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Semantan Bamboo Cellulose Nanofibrils (*Gigantochloa scortechinii*) Using Wet Disk Mill Treatment (WDM): Effect of Cycles on Their Yield, Physic-Chemical, Morphological and Thermal Behavior

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ABSTRACT

Cellulose nanofibril (CNF) was extracted from bamboo cellulose fibers (BCF) using a wet disk mill (WDM) treatment. This study focuses on understanding the impact on the physicochemical properties, yield across multiple milling cycles, morphology, and thermal behavior of the resulting bamboo cellulose nanofibrils (BCNF). Morphological analysis using transmission electron microscopy (TEM) revealed a reduction in fiber diameter, with increased milling cycles resulting in more uniform and finer nanofibers ranging from 5 to 15 nm. Remarkably, the yield of nanofibers was between 97.6% and 98.8%. The crystallinity index was found to be between 75% and 77%. Thermal analysis through thermogravimetric analysis (TGA) demonstrated that the thermal stability of NFC decreased with additional milling cycles. The wet disk milling process effectively produced bamboo cellulose nanofibrils from Semantan bamboo, with the number of cycles playing a crucial role in determining the yield and properties of BCNF. These findings suggest that careful control of milling parameters can tailor NFC properties for specific industrial applications, such as in nanocomposites and biodegradable materials.

HIGHLIGHTS

- WDM cycles increase nanofibrillated cellulose yield up to 98.8% as an optimal point.
- Increased WDM cycles enhance crystallinity, surface area, and fiber uniformity until optimal cycles are reached.
- Characterization results revealed a crystallinity index (CrI) ranging from 75% to 77%, with higher CrI correlating to improved mechanical properties.
- Proper WDM treatment enhances the thermal stability of nanofibrillated cellulose.

摘要

本研究利用濕式盤式研磨機 (WDM)處理，從竹纖維素 (BCF)中萃取出纖維素納米纖維 (CNF)。本研究著重於探討此處理過程對所得竹纖維素納米纖維 (BCNF)之物理化學性質、多重研磨週期產率、形態及熱行為的影響。透過傳輸電子顯微鏡 (TEM)進行的形態分析顯示，纖維直徑隨研磨週期增加而減小，研磨週期越多，所得的奈米纖維越均勻且越細，直徑範圍介於5至15奈米之間。值得注意的是，奈米纖維的產率介於97.6%至98.8%之間。結晶度指數則介於75%至77%之間。透過熱重分析 (TGA)進行的熱分析顯示，隨著研磨循環次數增加，NFC的熱穩定性隨之降低。濕式盤式研磨製程能有效從森曼坦竹中製備出竹纖維素奈米纖維，其中循環次數在決定BCNF的產率與性質方面扮演關鍵角色。這些發現表明，透過精確控制研磨參數，可針對特定工業應用 (如奈米複合材料與生物可降解材料)來調整NFC的性質。

KEYWORDS
Semantan bamboo; morphology; thermal behavior; physicochemical properties; cellulose nanofibrils; wet disk mill

KEYWORDS
Semantan竹; 形態; 熱行為; 物理化學性質; 纖維素納米纖維

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Introduction

The most popular subject in materials research currently is cellulose nanofibril (CNF), which are derived from plant fibers and possess remarkable inherent characteristics, including a large surface area (100 m^2 /g), a high aspect ratio of 100, and a nanoscale dimension (Asyraf et al. 2021; Syafri et al. 2022; Vilela et al. 2017), high crystallinity (Norfarhana, Ilyas, Ngadi, and Othman 2024; Yue and Qian 2018), decreased density, increased tensile strength, accessibility to specific morphologies, sustainability, and biodegradability (Amanda, Budiari, and Amelia 2024; Costa et al. 2024; Nurani et al. 2024; Sivanantham et al. 2024). Nanocellulose originates from cellulose, a linear homopolysaccharide composed of glucose units connected by β (1–4)-glycosidic linkages. Elementary fibrils, which are 2–10 nm thick and comprise both amorphous and crystalline regions, are found within cellulose aggregates. These fibrils can be further arranged into various polymorphs (I, II, III, and IV) based on unit cell dimensions and chain polarity. A variety of biomass sources can degrade cellulose to create nanocelluloses (NCs), which are distinguished by several unique characteristics, such as low density, low thermal expansion coefficient, high tensile strength and stiffness, high aspect ratio, and high specific surface area (Abral et al. 2021; Xie et al. 2018). Most mechanical defibrillation techniques including Wet Disk Milling (WDM), High-Pressure Homogenizing (HPH), and ball milling can separate CNF from plant cell walls (Davoudpour et al. 2015; de Barros et al. 2013; Norrrahim et al. 2021; Park, Han, Namgung, et al. 2017; L. Zhang, Tsuzuki, and Wang 2015). The CNF has been demonstrated to possess outstanding characteristics such a large specific surface area, a high strength, and exceptional thermal properties. Its diameter has been estimated to be between 10 and 30 nm (Davoudpour et al. 2015; Park, Han, Namgung, et al. 2017; L. Zhang, Tsuzuki, and Wang 2015; Zheng, Cai, and Gong 2014). In the past decades, to create CNF, a variety of materials have been employed, including cassava bagasse (Widiarto et al. 2019), oil palm (Ling et al. 2025; Razali et al. 2026), wheat straw (Kumar Trivedi, Kumar, and Gupta 2023) cotton cellulose (Theivasanthi et al. 2018), softwood wood (Zhao et al. 2017), rice straw (Shi and Liu 2021), kenaf (Taib et al. 2024), bamboo fiber (Othman, Ngadi, et al. 2025; Othman et al. 2024; H. Singh et al. 2023), sugar palm fiber (Asyraf et al. 2021; R. A. Ilyas, Sapuan, and Ishak 2018; R. Ilyas et al. 2021; R. A. Ilyas et al. 2018, 2019; Nazrin et al. 2021; Norfarhana, Ilyas, Ngadi, and Dzarfan Othman M 2024), ginger (Abral et al. 2020; Fahma et al. 2021; Rahmadiawan et al. 2022), water hyacinth (Asrofi et al. 2018; Syafri et al. 2019), and sugarcane bagasse (Mandal and Chakrabarty 2011; Mubarak, Ilyas, Ngadi, et al. 2024; Mubarak, Ilyas, Nordin, et al. 2024). The separation of CNF is done for the aim of providing reinforcement in the field of nanocomposite, which has attracted a lot of interest since it was initially investigated by (Syafri et al. 2022). Although cellulose nanofibrils derived from bamboo fibers using WDM treatment have been reported in the literature, existing studies predominantly focus on demonstrating the feasibility of nanofibrillation and describing basic morphological features. In contrast, systematic investigations that quantitatively correlate WDM cycle number with NFC yield, physicochemical composition, crystallinity evolution, surface charge characteristics, and thermal stability that are particularly for naturally derived bamboo cellulose remain limited. Furthermore, comprehensive studies addressing high-yield CNF production from specific bamboo species under well-controlled WDM processing conditions are still scarce. It is interesting to note that WDM are becoming increasingly popular due to their great energy efficiency compared to other milling methods. WDM efficiently breaks down cellulose into nanofibrils, producing CNF with exceptional surface qualities and unique characteristics (Norrrahim et al. 2021). Yasim-Anuar et al. (Yasim-Anuar et al. 2020b) CNF was effectively removed from wastepaper using the WDM technique. After ten milling cycles, their results show that this environmentally friendly method successfully shrank wastepaper fibers from micrometer size to 20–40 nm.

