. Further studies using XRD estimated the crystallinity index of cellulose and MCC at 62.8% and 78.9%, respectively. The different treatments applied to the isolated cellulose are linked to the variation in mass percentage. So, the purified MCC could be a useful ingredient in making environmentally friendly polymers, binders, adsorbents, and composites.

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The continuous and rapid expansion of industries has resulted in an increased need for a wide range of nano/micro materials that possess functional and structural properties. These materials are used for practical research and development in several fields. Due to the use of microtechnology, contemporary industrialized societies are utilizing more expensive materials for a wide range of new purposes. The rationale behind this is that advanced materials frequently exhibit superior characteristics in

comparison to the conventional materials already in use (Madukasi *et al.*, 2015).

Cellulose is a polysaccharide found in biomass that is made up of glucose molecules connected by glycosidic oxygen bridges. Each glucose unit, known as a monomer, is aligned at an 180° angle to its neighbouring unit (Haafiz *et al.*, 2017). Pectin, a type of plant-derived cellulose, retards the peristaltic flow of food in the gastrointestinal tract, so facilitating the absorption of essential nutrients by the body rather

\*Corresponding Mail Address: [oriekingsley81@gmail.com](mailto:oriekingsley81@gmail.com)

\*ORCID: <https://orcid.org/0000-0002-5110-7161>

\*Tel: +2348106148644

than their rapid elimination as waste. Similarly, indigestible fibres such as cellulose accelerate the process of moving food molecules through the digestive system, which is crucial for efficiently eliminating waste (Nwajiobi et al., 2019). Cellulose possesses unique characteristics that are essential for a range of industrial uses, including its role as a reinforcing component in composite materials like water and moisture, as well as in biomedical implants (Rasheed et al., 2020). Through the process of photosynthesis, plants and trees generate approximately 1011 to 1012 tonnes of cellulose year (Silva et al., 2024). It is a non-carbohydrate monomer present in plant cell walls. The presence of self-cohesive energy, non-thermoplastic material, and strong hydrogen bonds within and between molecule chains enables the creation of a well-organized three-dimensional crystal structure (Nsude et al., 2022).

Microcrystalline cellulose (MCC) is a particulate material that is produced from cellulose and occurs naturally. The material is a colourless crystalline powder that lacks any discernible odour. It has non-toxic characteristics, is compatible with living organisms, can be broken down naturally, and has exceptional mechanical durability. Moreover, it possesses a broad surface area and a low density. Because of these unique characteristics, it has attracted considerable attention in recent decades and has been used in different industries. It is widely employed in many areas, including culinary, cosmetic, and medicinal sectors. Microcrystalline cellulose is a substance commonly used as a binder and filler in culinary applications, pharmaceutical pills, and other goods. It is also utilized as a reinforcing agent in the manufacturing of polymer composites. Additionally, MCC has various other uses such as suspension stabilization, water retention, viscosity regulation, and emulsification in pastes and creams (Rashid et al., 2017; Zango and Imam, 2018; Donlawson et al., 2020). The African locust bean (*Parkia biglobosa*), is an angiosperm species that belongs to the Fabaceae family and is dicotyledonous. It is classified as a spermatophyte, which refers to vascular plants (Pouliot et al., 2012; Obetenb et al., 2020; Orié et al., 2021). The tree's pods, called locust beans, initially have a pink colour and darken to a deep brown when fully mature. These pods typically measure between 30 and 40 cm in length, although some can reach approximately 45 centimetres. Each pod can contain a maximum of 30 valuable seeds (Saleh et al., 2021; Damter et al., 2023). Conventional wisdom holds that *Parkia biglobosa*'s wood energy (in the form of fuel wood and charcoal) and other non-timber forest products (NTFP) are more valuable than its timber (Pouliot et al., 2012). Aside from timber, the *Parkia*

*biglobosa* forest produces a wide variety of non-timber goods, including food, medicine, animal feed, soil amendments, charcoal, and fuel. Nutrition is the primary end result of *P. biglobosa*. Because they depend on the availability of food and the timing of fruit ripening, *P. biglobosa* food items stand out. The seeds used to make dawadawa are rich in fat and protein. The vitamin C and carbohydrate-rich starchy mesocarp that surrounds the seed makes it a nutritious and tasty dietary supplement. Touré et al. (2022) note that dozim, a drink made from the dehydrated powder and water, is a popular beverage.

