J. Chem. Soc. Nigeria, Vol. 49, No. 6, pp 937 – 949 [2024]

SYNTHESIS AND CHARACTERIZATION OF NANOCRYSTAL CELLULOSE DERIVED FROM *Parkia biglobosa* POD

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ABSTRACT

The careless dumping of agricultural waste in Nigeria is a significant environmental issue that requires attention. Some states in Nigeria haphazardly dump the pod of Parkia biglobosa, a pod that should have served an essential purpose in the waste to wealth context. The study investigates the synthesis and characterization of nanocellulose derived from the Parkia biglobosa pod. Standard procedures were used to isolate nanocellulose and were characterised with FTIR, SEM-EDX, XRD, TGA, and DTG techniques. The FTIR analysis identified the existence of O-H, C-O-C pyranose rings, and cellulose β-glycosidic connections. The SEM showed a surface that was uneven and had small clusters of the NCC. The devolatilization of cellulose in the raw and NCC of ALBP was recorded at 43.205% and 71.90%, respectively, with the NCC exhibiting the highest value. At 380˚C and 400˚C, the thermal decomposition peaks for raw Parkia biglobosa and NCC caused the most weight loss. Nanocrystal cellulose has a higher crystallinity index than raw pod powder. Using EDX, the elemental analysis revealed the presence of raw C (49.21%) and O (50.79%), while the NCC contains C (46.03%) and O (53.93%). with some variations. Various treatments applied to the raw locust bean pod correlate with the change in percentage mass. Thus, the NCC might be employed as a reinforcing ingredient for the creation of green composites, binder, adsorbents, and polymeric polymers.

Keyword: Cellulose, Nanocrystal, Pod, Synthesis

INTRODUCTION

The worldwide effort to promote research and innovation in the renewable agriculture resources sector has facilitated the creation of novel, high-value lignocellulosic biomass products. A wide range of industrial products and non-food consumer items rely on cellulose as a valuable resource for their long-term growth [1]. Nature produces an extraordinarily high yearly output of cellulose, making it the most abundant organic material [2, 3]. Wood (which contains about 42%) and plant fibers (cotton, hemp, flax, and jute) are the primary sources of cellulose [4]. It is easily extracted from plants since it is insoluble in water and has other unique properties. Monomers, which are simple sugars, bind together very big molecules to form cellulose, a polymer [5, 6]. Cellulose, the most abundant natural polymer on Earth, primarily provides a

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plant's structural stiffness and strength. About one-third of the plant body contains cellulose, and natural processes generate one thousand metric tonnes of it annually [7, 8]. Biodegradability, biocompatibility, naturally renewable resources, and high availability have made cellulose a material of special interest to researchers and material developers [9, 10]. With these qualities, it could replace polymers made from petroleum, according to some proposals [11, 12].

Parkia biglobosa, more commonly known as the African locust bean, is a dicotyledonous angiosperm belonging to the Fabaceae family. According to Muhammed et al. [13] and Adedokun and AO [14], it is a member of the spermatophyte class of vascular plants. The northeastern and central regions of Nigeria revere this tree for its therapeutic properties [15]. The pod’s fibres, which include cellulose, hemicellulose, and lignin, make up 19% of the pod. Agricultural industries produce vast amounts of leftovers, which could potentially lead to the development of nanocrystalline cellulose (NCC) [16]. Glycosidic linkages connect the β-glucopyranose units that make up cellulose, a linear carbohydrate polymer. The NCC is crystalline because its structure resembles both rods and fibres [17]. Crystals are made up of hydrogen bonds that develop both within and between molecules [18]. Natural sources determine the length and characteristics of molecules [14]. The sizes of the particles range from 5 to 10 nm in width and 200 to 500 nm in length [19].

Some of the distinctive qualities that nanocrystalline cellulose displays include a high bending strength, a Young's modulus of about 150 GPa, a high aspect ratio, and a specific surface area. Because of this, it has found a place in reinforcing polymer matrices [20; 21]. The hydroxyl groups and other polar groups that cover the surface of NCC make it more reactive and better at absorbing water [22]. Cellulose nanoparticles can be used for many things, like stabilizing oil-water suspensions, strengthening, drug delivery, enzyme immobilization, and building blocks for tissue engineering [23, 24]. The most prevalent method for obtaining NCC involves acid hydrolysis of cellulose. Hydrolysis of the amorphous fraction using HCl and H2SO4 allows for the recovery of the crystalline portions using centrifugation [25]. Other documented mechanisms include enzymatic hydrolysis, ionic liquid therapy, and radiolysis [26].