Bamboo is among the fastest-growing plants in the world. Bamboo achieves maturity in around 4–5 years and is grown worldwide. It performs exceptionally well in low elongation, high stiffness, and strength (Abral et al. 2020; Asrofi et al. 2018; Fahma et al. 2021; Ganapathy et al. 2023; R. A. Ilyas, Sapuan, and Ishak 2018; R. Ilyas et al. 2021; R. A. Ilyas et al. 2018, 2019; Mandal and Chakrabarty 2011; Mubarak, Ilyas, Ngadi, et al. 2024; Mubarak, Ilyas, Nordin, et al. 2024; Nazrin et al. 2021; Norfarhana, Ilyas, Ngadi, and Dzarfan Othman M 2024; Nurazzi et al. 2022; Othman, Ngadi, et al. 2025; Othman et al. 2024; Radzi et al. 2022; Rahmadiawan et al. 2022; H. Singh et al. 2023; Syafri et al. 2019; Yasim-Anuar et al. 2020b). Hemicelluloses, lignin and cellulose are the three main parts of bamboo fiber, making up over 90% of its mass (Ganapathy et al. 2023; Md Shah et al. 2016). Bamboo serves as an important source of biomass made of lignocellulosic materials, with a high cellulose content for the synthesis of nanocellulose. Bamboo fibers have a cellulose concentration of nearly 74%, which is higher than that of other fibers (Cao et al. 2022). Semantan bamboo (*Gigantochloa scortechinii*) is characterized by high cellulose content (approximately 70–74%), moderate hemicellulose (15–20%), and lignin content (20–25%), which provide structural rigidity and mechanical strength (Hawanis et al. 2024). The fibers exhibit a well-aligned microfibrillar structure with high aspect ratio and relatively low microfibril angle, contributing to excellent tensile properties and stiffness (Hawanis, Ilyas, Al-Oqla, Jalil, Ibrahim, Abdul Majid, et al. 2025; Hawanis, Ilyas, Al-Oqla, Jalil, Ibrahim, Majid, et al. 2025; Hawanis, Ilyas, Jalil, et al. 2025; Othman, Ilyas, et al. 2025). Bamboo also possesses a hierarchical vascular bundle architecture that facilitates efficient fiber separation during mechanical processing. In addition, its low density, rapid growth cycle, and widespread availability in tropical regions make it an attractive and sustainable lignocellulosic resource for nanocellulose production. These compositional and structural characteristics further justify the selection of Semantan bamboo as a suitable precursor for nanofibrillation processes, particularly in mechanically driven systems where fiber integrity and hierarchical organization play a critical role in determining fibrillation efficiency (Norrrahim et al. 2025; Othman, Ilyas, et al. 2025).

The objective of this study is to systematically investigate the influence of wet disk milling (WDM) cycle number on the yield and physicochemical characteristics of bamboo cellulose nanofibrils (BCNF) derived from Semantan bamboo (*Gigantochloa scortechinii*). It is hypothesized that increasing the number of WDM cycles promotes progressive fibrillation of cellulose fibers, leading to a reduction in fibril diameter while maintaining high isolation yield and largely preserving the intrinsic crystalline structure of cellulose. In this context, the present work evaluates how repeated mechanical treatment governs the evolution of morphological, structural, and thermal properties, with particular emphasis on understanding whether intensified fibrillation can be achieved without substantial degradation of cellulose integrity. Such understanding is essential to establish a more efficient processing route, in which high-yield nanofibril production is attained primarily through mechanical disintegration rather than extensive chemical modification. To achieve this, the resulting BCNF samples were comprehensively characterized using transmission electron microscopy (TEM), Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), point of zero charge (pHpzc) analysis, and thermogravimetric analysis (TGA), thereby enabling a systematic evaluation of structure – property relationships as a function of WDM cycle number.

Materials and methods

Materials

Semantan bamboo fibers (*Gigantochloa scortechinii*), derived from culm-based lignocellulosic material, were collected from the Forest Research Institute Malaysia (FRIM), Kepong, Malaysia, and used as the primary raw material for cellulose isolation and subsequent nanofibrillation. The chemical reagents used were 80% pure sodium chlorite ($NaClO_2$) from Sigma, 99.8% pure acetic acid (CH_3COOH) from QRëc^{TM}, and 99% pure sodium hydroxide.

Isolation of cellulose

Delignification and mercerization are the two main methods for distinguishing cellulose fibers from Semantan bamboo fibers (BF) (Abral et al. 2020; Fahma et al. 2021; Rahmadiawan et al. 2022). Cellulose was isolated from Semantan bamboo fibers through successive delignification (bleaching) and alkaline mercerization steps to remove non-cellulosic components, including lignin and hemicellulose. Initially, the bamboo fibers were cut into small pieces, thoroughly washed with distilled water, and oven-dried at 60°C to remove moisture.

Delignification was carried out using a sodium chlorite – acetic acid bleaching system. Briefly, dried bamboo fibers were suspended in distilled water and heated to 70°C under continuous stirring. Sodium chlorite ($NaClO_2$, 80% purity) was then added at a concentration of 1.5 wt% relative to the fiber mass, followed by acetic acid to adjust the pH to approximately 4–5. The bleaching reaction was maintained for 1 h, after which fresh $NaClO_2$ and acetic acid were added, and the process was repeated until the fibers turned whitish, indicating effective lignin removal. The bleached fibers (holocellulose) were then washed repeatedly with distilled water until neutral pH was achieved.

The obtained holocellulose was subsequently subjected to alkaline mercerization to isolate α-cellulose. The fibers were treated with 5 wt% sodium hydroxide (NaOH) solution at room temperature (23 ± 2°C) for 2 h under gentle stirring to remove residual hemicellulose. After alkali treatment, the fibers were thoroughly washed with distilled water until neutral pH and then oven-dried at 103°C overnight. The resulting purified cellulose was designated as bamboo cellulose (BBC) and used for subsequent WDM.

The overall procedure follows the general principles outlined in ASTM D1104-56 and ASTM D1103-60, with experimental details explicitly described here for clarity and reproducibility.

Wet disk mill

Bamboo cellulose was ground to obtain nanocelluose by Wet Disk Milling (WDM) fitted with two ceramic nonporous disk grinders from Grow Engineering in Adachi-ku, Tokyo, Japan. The grinders were set to revolve at 1800 rpm and were adjusted to leave an average of 20–40 nm between the lower and upper grinders. Following the WDM method, bamboo nano-fibrillated cellulose (BCNF) was isolated from BBC, as shown in Figure 1. After adding around 2 l of fiber suspension with 2% concentration, the grinder was run through 5, 10, 15, 20 cycles. The final fibers were given a designation as BCNF-5, BCNF-10, BCNF-15, and BCNF-20, which

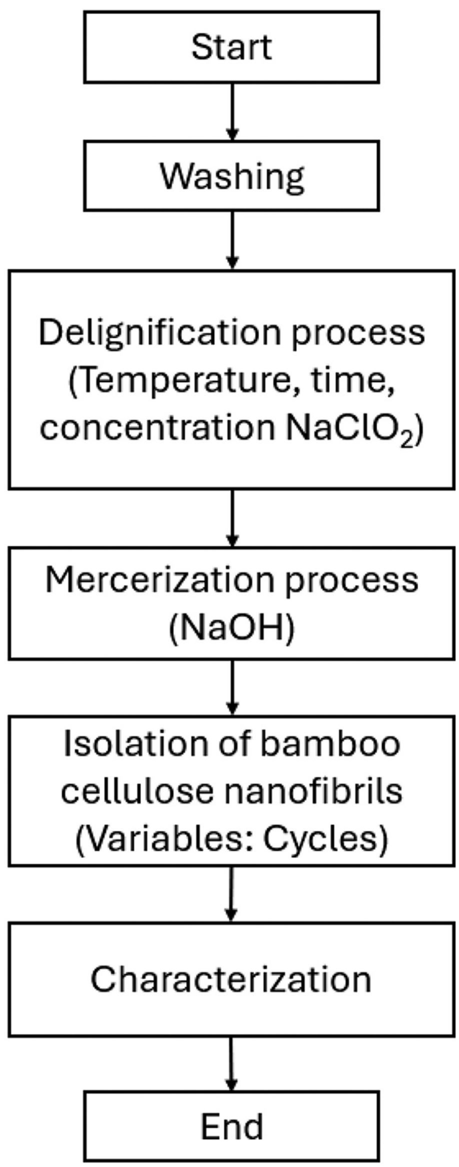

Figure 1. Flowchart illustrating the stepwise process for the isolation of bamboo cellulose nanofibrils.

correspond to bamboo cellulose nanofibrils subjected to 5, 10, 15, and 20 WDM cycles, respectively. All other operational parameters, including rotation speed (1800 rpm), disk gap (20–40 nm), suspension concentration (2 wt%), and processing volume (2 l) were kept constant throughout the experiments. For later usage, the BCNF slurry was stored with caution at 2°C in a covered beaker. For additional characterization, a slurry sample of 10 ml was freeze-dried. In this study, a controlled parametric investigation approach was adopted from (Othman, Ilyas, et al. 2025), where only the number of WDM cycles was varied while all other processing parameters were maintained constant.