The stem bark is thought to include a number of chemicals, including as tannins, phenols, terpenes, saponins, sterols, and reducing sugars, according to studies. Through the use of elemental analysis, elements like magnesium, calcium, iron, zinc, potassium, sodium, and copper were detected. Aboyeji et al. (2019) observed that the leaves of the African locust bean tree contain reducing sugars, alkaloids, tannins, saponins, and flavonoids. The tree also contains cardiac glycosides. The stem bark of *P. biglobosa* contains a mixture of long-chain ciferulates, a long-chain ester of trans-ferulic acid, and a variety of catechins and ferulates, according to Fayinminnu et al. (2017). Based on the results of the phytochemical research, the plant's root bark contains a modest amount of alkaloids, a lot of saponins, and a big quantity of glycosides and tannins (Aboyeji et al., 2019). Furthermore, it was demonstrated by Saleh et al. (2021) that *P. biglobosa* root contains tannins, saponins, polysaccharides, and flavonoids.

The pods are thrown away once the seeds are harvested, which contributes to the fast accumulation of waste. Eventually, this trash will start to annoy nature. They contribute to air pollution when burned because of the strong and unpleasant smell they release. Their decay released foul odours that were uncomfortable to the environment whenever it rained. Environmental management of these pods is problematic during both the rainy and dry seasons, and as a result, they are thrown out as useless trash in West African nations. Various research have demonstrated that cellulose may be sourced from a range of natural matter, including plants, algae, and bacteria (Haafiz et al., 2017; López-Malvar et al., 2021), suggesting that there is a potential stable supply of this material. Since primary resources like fossil fuels and agricultural byproducts are steadily dwindling, bio-wastes are being regarded as a potential replacement (Kamani et al., 2019; Nwajiobi et al., 2019) for the production of cellulose and its derivatives. As a result, more and more factories are switching to using recyclable biomass from natural sources, particularly non-edible

plant resources like processed cashew nut shells. Thus, the objective of this paper is to isolate and characterize cellulose and microcrystal cellulose obtained from the pod of African locust bean (*Parkia biglobosa*).

## MATERIAL AND METHODS

**Materials:** Analytical grade chemicals were used, together with natural *Parkia biglobosa* pod, KOH, HCl, ethanol, toluene, acetic acid, NaOH, and sodium chlorite. Every single chemical was applied exactly as it was given. All experiments using deionised water.

**Sample collection and Preparation:** The *Parkia biglobosa* pods were collected in Aku, Igbo Etiti Local Government Area, Enugu State, and then sent to the Department of Industrial Chemistry, Enugu State University of Science and Technology. To ensure that the sample was free of any contaminants, it was meticulously sorted. The sample was sun-dried for two to three weeks, rinsed with distilled water, and then chopped with a cutter in preparation for pulverization. Crushed and sieved to particle sizes of 0.07 mm, the sun-dried chopped pod sample of *Parkia biglobosa* pods was ground into a fine powder in order to enhance future treatment by increasing the surface area.

**Dewaxing of *Parkia biglobosa* pod:** The dewaxing technique used was consistent with Nsude *et al.* (2022). 954 g of the powdered locust bean pod were weighed out and put in a basin (stainless) which served as a digester. An aqueous solution of 4 % NaOH was added to the powdered locust bean pod in the stainless basin and stirred for a few minutes and then kept in a water bath maintained at a temperature of 80 °C for 3 hours with stirring at intervals to maintain uniform temperature of the mixture. After 3 hrs, the heating was stopped and the liquor was allowed to cool with the addition of distilled water and later sieved. The residue was thoroughly washed with distilled water to remove the sodium hydroxide completely. After drying, the colour of the pulp was brown and then bleached to brighten the colour.

**Bleaching of cellulose from *Parkia biglobosa* pod:** The air dried cellulose was bleached with sodium hypochlorite (7.5%) at 70 °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 made was then dried in an oven at 60 °C until the weight stayed the same. It was then put in a sample bottle and used later

**Isolation of Microcrystalline Cellulose (MCC) of *Parkia biglobosa* pod:** The method used was based on Rashid *et al.* (2017), with some adjustments. A glass container was used to hydrolyse 30 g of acellulose

from *Parkia biglobosa* pods. The mixture was heated to 105°C for 15 minutes with hydrochloric acid (2.5 M, 500 ml). After vigorously swirling the mixture with a spatula, 1.5 litres of cold tap water was added to the hot acid mixture. The combination was then left to stand overnight. After this process, the *Parkia biglobosa* pods were filtered, rinsed with water until they were neutral, filtered again, compressed, and then dried in a hot air oven set at 60 oC for 60 minutes.

