EXTRACTION AND CHARACTERIZATION ANALYSIS OF NANOCELLULOSE DERIVED FROM BAMBOO FIBERS

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ABSTRACT: Bamboo, a valuable and versatile resource in Malaysia, shows great potential for nanocellulose (NCC) production, a sought-after material across various industries. This study investigates a sustainable and cost-effective method to extract NCC from Phyllostachys Aurea bamboo fibers. Due to its high lignin content, bamboo requires a unique, time-intensive NCC synthesis process. The research focuses on acid hydrolysis using sulfuric (H_2SO_4) and hydrochloric (HCl) acids at concentrations ranging from 50-70 wt% for 120 minutes at 45°C. Results indicate hydrochloric acid as the optimal choice for NCC extraction, yielding well-dispersed, crystalline cellulose with minimal aggregation. Characterization techniques such as Field Emission Scanning Electron Microscope (FESEM), X-Ray Diffraction (XRD), and Raman Spectroscopy analysis confirm the quality of the extracted NCC. This method surpasses others by enhancing dispersion and crystallinity while reducing aggregation. The NCC derived from bamboo fibers presents a significant opportunity for the nanocomposites sector, promoting the use of bamboo, a sustainable material, in eco-friendly design and development initiatives.

KEYWORDS: *Bamboo Nanocellulose (NCC), Acid Hydrolysis, Phyllostachys Aurea, Sustainable Extraction, Nanocomposites*

1.0 INTRODUCTION

Nanocellulose, derived from plant materials, is gaining attention due to its unique properties and potential applications across various industries, such as packaging, military, design, and electronics. It acts as a strengthening agent and offers advantages like biodegradability, low density, and high mechanical strength. Bamboo, a notable source of nanocellulose, is economically renewable and can be harvested every 3 to 5 years, unlike timber which takes much longer [1-3]. Furthermore, because of its availability, green materials, and low cost, it provides another raw material option for cellulosic sources, and this a feature provides a sustainable cellulose source for nanocellulose production [4]. Thus, it is clear that the use of natural fibres, such as the raw material used in the production of bamboo fibres, has a minimal impact on human well- being and the environment, and it is critical to broaden research on the use of natural materials for present as well as future uses Nanocellulose exists in different forms like nanocrystal cellulose (NCCs), cellulose nanofibrils (CNFs), and bacterial cellulose (BC), each with distinct characteristics [5]. The extraction process involves both chemical and mechanical treatments, with acid hydrolysis being a key method. Acid hydrolysis effectively dissolves amorphous regions, yielding crystalline cellulose. Critical parameters such as temperature, hydrolysis duration, and acid concentration influence the quality of nanocellulose produced. Pre-treatment, often using alkali, is necessary to make the cellulose more accessible for the hydrolysis process. Bamboo-derived nanocellulose presents significant opportunities for sustainable and innovative applications, offering a balance between environmental conservation and meeting societal needs for diverse material resources [6].

This report examines the preparation and characterization of nanocellulose (NCC) from bamboo fibers through acid hydrolysis, resulting in high crystallinity and stable colloidal suspensions of cellulose nanocrystals. The process begins with alkaline treatment to extract NCC from bamboo fibers, followed by acid hydrolysis. Bamboo is chosen due to its rapid growth, broad availability, and sustainable properties. However, commercialization is hindered by the lack of standardized characterization methods, which complicates comparison and validation across different studies [7]. This standardization is crucial for maximizing the economic potential of NCC from bamboo and facilitating its broader application in industries such as textiles, packaging, construction, electronics, and biomedical fields. Focusing specifically on Phyllostachys Aurea bamboo fiber, the research aims to optimize extraction methods, enhancing the yield, purity, and quality of NCC. Due to high lignin content, extracting cellulose nanocrystals from bamboo requires significant energy and time, presenting challenges that must be addressed through careful optimization of acid hydrolysis parameters [8]. The study involves two main stages: an alkali pre-treatment to extract pure cellulose from bamboo fibers, followed by acid hydrolysis to produce nanocrystals. The process is optimized for factors like acid concentration, hydrolysis duration, and temperature, with the ideal setting identified at 50°C for maximum crystallinity. The research aims to establish standardized procedures for characterization and production, paving the way for the commercialization and industrial application of bamboo-derived nanocellulose. Table below shows a brief composition of bamboo fibres.

Table 1: composition of typical bamboo fibers.