Characterization

All analytical data were processed using the respective instrument software. Baseline correction was applied to FTIR spectra to remove background drift, and standard background subtraction was performed for XRD patterns. No additional normalization, smoothing, or peak deconvolution procedures were applied. Thermal analysis parameters were obtained directly from instrument-generated TG/DTG curves. Morphological measurements were conducted using Image-Pro Plus software without digital alteration of micrographs. All experimental measurements were conducted in triplicate and expressed as mean ± standard deviation to ensure reproducibility and reliability of the results. The analysis focuses on systematic trend evaluation as a function of WDM cycle number under controlled processing conditions. Given the parametric nature of the study, comparisons among samples were interpreted based on consistent directional changes supported by low variability in replicated measurements. In addition, basic comparative interpretation was performed to evaluate the relationship between WDM cycle number and the resulting physicochemical, morphological, and thermal properties of BCNF.

Morphology analysis

Transmission electron microscopy (TEM) was employed to observe the nanostructural morphology of BCNF. TEM analysis was conducted using a Philips Tecnai 20 instrument operated at an accelerating voltage of 200 kV and equipped with a standard inclined sample holder. Prior to observation, BCNF suspensions were prepared by dispersing the samples in distilled water, followed by ultrasonication for 10 min to ensure uniform dispersion of the nanocellulose. Subsequently, a drop of the diluted BCNF suspension was deposited onto a carbon-coated copper grid and allowed to dry naturally at room temperature. The obtained TEM images were digitally enhanced to improve image clarity and resolution. For diameter analysis, 100 individual fibrils were measured using ImageJ, and only clearly individualized fibrils were included in the calculation of mean diameter and standard deviation.

Bamboo fiber nanocellulose BCNF yield

Before being dried to a constant weight at 90°C in a halogen desiccator, the solid content of about 0.2% was diluted with distilled water and centrifuged at 4500 rpm for 20 min to separate the Nano fibrillated material (in the supernatant fraction) from the non-fibrillated and partially fibrillated ones, which settled down. Equation (1) was used to compute yield.

$$Yield\% = 1 - \left(\frac{weight\ of\ sediment}{weigth\ of\ diluted\ sample \times \%Sc}\right) \times 100 \quad (1)$$

Where % Sc is the solid content percentage. The result reflects the average of three replicates.

Fourier transform infrared (FT-IR) analysis

The PerkinElmer 2000 (USA) Spectrum GX model instrument is used for this spectroscopic analysis. The analysis spans from 4000 to 500 cm–, with a resolution of 4 cm^{-1} and a scanning speed of 20 kHz (R. A. Ilyas et al. 2019).

X-ray diffraction (XRD)

The consequences of the several treatments on the X-ray diffraction patterns of the BCNF sample were investigated using the Rigaku D/max 2500 X-ray powder diffractometer (Tokyo, Japan). With CuKα radiation (λ = 0.1541 nm) and 2.00/min scanning in the 2θ range of 5–40°, the device demonstrated the intricate possibilities of the patterns. The crystallinity index (CrI%) was calculated using Equation (2), which was derived by Segal et (Segal et al. 1952). Based on the intensity of the diffraction pattern.

$$Xc = \frac{I_{002} - Iam}{I_{002}} \times 100 \tag{2}$$

Where the peak intensities of amorphous materials and crystalline are denoted by I_{am}, and I_{002} respectively.

Point zero of charge

The BCNF's Point Zero of Charge (PZC) was ascertained by transferring 50 mL of 0.01 M NaCl solution into five separate 250 mL Erlenmeyer flasks. The pH of each solution was then increased from 2 to 10 by adding 0.1 M HCl and NaOH solutions. Each one received roughly 0.5 g of BCNF after the pH solution was adjusted. The ultimate pH of the prepared solutions was measured after they had been stored for 48 h. Plots of pH final vs pH initial were made, and the BCNF's pH_{pzc} was determined by finding the junction point of the two curves. The ideal pH was eventually identified. Equation (3) was utilized in the computation of pH_{PZC}.

$$pH = pH1 - pH2 \tag{3}$$

Where pH_1 is the pH value before the experiment and pH_2 is the pH value after the experiment. In the plot of ΔpH vs pH, the intersection points at which ΔpH = 0 specify pH_{PZC}.

Thermogravimetric analysis (TGA)

The thermal stability of Nano cellulose for weight loss from temperature increases was examined using the TGA analyzer. TGA was performed using a Q series thermal analysis equipment from TA Instruments (Mettler-Toledo AG, Schwerzenbach, Switzerland) to determine the potential for heat degradation of bamboo nanocellulose at various isolated stages. The study was carried out by placing fiber samples in aluminum pans. Typically, the operating conditions comprise a dynamic nitrogen environment in the temperature range of 25–900°C and a heating rate of 10°C/min (R. A. Ilyas et al. 2019).

Result and discussion

Morphology analysis

Fiber size and surface morphology may alter during wet disk mill cycles. Figure 2 displays the TEM images that were analyzed using ImageJ for BCNF. It is evident that when the number of wet disk mill cycles rises, the morphological structure of BCNF changes. The pictures showed that the nanocellulose was evenly distributed throughout the matrix, which is a sign of efficient processing methods. In addition, the BCNF-5 exhibits big individual BCNF web-formation and a greater nanofiber morphological diameter from other cycles. The integrated BCNF, made up of thousands of individual nanofibers joined by strong hydrogen bonds, may be responsible for this phenomenon. Furthermore, the surface morphology showed a generally smooth texture with sporadic fibrillar features projecting from the surface (Hayes et al. 2018). Following all treatments, isolated nanofibers with minimal aggregation were observed, indicating a trend toward fiber defibrillation. This outcome was consistent with past research findings published in the literature by (Saurabh et al. 2016). Besides that, this observation suggests a high aspect ratio of the nanocellulose fibers. The BCNFs-5 possess diameter of approximately 12.41 ± 14.88 nm, which presented reduction in fibril diameter as those of BCNFs-10 approximately 7.48 ± 12.70 nm, BCNFs-15 4.12 ± 7.91 nm, and BCNFs-20 with an average value around 4.76 ± 6.96 the results are shown in the Figure 2, and Table 1.

The microscopic drawing displays for BCNF a tangled structure and single fibers that indicate the fibrillation process during the mechanical process WDM, due to the rotating disk creating high shear forces that act on the fibers, pulling them apart longitudinally (Guélon, Mathias, and Stoodley 2011). This impact force helps to break down the fiber bundles further (Ning et al. 2019). The presence of water in the milling process aids in the separation of fibers by reducing friction, and the hydrodynamic forces also help to transport the fibers through the milling chamber effectively. The most recent findings in this TEM examination were

