# Surface Morphology and Thermal Properties of Cellulose from Three Species of Bamboo

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Bamboo is a rapidly growing biomaterial and a high-yielding renewable resource indigenous to Asia and Oceania. Bamboo and wood differ in a variety of ways, including macroscopic and microscopic features, chemical composition, physical and mechanical properties. For this reason, the purpose of this study was to characterize the surface morphology and thermal properties of cellulose in three bamboo species: *Schizostachyum brachycladum*, *Bambusa vulgaris*, and *Bambusa oldhamii*. The cellulose in bamboo was removed using a chemical process that comprised dewaxing, delignification, and mercerization. The surface morphology of the cellulose was characterised using Field Emission Scanning Electron Microscopy (FESEM) and Atomic Force Microscopy (AFM). The thermal properties were then analysed by Thermo-gravimetric Analysis (TGA). The FESEM study revealed that mercerization resulted in fibrillation and fibre separation, which increased the effective surface area accessible for reinforcement. From the TGA analysis, bamboo cellulose was found to be thermally stable. Based on these findings, more research is needed on the qualities of bamboo, as well as cost-effective technology and management strategies. With the use of innovative procedures and adaptable technologies, bamboo can be processed into a wide range of goods and compete successfully with wood and other raw materials in the future.

**Key words**: Bamboo; cellulose; morphological; thermal analysis

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Natural fibres have been used as reinforcement materials since the early twentieth century. Since 1950, there has been a growing desire for stronger, stiffer, and lighter composite materials in areas including aircraft manufacturing, transportation, and construction. As a result, high-performance fibres must be used as reinforcements. Advanced composites, like metals, have low specific gravity, greater strength, and a higher modulus of elasticity [1]. Like other plant-based natural fibres, bamboo has a diverse set of characteristics and properties, and is especially intriguing because of its tremendous potential, notably as a composite material for the manufacture of polymer composites [2].

Bamboo is an agricultural crop that can be used in the research and design of polymer composites [3]. Bamboo grows prolifically in Asia and the Middle East [4]. Bamboo fibres are not uniformly distributed, with heavier fibres on the outside and sparsely distributed fibres inside. Bamboo fibres have great strength because of the longitudinal reinforcement supplied by the strong fibres and the fact that it is multi-layered and hollow [3]. Bamboo is a popular plant fibre with great potential in the polymer composite sector [4]. Its structural diversity, mechanical qualities, fibre extraction, chemical manipulation, and thermal properties make it flexible for use in composites [5]. The main reason for making composites out of bamboo instead of synthetic fibres is to acquire its superior characteristics.

Bamboo fibres have been employed as a filler, and a twin-screw extruder has been used to mix bamboo with a biodegradable polymer to make bamboo reinforced polymer composites [5]. Bamboo's diversity is reflected in the number of species found worldwide; bamboo is a fast-growing grass with over 1000 species [4]. Bamboo fibre is mostly composed of cellulose (73.83%), hemi-celluloses (12.49%), and lignin (10.15%). Protein, fat, pectin, tannins, colours, and ash are the other components. These constituents are in the cell cavity or specific organelles of bamboo and play a vital role in its physiological function [6].

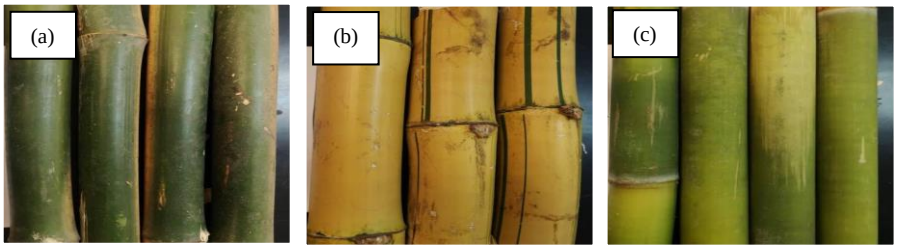


**Figure 1.** Three different species of bamboo *(a) Schizostachyum brachycladum; (b) Bambusa vulgaris; (c) Bambusa oldhamii.*

In this study, three bamboo species, namely *Schizostachyum brachycladum*, *Bambusa vulgaris*, and *Bambusa oldhamii* were extracted and characterized in terms of their surface morphology. Size and shape were determined using Atomic Force Microscopy (AFM) and Field Emission Scanning Electron Microscopy (FESEM). Finally, the thermal stability of natural bamboo fibres was analysed by thermogravimetric analysis (TGA).

## MATERIALS AND METHODS

### 1. Materials

As shown in **Figure 1**, samples of three different bamboo species, *Schizostachyum brachycladum*, *Bambusa vulgaris*, and *Bambusa oldhamii* were taken from the Ethno-botanical garden at the Sabah Museum (5°57′38″N 116°04′18″E) in Kota Kinabalu, Sabah, Malaysia. Sodium hydroxide (NaOH) and sodium hypochlorite solution (NaOCl) were purchased from Sigma Aldrich, USA. Laboratory-grade chemicals and reagents were utilized in the experiments. Deionized water was used throughout the study.

