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EXTRACTION, PHYSICOCHEMICAL, AND STRUCTURAL CHARACTERIZATION OF NANOCELLULOSE FROM PINEAPPLE (*Ananas comosus*) PEELS WITH POTENTIAL FOR SUSTAINABLE PAPER APPLICATIONS

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ABSTRACT

The heavy reliance on wood-based pulp for paper production accelerates significant loss of biodiversity, deforestation, and increased greenhouse gas emissions, highlighting the urgent need for sustainable non-wood alternatives. In this study, nanocellulose was extracted from pineapple (*Ananas comosus*) peels, an abundant and underutilized agro-industrial waste, and comprehensively characterized to evaluate its physicochemical and structural properties. Cellulose was isolated from powdered pineapple peels through sequential alkaline delignification and bleaching, followed by acid hydrolysis to obtain nanocellulose. The resulting nanocellulose was characterized by Fourier-Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), and X-ray Diffraction (XRD), while key hydration-related parameters were measured. A cellulose-based nanocellulose yield of 7.5 % was obtained, corresponding to a biomass-based yield of 12.9 %, with a bulk density of 0.55 g/mL and a water retention capacity of 0.25 g/g. FTIR spectra confirmed effective removal of lignin and hemicellulose, SEM revealed nanoscale fibrillar morphology, and XRD showed an increase in crystallinity index from 67.09 % (bleached cellulose) to 69.40 % in nanocellulose, indicating an enhanced structural order. Overall, the findings demonstrate that pineapple peel-derived nanocellulose exhibits desirable physicochemical and morphological characteristics suitable for sustainable paper applications. The observed structural features and hydration behavior suggest that pineapple peel-derived nanocellulose has characteristics that may be favorable for cellulose-based material development, supporting its potential use in sustainable paper-related applications.

Keywords: Nanocellulose, Agrowaste, Sustainable paper production, valorization, Crystallinity

INTRODUCTION

The global demand for paper continues to increase due to population growth and expanding packaging needs, placing significant pressure on forest resources and contributing to deforestation, biodiversity loss, and carbon emissions (FAO Codex Committee, 2008). This situation highlights the importance of developing sustainable alternatives to wood-based paper and adopting circular economy principles that promote resource efficiency and waste minimization. Valorization of agricultural residues into high-value materials offers a promising strategy for sustainable material development. Agricultural wastes such as crop residues and fruit peels are rich in cellulose, hemicellulose, and lignin. They can serve as feedstocks for producing nanocellulose—a renewable, biodegradable nanomaterial with excellent mechanical properties, high surface area, and versatile applications in papermaking, coatings, and composites (Khan *et al.*, 2024). Nanocellulose has been widely reported to reinforce paper sheets, improve barrier properties, and provide an environmentally friendly alternative to synthetic additives (Hashemzehi *et al.*, 2022).

Pineapple (*Ananas comosus*) processing generates large quantities of peel residues, which are often discarded or underutilized. Improper disposal of pineapple peel can contribute to environmental pollution, including methane emissions in landfills, leachate generation, and increased biochemical oxygen demand (BOD) in water bodies, highlighting the need for sustainable valorization strategies (Fouda-Mbanga & Tywabi-Ngeva, 2022; Hikal *et al.*, 2021). Utilizing pineapple peel not only provides a sustainable raw material for paper manufacturing but also contributes to waste reduction and the achievement of circular economy goals. The ability to extract high-purity cellulose from pineapple peels represents a significant step toward sustainable materials development. Agricultural waste is often discarded or burned, generating methane and contributing to greenhouse gas emissions. In contrast, its transformation into nanocellulose contributes to waste minimization, renewable raw material sourcing, and the reduction of deforestation pressures associated with wood-based pulp.