Analysis	**Composition (%wt)**
Moisture	10.99%
Volatile	83.70%
Ash	1.4%
Fixed Carbon	3%
Lignin	1.5%
Cellulose	47.2%
Hemicellulose	23.9%

2.0 MATERIALS AND METHODS

This study explored the use of bamboo as a sustainable and efficient raw material for nanocellulose production. Bamboo's rapid growth, minimal cultivation requirements, and high cellulose content make it an attractive green alternative feedstock [9]. Additionally, the inherent morphological and structural properties of bamboo fibers, including high aspect ratio and interconnected networks, contribute to the production of nanocellulose with desirable characteristics. The source of the bamboo was ethically obtained with permission from a local resident. This research utilized commercially available analytical-grade chemicals. Sodium hydroxide (NaOH) at a concentration of 5 wt% was employed in a pre-treatment stage to remove hemicellulose and lignin through an alkali treatment. Sulfuric acid (H_2SO_4) was also used in subsequent stages, however the specific concentration varied for the acid hydrolysis process. Distilled water was used throughout the experiments. There are several experimental processes to be carried out in order to produce nanocrystals cellulose from bamboo fibres, which include sample preparation, alkali pretreatment, and acid hydrolysis in separation of nanocrystals cellulose. The raw material used in this experiment is bamboo fibre specifically from 'Phyllostachys Aurea'. Approximately a total of 2 kilograms of bamboo was prepared. The bamboo was first cut into little pieces of roughly around 2-4 cm and cleaned four to five times with hot water. The bamboo sample was then kept at room temperature for 24 hours. The smaller bits of material were then crushed down using a high-speed blender to quickly and efficiently grind soft to medium-hard, brittle, and fibrous sample.

2.1 Pre-treatment by Alkali Treatment & Preparation of Nanocrystals Cellulose

The bamboo fibre sample was treated at room temperature for 24 hours with a 5% concentration of sodium hydroxide (NaOH) to remove lignin, hemicellulose and to disrupt the hydrogen bonds between various cellulose chains. The mixture was then filtered and washed numerous times with distilled water until the pH reached neutral. The resultant mixture was then dried for 3 hours in a drying oven set at 100°C. The acid hydrolysis was carried out using different concentrations of sulphuric acid (H_2SO_4) which were 50 wt%, 55 wt%, 60 wt%, 65 wt%, and 70 wt% while the hydrolysis period was 120 minutes. Temperature correlation was set at 45°C. According to the previous research, a concentration of 60-65 wt% is the ideal concentration for conducting the acid hydrolysis procedure, and the optimum concentration was determined to be 60 wt%. The cellulose-to-acid solution ratio was set at 1:14 (w/v). As a result, the new parameter was created to investigate if the concentration affects the hydrolysis time. The alkali-treated bamboo fibres have been mixed with sulphuric acid. The experiment was carried out using various hydrolysis times and concentrations. The mixture was continually stirred until the hydrolysis period was finished. The hydrolyzed cellulose was dialyzed multiple times with distilled water until the pH was 7. The sample was then dried in an oven at 50°C. The cellulose nanocrystals that were obtained will be maintained at room temperature for analysis.

Table 2: Arrangement of sample condition for acid hydrolysis

Sample	Acid Concentration (wt%)	Hydrolysis Temperature (°C)	Hydrolysis Temperature (°C)
H_2SO_4	50	120	45
	55	120	45
	60	120	45
	65	120	45
	70	120	45

2.1 Characterization of Nanocrystals Cellulose (NCCs) of Bamboo Fibres

The following part covers the characterization of nanocrystals cellulose of bamboo fibres in order to meet the research's second and third objectives. X-Ray Diffraction (XRD) Analysis, Fourier Transform Infrared Spectroscopy (FTIR), and morphological analysis using Field Emission Scanning Electron Microscopy (FESEM) were utilized in the characterization. Figure 1 shows a resultant nanocellulose after going through selected parameter.

Figure 1: A resultant nanocellulose after going through selected parameter.

3.0 RESULTS AND DISCUSSIONS

The crystallinity index (CrI) and crystallite size of cellulose that had been separated from bamboo fibre were measured in an experiment utilizing X-ray diffraction (XRD). The XRD of both untreated and alkaline-treated bamboo fibre is displayed in Figure 2. Whereas the peak intensity of diffraction of the amorphous area of cellulose is given by Iam, and the greatest peak intensity value for crystalline cellulose is provided by $I200$ at about $2\theta = 22.0°C$ to 24 [10]. By using X-ray diffraction to determine the crystalline behaviour of fibres, it was discovered that the bamboo nanocellulose had a greater degree of crystallinity. They also shows XRD results illustrate that treated alkaline bamboo fibre has a greater peak than untreated bamboo fibre [11]. The crystalline structure of cellulose in both the untreated and alkaline-treated bamboo fibre samples was responsible for their high peak intensity of 2 ϴ = 22, while the presence of a wide peak at around 16.69º suggests the amorphous arrangement [12].

