

Synthesis And Characterization Of Cellulose Nanoparticles From Carica Papaya Barks

Oshido B. A^a., Ubwa S. T.^A, Aondoaver S. A.^A And Oshido L. E^b.

¹department Of Chemistry, Benue State University, Pmb 102119 Makurdi. Benue State, Nigeria.

Becky Gyuse College, Makurdi Benue State, Nigeria.^B

Abstract

In this research work, cellulose nanoparticles (CNPs) were synthesized from *C. papaya* barks and characterized using SEM, TGA, XRD and FTIR. The analysis revealed a highly textured surface morphology characterized by closely packed spherical structures similar to carbon fullerene with notable empty cavities. The nanoparticles exhibit remarkable stability, retaining 99.7 % of its weight up to approximately 98 °C but degrade up to 78.90 % at 320 °C with a residual weight of 10.40 %. XRD of the nanoparticles showed two major diffraction peaks at $2\theta = 22^\circ$ and 35° , corresponding to crystallographic indices of (002) and (110) respectively. The chemical nature of surface functional groups in CNP was further investigated by FTIR spectroscopy. Peak at 3437 cm^{-1} was attributed to stretching of the O-H band, the peaks at $2919\text{-}2356\text{ cm}^{-1}$ show the aliphatic saturated C-H stretching vibration, the band at 1604 cm^{-1} shows the C=C stretching, the peak at 1373 cm^{-1} was assigned to O=S=O symmetric stretching vibration and the peak at 1034 cm^{-1} indicates C-O stretching of cellulose nanoparticles. These findings compared well with literature and indicate that cellulose nanoparticles can be synthesized from *C. papaya* barks. The research findings showed the potential for utilization of the *C. papaya* barks for synthesis of CNPs which will reduce the risk of pollution posed by this biomass.

Keywords: Cellulose, *Carica papaya*, nanoparticles, biomaterial and biomass.

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I. Introduction

Particles in nanoscale dimensions have high surface area to volume ratio which contributes to enhance chemical activity, enhanced Rayleigh scattering and surface enhanced Raman scatterings compared to their bulk materials (Banica, 2012). These are particles of materials whose size ranges from one nanometre to hundred nanometres (1-100 nm) (Ankamwar, *et al.*, 2005). In recent years, the synthesis of nanoparticles using natural sources has garnered significant attention due to its eco-friendly nature and potential applications in various fields including medicine, catalysis, and environmental remediation (Dufresne, 2017). These materials from natural sources are commonly called biomaterials and abound in nature and also have applications in bioremediation, water treatment and biomedical field (Jana *et al.*, 2022). Among its myriad applications, cellulose nanoparticles (CNPs) have emerged as a promising frontier, owing to their biocompatibility, renewable sourcing, and versatile properties (Hubbe *et al.*, 2008). Derived from abundant natural resources such as wood pulp and agricultural residues, CNPs offer a sustainable alternative to conventional nanomaterials (Iwamoto *et al.*, 2011). Their ease of synthesis through environmentally benign methods further enhances their appeal, promising scalable production without compromising ecological balance.

Among the plethora of natural resources explored for nanoparticle synthesis, *Carica papaya* (papaya) has emerged as a promising candidate. The *papaya* tree, native to tropical regions, is not only celebrated for its succulent fruit but also for its versatile bark which has been traditionally utilized in folk medicine for its pharmacological properties (Mariano *et al.*, 2014). The bark of *Carica papaya* is rich in bioactive compounds such as alkaloids, flavonoids, and phenolics, which possess inherent reducing and stabilizing capabilities essential for nanoparticle synthesis. Nanoparticles synthesized from plants have shown enhanced biocompatibility and reduced toxicity compared to their chemically synthesized counterparts, making them highly attractive for biomedical applications (Elazzouzi *et al.*, 2008). Furthermore, the use of *papaya* bark for nanoparticle synthesis aligns with the principles of green chemistry, offering a sustainable alternative to conventional methods that often involve hazardous chemicals and generate toxic by-products.

Synthesis of CNPs from cellulose sources involves controlled degradation and modification processes, optimized to yield nanoparticles with precise dimensions and tailored surface chemistries. This facile synthesis route not only ensures cost-effectiveness but also minimizes environmental impact, aligning with sustainable development goals. Furthermore, the scalability of CNP production from cellulose sources underscores their potential to meet the escalating demand for high-performance nanomaterials without straining natural resources

(Kimura and Itoh, 1996). By harnessing their exceptional mechanical strength, high surface area, and functionalization capabilities, CNPs hold promise in enhancing drug delivery systems, reinforcing biocompatible polymers, and developing sensors for environmental monitoring (Huntley *et al.*, 2015). Moreover, their compatibility with biological systems underscores their potential in biomedical applications, ranging from tissue engineering to targeted drug delivery, offering novel solutions to pressing global health challenges.

