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Isolation And Characterization Of Nanocellulose From Corncobs

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Abstract
In this research work, cellulose was isolated from corn cobs, a solid agricultural waste and used to prepare nanocellulose which was characterized using SEM, TGA, XRD and FTIR. The results from SEM analysis showed that the nanoparticles from this source are amorphous with porous surfaces. The TGA showed that the nanoparticles decomposed on heating in three phases; the zone of loss of volatile organic compounds, the zone of loss of water of crystallization and the zone of decomposition of the nanocellulose. The XRD showed two major diffraction peaks at $2\theta = 16^o$ and 22^o with corresponding indices of (101) and (002) respectively. The functional groups in nanocellulose were further investigated by FTIR spectroscopy. The peak at 3457 cm^{-1} was attributed to stretching of the O-H band, peak at 1712 cm^{-1} showed the aliphatic saturated C–H stretching, the band at 1632 cm^{-1} shows the C=C stretching, the peak at 1387 cm^{-1} was assigned to O=S=O symmetric stretching vibration and the peak at 600 cm^{-1} indicates C–O stretching of nanocellulose. The result showed that nanocellulose was successfully synthesized from corn cobs.
***Keywords**: Corncobs, Nanocellulose, Solid waste, Isolation and Cellulose*

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I. Introduction

Agricultural activities produces many bio-wastes such as rice husk, sugar cane bagasse groundnut shells and corncobs. This produced bio-waste can be utilized as a potential source of biomaterials. However, the bio-waste is most times disposed into the environment to biodegrade (Mullen et al., 2010; Sartika et al., 2023). Among this biowaste, corn is one of the highest produced in Nigeria with over 12 million metric tons produced in 2021 (Onumah et al., 2021; Oyeleke et al., 2012). In Nigeria, most factories sort out corn seeds for further processing, leaving out corn cobs as industrial waste. Biodegradation of corn cobs by microorganisms takes a long time and release gases into the environment. Incineration is simple and fast, however, it releases greenhouse gas (CO_2) into the atmosphere which can cause global warming. Moreover, smoke from the process can also cause direct effect to public health (Abrahama, *et al.,* 2013). Due to these reasons, the corn industry ineffective waste disposal is causing a great concern due to environmental pollution.

A suitable process to dispose or convert corn cobs to valuable product is crucial in curtailing its effects on the environment. The large quantities of corn cob found in Nigeria, have a great potential to be employed as useful renewable source of energy through gasification and can also be isolated as cellulose (Oyeleke et al., 2012). The conversion of corn cobs into an energy carrier gas known as syngas through gasification is a viable alternative to electricity generation needed to meet the ever-escalating energy demands of remote settlements (Rabah *et al.,* 2011). However, the technology of gasification and the complex process makes it not viable for adoption in developing countries such as Nigeria. Therefore, the facile method involved in isolation of cellulose from corn cobs makes it's a viable process of waste treatment and biomaterial production which can be used in many applications.

Cellulose is a semi crystalline polycarbohydrate composed of anhydroglucose units (AGUs) linked chemically by β-1, 4-glycosidic bonds. It is found in abundance in the tissues of plant materials. It is usually extracted by different methods such as acid or base hydrolysis to breakdown the complex structures to simpler ones (Theivasanthi et al., 2018; Wang et al., 2019). Nanocellulose are produced by further breakdown of the cellulose material into nanoscale. Nanocellulose are low weight, biodegradable, thermally stable and non-hazardous materials that are highly reactive due to high surface tension, large surface area and broad surface functionality. These properties combined makes it highly promising in different applications such as water treatment, chemical industries, biomedical and pharmaceutical industries (Adejumo et al., 2020; Chan et al., 2015).

Therefore, this research is aimed at isolation of cellulose from corn cobs and converting to nanocellulose. The nanocellulose produced would be characterized using analytical techniques such as scanning electron microscopy (SEM), Thermogravimetric analysis (TGA), X-ray diffraction (XRD) and Fourier Transformed infrared Spectroscopy (FTIR).