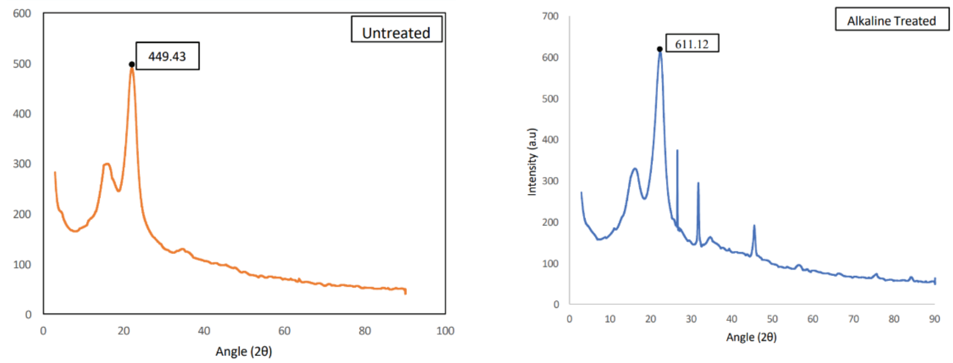

Figure 2: X-ray diffraction pattern of alkaline untreated and treated bamboo fibre.

While, the FTIR spectrum shows that treated bamboo fibres differ more from untreated bamboo fibres in terms of their spectra's pattern. This has to do with the alkaline process that was employed to get rid of lignin and hemicellulose prior to the chemical process, which is acid hydrolysis. The vibration peak at 3315cm^{-1} area for untreated bamboo fibres indicates that the hydrogen bonds O–H were bending due to absorbed water in the cellulose, but the vibration peak at 3328cm^{-1} for alkaline-treated bamboo fibres indicates that the hydrogen bonds O–H [13-15]. The C-H stretching of untreated bamboo fibres is shown by the peaks at 894.90 cm^{-1}, whereas alkaline treated bamboo fibres have peaks around 894.01cm^{-1}. In addition, it was discovered that the alkaline treatment's spectra included peaks about 1024.02cm^{-1}, which corresponds to the C-O stretching peak. These peaks were marginally smaller than those of the untreated bamboo fibres, and 1020.16cm^{-1} indicates the majority of the hemicellulose and lignin had been eliminated. Figure 3 shows untreated and alkaline treated bamboo fibres under FTIR Spectra.

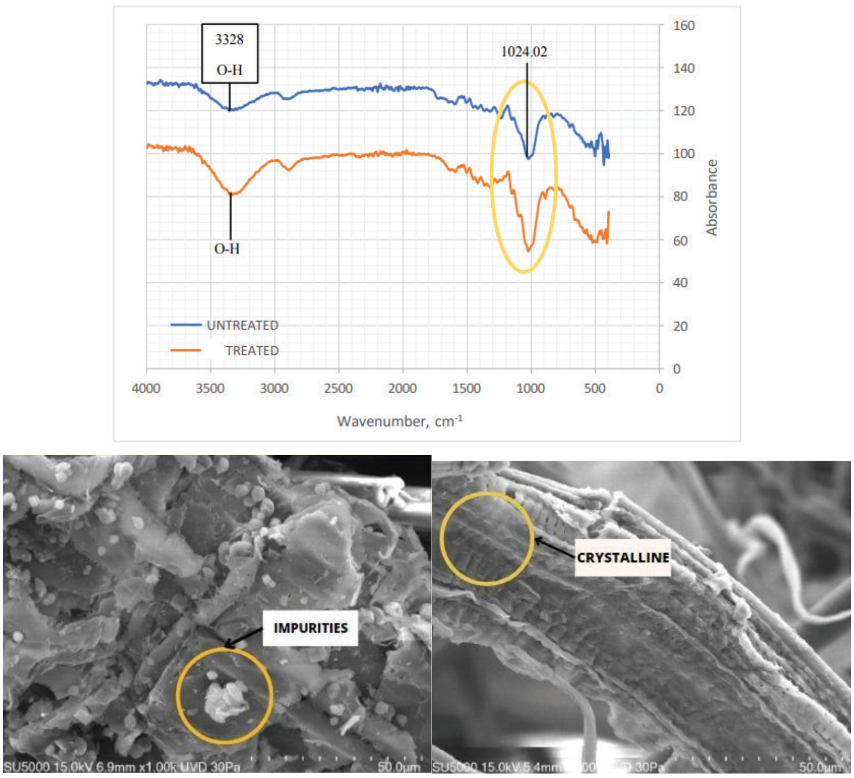

Figure 3: Untreated and Alkaline treated bamboo fibres under FTIR Spectra and SEM images.