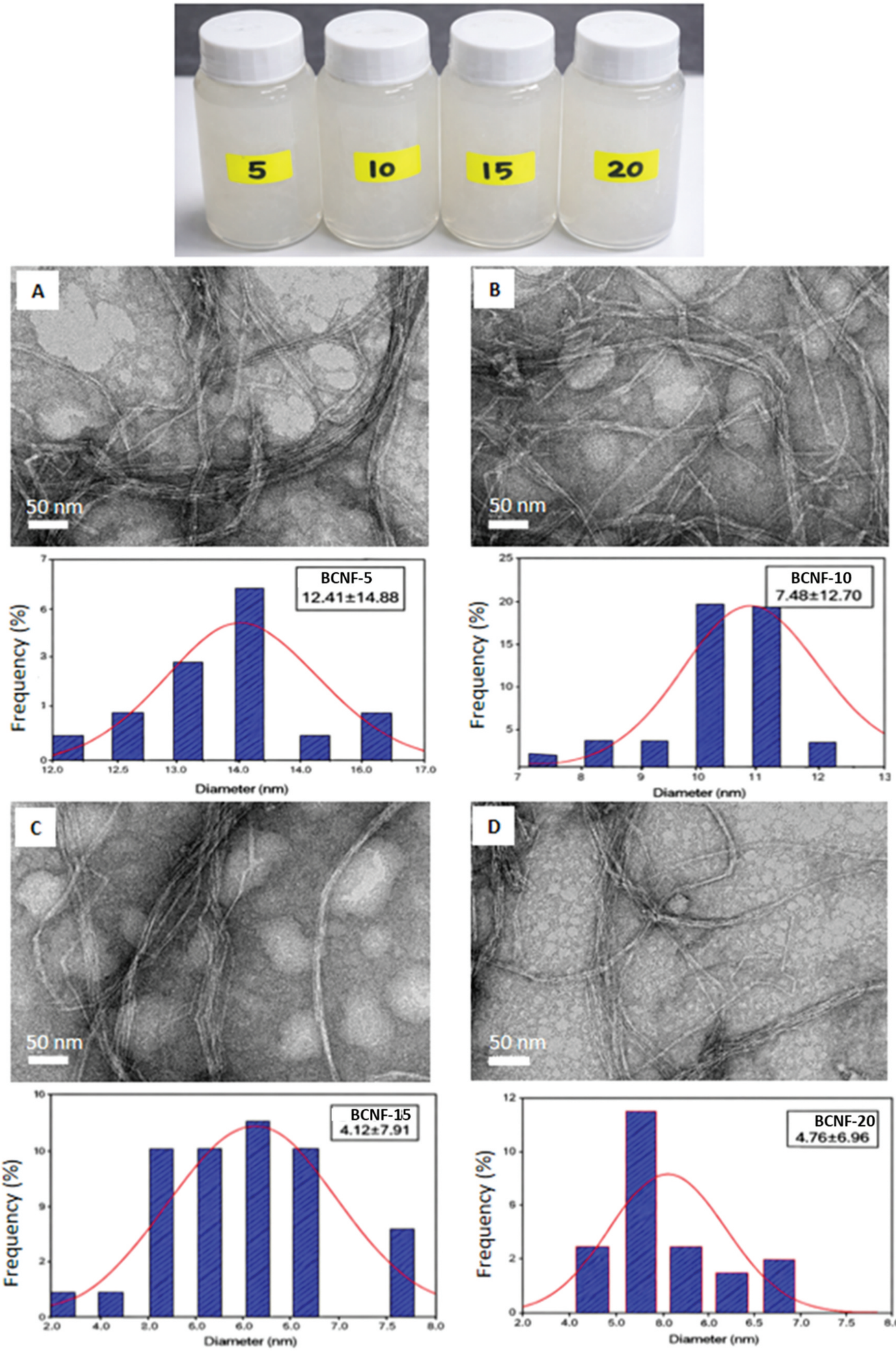

Figure 2. Microscopic images and corresponding diameter-distribution histograms of BCNF-5, BCNF-10, BCNF-15, and BCNF-20, illustrating variations in fiber morphology and fiber diameter distribution among the samples.

Table 1. Dimension a size of BCNFs-5, BCNFs-10, BCNFs-15, and BCNFs-20.

Sample	Diameter size means ± SD (nm)
BCNF-5	12.41 ± 14.88
BCNF-10	7.48 ± 12.70
BCNF-15	4.12 ± 7.91
BCNF-20	4.76 ± 6.96

corroborated by earlier research using an estimated diameter size of 5 nm for banana nanofibrils (M. Zhang et al. 2023; Zuluaga et al. 2007), and flax fiber (5 nm) (Doineau et al. 2020), and wider than prickly pear fruit (2–5 nm) (Xu et al. 2023), and smaller than raw cotton linter (*Gossypium hirsutum*) (12 nm) (Morais et al. 2013), alfa (*Alfa tenassissima*), pineapple leaf (30 nm) (Balakrishnan et al.), pineapple leaf fiber (32.5 nm) (Cherian et al. 2010), sugarcane bagasse (35 nm) (Gond, Gupta, and Jawaid 2021), wheat straw (30–70 nm) (Kumar Trivedi, Kumar, and Gupta 2023), hemp fiber (10–60 nm) and rutabaga (80 nm) (Panthapulakkal and Sain 2013). Other research on CNF from various bamboo species showed lateral sizes ranging from 2 to 30 nm (He et al. 2013b). As a result, the nanofibers produced in this work were smaller in diameter than the CNF of previously published literature. After being subjected to WDM, the bamboo cellulose exhibits an irregular structure that points to nano-fibrillation. This emerges from the mill stones' placement before the WDM process generates shearing and frictional difficulties to the cellulose fibers' outermost layer. Table 2 compares the morphological and physical properties of BCNF with other common lignocellulosic fibers.

The results indicate a clear inverse relationship between WDM cycle number and average fibril diameter, demonstrating that increasing mechanical treatment intensity promotes progressive fibrillation and nanofiber individualization. The observed reduction in fibril diameter together with decreased variability at higher cycles further supports the effectiveness of repeated WDM processing in refining bamboo cellulose into nanoscale fibrillar structures.

Table 2. Comparison of the morphological and physical properties of BCNF with other common lignocellulosic fibers.

Lignocellulosic biomass	Extraction method	Morphological properties: Shapes	Morphological properties: Diameter (nm)	Crystallinity (%)	References
Soybean straw	- Alkali treatment+ bleaching	Thread-like structures	9.4	50	Michelin et al. (2020)
Sugar palm fiber	- Pretreatment of ILs - H_2O_2 bleaching - WDM treatment	Thread-like structure	3.30 ± 0.68	61.62	Norfarhana, Ilyas, Ngadi, and Dzarfan Othman (2024)
Jack fruit peduncle	- Acidified chlorination - Alkalization treatment - Acid hydrolysis	Cylindrical structure	27300	79.6	Devarajan et al. ()
Cuscuta reflex	- Alkaline treatment - Steam explosion treatment	Thread network structure	10–30	67	Dominic et al. (2020)
Agave gigantea	- Alkaline treatment - (NaOH+ CH3COOH+ $NaClO_2$ bleaching - Ultragrinding + ultrasonication treatment	Fibril -fibril structure	4.07	65.21	Syafri et al. (2022)
Rice husk	- Alkaline treatment - H2O2 bleaching - HPH treatment	Fiber networks	10.15–12.42	62–72	Samsalee, Meerasri, and Sothornvit (2023)
Sugarcane bagasse (This study)	- Alkaline treatment - Acid treatment - Alkaline treatment - High speed grinding	Spherical/ rod like structure	57.07	58.32	Gond, Gupta, and Jawaid (2021)
Timoho	- NaClO2 bleaching - Alkaline treatment - Acid hydrolysis	Glycosidic network	68.94 ± 8.36	71.14	Devarajan et al. ()
Wheat straw	- Steam explosion+ microwave assisted - alkali hydrolysis treatment	Spherical or irregularly structure	10–40	58.62	Espinosa et al. (2019)
Banana peel	- Enzymatic hydrolysis using xylanase	Entangled networks	9–26	66.2	Tibolla et al. (2019)
Bamboo	- Delignification+ mercerization - Wet disk mill	Bundles of slender	4.76–14.88	77	Current study

BCNF yield

The isolated BCNF yields from bamboo fiber were compiled according to each of the 5, 10, 15, and 20 WDM cycles in Figure 3. It can be observed that BCNF-5 dominates the lowest yield yielded 97.6%, BCNF-10 yielded 97.8%, BCNF-15 yielded 98.8% and BCNF-20 also yielded 98.8%. A similar trend was seen throughout the extended number of cycles. The consistently high yield values observed across increasing WDM cycles suggest that intensified mechanical fibrillation does not substantially compromise material recovery, indicating a positive relationship between fibrillation efficiency and nanocellulose yield retention. This is due to uniform particle size distribution resulting in a high yield of nano-sized particles by wet disk mill method (Cho et al. 2024). During the wet disk mill process, an intense process of breaking action happens, impact, and compression was used to separate the fibers from micro-size to nano-size. A cellulose suspension was prepared in a liquid medium, usually water, in a chamber that contained a stationary cylindrical vessel, and a rotating disc equipped with grinding elements, such as pins or rods. As a result, the yield of nanofibers rose as the wet disk mill's cycles increased because the nanofibers were frequently exposed to cavitation, impact, and shear stresses, which caused more microfiber to become nanofiber (Kumagai et al. 2015). The consistently high yield across all cycle numbers indicates that the WDM process enables efficient fibrillation with minimal material loss, thereby supporting its suitability as a predominantly mechanical approach for nanocellulose production.