II. Methodology

Sample collection and preparation

Corn cobs were collected from a local farm in Makurdi, Benue State, Nigeria. The cobs were washed with tap water to remove impurities and sundried for two weeks after which it was rewashed using distilled water and dried. The dried cobs were grounded into powder and stored for further analysis. Exactly 50 g of dried corn cobs powder was dissolved in 500 mL of 4 % NaOH solution and stirred at 80 °C for 3 hours. This gave a brownish yellow solution which was cooled and then washed with distilled water for 72 hours to pH of 7.0. The sample was filtered and dried at 60 °C for 2 hours.

Bleaching

Bleaching of the sample was done using sodium tetraborax solution by immersion. The sample was immersed in 250 mL of 4 % sodium borax solution and heated at 70 °C for 3 hours with continuous stirring. The bleaching process was repeated three (3) times and the bleached fibers were filtered and cleaned in each cycle with distilled water till the pH becomes neutral. Bleached fibers were dried at 45 °C for 12 hours in a hot air oven. The majority of lignin was removed during the bleaching process.

Acid Hydrolysis

The bleached fibers were soaked in 250 mL of 20 % H_2SO_4 solution at 40 °C on the hot plate for 1 hour. About 500 mL distilled water was added to the solution to stop the reaction. After the acid hydrolysis, the acid-treated mixture solution was centrifuged at 2500 rpm for 24 minutes and washed with distilled water until the pH became neutral. The suspension was sonicated for 2 hours at 45 °C to improve the texture and breakdown the cellulose into nanocellulose, after which it was dried and crushed into fine powder then stored in sample bottles for characterization (Huntley *et al.,* 2015).

Characterization of the synthesised nanocellulose

SEM analysis

SEM analysis was done using SEM machine to determine the morphological features of the synthesized nanocellulose. The films of the sample were prepared on a carbon coated copper grid by just dropping a very small amount of the sample and the extra solution was removed using a blotting paper. The film on the SEM grid was allowed to dry by putting it under a mercury lamp for 5 minutes. The sample was run in the SEM instrument at an accelerating voltage of 15 kV.

Thermogravimetric analysis (TGA)

The TGA was conducted on an instrument referred to as a thermogravimetric analyzer. The thermogravimetric analyzer continuously measured the mass loss as the temperature of the sample changes over time. Thermal stability of the sample was analyzed using a thermogravimetric analyzer with a STARe software (version 9.01). Sample was heated from 98 - 332 °C at a rate of 5 °C/min under nitrogen gas flowing at 30 mL/min.

X-ray diffraction analysis (XRD)

Powdered samples were pelletized and sieved to 0.074 mm. This was dropped on an aluminum alloy grid (35 mm x 50 mm) glued to a flat glass plate and covered with a paper. Wearing hand gloves, the sample was compacted by gently pressing it with hand. Each sample was run through the Rigaku D/Max-lllc X-ray diffractometer developed by the Rigaku Int. Corp. Tokyo, Japan and set to produce diffractions at scanning rate of 2 ^{0}/min in the 2 to 50^0 at room temperature with a Cu kα radiation set at 40 kV and 20 mA. The diffraction data (d value and relative intensity) obtained was compared to that of the standard data of minerals from the mineral powder diffraction file

FTIR analysis

The chemical functional groups of the synthesized cellulosic nanoparticles was studied using FTIR spectrometer. The samples data was collected at a wavenumber range of 4000 cm^{-1}- 400 cm^{-1} at an interval of 2 cm^{-1}.

III. Results And Discussion

In this research work, nanocellulose were synthesized from corn cobs using acid hydrolysis. From Figure 1, it can be seen that the nanoparticles are amorphous with porous surfaces. The SEM micrographs of the corn cobs nanocellulose are similar with that reported by Asrofi *et al.,* (2017).