Environmentalists need to address the important issue of careless dumping of agricultural waste, particularly fruit pods, in Nigeria [22]. Preventing accumulation, breakdown, or combustion, as well as maintaining a clean environment, are two major advantages of managing agricultural wastes, as highlighted by Adeleke et al. [27] and Nna et al. [15]. Pods of Parkia biglobosa can be hydrolyzed to yield NCC and cellulose, as described in this article. In addition to determining the elemental content and shape of

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NCC using SEM/EDX, the degradation temperature was evaluated using TGA, and the chemical composition and degree of crystallinity were assessed using FTIR.

MATERIAL AND METHODS

Materials

The natural Parkia biglobosa pod, potassium hydroxide (KOH), hydrochloric acid (HCl), ethanol, toluene, acetic acid, sodium chlorite, NaOH and other chemicals were analytical grade. All the chemicals were used as received. Deionized water was used in all experiments

Sample collection and Preparation

The material for Parkia biglobosa pod (locust bean pod) was sourced from different farms in Aku, Igbo-Etiti Local Government Area, Enugu State. The material was later transported to the department of Industrial Chemistry laboratory, University of Science and Technology, Enugu. The seeds in the locust bean pod were removed and the pods were carefully sorted to eliminate foreign materials from the sample. The locust bean pod were first washed with tap water and thereafter washed with distilled water to get rid of suspended impurities and then dried under the sun using wooden plank for about three weeks until constant weight. To increase the surface area and improve further treatment, the sun-dried pod samples were grounded into fine powder and sieved to particle sizes of 0.07 mm and kept in an airtight container for use.

Production /Isolation of Nano Particles from the Parkia biglobosa pod

About 100 g of Parkia biglobosa pod powder was added into 4 % w/v NaOH solution and digested at 80 0C for 3 hours using a thermostated hot plate. This removes lignin in the form of soluble complexes. The samples were then washed severally with distilled water and filtered using sieve number 40 mm mesh size. The samples were bleached with 10 % aqueous dilution of sodium hypochlorite for 30 minutes at 105 0C, washed and then filtered again. The samples were further treated with 17.5 % w/v sodium hydroxide at 80 0C for 1 hour and the resulting samples were washed thoroughly with water and subjected to bleaching process with 10 % sodium hypochlorite for 15 minutes at 80 0C. Finally, they were washed with water until neutral. The dried samples obtained were milled until very fine particles were obtained [22].

Characterizations of Nano crystal Cellulose of Parkia biglobosa pod

Scanning Electron Microscope

The morphological feature of the cellulose wasobserved using scanning electron microscope (SEM, FEI,Quanta 200, USA),transmission electron microscope(TEM, FEI, Tecnai G20, USA) at NARICT Zaria

Fourier-Transform Infrared

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Fourier transform infrared spectra were obtained from the FTIR-8400S spectrophotometer at National arbovirus research center Enugu using an ATR disc for untreated locust bean pod and acid-hydrolyzed locust bean pod samples.

X-ray Diffraction

XRD analysis was carried out using a Bruker D8 ADVANCE Powder XRD instrument with CuK-α radiation of λ = 1.5404 nm and the X-ray diffractometer was operated at a voltage of 40 kV and a current of 30 mA at University of Ibadan Research Center. The crystalline structures of cellulose samples were determined by XRD technique. XRD data were collected within the range of scattering angles (2 θ) of 10 to 40 ° at room temperature.

Crystallinity index (CrI) was calculated using the formula as stated in Equation 1:

$$Crl = \frac{(l200 - lam)}{l200} \times 100 \qquad 1$$

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

Thermogravimetric Analysis

TG/DTG curves were obtained using Seiko EXSTAR 6000 TG/DTA 6300 thermal analyzer at NARICT ZARIA. Approximately 10.2 mg of samples were placed on an aluminium pan for testing. This test was carried out from 30 to 900 °C in dynamic nitrogen atmosphere with the flow rate of 10 ml/min and heating rate of 10 °C/min.