### 2. Extraction of Bamboo Cellulose

Alkali treatment was used to remove bamboo cellulose [7]. Bamboo tree internodes were cut into 10.0 cm pieces (the node portions and thin layers of exodermis and endodermis bark were removed). The cylindrical section of the culm was peeled longitudinally into strips that were roughly 0.2 cm thick, 12.0 - 15.0 cm long, and 3.0 cm wide. In order to remove dust and contaminants from the surface of the bamboo strips, they were washed using distilled water at room temperature. Following that, they were gently rolled to extract the bamboo fibre.

Next, the bamboo fibres were ground to a size of around 500 µm. Other components of bamboo fibres, such as lignin, hemicellulose, and pectin, had to be removed before the cellulose fibres could be isolated. The completion of this process involved soaking the bamboo fibres in a sodium hydroxide (NaOH) solution of 4 %, 6 % and 8 % by volume of water at a temperature of 30 °C for a period of 72 h [8]. Then, the bamboo fibres were treated with a bleaching process using 5 wt % sodium hypochlorite solution (NaOCl) at 80 °C for 3 h. Subsequently, the bamboo fibres under-went an acid hydrolysis process where the dissolved components were treated with 2.0 mol/ L of hydro-chloric acid (HCl) at 80 °C. The overall extraction process is simplified in **Figure 2**. The percentage yield of the bamboo cellulose collected overall was calculated using Equation (1).

$$Percentage\ yield\ (\%) = \frac{weight\ after\ hydrolysis}{Initial\ weight} \times 100\ \% \qquad (1)$$

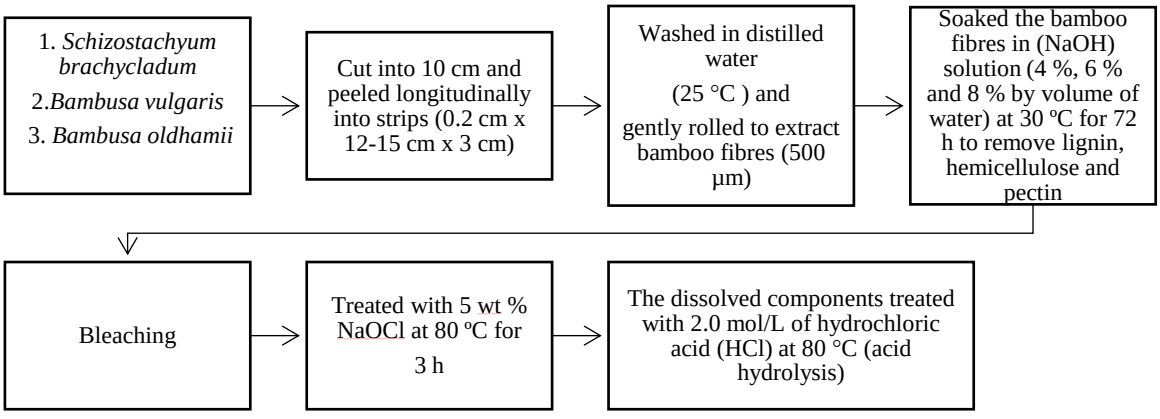


**Figure 2.** Flow diagram of bamboo cellulose extraction

## 3. Characterization of Bamboo Cellulose

### 3.1. Field Emission Scanning Electron Microscopy (FESEM)

A LEO Supra 50 Vp FESEM equipped with secondary, backscattered electron, and EDX detectors was used to track morphological changes in the bamboo cellulose. The FESEM system used changing $N_2$ gas pressure to function. Before analysis, samples were gold-sputtered using a Polaron SC 515 sputter coater with a sample thickness of 20 nm [9].

### 3.2. Atomic Force Microscopy (AFM)

A Burker Icon atomic force microscope was used to examine the samples (CA, USA). The experiment was carried out by consistently dropping aqueous cellulose nanocrystals on a glass slide, which were then dried in air before analysis. At ambient conditions, pictures were captured by tapping mode on the bamboo cellulose samples at a scan rate of 4.80–6.00 m/s [7].

### 3.3. Thermogravimetric Analyzer (TGA)

A thermogravimetric analyzer was used to test the sample's thermal stability. Thermal analysis (TA) was performed on 3–5 mg samples by permitting thermal degradation in the TA Instruments Q500 thermogravimetric analyzer and TGA. The samples were scanned at a rate of 20 °C/min and a temperature range of 20–1000 °C in a flowing nitrogen gas environment to obtain a TGA curve. A derivative thermogravimetric (DTG) curve was plotted using TA Universal Analysis software [7].

## RESULTS AND DISCUSSION

### 1. Extraction of Bamboo Cellulose

The isolation of cellulose from bamboo in this work was accomplished by acid hydrolysis. It is worth noting that the chosen technique is easily scalable and can be regulated by adjusting the concentration of solvents/chemicals employed, the degree of polymerization, the crystallinity, the particle size distribution, and the thermal characteristics, among other parameters. **Figure 2** illustrates the multiple steps involved in isolating cellulose from bamboo fibres, including an active alkali treatment accompanied by bleaching with NaOCl at 80 °C and acid hydrolysis. The final product of bamboo cellulose, as shown in **Figure 3**, had an average yield of 60.2 %. The yield obtained was slightly higher than the 54.61 % obtained in a previous study by Rasheed et al. [7], as there was a difference in the concentration of acid used.