**Characterizations of Microcrystalline Cellulose (MCC) of *Parkia biglobosa* pod**

**Scanning Electron Microscope:** At NARICT Zaria, the morphological properties of the cellulose were examined by scanning electron microscopy (SEM, FEI, Quanta 200, USA) and transmission electron microscopy (TEM, FEI, Tecnai G20, USA).

**Fourier-Transform Infrared:** For both the untreated and acid-hydrolyzed samples of locust bean pods, Fourier transform infrared spectra were acquired using an ATR disc at the National Arbovirus Research Centre in Enugu's FTIR-8400S spectrophotometer.

**X-ray Diffraction:** The Bruker D8 ADVANCE Powder XRD equipment, which emits CuK- $\alpha$  radiation with a wavelength of  $\lambda = 1.5404$  nm, was used for XRD analysis at the University of Ibadan Research Centre. The X-ray diffractometer was operated at 40 kV and 30 mA. The XRD method was used to ascertain the crystalline structures of cellulose samples. At room temperature, XRD data were acquired over a scattering angle ( $2\theta$ ) range of 10 to 40 °. Crystallinity index (CrI) was calculated using the formula as stated in Equation 1:

$$CrI = \frac{(I_{200} - I_{AM})}{I_{200}} \times 100 \quad (1)$$

Where  $I_{200}$  and  $I_{AM}$  are the maximum peak intensities of crystalline and amorphous regions, respectively

**Thermogravimetric Analysis:** At NARICT ZARIA, the Seiko EXSTAR 6000 TG/DTA 6300 thermal analyser was used to obtain TG/DTG curves. A pan made of aluminium was used to examine about 10.2 mg of samples. Using a flow rate of 10 ml/min and a heating rate of 10 °C/min, this test was conducted in a dynamic nitrogen environment from 30 to 900 °C.

## RESULT AND DISCUSSION

**FTIR Studies of Cellulose of *Parkia biglobosa*:** Fig. 1 displays the FTIR spectra of microcrystals cellulose isolated cellulose from *Parkia biglobosa* pod. The hydroxyl group stretching vibration in cellulose, hemicellulose, and lignin is corresponding to the wide

transmission band in the spectra between 3500 and 3200  $\text{cm}^{-1}$  (Mandal & Chakrabarty 2011; Nsude, & Orié 2022). The acid hydrolysis that liberated part of the OH group is linked to this conspicuous band (OH group) for MCC. Donlawson et al. (2020) identified the C-H stretching vibration of alkyl groups in aliphatic bonds of cellulose, lignin, and hemicelluloses as the distinctive band at 2891  $\text{cm}^{-1}$ , which is present in all spectra.

A band located at approximately 1640  $\text{cm}^{-1}$  is associated with the O-H bending of water absorbed into the structure of the cellulose fibre and MCC (Kian et al. 2017; Tkachenko et al. 2022). The bands at 1430-1420  $\text{cm}^{-1}$  are due to CH<sub>2</sub> scissoring vibrating motion in cellulose, and are more pronounced in MCC (Lourdin et al., 2019; Barkane et al., 2021), 1382-1375  $\text{cm}^{-1}$  (C-H bending), 1336  $\text{cm}^{-1}$  (O-H in plane bending), 1054  $\text{cm}^{-1}$  (C-O-C pyranose ring), 902-893  $\text{cm}^{-1}$  (cellulosic  $\beta$ -glycosidic linkages), 1150  $\text{cm}^{-1}$  (C-C ring stretching band), and at 1105  $\text{cm}^{-1}$  (the C-O-C glycosidic ether band) (Nsude et al., 2022; Orié et al., 2021). The presence of MCC is associated with a somewhat elevated level of the cellulose band at 895  $\text{cm}^{-1}$  (Dinand et al., 2002; Spiridon et al., 2011). A higher absorbance in this band indicates that more of the crystalline cellulose polymer's crystallites are available once the amorphous cellulose is removed (Barkane et al., 2021; Orié et al., 2021).