RESULTS AND DISCUSSION

The process began with the removal of lignin and other compounds, such as proteins, by means of NaOH and heat. Natural fibre cellulose contains less lignin and hemicellulose after the bleaching process that leads to nanocellulose. This study found a similar pattern when it reran the tests that had previously been run to confirm the percentage composition of the presence of the main components of the Parkia biglobosa pod—namely, cellulose, lignin, and hemicellulose—after nanocellulose had been isolated. The increase in the crystalline fraction was caused by the removal of the amorphous content from the Parkia biglobosa pod. Liu et al. [28] found that when the percentage of crystalline cellulose is increased, thermal stability is also increased.

X-ray diffraction (XRD) study of Parkia biglobosa pod powder

The XRD diffractograms of raw Parkia biglobosa pod powder (PBP), and nanocrystals cellulose (CNC) is shown in Figure 1. The main reflective peak in raw pod and nanocrystals cellulose is centred on 2θ = 15.82o, 2θ = 22.48o and 34.63o corresponding to (110), (002) and (023) crystallographic planes of cellulose I polymorph thus indicating that CPC has typical cellulose I structure (Rosli et al., 2013). The aforementioned peaks in the raw pod is not pronounce, especially,

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the peak located 2θ =34.63o. This could be associated to the presence of high lignin, pectin and hemicelluloses in the raw pod. The NCC isolated by acid hydrolysis, shows three prominent peaks with at 2θ = 22.48o having the highest intensity, and a slight change in position, as well as increased intensity and narrowing of peaks. The amorphous background is characterized by low diffraction intensity around 2θ = 18o) [29]

The crystallinity index, CrI, of raw pod powder, and nanocrystals cellulose Parkia biglobosa were found to be 30.19% and 70.60% respectively. The presence of lignin in the fibre after chemical treatment and acid hydrolysis could be responsible for the low crystallinity of CPC and CNC obtained from XRD calculations [18]. This clearly shows that the crystallinity of the isolated NCC materials increases with progressive chemical treatments. This increase in crystallinity index is due to the removal of the amorphous constituents and the rearrangement of the crystalline regions into more ordered structure [22]. The progressive increase in CrI seen in this study is similar to the trend noticed during the isolation of CNC from mulberry bark [30], agave angustifolia [31], and oil palm trunk [32].