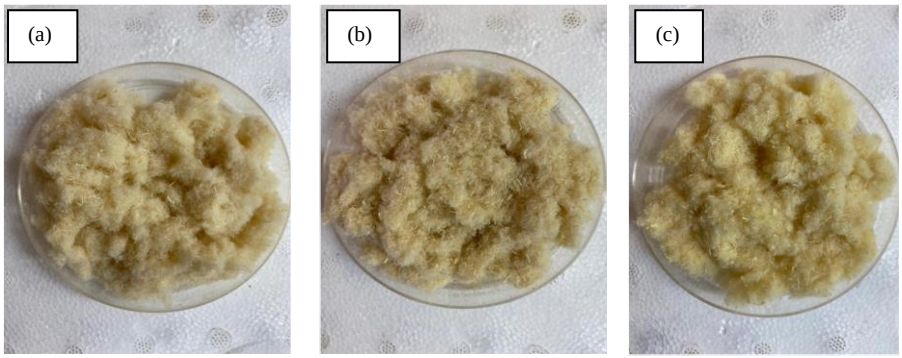


**Figure 3.** Bamboo cellulose extracted from three different species of bamboo (a) *Schizostachyum brachycladum*; (b) *Bambusa vulgaris*; (c) *Bambusa oldhamii*.

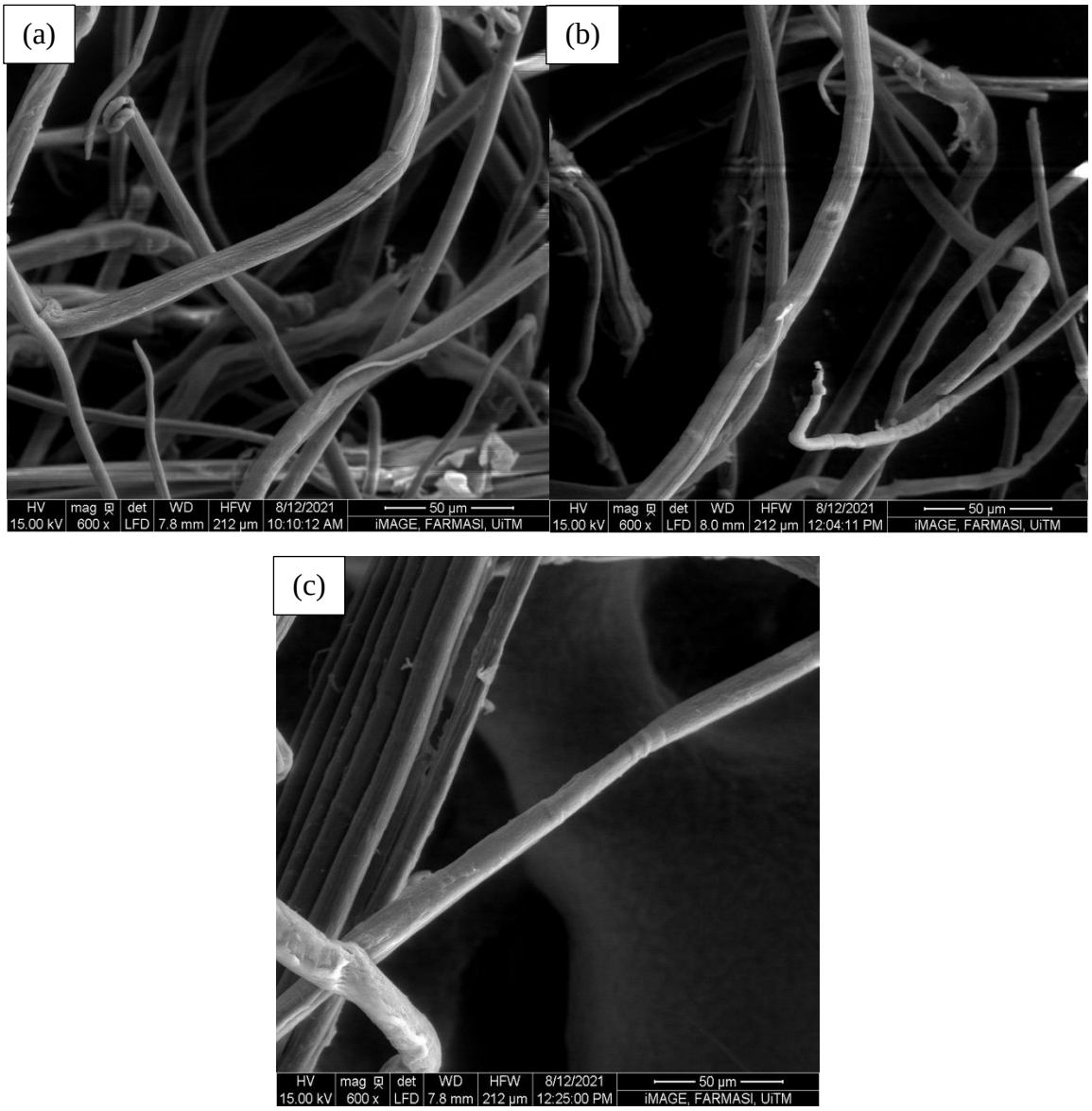


**Figure 4.** Field Emission Scanning Electron Micrographs of bamboo celluloses at 600x magnification (a) *Schizosrachyum brachycladum* (b) *Bambusa vulgaris* (c) *Bambusa oldhamii*