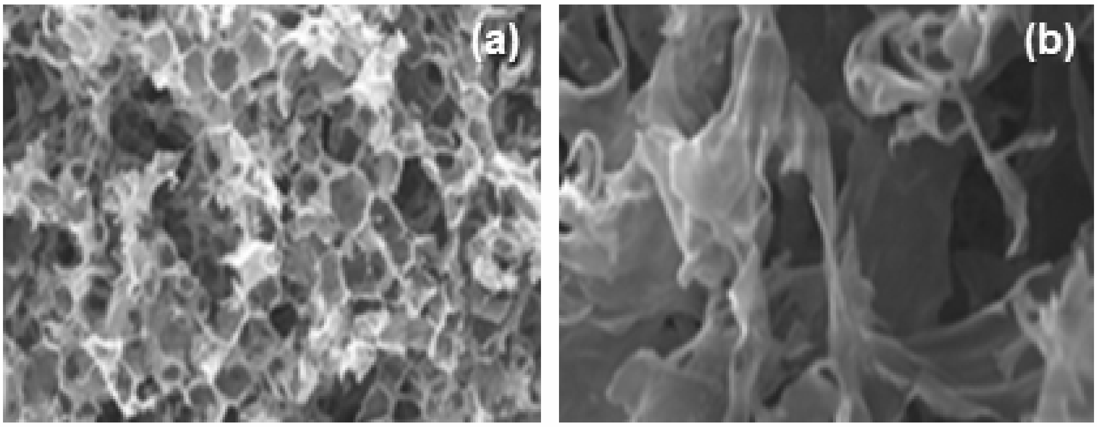

Figure 1. The SEM image of cellulose nanoparticle produced from corn cobs.

Thermogravimetric analysis (Figure 2) was carried out to obtain information on the thermal stability of the synthesized cellulose nanoparticles. The TGA is divided into three regions. It can be seen that the initial weight of the sample was 99.20 % and decreases by about 24 % at the temperature range of 98 – 250 °C (first region-horizontal). This signifies the combustion of volatile organic molecules. The second region (vertical fall) that decreases by about 68.50 % at a narrow temperature range of 250 – 255 °C indicates the rapid decomposition of the organic groups derived from the synthesis process. Similar observation has been reported by Cao *et al*., (2019). The last region (horizontal) indicates residual particles of the nanocellulose after decomposition.