Despite the abundance of agro-residues such as pineapple peel, their potential for nanocellulose production remains underexplored. Current research has largely concentrated on conventional agricultural wastes including sugarcane bagasse, rice husk, and corn stalks, which are widely recognized as reliable feedstocks for nanocellulose extraction (Picot-Allain & Emmambux, 2023). Within the pineapple biomass stream, scholarly attention has predominantly focused on the leaves, which are already exploited in textile manufacturing and polymer composite applications due to their favorable fiber characteristics (Sethupathi *et al.*, 2024; Nguyen *et al.*, 2021). This emphasis on leaves has inadvertently overshadowed the peel, a residue generated in substantial quantities during fruit processing but often discarded or relegated to low-value uses such as animal feed or compost. Pineapple peel, therefore, represents a promising yet underutilized resource for nanocellulose research. Addressing this gap could not only diversify raw material sources but also contribute to circular economy strategies by transforming waste into high-value bioproducts.In contrast, systematic investigations of pineapple peel-derived nanocellulose remain scarce, particularly with respect to its physicochemical and structural properties relevant to paper reinforcement. Existing peel-focused studies have primarily emphasized film formation or general material characterization (Dai *et al*., 2018; Esquivel-Alfaro *et al*., 2025), leaving a gap in the comprehensive evaluation of peel nanocellulose. This present research systematically investigates the yield, density, water retention capacity, crystallinity, and fibrillar morphology of nanocellulose extracted from pineapple peel residues. By establishing a detailed physicochemical and structural profile, this study provides foundational knowledge that highlights the potential of peel-derived nanocellulose as a sustainable raw material for future applications, while simultaneously valorizing an underutilized waste stream and addressing environmental concerns associated with improper peel disposal. To the best of our knowledge, this study represents one of the few comprehensive evaluations of nanocellulose derived exclusively from pineapple peels, combining yield analysis, hydration behavior, crystallinity, and nanoscale morphology in a single framework.

MATERIALS AND METHODS

Materials

Fresh pineapple (*Ananas comosus*) peels were obtained from local fruit vendors in Asaba, Delta State, Nigeria. Sodium hydroxide (NaOH), sodium hypochlorite (NaOCl), acetic acid (CH_3COOH), and sulfuric acid (H_2SO_4) were all of analytical grade and used without further purification. Distilled water was used throughout the study. Major instruments employed included an oven (WH-43 Electric Thermostatic Heated Dry Box), pH meter (Labman-2601), centrifuge (Omack Scientific 800D), diaphragm vacuum pump (GM-0.33II), Fourier-transform infrared (FTIR) spectrophotometer (Agilent CARY 630), scanning electron microscope (SEM) (JEOL JSM-7000F), and X-ray diffractometer (Rigaku Miniflex 600).

Methods

Sample preparation

Collected pineapple peels were washed thoroughly with tap and distilled water to remove dirt and sugars, cut into small pieces, and oven-dried at 80 °C until a constant weight was achieved. The dried peels were ground into fine powder using a blender and stored in airtight containers for subsequent analysis.

Extraction of cellulose

Cellulose was extracted following the method of Zuluaga *et al.* (2009) with modifications. Fifty grams of dried pineapple peel powder was treated with 500 mL of 10 % NaOH solution at room temperature for 1 h, then heated at 60–80 °C for 3 h to remove hemicellulose and lignin. The mixture was stirred intermittently, filtered, and washed with distilled water until neutral pH was achieved. The residue was oven-dried at 80 °C for further use. The delignified material was bleached using the procedure described by Madureira *et al.* (2018). The cellulose was soaked in 3.5 % NaOCl solution containing 1 mL acetic acid for 1 h at room temperature with intermittent stirring. The bleached cellulose was filtered under vacuum, washed with distilled water until neutral, and oven-dried at 60 °C.