## 2. Field Emission Scanning Electron Microscopy (FESEM)

**Figure 4** depicts the surface morphology of three different bamboo species. From these photographs, the cellulose fibres displayed rod-like structures with lengths ranging from 5.43 – 13.29 µm, as indicated by the FESEM images in **Figure 5.** The nanostructures of bamboo cellulose appear to be uniformly distributed and not aggregated [9]. The bamboo cellulose samples (see **Table 1)** had diameters of less than 10 nm, smaller than the 20 nm nano-cellulose generated from bamboo in a previous study by Yu et al. [10]. This difference may be due to the different extraction methods utilizd. Additionally, as shown in **Figures 4** and **5**, the isolated bamboo cellulose was homogenous and well-dispersed, indicating excellent colloidal suspension stability [11].

**Table 1**: Cellulose sizes for three different bamboo species

| Bamboo Species | Cellulose Size (µm) |
|---|---|
| *Schizostachyum brachycladum* | 7.69-10.82 |
| *Bambusa vulgaris* | 5.43-11.15 |
| *Bambusa oldhamii* | 9.53-13.29 |

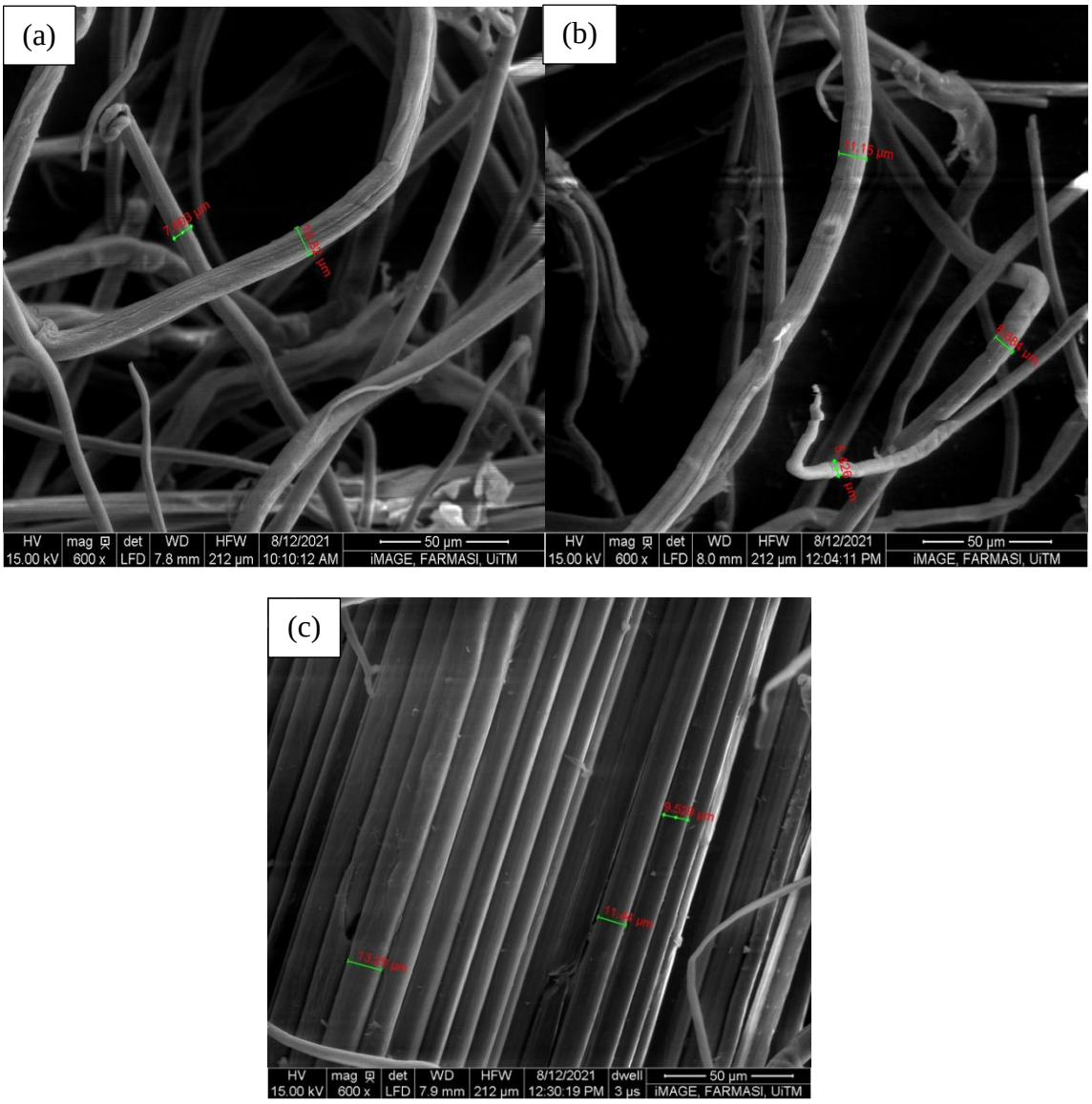


**Figure 5.** Field Emission Scanning Electron Micrographs of bamboo celluloses at 600x magnification (a) *Schizosrachyum brachycladum* (b) *Bambusa vulgaris* (c) *Bambusa oldhamii*

As illustrated in **Figure 5**, the bamboo cellulose had a smooth surface, indicating that imperfections in the outer layer of the fibre had been removed as a result of the alkali treatment during the delignification process [12].