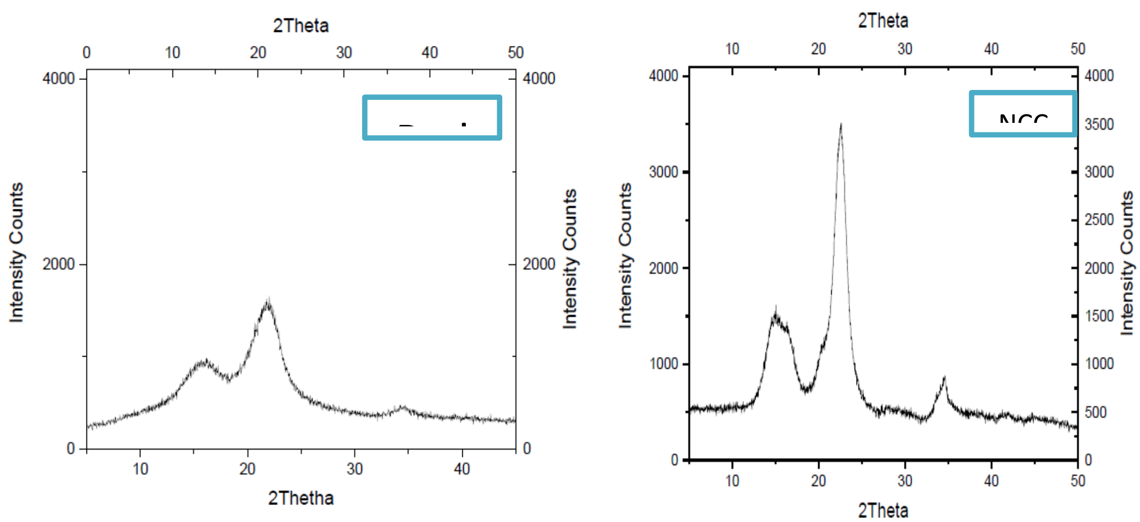

Figure 1: XRD diffractograms of raw PBP and nanocrystals cellulose

FTIR spectroscopy analysis of Parkia biglobosa pod powder

Figure 2 shows the FTIR spectra of the raw pod and nanocrystal cellulose (NCC). The raw pod's spectrum shows a band at 1744 cm−1. This is due to the stretching vibration of the acetyl carbonyl group of pectin, hemicelluloses, or the ester linkage of the carboxylic group of ferulic and p-coumaric acids of lignin and/or hemicelluloses [33, 34]. This band is absent in the spectra of CNC obtained after chemical treatment and acid hydrolysis, respectively. The alkali treatment cleaved all ester linkages of the hemicelluloses, as evidenced by the band's disappearance.

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However, this treatment does not affect the linkages between lignin and hemicelluloses [32]. The band at 1057 cm−1 seen in the raw and NCC spectra is caused by C-O-C pyranose stretching skeletal vibration, which proves that cellulose is present in all three fibres. Progressive chemical treatments increase the intensity of this band, indicating an increase in the crystallinity of the samples [31]. All of the samples have a band at 2920 cm−1 that is caused by the C-H stretching vibration, which shows that there are organic molecules in the samples. The bands at 854 cm−1 and 1624 cm−1 are caused by the C-H rocking vibration of carbohydrates and the O-H bending vibration of water. The broad band with maxima at 3359 cm−1 is due to the O-H vibration of the adsorbed water molecule [18, 35].

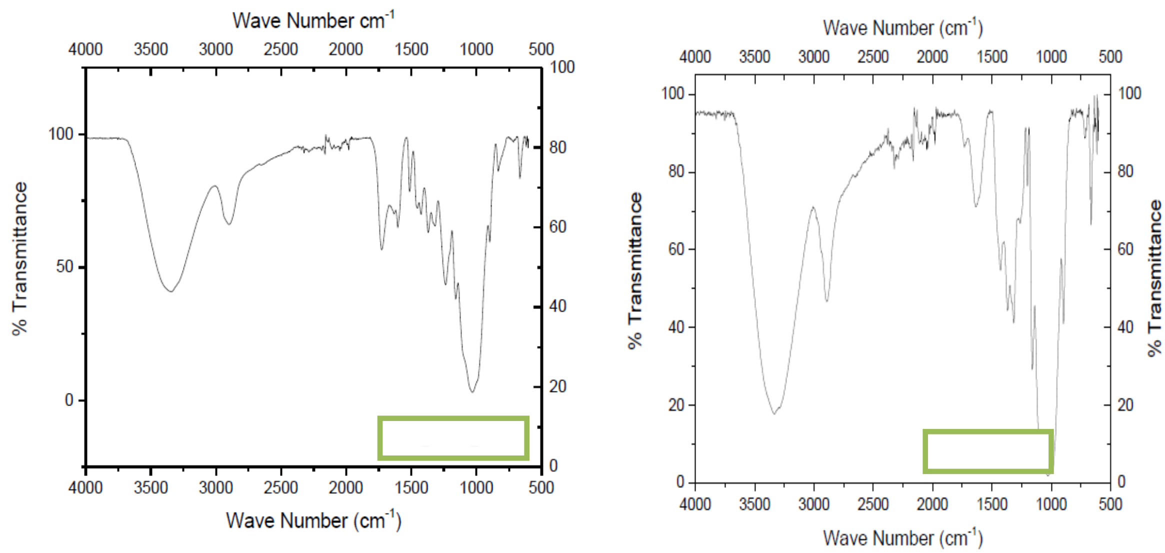

Fig. 2: FTIR spectra of cellulose and microcrystalline cellulose of *Parkia biglobosa* pod

Thermogravimetric analysis and derived (DTG) analysis

TGA was performed to check the thermal characteristics of the biomass raw pod and NCC of *Parkia biglobosa*. It also provides information about the amount of moisture content, volatiles and biomass composition. Raw pod and NCC thermogravimetric (TG) analyses of the African locust bean as a function of temperature is illustrated in Table 1,