3.1 Characterization of Sulphuric Acid Hydrolysis of Bamboo Fibre

Figure 4 demonstrated acid hydrolysis, H_2SO_4 with a concentration of 50–70wt%, a set duration of 120 minutes, and a constant temperature of 45°—were compared. The XRD graph indicates that after acid hydrolysis and alkaline purification, the crystallinity peak rises [16]. It is possible to conclude that the acid has cellulose type I structure based on the diffraction pattern, which shows the amorphous material to have a wide peak at about 17˚. The result indicates that there are three diffraction peaks for nanocellulose: 2θ = 5°, 17°, and 22° [17]. The sample with acid concentration of 50-60wt% does not show presence of peak for the amorphous region, make the result for crystallinity index could not be obtained. Figure 4 below shows the XRD patterns.

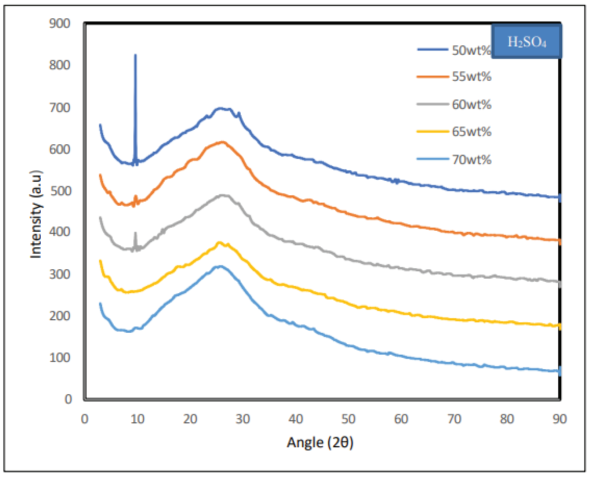

Figure 4: XRD analysis of H_2SO_4

The morphological surface in Figure 5 has a rod-like form, which may be related to the amorphous cellulose's primary breakdown. The most popular method for acid hydrolysis is H_2SO_4, as it produces highly crystalline, rigid, and effective results when amorphous materials are eliminated [18].

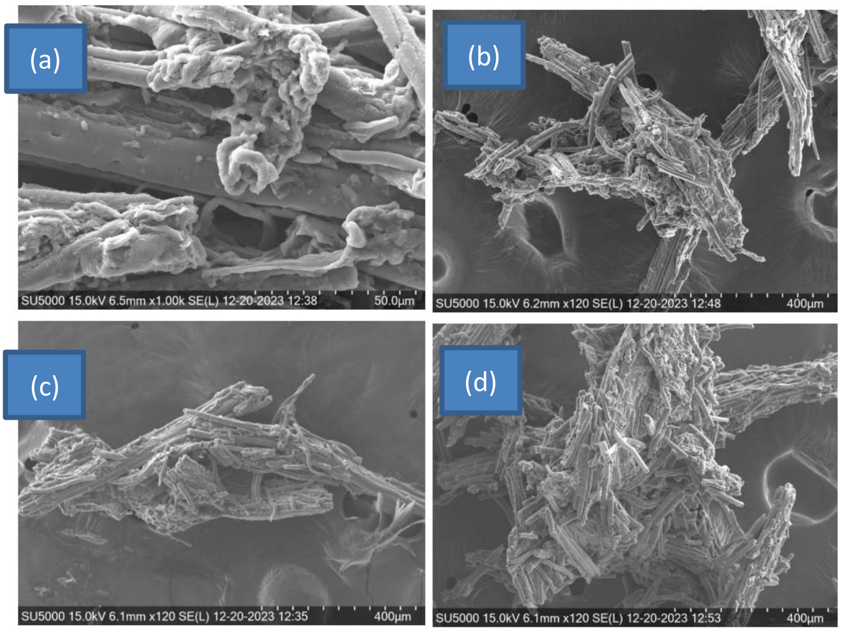

Figure 5(a) and (b): 50wt% surface morphology. (c) and (d): 70wt% surface morphology, on scale 10μm and 100μm

4.0 CONCLUSION

The study successfully produced nanocellulose from bamboo fibers using acid hydrolysis. By evaluating different parameters, the optimal conditions were identified as hydrochloric acid (HCl) at a concentration of 60 wt%, a temperature of 45°C, and a duration of 120 minutes. These conditions yielded nanocellulose with a high crystallinity index and good crystallite size, as confirmed by X-ray diffraction (XRD) analysis. Fourier transform infrared spectroscopy (FTIR) analysis indicated the successful removal of lignin and hemicellulose during the pre-treatment stage with 5 wt% sodium hydroxide (NaOH). Finally, scanning electron microscopy (SEM) visually confirmed the removal of impurities from the bamboo fibers after the pre-treatment process.

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