Preparation of nanocellulose

Nanocellulose was prepared from bleached cellulose following the method of Velázquez *et al.* (2022) with slight modification. The bleached cellulose was first partially hydrolyzed with 17 % (w/w) HCl at 60 °C for 2 h to obtain microcrystalline cellulose. After thorough rinsing with cold distilled water to remove excess acid, the sample was subjected to further hydrolysis using 35 % (w/w) H_2SO_4 at 45 °C for 45 min under constant stirring to form cellulose nanocrystals. The reaction was quenched with cold distilled water and repeatedly washed until a neutral pH (6.5–7.0) was achieved. The resulting suspension was filtered and oven-dried at 50–60 °C until a constant weight was obtained. The sequential use of HCl followed by H_2SO_4 was adopted to achieve controlled cellulose size reduction. Initial HCl treatment minimizes sulfate esterification, while subsequent H_2SO_4 hydrolysis promotes removal of amorphous regions, yielding nanocellulose with improved crystallinity and dispersion stability (Beck-Candanedo *et al*., 2005; Velázquez *et al*., 2022).

Physicochemical characterization of the pineapple nanocellulose

The physicochemical properties of the isolated nanocellulose were analyzed following the method described by Okeke *et al.* (2021) with minor modifications.

The extracted nanocellulose was dried in a hot-air oven at 60 °C to constant weight and the percentage yield was calculated using Equation (1):

$$\%\,Yield = \frac{M_N}{M_P} \times 100 \qquad (1)$$

where M_N is the weight of dried nanocellulose, and M_P is the weight of the original dried pineapple peel powder. Yield was calculated based on biomass weight as well as cellulose weight.

Bulk density

Bulk density was determined according to Prakhongpan *et al.* (2002). A clean, dry 100 mL graduated cylinder was weighed and filled with 50 g of PPNC without compaction. The cylinder was gently tapped three times from a height of approximately 2 cm to level the sample, and the occupied volume was recorded. Bulk density was expressed as mass per unit volume (g/mL) using Equation (2):

$$Bulk\ density\ (g/mL) = \frac{W}{V} \qquad (2)$$

where W is the weight of the sample (g) and Vis the bulk volume (mL)

Packed density

Packed density was measured using a calibrated 10 mL graduated syringe as described by Prakhongpan *et al.* (2002). A known mass of sample was introduced into the syringe, and compression was applied manually in repeated cycles until no further volume reduction was observed after three consecutive compressions. Packed density was calculated as the ratio of sample mass to its minimum compressed volume (g/mL) using Equation (3):

$$Packed\ density(g/mL) = \frac{W}{V_P} \qquad (3)$$

where W is the weight of the sample (g), and Vp is the packed volume (mL)

Hydrated density

Hydrated density was determined using the water displacement method. A calibrated 10 mL graduated cylinder was filled with distilled deionized water, and approximately 1.0 g of sample was carefully added to avoid adhesion to the cylinder walls. The increase in water volume was recorded as the displaced volume. Measurements were conducted at room temperature (25 ± 2 °C). Hydrated density was expressed as grams of sample per milliliter of displaced water (g/mL) and calculated using Equation (4):

$$Hydrated\ density(g/mL) = \frac{W}{V_d} \qquad (4)$$

where W is the weight of the sample (g), and V_d is the volume of water displaced (mL)

Water retention capacity (WRC)

The water retention capacity was determined gravimetrically, following the FAO Codex Committee on Codex Specifications (2008). Briefly, 2 g of the sample was dispersed in 30 mL of distilled water in a 50 mL centrifuge tube and allowed to stand for 10 min. The mixture was centrifuged at 2000 rpm for 15 min using a fixed-angle rotor. After centrifugation, the supernatant was carefully decanted, and the residue was allowed to drain for 2 min without blotting before weighing. WRC was expressed as grams of water retained per gram of dry sample using Equation (5):

$$WRC = \frac{W_w}{W_d} \qquad (5)$$

where Ww is the weight of the hydrated sample and W_d is the weight of the dry sample

Functional group and morphological characterization

Fourier-transform Infrared Spectroscopy (FTIR) was used to identify surface functional groups in pineapple peel (PP), pretreated cellulose (PPCP), and nanocellulose (PPNC) samples using an Agilent CARY 630 spectrophotometer. Spectra were recorded within 650–4000 cm^{-1} at 4 cm^{-1} resolution over 32 scans.