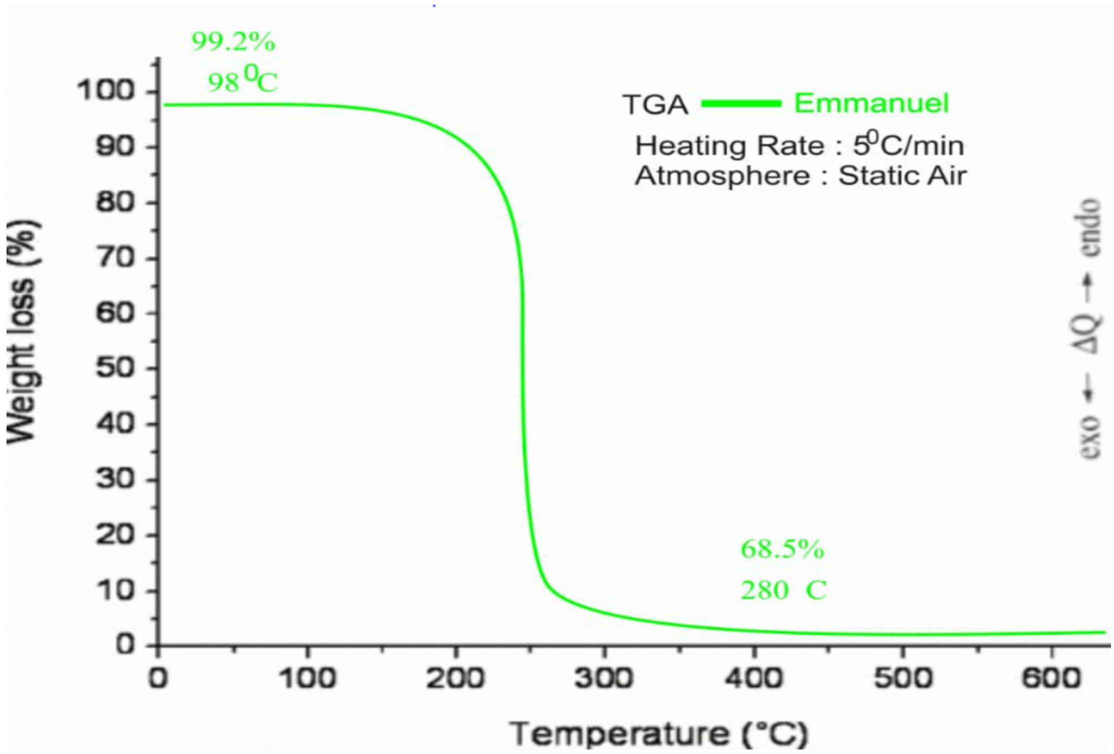

Figure 2: Thermogravitric Analysis (TGA) of cellulose nanoparticles

The XRD analysis performed on the nanocellulose showed the formation of crystals with two major diffraction peaks at 2θ = 16º and 22º and corresponding to crystallographic planes of (101) and (002) respectively (Figure 3). Ford *et al.,* (2010), reported XRD peaks about 2θ =16 º, 22º, and 35º and deduced that the extracted nanocellulose crystals adopt a crystalline structural formation. The broad nature of the peak indicates the nanoscale nature of the prepared nanocellulose.

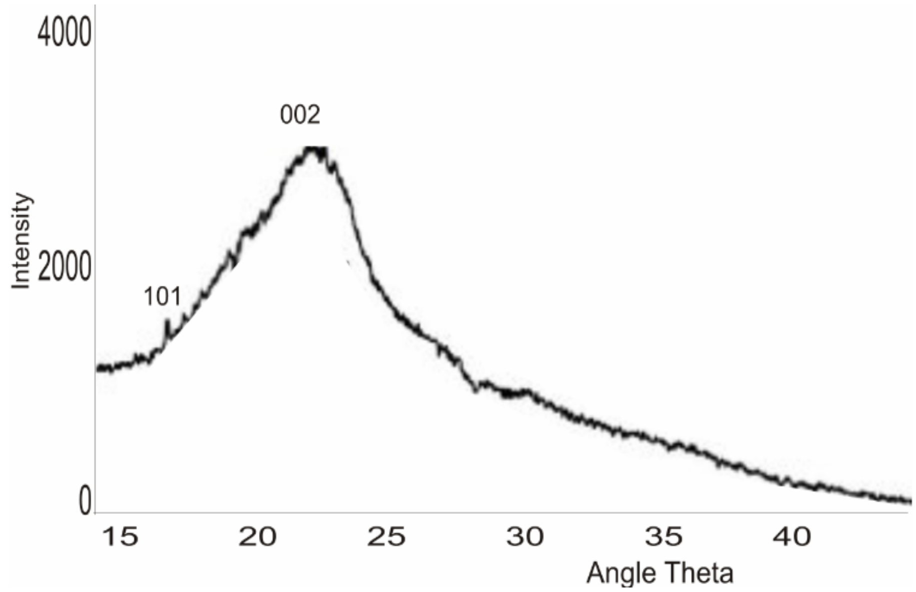

Figure 3: X-ray diffraction (XRD) of corn cobs synthesised cellulose nanoparticles

The Fourier Transform-Infrared Spectroscopy (FT-IR) was used to study the chemical functional groups present in the nanocellulose. The graph of the FTIR is shown in Figure 4 and the specific vibrational modes and wavenumber is presented in Table1.