The materials' crystallinity may be determined by analysing the infrared bands spanning 1500 to 850  $\text{cm}^{-1}$  (Chen et al., 2012; Haafiz et al., 2017). This is solely

relevant for samples that contain either crystalline cellulose, amorphous cellulose, or a combination of the two (Santa-Maria and Jeoh, 2010). There is a strong correlation between the crystal structure of the cellulosic material and the aforementioned IR area. The crystal structure of cellulosic material can be best explained by examining the spectra at 1420-1430  $\text{cm}^{-1}$  and 893-897  $\text{cm}^{-1}$  (Barkane et al., 2021). These IR ratios illustrate the process of calculating the total crystallinity index (TCI) and the lateral orientation index (LOI). Using the associated FTIR spectra, the ratio for each sample was computed.

$$\text{LOI} = \frac{1430\text{cm} - 1}{890\text{cm} - 1} \quad (2)$$

$$\text{TCI} = \frac{1375\text{cm} - 1}{2900\text{cm} - 1} \quad (3)$$

Where LOI = Lateral Orientation Index; TCI = Total Crystallinity Index

Cellulose had an estimated LOI of 1.24 and a TCI of 1.43, whereas microcrystalline cellulose had a LOI of 0.920 and a TCI of 0.978. (Sainorudin et al., 2018; Donlawson et al., 2020) Crystalline cellulose is readily available due to the low values of these ratios, while amorphous cellulose is present in trace amounts. Chemical processing of cellulose to produce microcrystal cellulose is responsible for the discrepancy between the two types of cellulose's crystallinity index.

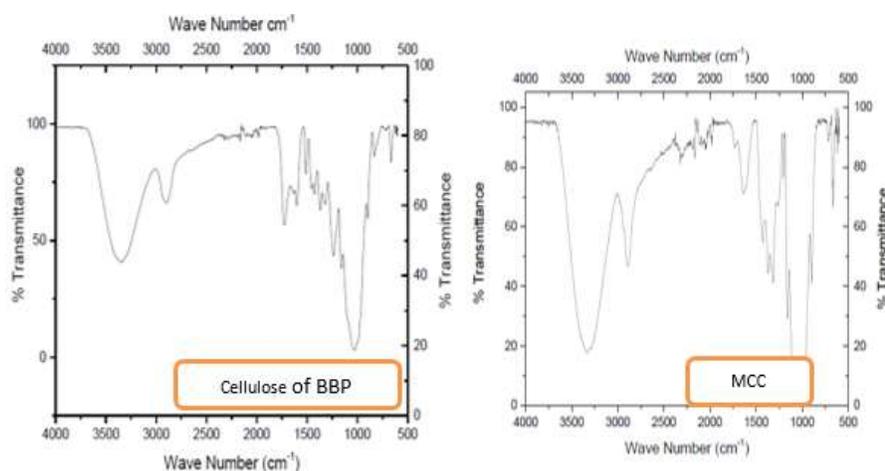


Fig. 1: FTIR spectra of cellulose and microcrystalline cellulose of Parkia biglobosa pod

*X-ray diffraction (XRD) analysis:* Fig. 2 shows the XRD diffractogram that was produced from the broad angle X-Ray Diffraction analysis conducted on the cellulose and MCC of Parkia biglobosa pods. According to Sheltami et al. (2012) and Nsude et al.

(2022), the cellulose-I structure was demonstrated by the sample's notable diffraction peaks at  $2\theta = 15^\circ$ ,  $22.5^\circ$ , and  $34.5^\circ$ , which map onto the crystallographic planes of (110), (200), and (040), respectively. The findings demonstrated the presence of cellulose-I, a

crystalline structure, in both the cellulose and MCC samples. The principal crystalline peak (index = 200) is indicated by  $2\theta = 22.5^\circ$ , while the amorphous peak is shown by  $2\theta = 15$ . Cellulose crystallinity revealed by the peaks' width and intensity in comparison to the amorphous peaks. Rosa *et al.* (2012) observed comparable results when utilising chlorine-free cellulose obtained from rice and whisker. Parkia biglobosa pod cellulose had an estimated crystallinity index of 62.8% and MCC 78.9%. The cellulose content is lower than what has been found in rice straw (69.2%; Ibrahim *et al.*, 2013), banana stem (74.55%), coconut coir (72.73%), and sugarcane bagasse (66.50%; Rosa *et al.*, 2012; Sainorudin *et al.*, 2018; Zhang *et al.*, 2018). An average crystallinity index of 78.9% was observed in the microcrystal cellulose of Parkia biglobosa pod sections that were analysed (Alotaibi *et al.*, 2019). In comparison to the study's