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Table 1: Thermogravimetric Studies of Raw and NCC of African Locust Bean

Temperature (^{0}C)	Weight (%) of Raw	Weight (%)of NCC	Remark
80-150	5.013	3.014	Moisture
150-350	31.333	13.819	Devolatilization of hemicellulose
350-500	43.205	71.90	Devolatilization of cellulose
500 and above	4.718	6.950	Fixed carbon

The percentage weight loss of moisture from raw African locust bean within the temperature range of 80–1500C was estimated at 5.013%, and that of NCC was recorded at 3.014%. At the temperature of 150-350, the weight loss was recorded at 31.333% and 13.819% for raw and NCC. At the temperature of 350-500 ^{o}C, weight loss of 43.205% and 71.9% was estimated for raw and NCC. The active pyrolytic phase occurs at the temperature of 150-500 ^{o}C, known as the second phase of the thermal degradation stage. It involves the devolatilization of both hemicelluloses (150–350 ^{o}C) and cellulose (350–500 ^{o}C).

Both raw and NCC ALBP recorded fixed carbon estimates of 4.718% and 6.950%. The moisture content and devolatilization of hemicelluloses were for raw ALBP, whereas the devolatilization of cellulose was high for NCC. Researchers have proposed the vaporisation of adsorbed moisture on sample surfaces and the presence of chemisorbed and H-bonded water molecules within the samples as potential explanations for the observed weight drop in this region [31, 33, 34]. The residual hemicellulose in the pod or the volatilization of organic compounds with low molecular weights in the raw pod could potentially be the cause [35]. Monosaccharides (pentoses), primarily composed of anhydroxylose units, compose hemicellulose; their decomposition commences at low temperatures between 150 and 350 °C. Cellulose, being a crystalline polymer of high molecular mass, decomposes at high temperatures ranging from 350 to 500 °C [33, 36].

The breakdown of the remaining lignin starts at low temperatures and goes up to about 1000 °C. This makes about 4.718% and 6.950% of carbon residue and inorganics in raw and NCC, respectively. The decomposition of residual lignin and other components occurred at over 400 °C. Compared to hemicellulose and cellulose, lignins, composed of three types of phenylpropane units with high molecular mass and higher thermal stability, decompose at a slower rate [33, 37, 38]. Additionally, Figure 3 illustrates the DTG curves, which show the maximum weight loss of the thermal decomposition peaks for raw ALBP and CNC, respectively, at 380 oC and 400 oC. It indicates that the NCC samples possess higher thermal stability as compared to raw ALBP.