The present research presents an impressive yield compared to the CNF obtained by TEMPO-oxidation of wood fibers; it exceeds the 30.46% and 44–55% yield reported by Masruchin et al. (Masruchin et al. 2020) and Jonasson et al. (Jonasson et al. 2020), respectively. Interestingly, compared to earlier studies that produced 32.9%, 33.9%, 31.4%, and 30.5% from various species of bamboo *(Thyrsostachys siamesi Gamble, Dendrocalamus sericeus Munro, Bambusa logispatha, and Bambusa sp)* using chemical – mechanical methods, this study demonstrates a far higher yield of nano-fibrillated cellulose (NFC) from Semantan bamboo fiber BF (Thipchai et al. 2023), and 65% from Bamboo Sawdust by the experimental acid treatment methods are sulfuric acid hydrolysis for bamboo fiber nanocrystal cellulose (BBF-NCC) (Pantamanatsopa et al. 2023). This result was high yield compared with others which was 81.37% yield nanofiber cellulose via cellulose extracted from oil palm (*Elaeis guineensis*) empty fruit bunch (OPEFB) hydrolyzed with Ni(II) (Yahya et al. 2019). The material's source, pre-treatment procedures, the number of processing cycles, and the temperature employed in the mechanical techniques may be linked to these yield disparities (Khan et al. 2020).

Fourier transform infrared (FT-IR) analysis

FTIR analysis was used to investigate how the chemical structure of BCNF changed following many wet disk mill treatments. Figure 4 shows the physicochemical analysis of the FTIR spectra of BCNF-5, BCNF-10, BNC-15, and BCNF-20. FTIR analysis is an essential approach for directly getting information on the changes in the chemical structure of nanofibers after they have undergone numerous cycles of WDM

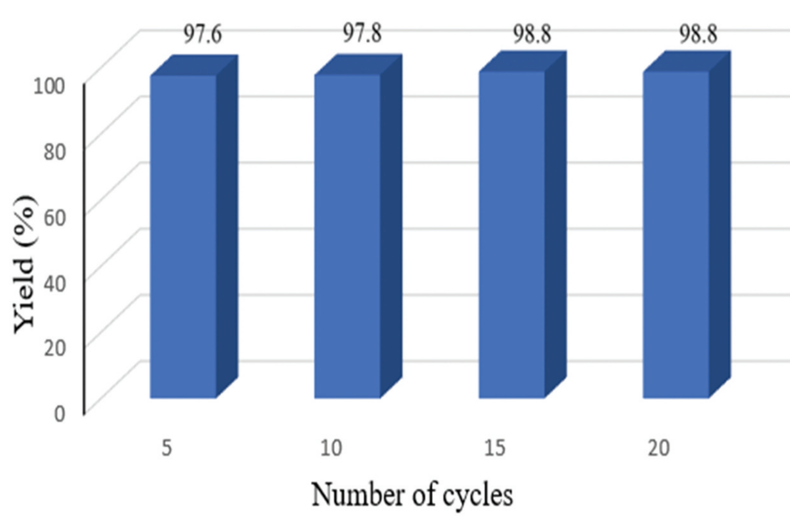

Figure 3. A bar graph showing yield across number of cycles.

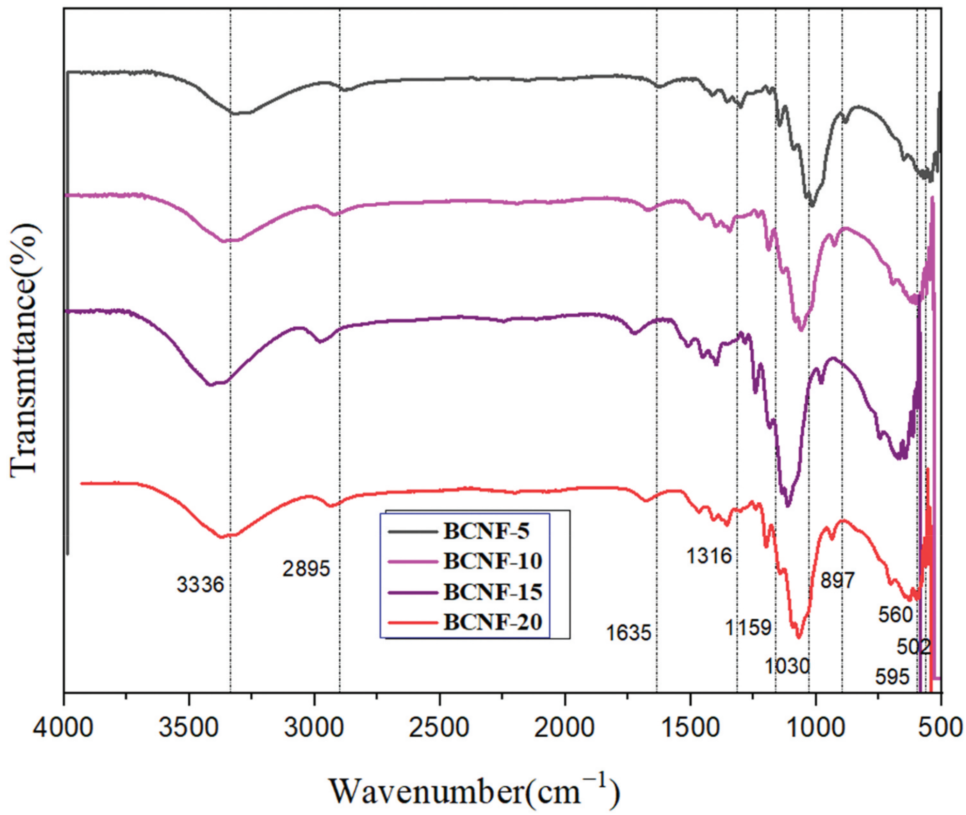

Figure 4. Fourier Transform Infrared (FT-IR) spectra of BCNF-5, BCNF-10, BCNF-15, and BCNF-20 showing characteristic functional groups.

mechanical treatment. According to FTIR analysis, all spectra detected in all samples are identical, indicating a similar chemical makeup in all the nanofibers.

The Semantan bamboo nanocellulose showed clear bands at 3331 and 2895 cm^{-1}, respectively. These bands corresponded to the stretching vibrations of the hydroxyl group (if there are H-bonds) and the C–H bond in the pyrenoid ring (Mo et al. 2009). The band at 1635 cm^{-1} was caused by the absorbed water's H–O–H bending (Razalli et al. 2017). The distinct bands that emerged at 3331 cm^{-1}, 2895 cm^{-1}, and 1635 cm^{-1} demonstrated a strong correlation with the findings documented by Liu et al. (Liu and Kim 2015). They used the acid hydrolysis approach to make nanocellulose from flax yarn. The –OCH in-plane bending, C–H deformation, and C–O–C stretching are shown by the bands at 1316 cm^{-1}, 1321 cm^{-1}, 1159 cm^{-1}, and 1030 cm^{-1}, respectively (Razalli et al. 2017). These bands are typical of the cellulose's β-glycosidic connections. Their existence in every sample suggests that WDM treatment preserves cellulose's basic glycosidic bond structure. 800–1500 cm^{-1} is indicated several bands that represent the C–O, C–C, and C–H vibrations, among other complex vibrational modes of the cellulose backbone. Variations in the number of milling cycles do not appear to appreciably alter the chemical structure of cellulose, as indicated by the similarities in this region throughout the spectra (Brizuela et al. 2012; Y. P. Yang et al. 2017). The FTIR spectra verify that the nanocellulose's physical form is the main effect of wet disk milling, with few changes to its chemical structure. The intrinsic qualities of cellulose, such as its mechanical strength and thermal stability, are critical for a variety of industrial uses, and their preservation depends on its chemical integrity. A dependable material for additional functionalization and application development, cellulose's chemical structure is resilient, as seen by the persistent bands over many WDM cycles.