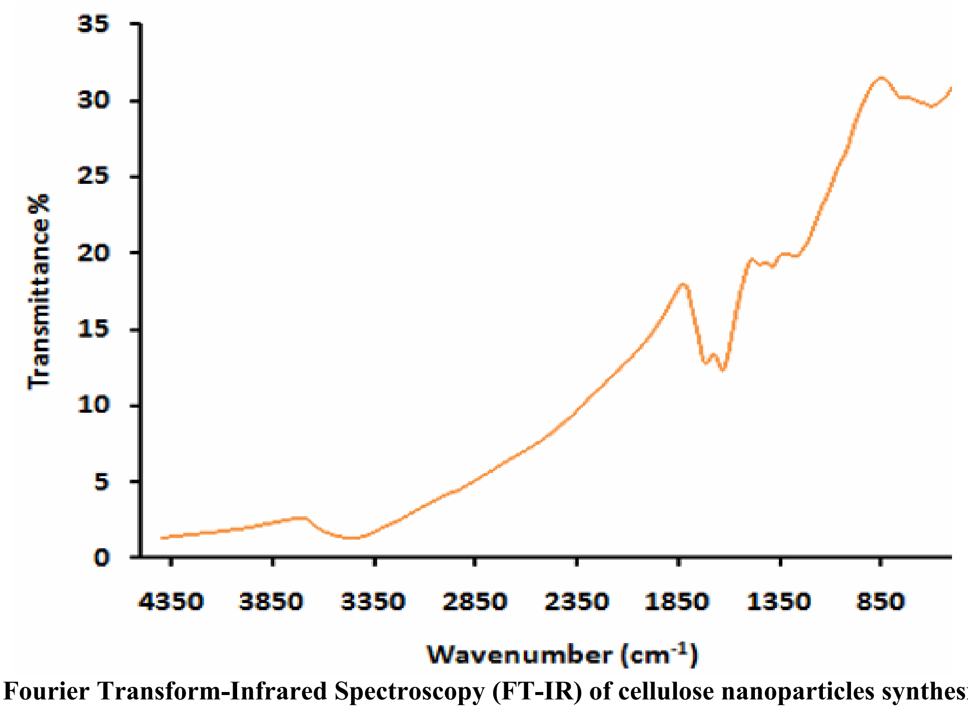

Figure 4: Fourier Transform-Infrared Spectroscopy (FT-IR) of cellulose nanoparticles synthesised from corn cobs

Table 1: Wavelengths of the main bands obtained from cellulose nanoparticles

Vibrational mode	Wavenumber (cm^{-1})
CO stretching	600
O=S=O stretching	1387
C=C vibration	1632
C-H vibration	1712
OH stretching	3457

From the spectral, a broad band was observed at 3457 cm^{-1} which can be attributed to the O-H stretching, which are due to OH groups attached to the benzene ring from glucose Units. It could also be as a result of loosely attached water molecule on the surface of the nanocellulose. Similar result was also observed by Plazonic *et al.,* (2016), and Johar *et al.,* (2012). The peaks at 1712 cm^{-1} showed the presence of aliphatic saturated C–H stretching in nanocellulose. A similar peak was also observed by Huntley *et al.,* (2015) in cellulose prepared from wheat straw. The band at 1632 cm^{-1} is attributed to the C=C stretching while the peak at 600 cm^{-1} indicates C–O stretching. The peak at 1387 cm^{-1} was assigned to O=S=O symmetric stretching, this peak showed the presence of SO_3H group from acid hydrolysis treatment. Jianguo *et al.,* (2007) in their work on "Facile synthesis of spherical cellulose nanoparticles" reported a similar infrared vibrational bands.

IV. Conclusion

In the present research, nanocellulose was synthesized from corn cobs using acid hydrolysis. The synthesised nanocellulose was characterized using SEM, TGA, XRD and FTIR. It was found to be amorphous with porous surfaces as shown in the SEM images and the thermal decomposition was observed from the TGA spectrum to have occurred in three stages. They nanocellulose showed two crystallographic peaks at $2\theta = 16^o$ and 22^o characteristic of nanocellulose. The IR spectra showed the key functional groups found in the nanocellulose. The findings therefore indicates that nanocellulose can be synthesized from corn cobs.

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