Surface morphology was analyzed using a JEOL JSM-7000F scanning electron microscope at an accelerating voltage of 10–20 kV. Samples were mounted on aluminum stubs with carbon tape and sputter-coated with platinum before imaging at 9000× magnification.

Crystallinity was assessed by X-ray diffraction (XRD) using a Rigaku Miniflex 600 diffractometer equipped with Cu Kα radiation (λ = 1.5406 Å). Operating parameters were 30 kV and 30 mA, with scans collected over a 2θ range of 5°–60° at 2° min^{-1} to determine crystallinity indices of the samples.

RESULTS AND DISCUSSION

Extraction of Cellulose and Nanocellulose from Pineapple Peels

The sequential alkaline, bleaching, and acid hydrolysis treatments successfully yielded purified cellulose (PPC) and nanocellulose from pineapple peels. The visual transformation from brownish raw peels to white cellulose and translucent nanocellulose was observed, reflecting efficient delignification and decolorization, confirming the removal of non-cellulosic components.

Physicochemical characterization of Nanocellulose from Pineapple Peels (PPNC)

The physicochemical properties of pineapple peel nanocellulose (Table 1) reflect its potential for papermaking. The nanocellulose yield obtained from pineapple peel in this study was 7.5 %, which is consistent with the yields reported by Kian *et al.* (2019), who extracted nanocrystalline cellulose from roselle-derived microcrystalline cellulose using sulfuric acid hydrolysis and achieved yields in the range of 5–7 %. Similarly, Esquivel-Alfaro *et al.* (2025) documented yields of 3.65–15% from pineapple peel using sulfuric acid hydrolysis. Comparable ranges have also been observed for other agro-residues: Zuluaga *et al.* (2009) reported 7–10 % yields from banana rachis following alkaline and acid treatments, and Velázquez *et al.* (2022) obtained 8–14 % yields from Paraguayan agro-industrial biomass. These values demonstrate that the yield achieved in the present work falls within the established efficiency window for lignocellulosic feedstocks, confirming the suitability of pineapple peel for nanocellulose production.

The moderate bulk (0.55 g/mL) and packed (0.44 g/mL) densities of pineapple peel nanocellulose indicate a porous structure with good water dispersibility, while the hydrated density of 1.25 g/mL confirms its hydrophilic nature (Esquivel-Alfaro *et al.*, 2025). These values are comparable to those reported for other agro-waste celluloses; Pelissari *et al.* (2014) reported bulk densities of 0.646 g/mL for banana peel cellulose, while Bigi *et al.* (2023) reported values around 0.305 g/mL for orange peel cellulose, placing pineapple peel nanocellulose within a competitive range. The obtained yield demonstrates that pineapple peel, an abundant agro-waste, can be valorized into high-value cellulose nanomaterials using simple, scalable chemical routes.

Table 1: Physicochemical properties of pineapple nanocellulose

Parameters	PPNC
Biomass-based Yield (%)	12.90
Cellulose-based Yield (%)	7.50
Bulk Density(g/ml)	0.55
Packed Density (g/ml)	0.44
Hydrated Density(g/ml)	1.25
Water Retention Capacity (WRC) g of water/g of sample	0.25

The hydrated density (1.25 g/mL) and water retention capacity (0.25 g/g) of pineapple peel nanocellulose indicate that the material retains water effectively without forming excessive gels. This balance ensures even dispersion during papermaking while maintaining fiber bonding strength. Water retention capacity (WRC) is a critical parameter influencing fiber-fiber bonding during drying, which directly affects paper tensile and tears strength. The WRC obtained in this study is comparable to values reported by Tiwari and Sanjog (2023), who found WRCs between 0.22 and 0.28 g/g for agro-waste-derived nanocellulose. These hydration properties are therefore essential for paper applications requiring moisture interaction and dimensional stability. In comparison with commercial microfibrillated cellulose, the obtained WRC values are favorable, suggesting that pineapple peel nanocellulose can impart comparable bonding enhancement at lower dosage levels. This translates to reduced pulp consumption and energy cost, aligning with sustainable manufacturing objectives as highlighted by Okeke *et al.* (2021), who emphasized the role of fibrillated cellulose in advancing greener fabrication methods for pulp and paper industries. Although direct paper-making or mechanical performance tests were not conducted, the observed yield, hydration behavior, density, crystallinity, and fibrillar morphology are widely recognized as key indicators of nanocellulose performance in paper-related systems, as these properties govern fiber-fiber bonding and sheet consolidation (Hashemzehi *et al.*, 2022; Velázquez *et al*., 2022; Tiwari & Sanjog, 2023).