Thus, this research endeavors to elucidate the burgeoning prospects of CNPs in the realm of nanotechnology, underscoring their synthesis feasibility from biomass like *papaya* barks, characterization and expansive application potentials.

II. Methodology

Sample Collection

Carica papaya barks were collected at a local market in Makurdi and taken to the Department of Chemistry, Benue State University. The barks were washed with tap water to remove impurities and sundried for two weeks after which they were rewashed using distilled water and dried. The dried barks were pulverized into powder using mortar and pestle and stored for further analysis.

Alkaline Treatment: Exactly 50 g of dried *papaya* bark powder was measured into 500 mL beaker and 300 mL of 4 % (w/v) of sodium hydroxide solution was added and stirred at 80 °C for 3 hrs. The mixture was cooled and then washed with double distilled water till the pH of the solution became neutral. The mixture was filtered and dried for further treatment.

Bleaching: After the alkali treatment, the powder was bleached with 5 % (w/v) disodium tetraborate decahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) solution at 70 °C. The bleaching process was repeated three (3) times. The bleached fibers were filtered and cleaned in each cycle with double distilled water till the pH becomes neutral. The fibers was then dried at 45 °C for 12 hrs in a hot air oven. The bulk of lignin was removed during the bleaching process.

Acid Hydrolysis: The bleached fibers were soaked in 20 % (w/v) sulphuric acid solution at 40 °C on the hot plate for 1 h. Exactly 300 mL distilled water was added to the solution to stop the reaction. After the acid hydrolysis, the acid-treated mixture was centrifuged at 4000 rpm for 15 min and washed with double distilled water until the pH becomes neutral. The suspension was sonicated for 2 hrs at 45 °C to improve the texture of cellulose after which it was dried and crushed into fine powder and then stored in sample bottles for characterization (Huntley *et al.*, 2015).

Characterization of the Synthesized Cellulose Nanoparticles

Characterization of the synthesized nanocellulose was done to confirm their synthesis and to check some of their characteristics.

SEM analysis: Scanning electron microscopy (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 removed using a blotting paper and then film on the SEM grid were allowed to dry by putting it under a mercury lamp for 5 min. the sample was characterized in the SEM at an accelerating voltage of 15 kV.

Thermogravimetric analysis (TGA): Thermogravimetric analysis (TGA) was conducted on a thermogravimetric analyzer. A thermogravimetric analyzer continuously measures mass loss while the temperature of a sample is changed over time. Mass, temperature, and time are considered base measurements in thermogravimetric analysis while many additional measures may be derived from these three base measurements. Thermal stability of the sample was analyzed using a thermogravimeter 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. These were later taken in an aluminum alloy grid (35 mm x 50 mm) on a flat glass plate and covered with a paper. Wearing hand gloves, the samples were compacted by gently pressing them with the hand. Each sample was run through the Rigaku D/Max-IIIc X-ray diffractometer developed by the Rigaku Int. Corp. Tokyo, Japan and set to produce diffractions at scanning rate of 2^o/min in the 2 to 50^o at room temperature with a $\text{CuK}\alpha$ radiation set at 40 kV and 20 mA.

FTIR analysis: The chemical functional groups of the synthesized cellulosic nanoparticles was studied using FTIR spectrometer. The samples were characterized in the range of 4000 cm^{-1} - 400 cm^{-1} .

III. Results And Discussion

The synthesis and characterization of cellulose nanoparticles using *Carica papaya* barks was done and the results presented in the figures and table below.