Point of zero charge

A substance's point of zero charge (PZC) is the pH at which it has no net charge. Because of electrostatic forces, it is important to understand how charged particles interact with surfaces. If the pH is less than

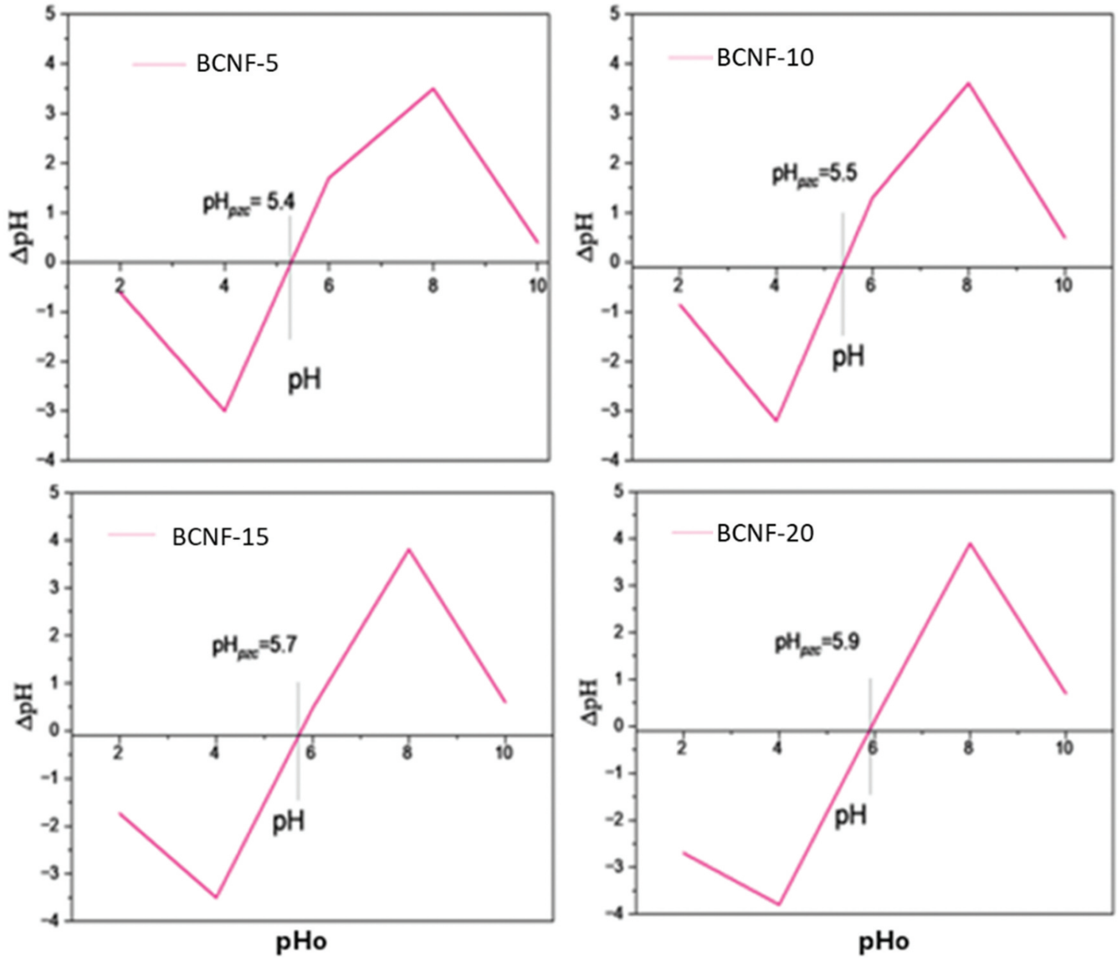

Figure 5. Line graphs of ΔpH versus initial pH for BCNF-5, BCNF-10, BCNF-15, and BCNF-20, illustrating the determination of the point of zero charge (pHpzc) for each sample.

pH*pzc*, the BCNFs surface area is positively charged; if it is higher than pH*pzc*, it is negatively charged. Most ions in the solution are positively charged when the pH falls below pH*pzc* at specified values. The surface area of the BCNF becomes negatively charged when the pH exceeds the point of zero charge or pH*pzc* (Raj et al. 2016; S. Singh et al. 2024).

From this a point, it can be concluded that the Semantan nanocellulose bamboo, treated with different numbers of wet disk milling (WDM) cycles (5, 10, 15, and 20 cycles), was pH 7.80 and the point of zero charge (PZC) for each cycle as shown in Figure 5, where BCNF 5,10,15, and 20 was 5.4, 5.5, 5.7 and 5.9 respectively. Because hydroxyl groups are present in nanocellulose, this technique will strengthen the electrostatic interaction between nanocellulose particles and dye ions as well as the capacity of nanocellulose to remove anionic dyes (Bassyouni et al. 2022), the pH was such as the result of nanocellulose to removal fluoride which was pH_{pzc} 5.6 (Das, Das, and Bhowal 2023), and in other study of adsorption of Safranin-O dye was pH_{pzc} 7.3 (Shaltout et al. 2024). By using batch adsorption studies, the effect of the biopolymer composite film's constituent materials and pH on the elimination of methylene blue (MB) dye from an aqueous testing solution were investigated. The pH values used in these studies were based on chitin/nanocellulose and ranged from 8.05 to 8.55 (Mok et al. 2020).

The pH appears to be slightly shifted toward higher values as the number of milling cycles is increased. This may result from increased surface functional group exposure or from minor chemical changes brought about by rigorous mechanical treatment.

X-ray diffraction (XRD)

The material's mechanical and thermal properties are significantly influenced by the crystallinity of individual fibers, which was determined using XRD (R. A. Ilyas, Sapuan, and Ishak 2018). Figure 6 shows the X-ray diffractograms of Nano cellulose bamboo of different cycles 5,10, 15, and 20 using a wet disk mill. The Crystallinity Index (CI) of the samples under investigation was determined by analyzing their diffraction patterns and changes in the ratio of their crystalline to amorphous components. All samples under study have crystalline and amorphous domains, as revealed by X-ray diffraction patterns, which tend to affect the observed pattern and crystallinity index (CI) values. The nanocellulose bamboo diffraction pattern, separated from the cellulose bamboo, is recognized for its typical polymorphs cellulose1 structure (typical peaks at $2\theta =$ 16.70° and 22.24°) (Cui et al. 2018), indicating preservation of the native crystalline structure after WDM processing. The calculated crystallinity index (CrI) values showed only minor variation with increasing milling cycles, suggesting that mechanical fibrillation primarily affects morphological structure rather than inducing crystalline polymorphic transformation. These findings are consistent with previous studies on nanocellulose derived from bamboo, wood pulp, cotton, and various agricultural residues, where mechanical treatments were reported to preserve cellulose I structure with only slight fluctuations in CrI. In contrast, acid-hydrolyzed cellulose nanocrystals (CNC) typically exhibit higher crystallinity (often >80%) due to preferential removal of amorphous domains (T. Yang et al. 2023). The relatively stable CrI values observed in the present study therefore confirm that wet disk milling enhances fibrillation efficiency while maintaining the intrinsic crystalline framework of cellulose. The relatively stable crystallinity index across different WDM cycles indicates that progressive fibrillation primarily affects fiber morphology rather than significantly disrupting the intrinsic cellulose crystalline domains. This observation suggests a controlled structural evolution during mechanical treatment. The results are shown in Table 3. Aside from that, the crystallinity values for each cycle of BCNF-5,10,15, and 20 were 75%, 76.2%, 76.9%, and 77%, respectively, with the acute intensity peaks situated at approximately (22.45°–22.78°) and minor intensity peaks at (15.44°–17.42°). The results obtained indicated a negligible rise in the nanofibers' crystallinity index. According to (He et al. 2013a), This may happen due to WDM breaking up the amorphous areas while simultaneously reorganizing and enhancing the semi-crystalline cellulose regions. The increased surface area and size reduction could be the cause of these improvements (Jang et al. 2015; Xiao et al. 2016).