findings, Galiwango *et al.* (2019) demonstrated that MCC isolated from rice straw had a relatively low crystallinity index of 71% and that MCC isolated from potato tuber had a relatively low index of 68%. The study conducted by DeAvila-Delucis *et al.* (2021) discovered that cotton and softwood exhibited  $\alpha$ -cellulose crystallinity indices between 50% and 65%, together with a high MCC of 75-87%. The  $\alpha$ -cellulose crystallinity indexes of cotton linter varied from 98.1% to 99.0% following acid treatment and electron beam irradiation, according to Galiwango *et al.* (2019). The bleaching and alkaline-extraction procedures of cellulose samples are associated with variability in crystallinity. The higher crystallinity peak seen in the MCC and recovered cellulose samples from various agricultural waste sources corroborated this finding (Salem *et al.*, 2023).

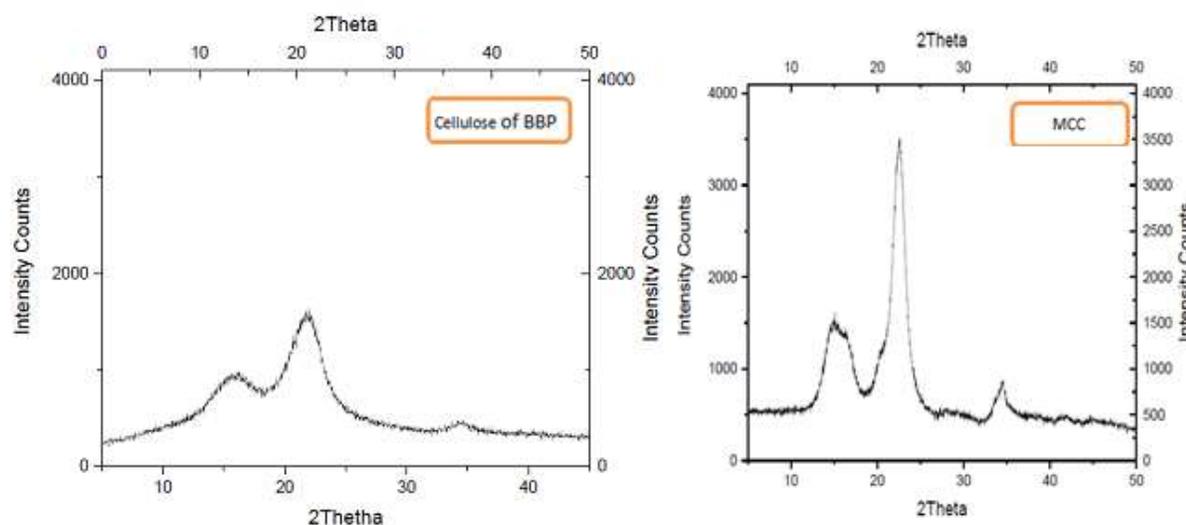


Fig. 2: XRD diffractogram of cellulose and MCC of Parkia biglobosa pod

*Scanning Electron Microscopy (SEM)/Electron diffraction X-ray (EDX):* Fig. 3 depicts the microstructure of isolated cellulose and MCC as detected using a SEM/EDX. The morphology of the cellulose types was investigated using SEM, and the resulting micrographs revealed that the cellulose samples are fibrous, as is typical of cellulose, with fibres that are rough and irregularly formed. Acid hydrolysis could account for the MCC's superior regularity. Both micrographs (cellulose and MCC) had a porous structure; however, the cellulose structure was caused by lignin breakdown by alkali treatment, whereas the MCC structure was caused by further acid hydrolysis. A similar result was seen for walnut (*Juglans regia*) shell treated with an acetate buffer/(NaClO<sub>2</sub>) solution (Alotaibi *et al.*, 2019).