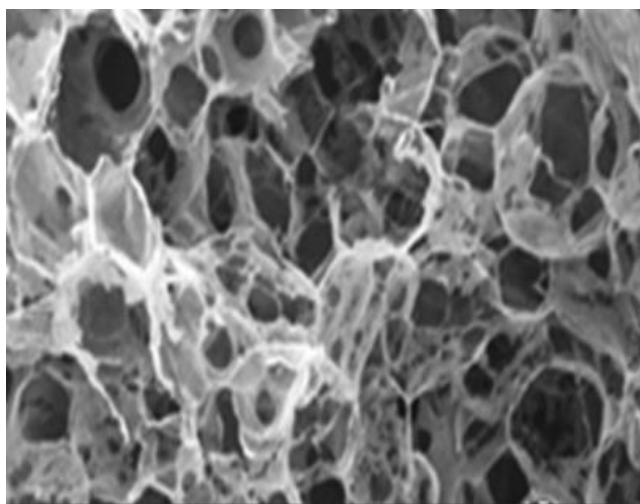


Figure 1.0: Scanning electron microscopy (SEM) of cellulose nanoparticles

Scanning electron microscopy (SEM) was used to study the surface characteristics of the synthesized cellulose nanoparticles. The SEM micrograph revealed a highly textured surface morphology characterized by closely packed spherical structures similar to carbon fullerene with notable empty cavities (Figure 1.0). These complex structures suggest the nanoparticles have a significant surface area, which is advantageous for applications in adsorption technology, encapsulation technology as carrier agents in composite delivery and catalysis (Asrofi *et al.*, 2017).

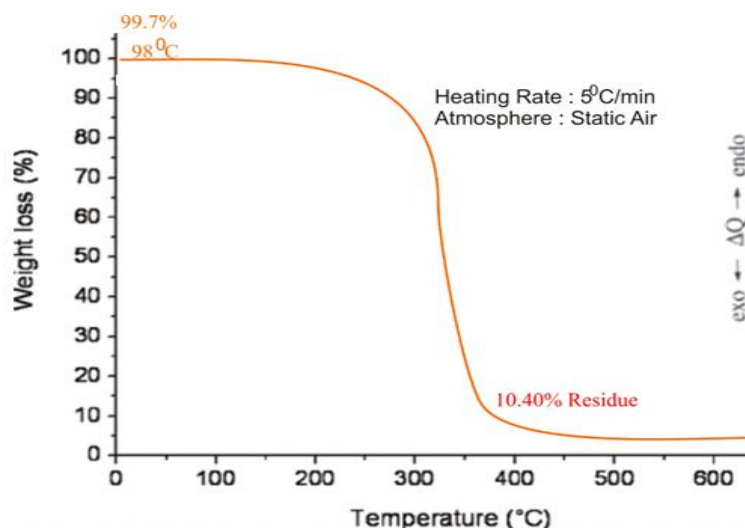


Figure 2.0: Thermogravimetric Analysis (TGA) of cellulose nanoparticles

Thermogravimetric analysis was carried out to obtain information on the thermal stability of the synthesized cellulose nanoparticles with increasing temperature. The plot provides a comprehensive overview of how the material responds to increasing heat. Initially, the nanoparticles exhibit remarkable stability, retaining 99.7 % of its weight up to approximately 98 °C (Figure 2.0). This suggests minimal presence of moisture that would evaporate at lower temperatures, making nanoparticles suitable for applications requiring thermal stability within this range. A significant thermal event is observed starting at around 320 °C and continuing up to approximately 365 °C, indicating a phase of major decomposition of the materials. This signifies the combustion of volatile organic components within the material with liberation of small molecules like CO₂ (Cao *et al.*, 2019). The rapid weight loss during this phase suggests a substantial breakdown of the nanoparticles structures under increasing temperature, with the endpoint marking the completion of significant thermal decomposition processes (Cheng *et al.*, 2010). This phase highlights critical temperature thresholds that impact the material's thermal

stability and degradation characteristics. The residue analysis after complete thermal processing reveals a remaining 10.40 %, indicating that 89.60 % of the sample decomposed or vaporized above 365 °C, leaving behind non-volatile components like ash or stable inorganic materials. This detailed understanding from TGA analysis is important for material scientists and engineers involved in optimizing and developing materials for applications requiring robust thermal performance and stability.