### 3. Atomic Force Microscopy (AFM)

On the one hand, AFM can be used in place of scanning electron microscopy (SEM) for examining fibre morphology in ambient circumstances. On the other hand, AFM can be used in force spectroscopy mode to quantify the forces associated with surface interactions. The approach has been widely utilized in the pulp and paper industry to investigate the interactions between cellulose surfaces [13]. AFM is a precise technique that provides qualitative and numerical information about the surface topography of natural fibres with uneven surfaces containing lignin, a wax-like impurity substance [12]. **Figures 6-8** illustrate the two-dimensional and three-dimensional topographical structures of cellulose in the three different bamboo species, as determined by AFM. The three-dimensional topographical structure of bamboo cellulose demonstrates that there has been a significant improvement in the surface structure [14]. The three critical surface roughness parameters (Ra – the mean value of the surface about the centre plane, Rq – the root mean square of the Z data, and Rz – the mean difference between the highest peaks and lowest valleys) were determined using these AFM images, and the results are summarised in **Table 2**. Colloidal fouling is inversely proportional to the surface roughness of the membranes, implying that as surface roughness reduces, colloidal fouling decreases [15]. Numerous

studies demonstrate that changes in the roughness parameters of polymeric membranes are proportional to changes in pore size which promote hydrophobic properties [15]. According to earlier research, the average thickness or size range of bamboo cellulose is approximately 32 nm, which leads to better interfacial bonding between fibre and matrix [7,8].