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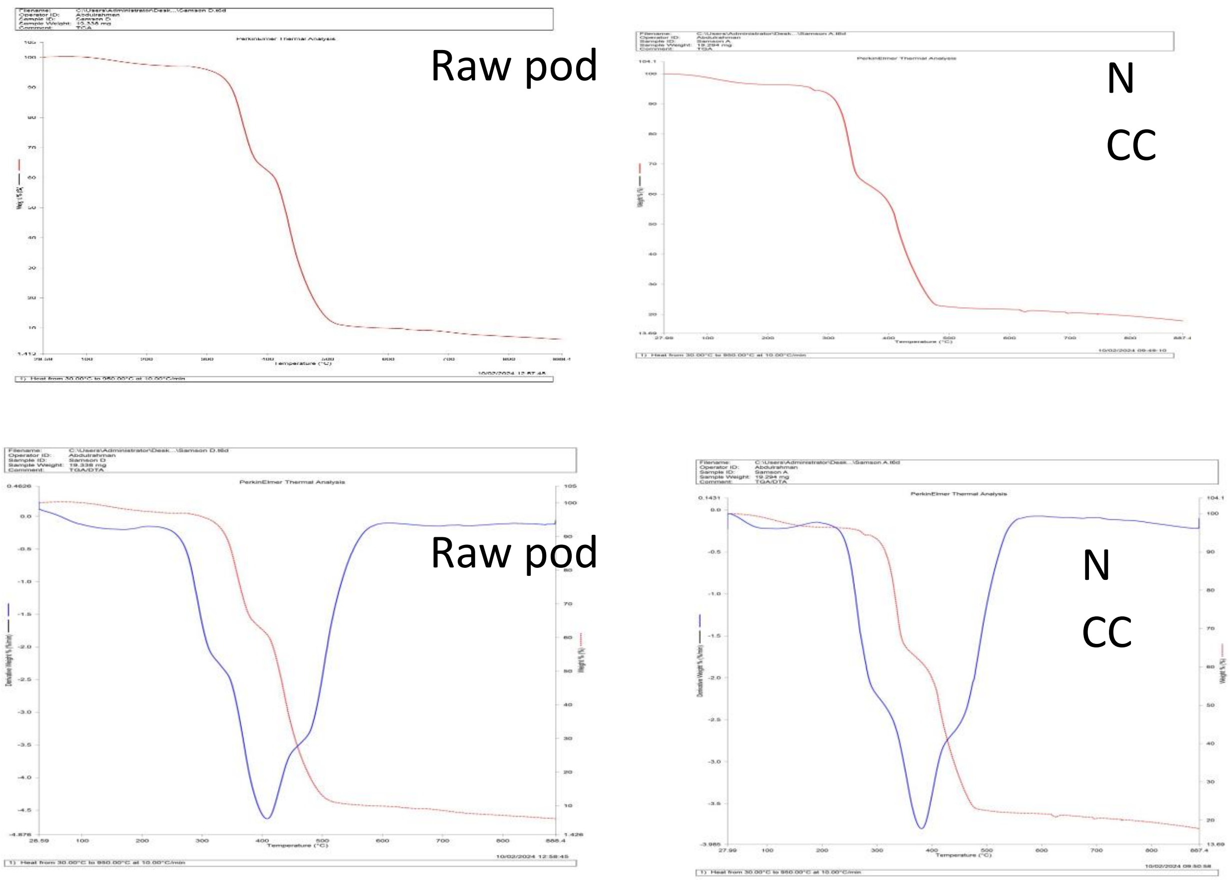

Figure 3: Thermogram curves of raw pod and NCC of Parkia biglobosa pod

Scanning Electron Microscopy (SEM)/Electron diffraction X-ray (EDX)

The morphology of the ALB biomass at different stages of the chemical treatment was investigated by scanning electron microscopy (SEM) and micrograms (Figure 4). Progressive chemical treatments lead to a noticeable change in the morphology of the NCC in ALBP. The untreated sample of the locust bean pod has a rough surface because it contains non-fibrous components like lignin, hemicellulose, wax, pectin, and oil. Following treatment, Fig. 3 clearly shows the removal of hemicellulose and lignin. An acetate buffer/(NaCIO2) solution treatment of walnut (Juglans regia) shell produced a similar result [35].

Furthermore, EDX analysis of both raw and nanocrocrystal cellulose indicated the presence of several elements and their weight concentrations. Some of the components detected in the cellulose had quantitative percentages of C (49.21%) and O (50.79%). The elemental analysis of NCC revealed the existence and quantity of the following elements: C (46.03%) and O (53.93%).

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The fluctuations in the percentage of elements may be attributed to successful chemical treatment, which transforms them into cellulose nonocrocrystals. These elements are easily metabolized due to their organic source [22, 31, 32].