Fourier Transform Infrared (FTIR) Spectroscopy

The FTIR spectra (Figure 1a-c) confirm the progressive removal of hemicellulose and lignin and the successful isolation of cellulose and nanocellulose. In the raw pineapple peel spectrum (Figure 1a), the broad O–H stretching vibration at 3330 cm^{-1} and C–H stretching near 2920 cm^{-1} were associated with hydroxyl and aliphatic groups of polysaccharides. The strong absorption at 1735 cm^{-1}, corresponding to the carbonyl stretching of acetyl and uronic ester groups in hemicellulose, disappeared after alkaline treatment, confirming its removal.

Similarly, the band around 1510 cm^{-1}, characteristic of lignin's aromatic skeletal vibrations, was no longer observed after bleaching, indicating effective delignification (Figure 1b). The persistence of the cellulose-specific bands at 3330 cm^{-1} (O–H), 2890 cm^{-1} (C–H), and 1050 cm^{-1} (C–O–C) confirms that the cellulose backbone remained structurally intact throughout treatment. The observed increase in peak sharpness and intensity after hydrolysis indicates enhanced order and crystallinity (Hachaichi *et al.*, 2021). These findings align with Chen *et al.* (2021), who observed similar spectral transitions during nanocellulose extraction from pineapple peel, confirming the removal of non-cellulosic components and enrichment of cellulose-specific bands. The intensification of peaks at 1162 and 897 cm^{-1} in this study indicates increased crystallinity and purity, consistent with observations by Hachaichi *et al.* (2021) in date palm fiber-derived nanocellulose. The FTIR data substantiate the conversion of lignocellulosic biomass into high-purity cellulose nanostructures. The improved hydroxyl intensity suggests greater hydrogen-bonding capacity—critical for interfiber adhesion and strength in papermaking (Madureira *et al*., 2018). This chemical profile supports the suitability of pineapple peel nanocellulose as an eco-friendly reinforcement material capable of enhancing the bonding and mechanical integrity of sustainable paper products.