Consequently, it may be concluded that the wet disk mill's excellent defibrillation capability contributed to the BCNF's higher crystallinity index. But after a few cycles, ranging from 10 to 15, the crystallinity index progressively rises (Guimarães et al. 2015; Norfarhana, Ilyas, Ngadi, and Dzarfan Othman 2024). This might

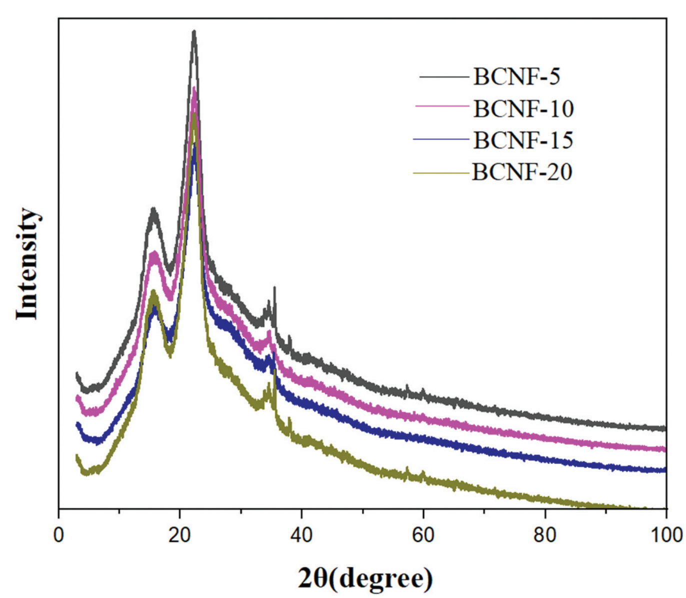

Figure 6. X-ray diffraction (XRD) patterns of BCNF-5, BCNF-10, BCNF-15, and BCNF-20 showing characteristic peaks and variations in crystallinity.

Table 3. XRD analysis data of BCNF-5, BCNF-10, BCNF-15, and BCNF-20.

Samples	2θ (am) (°)	2θ (0 0 2) (°)	Xc%
BCNF-5	15.44°	22.45°	75%
BCNF-10	16.1°	22.53°	76.2%
BCNF-15	16.70°	22.70°	79.9%
BCNF-20	17.42°	22.78°	77%

be because WDM strengthens and restructures the semi-crystalline cellulose areas in addition to breaking the amorphous ones. Consequently, it can be concluded that the wet disk mill's vigorous defibrillation process is what gave bamboo cellulose nanofibrils its higher crystallinity index. This extended WDM process results in the degradation of cell walls into nanofibrils (Park, Han, Namgung, Seo, et al. 2017). For unprocessed bamboo and cellulose, the computed values of the crystallinity index (CrI) were 46% and 72.5%, respectively. The computed crystallinity index was comparable to the nanostructures recovered from other agricultural residue sources, as the 81.5% kenaf bast fiber *(Hibiscus cannabinus)* (Karimi et al. 2014), and surpassing 54% of pineapple leaves (Mahardika et al. 2018), 60% of it is eucalyptus Kraft pulp (Cebreiros2021), pinecone (*Pinus Banksiana Lamb*) (70%) (Rambabu et al. 2016), pine (75%) (Coelho et al. 2020), and pineapple leaf (75.38%) (Balakrishnan et al.). It was, nevertheless, less than cotton linter (90%) (Morais et al. 2013), eucalyptus (86%) (Raju et al. 2023). This indicates, albeit indirectly, that WDM has superior CNF formability when compared to alternative fibrillation techniques. This behavior indicates that although repeated milling promotes fibrillation at the nanoscale, it does not substantially disrupt the underlying crystalline domains, thereby preserving the structural integrity of cellulose despite intensive mechanical processing.

Thermogravimetric analysis (TGA)

The thermal stability of bamboo fiber nanocellulose was examined using thermogravimetric analysis (TG). Figures 7 shows that the TG and DTG thermograms generated from bamboo cellulose showed a little weight loss, mostly caused by the water from CNF evaporating (Yasim-Anuar et al. 2020b). The temperature range in which the largest weight losses were seen for all samples was 200–370°C. The outcome implies that cellulose decomposes more quickly at higher temperatures, which is consistent with the breakdown of cellulose. Of all the BCNF samples, BCNF-20 had the lowest beginning thermal decomposition (250°C), based on the thermogravimetric (TG) study data shown in Table 4. It should be noted that slight variations in onset temperature (T_onset) and residual char percentage can also be influenced by differences in initial solid content, including minor variations in residual moisture and sample packing during TGA pan loading. Although all samples were dried

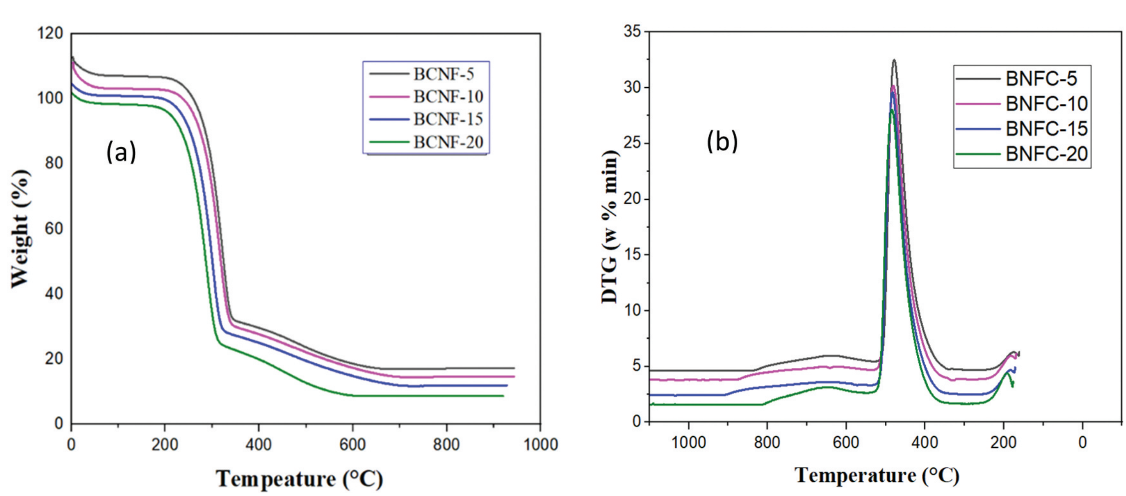

Figure 7. (A) Thermogravimetric (TG) curves and (B) derivative thermogravimetric (DTG) curves of BCNFs-5, BCNFs-10, BCNFs-15, and BCNFs-20.

Table 4. The TG and DTG curves were used to determine the onset temperature (T_{Onset}), degradation temperature on maximum weight-loss rate (T_{Max}), weight loss (WL), and char yield for BCNF-5, BCNF-10, BCNF-15, and BCNF-20.

Samples	Water Evaporation			1st thermal degradation			Charyield
	T_{Onest} (°C) (%)	T_{Max}	WL (°C)	T_{Onest} (°C)	T_{Max} (°C)	WL (%)	W (%)
BCNF-5	25	116.41	5.67	243.36	359.37	72.43	22.1
BCNF-10	22.5	113.12	6.96	257.31	355.81	70.07	21.5
BCNF-15	21.4	162.38	9.12	253.76	345.41	69.08	20.2
BCNF-20	25.3	155.26	9.66	250.20	352.25	72.43	19.1

prior to analysis and tested under identical nitrogen atmosphere and heating rate conditions, small differences in solid content may contribute to marginal shifts in T_onset and char yield. Therefore, the observed variations in thermal behavior should be interpreted primarily in conjunction with structural changes induced by wet disk milling rather than attributed solely to intrinsic chemical modification. In addition, the slight reduction in onset degradation temperature observed for higher milling cycles may be attributed to increased surface area and enhanced exposure of thermally accessible regions following fibrillation, as previously reported for mechanically processed cellulose nanofibrils (Siró and Plackett 2010; Wang and Sain 2007). This phenomenon could be brought on by the pressure and friction that disrupt chemical bonds in the nanocellulose during the milling process, causing it to disintegrate into crystalline formations (Solhi et al. 2023). Furthermore, the reduction in diameter size and increase in surface area may cause heat degradation to start earlier (Abraham et al. 2013). The slight reduction in onset degradation temperature with increasing WDM cycles demonstrates a correlation between fibrillation intensity and thermal response, which is likely associated with increased surface area and exposure of less ordered cellulose regions after repeated mechanical treatment. In addition, the main decomposition temperature of all samples of nanofibers ranges very little due to the strong hydrogen bonds that hold the crystalline structure of cellulose together, largely even after nanofibrillation (Lichtenstein and Lavoine 2017).