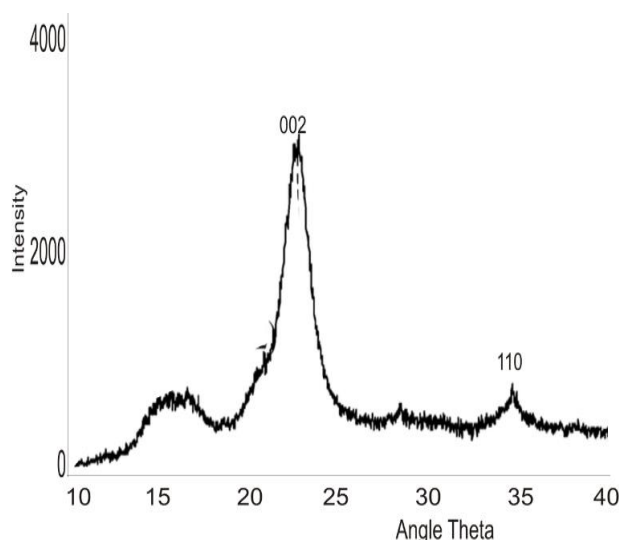


Figure 3.0: X-ray diffraction (XRD) of cellulose nanoparticles

X-ray diffraction (XRD) pattern is a fundamental tool for analyzing the crystalline structure of materials. The graph illustrates the relationship between intensity and the angle theta (2θ), where each peak signifies a specific atomic plane within the crystal lattice that diffracts X-rays constructively. The X-ray powder diffraction (XRD) analysis performed on the CNPs pointed out amorphous structure of the nanoparticles with few crystals diffracting X-rays at $2\theta = 22^\circ$ and 35° , corresponding to crystallographic planes of (002) and (110) respectively (Figure 3.0). The result is similar with Johar *et al.*, (2012) who reported XRD peaks of nanocellulose at $2\theta = 16^\circ$, 22° , and 35° from rice husk. XRD pattern provides crucial insights into the structural properties of nanoparticles. Sharp and distinct peaks suggest a high degree of crystallinity, in contrast to broad peaks reported here which would indicate an amorphous structure (Klemm *et al.*, 2005).

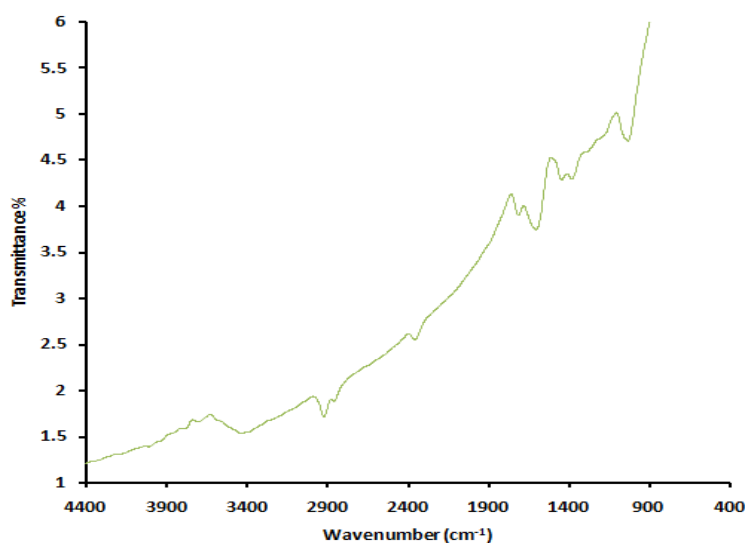


Figure 4.0: Fourier Transform-Infrared Spectroscopy (FT-IR) of cellulose nanoparticles

Table 1.0: Wavelengths of the main bands obtained from cellulose nanoparticles

Vibrational mode	Wavenumber (cm ⁻¹)
CO stretching	1034
O=S=O stretching	1373
C=C vibration	1604

C-H vibration	2919
OH stretching	3437

The functional groups in the CNPs were further investigated by FTIR spectroscopy (Figure 4.0) and the vibrational modes of main bands are outlined in Table 1.0. Peak at 3437 cm⁻¹ was attributed to stretching of the O-H band, which arises from anhydroglucopyranose unit of cellulose. Similar results have been observed by Plazonic *et al.*, (2016). The peaks at 2919-2356 cm⁻¹ show the aliphatic saturated C-H stretching vibration and similar results have been observed by Huntley *et al.*, (2015). The band at 1604 cm⁻¹ was assigned to C=C stretching. The peak at 1373 cm⁻¹ was assigned to O=S=O symmetric stretching vibration, this peak shows the presence of SO₃H group arising from acid hydrolysis treatment. The peak at 1034 cm⁻¹ indicates C-O stretching of nanocellulose (Ciolacu *et al.*, 2011).

IV. Conclusion

Cellulose nanoparticles were synthesized using *C. papaya* as starting material and characterized to check their properties. The nanoparticles were however amorphous with high thermal stability up to 98 °C, forming spherical cavities similar to carbon fullerene. These findings compared well with literature and indicate that cellulose nanoparticles can be synthesized from *C. papaya* barks. The research findings showed the potential for utilization of the *C. papaya* barks for synthesis of CNPs which will reduce the risk of pollution posed by this biomass.

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