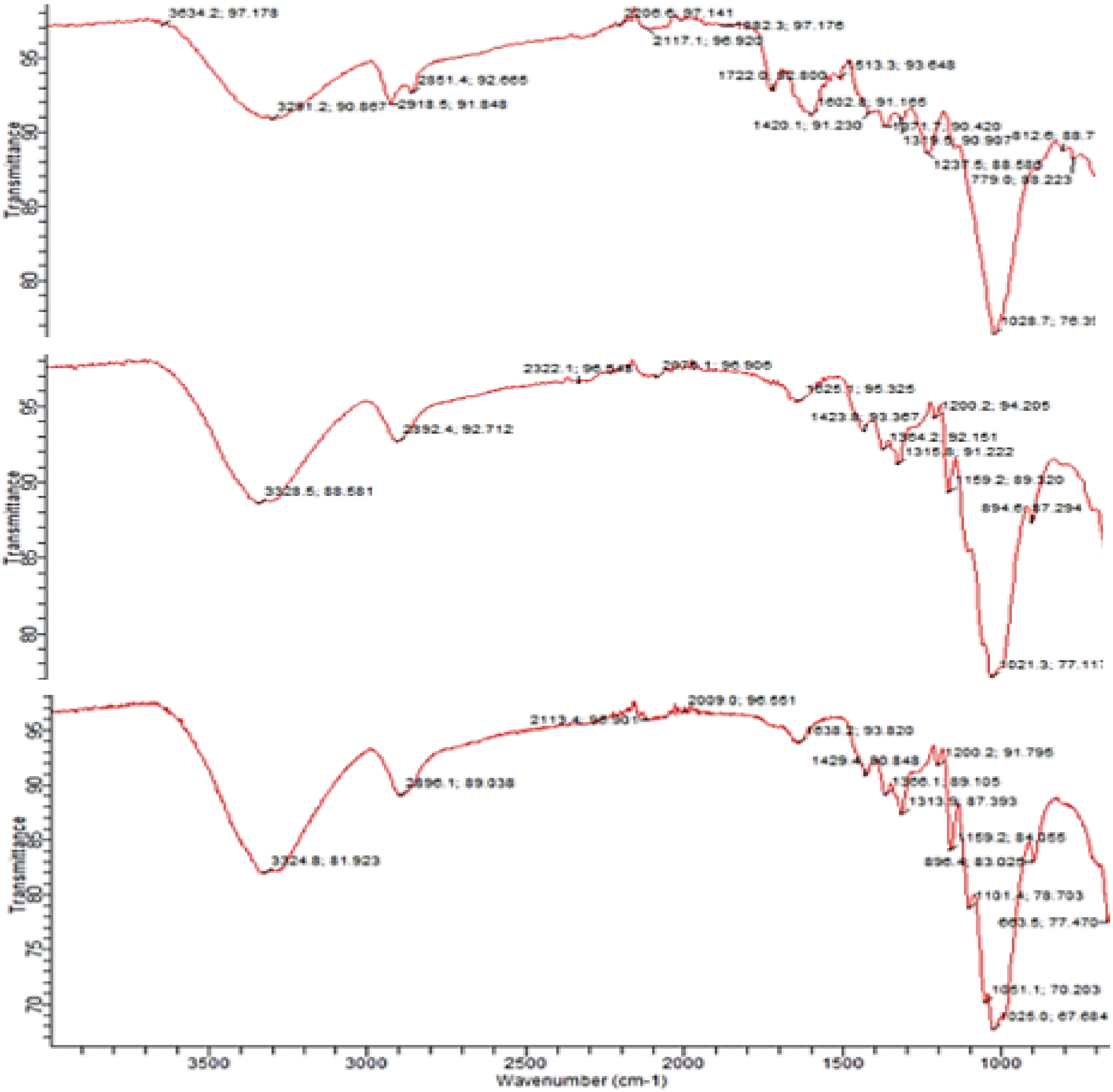

Figure 1: FTIR Spectrum of (a) raw pineapple peel (b) Pineapple peel cellulose (c) Pineapple peel nanocellulose