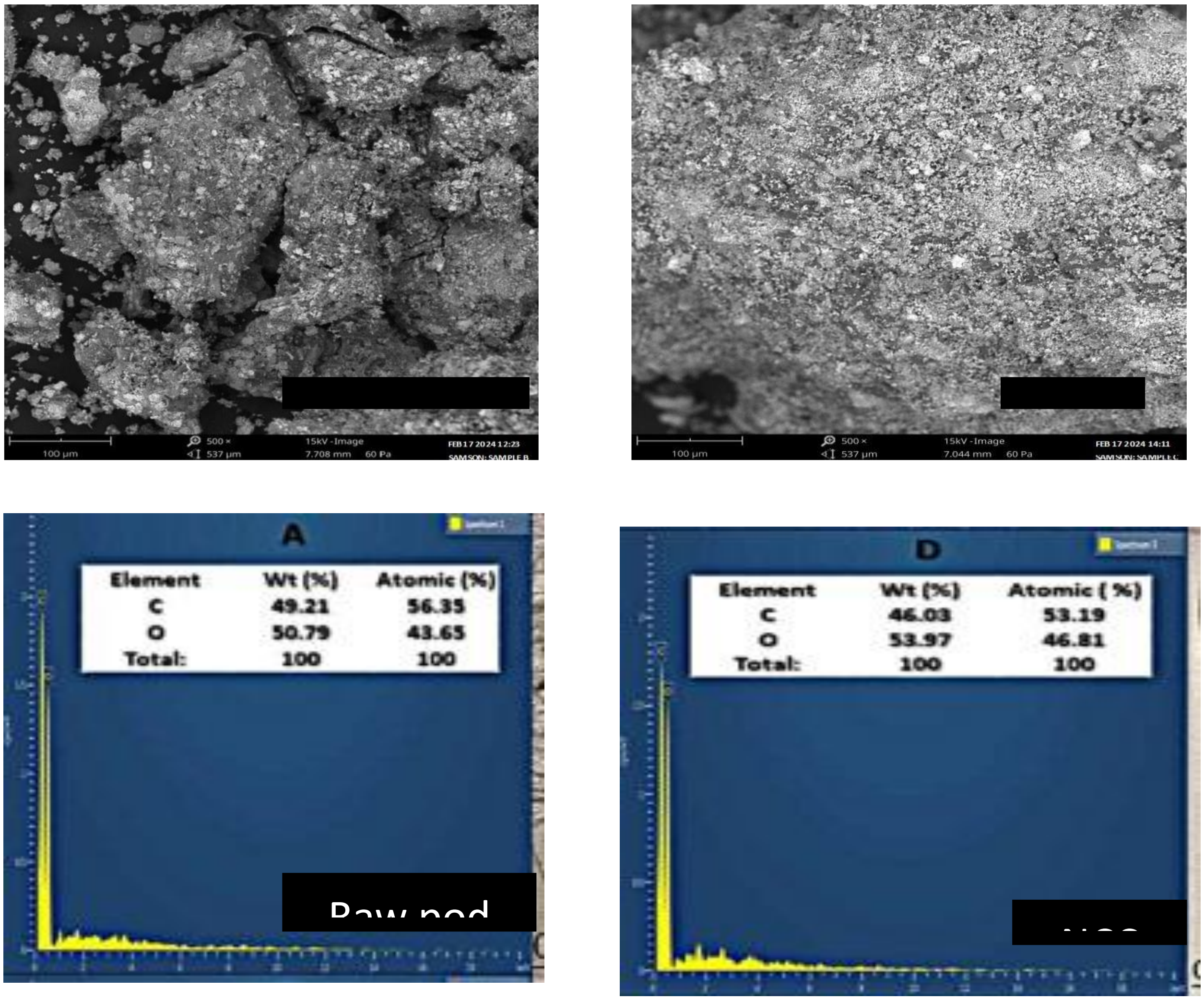

Figure 4: Micrographs Analysis of Raw and NCC of Parkia biglobosa pod

CONCLUSION

The study explains the synthesis and characterization of nanocrocrystal cellulose from locust bean pod biomass (waste) using de-waxing, bleaching, and acid hydrolysis. The FTIR, SEM-EDX, XRD, TGA, and DTG techniques were employed to analyse and describe the properties of both raw and nanocrocrystalline cellulose.

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The FTIR analysis identified the existence of O-H, C–O–C pyranose rings, and cellulose β-glycosidic connections. The SEM showed a surface that was uneven and had small clusters of the NCC. The cellulose devolatilization NCC was higher than that of the raw. At 380˚C and 400˚C, the thermal decomposition peaks for raw ALBP and NCC caused the most weight loss. Nanocrystal cellulose has a higher crystallinity index than raw pod powder.

The elemental analysis, conducted using EDX, identified the following elements in the raw C (49.21%) and O (50.79%), as well as in the NCC: C (46.03%) and O (53.93%). with some variations when compared to cellulose. Various treatments applied to the raw locust bean pod correlate with the change in percentage mass. Thus, the NCC might be employed as a reinforcing ingredient for the creation of green composites, binder, adsorbents, and polymeric polymers.

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