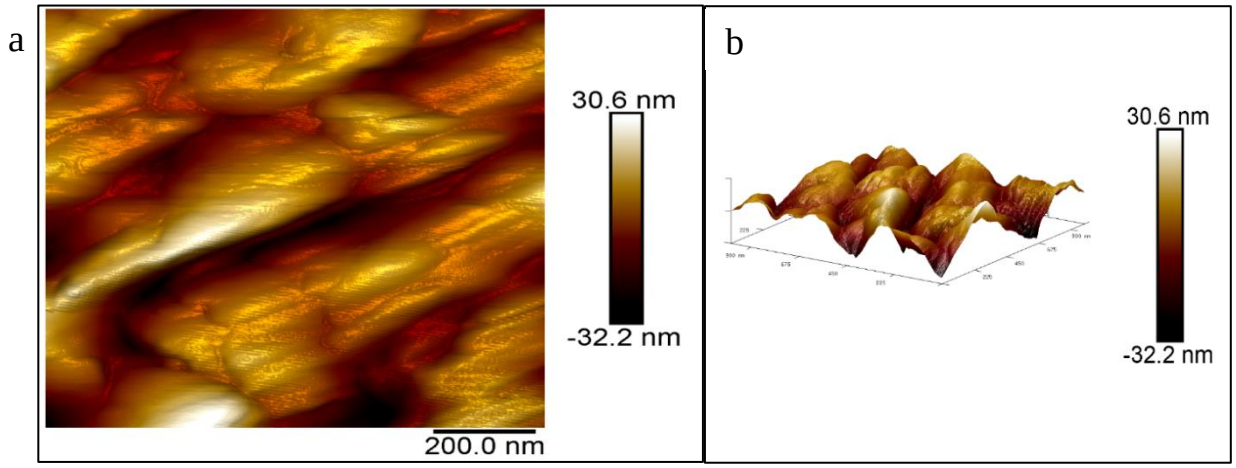


**Figure 6:** (a) 2D and (b) 3D AFM micrographs of cellulose from *Schizostachyum brachycladum* bamboo

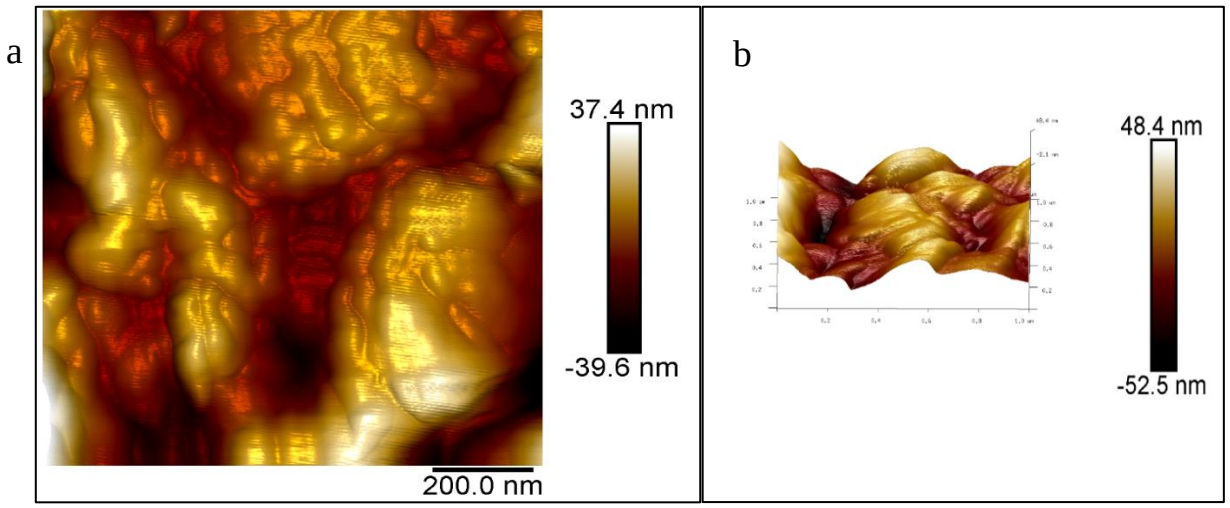


**Figure 7:** (a) 2D and (b) 3D AFM micrographs of cellulose from *Bambusa vulgaris* bamboo

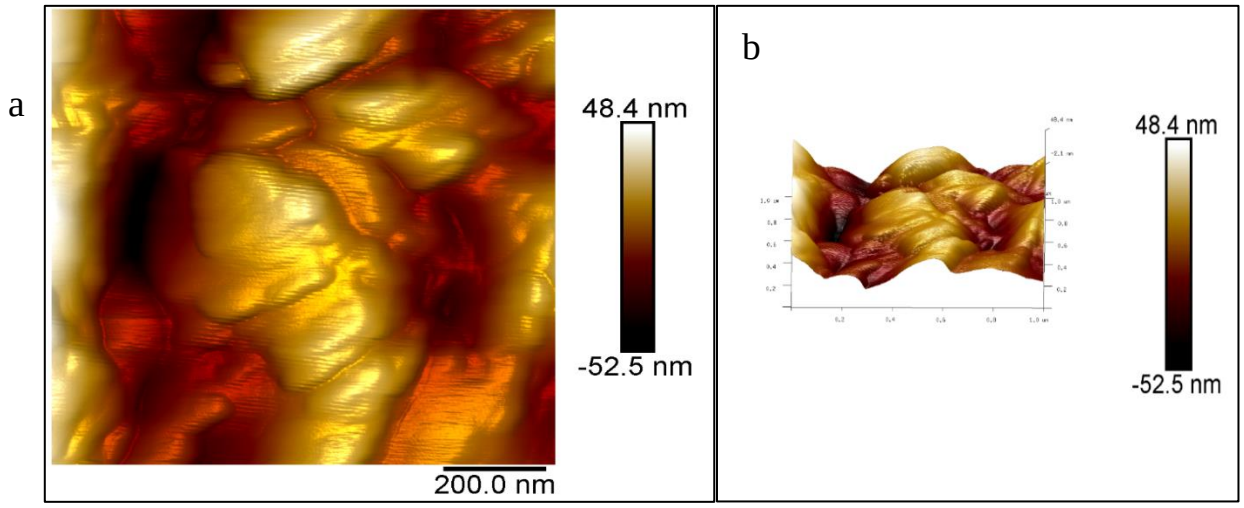


**Figure 8:** (a) 2D and (b) 3D AFM micrographs of cellulose from *Bambusa oldhamii* bamboo

**Table 2.** Summarized AFM data of cellulose from different species of bamboo

| Sample | Average Width | Average Height | Surface Roughness, Rq | Surface Roughness, Ra | Surface Roughness, Rmax |
|---|---|---|---|---|---|
| ***Schizostachyum brachycladum*** | 294.556 (nm) | 25.235 (nm) | 32.8 nm | 25.4 nm | 212 nm |
| ***Bambusa vulgaris*** | 452 (nm) | 329 (nm) | 24.3 nm | 19.1 nm | 133 nm |
| ***Bambusa oldhamii*** | 602.282 (nm) | 329.670 (nm) | 36.1 nm | 28.3 nm | 176 nm |

## 4. Thermogravimetric Analyzer (TGA)

The heat stability of cellulose is critical for its usage as a reinforcement material in the manufacture of bio-composites. **Figures 9**, **10**, and **11** illustrate the TGA and differential thermogravimetry (DTG) curves for cellulose from three different species of bamboo. The first degradation process for bamboo cellulose began at 70 °C and continued until 100 °C due to the evaporation of the remaining water in the cellulose components [16,17] resulting in a weight loss of around 95 %. The second degradation step occurred between 200 and 410 °C. These bamboo celluloses displayed only minor temperature changes related to the destruction of cellulosic chains and depolymerization of cellulosic compounds extracted from bamboo fibre [12]. Most of the degradation occurred between 300 and 400 °C, consistent with cellulose pyrolysis [16]. At 70 °C, the initial weight loss began, while the decomposition rate of bamboo cellulose remained constant at 5-10 %. Due to the breakdown temperature of hemicellulose, lignin, and pectin, free water evaporation resulted in weight loss in the samples. Due to the limited size range of the chains and the significant surface area of the free ends in the bamboo cellulose sample, isolated bamboo cellulose exhibited lower thermal stability [18]. Additionally, as shown in **Figures 9-11** and **Table 3**, cellulose from *Schizostachyum brachycladum*, *Bambusa vulgaris*, and *Bambusa oldhamii* recorded significant peaks at 366 °C, 367 °C, and 370 °C, respectively. Additionally, **Table 3** demonstrates that the weight residues for bamboo cellulose were 20.15 %, 21.38 %, and 19.96 % for *Schizostachyum brachycladum*, *Bambusa vulgaris*, and *Bambusa oldhamii*, respectively. The presence of char-forming flame retardant compounds might be interpreted as an indication of the elevated char residue achieved with bamboo cellulose [19,20]. It was previously observed that when bamboo fibres were treated with 6-10 % NaOH solutions, the thermal stability of the fibres increased. This is likely due to the chemical composition and microstructure discrepancies between fibres extracted using chemical and mechanical approaches [21]. The DTG graphs followed a similar trend to the TGA graphs for all samples.