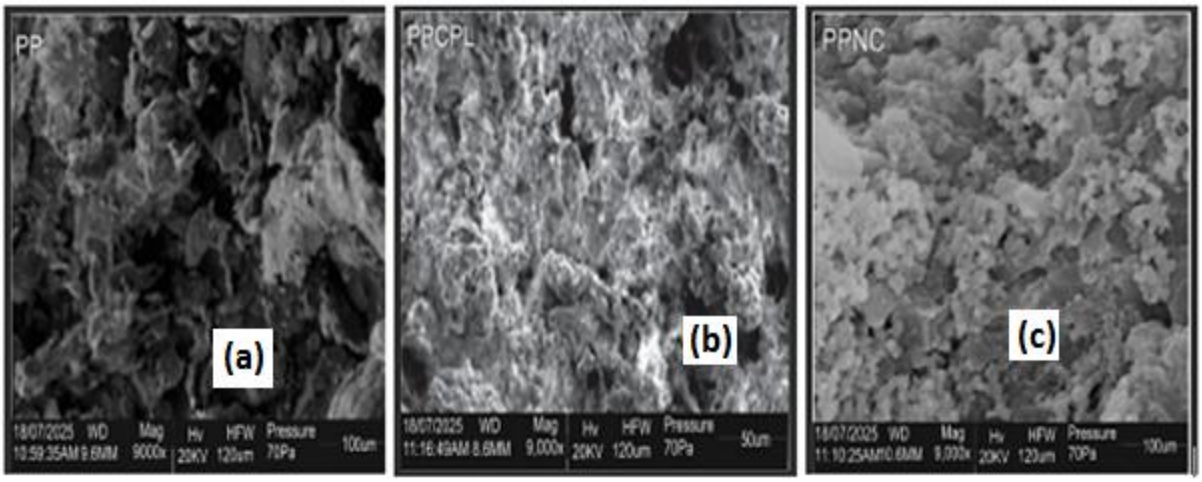

Figure 2: Scanning Electron micrographs of (a) raw pineapple peel (b) Pineapple peel cellulose (c) Pineapple peel nanocellulose

Scanning Electron Microscopy (SEM) Analysis

The SEM micrographs (Figure 2a-c) revealed the morphological evolution of pineapple peel fibers during treatment. SEM analysis revealed distinct structural changes during the transformation of raw pineapple peel to nanocellulose. The raw pineapple peel (PP) exhibited a rough, heterogeneous surface with loosely arranged fibers and residual plant debris. After alkaline and bleaching treatments, the surface appeared cleaner and more fibrous indicating the removal of non-cellulosic components such as hemicellulose and lignin. Further processing into nanocellulose (PPNC) resulted in a dense, highly porous structure with nanoscale fibers, reflecting significant size reduction and surface area increase. These structural modifications confirm the successful isolation and refinement of cellulose into nanocellulose, suitable for applications requiring high surface area and uniform morphology.

The nanocellulose exhibited a distinct nanoscale fibrillar network, with entangled, needle-like structures typical of cellulose nanocrystals. These nanosized fibrils possess a high surface-to-volume ratio and abundant hydroxyl groups, enabling strong hydrogen bonding when incorporated into pulp matrices. Such morphology is critical in papermaking, as nanocellulose can fill voids between macrofibers, improving fiber cohesion, tensile strength, and sheet smoothness (Velázquez *et al*., 2022).The formation of this fine, interconnected network aligns with findings by Madureira *et al*. (2018), who reported similar structural refinement in pineapple leaf-derived nanocellulose. The morphology suggests that pineapple peel nanocellulose can serve both as a strength enhancer and as a coating agent for biodegradable paper, improving surface uniformity and reducing porosity without increasing basis weight—a key criterion for sustainable paper engineering.

X-ray Diffraction (XRD) Analysis

XRD patterns (Figure 3a and b) showed sharp diffraction peaks of PPCP and PPNC. Both samples exhibit prominent diffraction peaks at approximately 16.4° and 22.58° 2θ for cellulose, and 16.10° and 22.66° 2θ for nanocellulose. These peaks correspond to the (110) and (200) crystallographic planes, respectively, indicative of the cellulose Iβ crystalline structure (Bano & Negi, 2017).

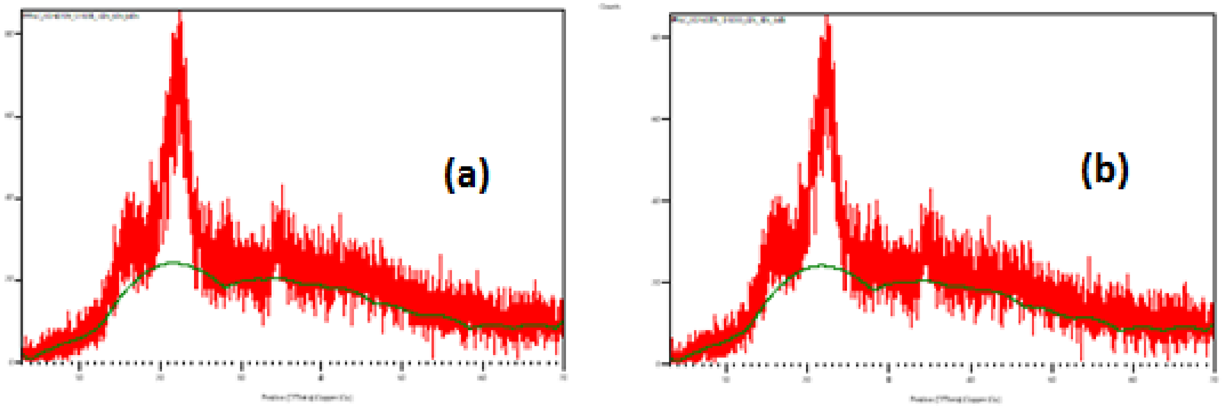

Fig 3: Xray diffractogram of (a) Pineapple peel cellulose (b) Pineapple peel nanocellulose