Furthermore, EDX analysis of both cellulose and microcrystal cellulose indicated the presence of several elements and their weight concentrations. Some of the components detected in the cellulose with quantitative percentages C (80.42%), Na (4.21%), O (15%), N (10.12%), Al (5.66), Mg (2.73%), Si (2.11%), and Na (4.21%). The elemental analysis of MCC revealed the existence and quantity of the following elements: C (61.32%), Na (19.59%), O (17.43%), Cu (30.67%), Si (9.23%), Mn (6.15%), and Na (4.21%). Sheltami *et al.* (2012) and Nsude *et al.* (2022) found that elements derived from organic sources are easily metabolisable, and that percentage changes in element quantity may be associated with successful bleaching and subsequent transformation into cellulose microcrystals.

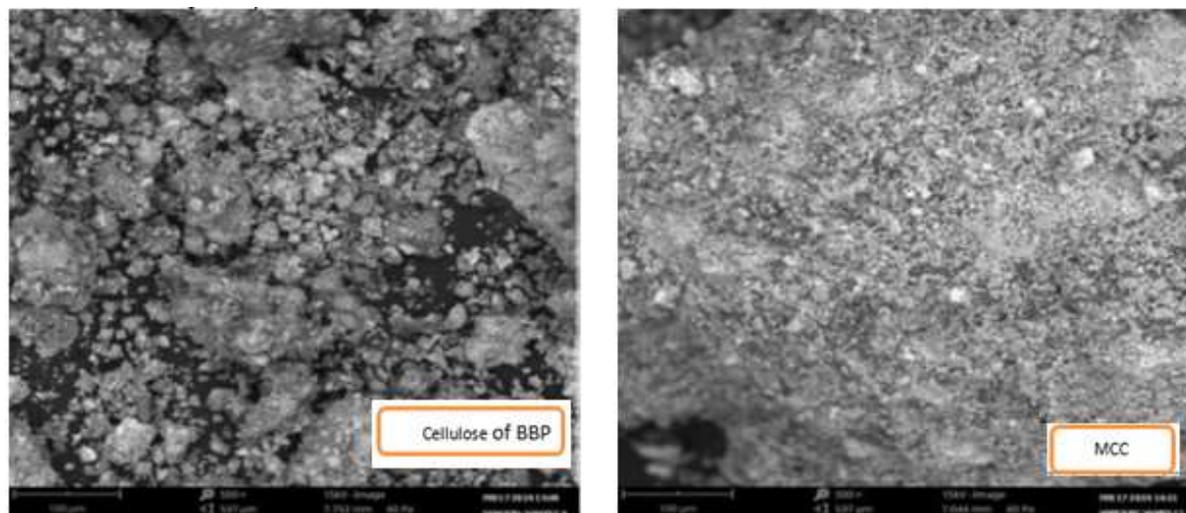


Fig. 3: SEM of Cellulose and MCC of of Parkia biglobosa pod

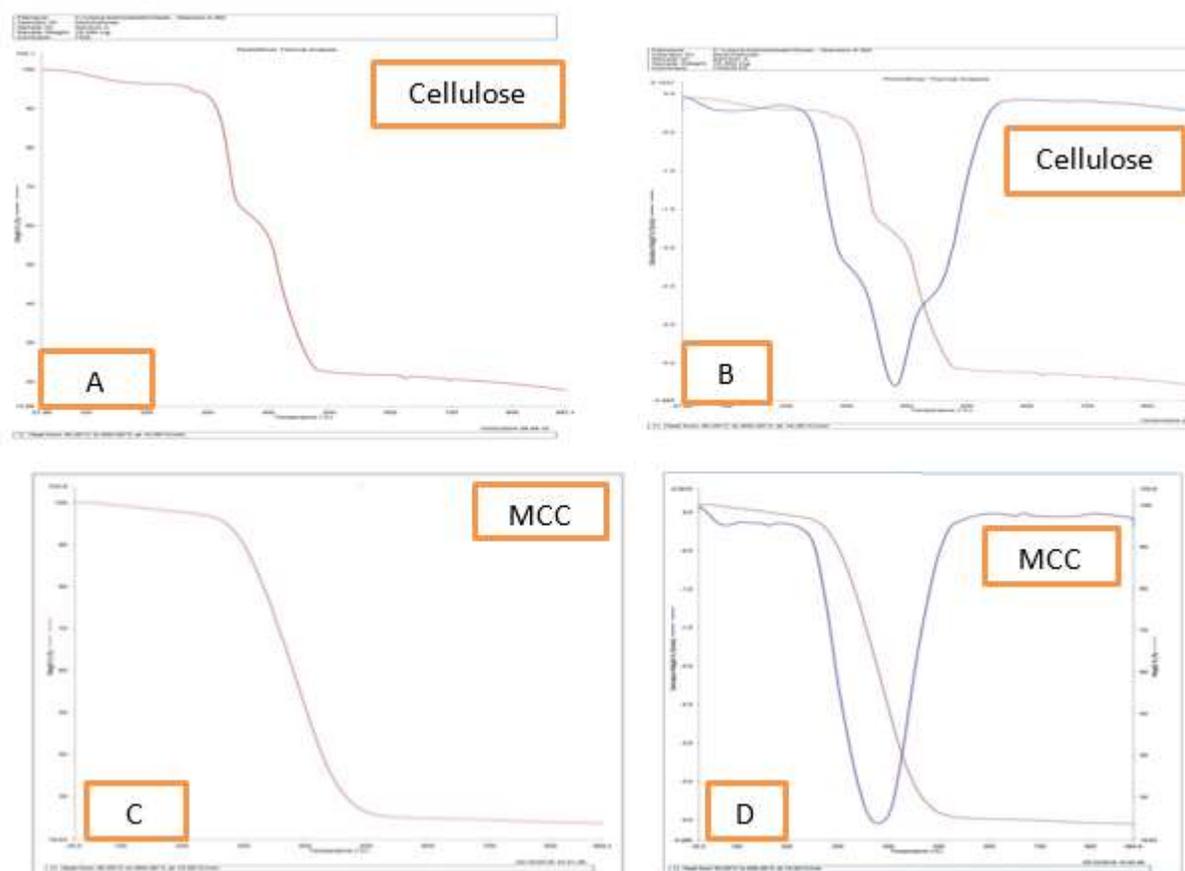


Fig. 4: Thermogram curves of cellulose and MCC of Parkia biglobosa pod

*Thermogravimetric Analysis (TGA) and Derivative Thermogravimetry (DTG):* The TGA clearly illustrates the thermal stability of isolated cellulose and MCC from Parkia biglobosa pods. Fig. 4 depicts the thermogram curves for cellulose and MCC, and the weight loss (%) in the thermogravimetric curve between 30 and 100 °C in figures 4a and c can be

attributed to the evaporation of absorbed moisture in the separated cellulose and MCC. The weight losses at the aforementioned temperature were 0.79% and 0.74% for cellulose and MCC, respectively. This indicates that the cellulose sample and MCC had almost identical moisture content (Bohrer *et al.*, 2023). The derived cellulose and MCC