**Table 3.** Thermal analysis data for bamboo cellulose

| Samples | Obtained peaks | | | |
|---|---|---|---|---|
| | Initial degradation temperature (°C) | DTG Peak temperature (°C) | Maximum Weight loss (%) | Weight residue (%) |
| ***Schizostachyum brachycladum*** | 70 | 366 | 94 | 20.15 |
| ***Bambusa vulgaris*** | 70 | 367 | 95 | 21.38 |
| ***Bambusa oldhamii*** | 70 | 370 | 94 | 19.06 |

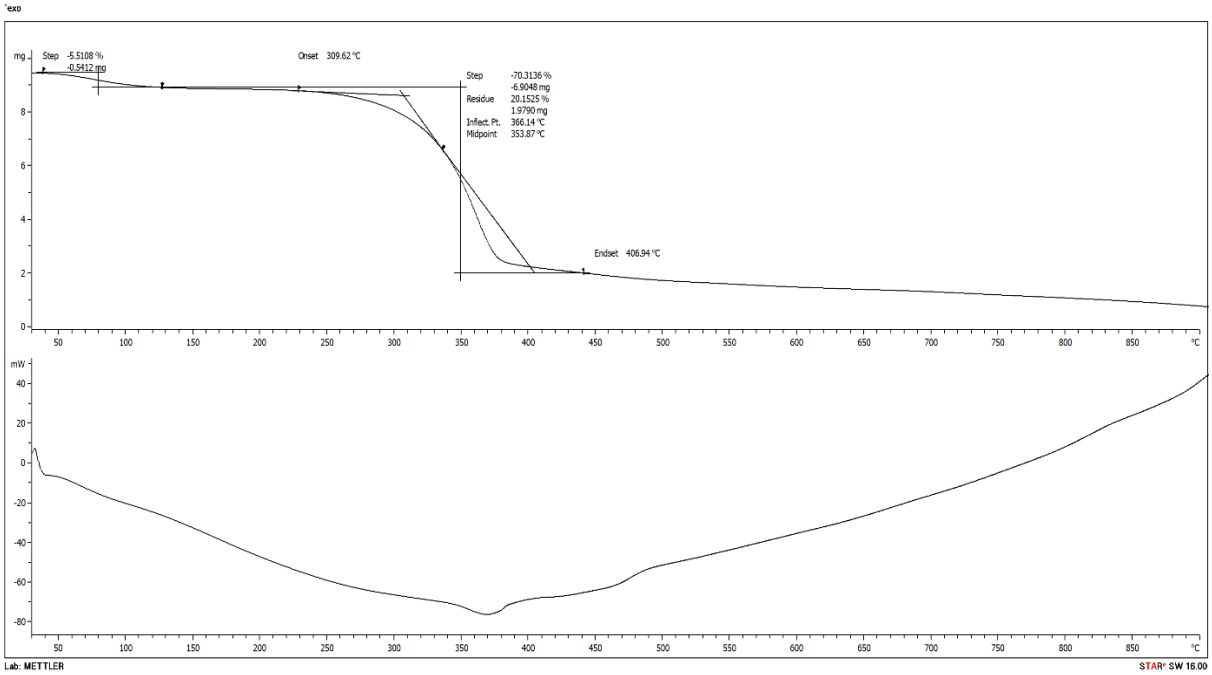


**Figure 9.** TGA and DTG curves for *Schizostachyum brachycladum* bamboo cellulose

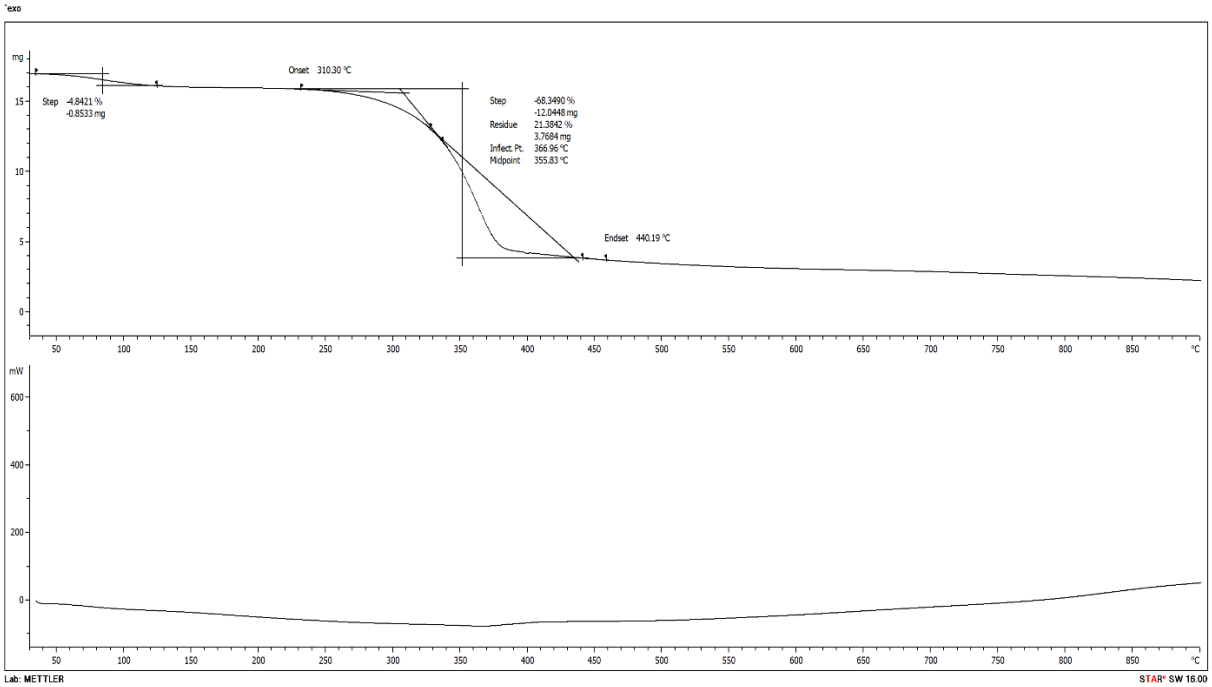


**Figure 10.** TGA and DTG curves for *Bambusa vulgaris* bamboo cellulose

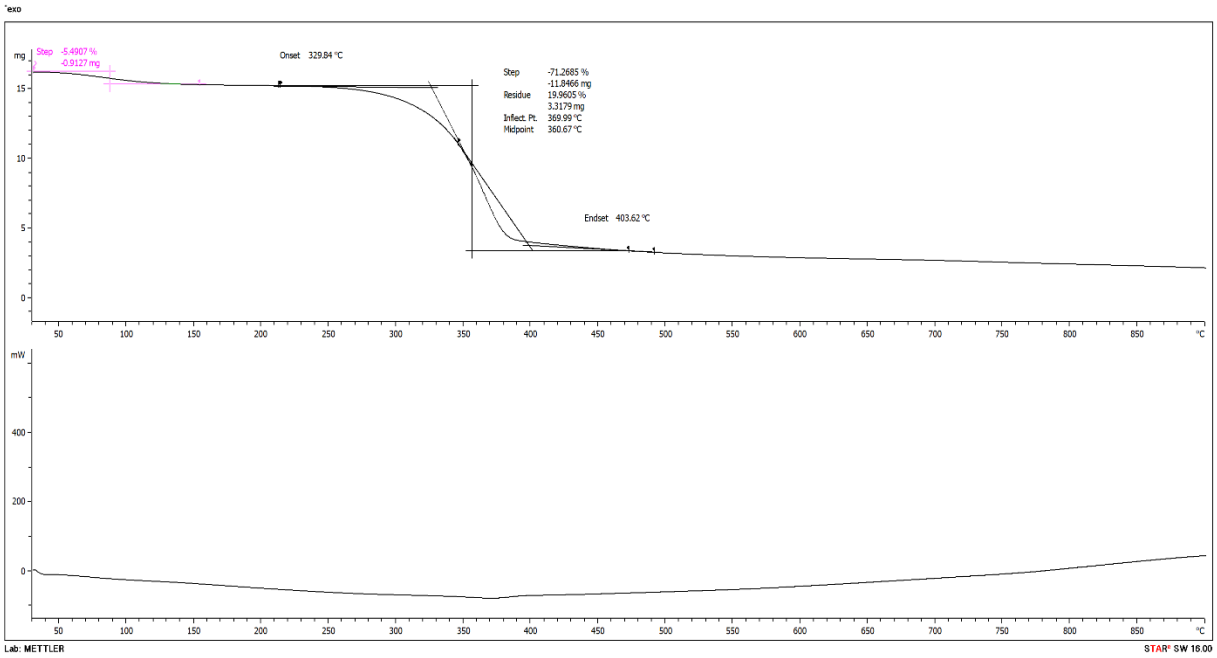


**Figure 11.** TGA and DTG curves for *Bambusa oldhamii* bamboo cellulose

## CONCLUSION

Based on the results, it is evident that the bamboo cellulose developed in this study has superior traits and properties. Cellulose was discovered to be the primary ingredient of the final products. FESEM photographs revealed that the diameter of the fibres ranged between 5 and 13 nm. Surface roughness is critical for achieving super hydrophobicity while bamboo cellulose had a low surface roughness, as determined by AFM. TGA analysis revealed that bamboo cellulose had lower thermal stability due to its smaller size and high surface area. Thus, these materials can be used as reinforcements in bio-composites for various applications, including biomedicine and packaging. These composites can also be used in synthetic materials and may emerge as an environmentally friendly and sustainable material in the future.

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