The XRD patterns reveal that the bleached cellulose maintains a semi-crystalline cellulose I structure, with a crystallinity index (CrI) of approximately 67.09 %. Following nanocellulose synthesis, an increase in CrI to 69.4 % is observed, likely due to the removal of amorphous regions during acid hydrolysis (Table 2).

Table 2: Crystallinity indices of PPCP and PPNC from XRD analysis

Sample	I_{200I}	I_{am}	CrI (%)
Bleached Cellulose	629	207	67.09
Nanocellulose	540	165	69.4

This enhancement in crystallinity aligns with findings by Nagarajan *et al*. (2019), who reported similar improvements in acid-hydrolyzed nanocellulose derived from agro-wastes. It also aligns with previous findings where amorphous fractions were selectively removed, enriching crystalline cellulose domains and improving mechanical properties (e.g., tensile strength and stiffness)—a key quality for reinforcement in paper (Hashemzehi *et al*., 2022). Increased crystallinity is directly correlated with improved mechanical rigidity, dimensional stability, and reduced moisture sensitivity—properties vital for high-quality paper production (Hachaichi *et al*., 2021). The crystallinity values obtained are comparable to those reported for nanocellulose from sugarcane bagasse (Velázquez *et al*., 2022), confirming the structural competitiveness of pineapple peel-derived nanocellulose. Moreover, the moderate crystallinity of pineapple nanocellulose implies improved flexibility and fibril interlacing, promoting dense paper sheet formation and tensile strength. These findings highlight that pineapple peel nanocellulose possesses the structural integrity, morphology, and hydration behavior required for sustainable paper manufacturing. In conclusion, the

XRD analysis suggests that PPNC, with its higher crystallinity, holds promise for sustainable paper production. The increased crystalline properties imply suitability for reinforcing recycled paper, functional paper coatings, and the development of biodegradable or moisture-resistant paper products. The inferred suitability of PPNC for paper-related applications is based on physicochemical and structural characteristics such as yield, hydration behavior, density, crystallinity, and fibrillar morphology, despite the absence of mechanical and sheet-forming tests. These parameters fall within reported ranges for commercial and agro-waste-derived nanocellulose, and they are commonly used to benchmark nanocellulose for paper systems (Hashemzehi *et al*., 2022).

CONCLUSION

This study successfully demonstrated the extraction and detailed physicochemical and structural characterization of nanocellulose from pineapple (*Ananas comosus*) peel residues using chemical pretreatment and acid hydrolysis. The sequential alkaline and bleaching treatments effectively removed non-cellulosic components, as confirmed by FTIR analysis, while subsequent acid hydrolysis produced nanocellulose with a well-defined fibrillar morphology, as revealed by SEM. The increase in crystallinity index observed by XRD analysis indicates improved structural organization of cellulose at the nanoscale. The measured yield, bulk density, and water retention capacity further provide insight into the physical behavior of the extracted nanocellulose and its interaction with water, which are important parameters for cellulose-based material development. Overall, the results confirm that pineapple peels, often regarded as low-value agro-waste, can be transformed into nanocellulose with favorable structural and physicochemical properties.

This work contributes to the growing body of research on non-wood cellulose resources and supports the valorization of agricultural residues within a circular economy framework. While the present study focused on extraction and characterization, the generated data establish a strong foundation for future investigations into processing, modification, and application-oriented evaluation of pineapple peel-derived nanocellulose in cellulose-based materials.

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