As an instance, the thermal resistance of nanofibers toward beginning temperature decomposition can be influenced by the well-aligned crystallite structure of BCNF-5 and the rearranged crystallite structure of BCNF-10 and BCNFs-15 (Taflick et al. 2017). The thermal decomposition investigation revealed that, in comparison to BCNF-20, BCNF-5, BCNF-10, and BCNF-15 had higher thermal decomposition temperatures of 359.37°C, 358.81°C, and 355.41°C, respectively. Although BCNF-20 exhibited a slightly lower onset temperature, the overall degradation profile remained comparable to the other samples, indicating that the primary cellulose backbone structure was preserved. This is explained by the crystalline structure of BCNF-20 having a robust and unaltered native crystal order (French and Santiago Cintrón 2013; Yasim-Anuar et al. 2020a). This occurrence may have resulted from longer wet disk milling times that broke down the amorphous regions, which are hemicellulose, and lignin from improved accessibility during the creation of cellulose crystallinity in the nanofibers. The lack of nanocellulosic components in the system was the reason for the nanofibers' reduced char yield (Qing et al. 2013). This trend suggests that increased fibrillation enhances surface accessibility and exposure of less ordered regions, which slightly influences the initial thermal response, while the fundamental cellulose backbone remains largely unaffected.

Limitations of study

Although the present study provides a systematic investigation of the influence of wet disk milling (WDM) cycle number on the structural, physicochemical, and thermal properties of bamboo cellulose nanofibrils (BCNF), several limitations should be recognized, which also open avenues for future research.

First, the study focused exclusively on the effect of milling cycle number while maintaining other operational parameters constant. While this approach enables clear interpretation of cycle-dependent structural evolution, it does not explore the potential synergistic effects of rotational speed, disk gap distance, fiber concentration, or processing temperature. Future work incorporating multivariable optimization strategies could provide a more comprehensive understanding of process – structure relationships and energy efficiency. Additionally, the present study did not include quantitative compositional analysis of the removed lignin and hemicellulose fractions after delignification and alkaline treatment. Although the effectiveness of non-cellulosic component removal was indirectly supported by FTIR analysis and the improvement in crystallinity index, detailed chemical composition determination (e.g., Klason lignin content, hemicellulose quantification, or elemental analysis) was not performed. Furthermore, morphological characterization of the separated lignin and hemicellulose fractions using advanced techniques such as TEM or AFM was beyond the scope of this work. Future investigations should incorporate comprehensive compositional analysis and structural evaluation of the extracted fractions to better understand fractionation efficiency, optimize pretreatment conditions, and elucidate the relationship between biomass purification and subsequent nanofibrillation performance. Second, morphological characterization was primarily performed using TEM. Although sufficient to evaluate fibrillation behavior and diameter distribution, advanced nanoscale techniques such as TEM or AFM could offer deeper insights into fibril individualization, surface roughness, and nanofibril network formation. Third, thermogravimetric analysis was conducted under non-isothermal conditions, and minor variations in residual moisture or sample packing may influence onset degradation temperature and char yield. Future studies may incorporate kinetic modeling approaches or isothermal degradation analysis to further elucidate the thermal decomposition mechanism of BCNF.

Furthermore, yield determination was based on centrifugation separation between fibrillated and non-fibrillated fractions, which may introduce minor uncertainties. The implementation of complementary quantification techniques, such as rheological analysis or turbidity-based methods, could improve accuracy. Finally, the present work emphasizes physicochemical characterization without extending to application-specific performance evaluation. Future investigations should explore the functional performance of BCNF in areas such as polymer nanocomposites, barrier films, rheology modifiers, or adsorption systems to fully establish its industrial relevance. Overall, these limitations do not detract from the findings of the present study; rather, they provide a foundation for continued research toward process optimization, mechanistic understanding, and application-driven development of bamboo-derived nanocellulose.

The present work emphasizes physicochemical characterization, and therefore future investigations should extend toward performance-based evaluation in specific application systems. The fine nanoscale fibril diameter obtained after repeated WDM cycles suggests potential for enhanced stress transfer in polymer-reinforced composites, while the high isolation yield indicates favorable material efficiency for scalable production. In addition, the preserved crystallinity implies that structural rigidity and dimensional stability may be retained in composite matrices, whereas the observed thermal behavior indicates that the material can withstand moderate processing temperatures. These combined characteristics highlight the potential applicability of BCNF in polymer reinforcement, biodegradable composites, barrier materials, and other cellulose-based functional systems, although further validation under application-relevant conditions is required.

Conclusions

The present study systematically evaluated the influence of wet disk milling (WDM) cycle number on the yield and physicochemical properties of bamboo cellulose nanofibrils (BCNF). The morphological observations showed a progressive reduction in average fibril diameter from approximately 15 nm (BCNF-5) to 5 nm (BCNF-20), indicating continuous fibrillation with increasing milling cycles. Although the reduction became less pronounced beyond 15 cycles, the overall trend confirms that repeated mechanical treatment enhances fiber disintegration and promotes the formation of finer nanoscale structures. At the same time, the nanocellulose isolation yield remained consistently high, ranging from 97.6% to 98.8%, with only a slight increase observed at higher cycle numbers, demonstrating that the WDM process enables efficient fibrillation with minimal material loss.

The XRD analysis revealed that the crystallinity index remained within a narrow range of approximately 75% to 77% across all samples, indicating that the cellulose I crystalline structure was largely preserved despite intensive mechanical processing. In parallel, thermogravimetric analysis showed a gradual decrease in onset degradation temperature (Tonset) from approximately 339°C to 332°C with increasing cycle number, whereas the maximum degradation temperature (Tmax) remained relatively stable in the range of approximately 360°C to 365°C. This behavior suggests that increased fibrillation slightly influences the initial thermal response, likely due to enhanced surface area and exposure of less ordered regions, while the fundamental cellulose backbone and hydrogen-bonded network remain largely intact.

Taken together, these findings demonstrate that WDM provides an effective mechanical route for producing BCNF with refined nanoscale morphology, high yield, preserved crystallinity, and acceptable thermal stability. The successful utilization of Semantan bamboo (*Gigantochloa scortechinii*) further highlights its potential as a renewable and readily available feedstock for nanocellulose production. From an application perspective, the combination of fibril diameters within the 5–15 nm range, high material recovery, and retained structural integrity suggests that the produced BCNF is particularly suitable for polymer reinforcement, biodegradable composites, barrier materials, and other cellulose-based functional systems. Overall, the study provides meaningful insight into the relationship between WDM processing intensity and BCNF properties, while offering a practical basis for optimizing process conditions toward scalable and resource-efficient nanocellulose production.

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Author contributions

CRediT: **Jameelah Alhadi Salih Othman:** Conceptualization, Data curation, Funding acquisition, Methodology, Writing – original draft; **R.A. Ilyas:** Conceptualization, Formal analysis, Investigation, Methodology, Resources, Writing – original draft; **Norzita Ngadi:** Conceptualization, Data curation, Formal analysis, Investigation, Validation, Writing – original draft; **Abu Hassan Nordin:** Conceptualization, Formal analysis, Investigation, Validation, Writing – original draft; **M.F.M. Alkbir:** Conceptualization, Data curation, Investigation, Validation, Writing – original draft; **Nadlene Razali:** Conceptualization, Formal analysis, Investigation, Validation, Writing – original draft; **S.M. Sapuan:** Conceptualization, Funding acquisition, Methodology, Validation, Writing – original draft; **Mohd Nor Faiz Norrrahim:** Data curation, Investigation, Software, Writing – original draft; **Victor Feizal Knight:** Conceptualization, Data curation, Formal analysis, Writing – original draft.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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Availability of data and materials

All data and materials are available within this research article.

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