degradation proceeded in a single stage, with temperatures ranging from 300 to 400°C. The degradations were primarily caused by one component connected to cellulose and microcrystal deterioration in Figures 4A and C. The mass percentage lost related to cellulose was 33.75%, while MCC was 29.685%. This suggests that the MCC of *Parkia biglobosa* pods is more stable than conventional cellulose.

Previous research, such as Ibrahim *et al.* (2013) and Boukir *et al.* (2019) have indicated a comparable range for cellulose degradation; however, the stated range shows glycosyl breakdown in cellulose fibre and microcrystal cellulose. The narrow range found in DTG curves in Figures 4b and d indicates excellent lignin removal from the cellulose sample and better cellulose treatment to create microcrystal cellulose (Beroual *et al.*, 2021). Both cellulose and MCC show a dramatic peak around 400 °C, with a mass loss of roughly 66.584% and a little variation in weight masses across the temperature range of 30-100 °C (Zhang *et al.*, 2018; Abu-Thabit *et al.*, 2020).

**Conclusion:** The study details the process of extracting and characterizing microcrystalline cellulose from locust bean pod biomass (waste) using de-waxing, bleaching, and acid hydrolysis. The FTIR analysis identified the existence of O-H, C–O–C pyranose ring, and cellulose β-glycosidic connections. The SEM showed a surface that was uneven and had small clusters of the MCC. The change in percentage mass is connected with varied treatment applied to the isolated cellulose. Thus, the isolated MCC might be employed as a reinforcing ingredient for the creation of green composites, binder, adsorbents, and polymeric polymers.

**Declaration of Conflict of Interest:** The authors declare no conflict of interest

**Data Availability Statement:** Data are available upon request from the first author or corresponding author or any of the other authors

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