



RESEARCH ARTICLE

SURFACE MORPHOLOGY, COMPOSITIONS, CRYSTALLINITY, AND THERMAL STABILITY OF MICROCRYSTALLINE CELLULOSE DERIVED FROM *DENDROCALAMUS ASPER* BAMBOO FIBER**Muhammad Aqil Asyraf Mohd Mahadi¹, Nurul Syarima Nadia Sazman¹, Nik Fatin Nabihah Atiqah Nik Ramli², Hamizah Mokhtar², Megat Ahmad Kamal Megat Hanafiah¹, Zul Adlan Mohd Hir^{1,*}**¹Faculty of Applied Sciences, Universiti Teknologi MARA Pahang Branch, 26400 Bandar Tun Abdul Razak Jengka, Pahang, Malaysia.²Faculty of Civil Engineering, Universiti Teknologi MARA Pahang Branch, 26400 Bandar Tun Abdul Razak Jengka, Pahang, Malaysia.

Abstract. Microcrystalline cellulose (MCC) is a widely applied material in pharmaceutical, food and composites due to its biocompatibility, renewability and desired properties. Nevertheless, the conventional process for preparing MCC is wood or cotton cellulose based using alkaline and bleaching process which may not be sustainable in terms of resource depletion, deforestation, and environmental stability. Finding a renewable, sustainable source of MCC can help reverse this trend and encourage more eco-friendly manufacturing processes. Bamboo, especially fast-growing species such as *Dendrocalamus asper* (*D. asper*), provides an environmentally sound source with rich cellulose resources and is one of the promising raw materials for green preparation of MCC. Therefore, the present work is to prepare MCC from *D. asper* fiber and investigate its physicochemical characteristics. The MCC was prepared using alkali treatment of bamboo with NaOH, followed by acidified bleaching using NaClO₂ and HNO₃ and finally acid hydrolysis with H₂SO₄. Then, the MCC was taken for further characterizations using scanning electron microscope (SEM) equipped with energy dispersive x-ray (EDX), fourier transform infrared spectrophotometer (FTIR), x-ray diffractometer (XRD), as well as thermogravimetric analysis (TGA) to investigate its surface morphologies, elemental composition, functional groups, crystallinity and thermal stability. The recovered MCC has rod-like structures and contains 43 % of carbon and 57 % of oxygen. The FTIR spectrum obtained is consistent with other MCC sources. The crystallinity index calculated for the MCC is 58 %. The MCC reveals initial weight loss and is thermally stable as temperature increases to 500 °C. DTG analysis confirms MCC has thermal stability around 180-210 °C. The MCC extracted using bamboo fiber offers different properties compared to MCC extracted using wood or cotton fibers, offering new opportunities for green industry development that aligns with the United Nations' Sustainable Development Goals (SDG).

Keywords: Bamboo, *dendrocalamus asper*, extraction, microcrystalline cellulose.

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1. INTRODUCTION

Microcrystalline cellulose (MCC) is a versatile material used in pharmaceuticals, food, and composites. It consists of disordered crystalline-amorphous regions, and its structure depends on the extraction process [1]. In the pharmaceutical industry, MCC is used as an excipient for tablet binding, disintegration, and controlled drug delivery, while in food it serves as a bulking and stabilizing agent. Its reinforcing and compressive properties in composites further highlight its potential as a sustainable, lightweight alternative to petroleum-based materials [2]. MCC can be widely used due to its biocompatibility, renewability and desirable physical properties, including stability, non-toxicity, and easy handling [3]. Although MCC has many applications, its conventional production relies on cellulose from wood and cotton, contributing to significant environmental issues. Sourcing from cotton [4], and especially hardwood can lead to deforestation, land degradation, and resource depletion. [5,6]. Moreover, these practices release greenhouse gases and drive biodiversity loss. Consequently, there is growing demand for sustainable, high-quality alternative cellulose sources to meet increasing global MCC needs without environmental harm. As the world shifts toward sustainable and circular manufacturing, reducing reliance on finite resources and minimizing environmental impact are essential objectives.

Bamboo is a fast-growing, eco-friendly alternative for cellulose extraction. Species such as *Dendrocalamus asper* grow rapidly and thrive on degraded land. Rich in cellulose, bamboo is an excellent raw material for MCC. Native to Southeast Asia, including Malaysia, Thailand, Indonesia, the Philippines, and India, its exact origin is unknown. Considered a “green resource,” it requires minimal fertilizers, pesticides, and irrigation, making it less resource-intensive than conventional crops. It is widely used in furniture, traditional instruments, and handicrafts [7]. In addition, bamboo trees have high capacity to isolate carbon at a high rate, which represents a possible approach to counteract climate change [8]. Given its rapid growth and low-emission production, bamboo is a promising, sustainable source for MCC. However, transitioning to bamboo as a primary feedstock poses challenges, particularly in developing efficient extraction and processing methods to produce high-quality, market-ready MCC. While widely used in traditional sectors like paper and textiles, bamboo remains underexplored as a source of microcrystalline cellulose [3]. Bamboo’s composition and its conversion to pure cellulose differ from wood and cotton, especially in alkali strength during treatment and bleaching. Understanding these differences is key to optimizing the yield and quality of bamboo-derived MCC. Moreover, scaling up production requires not only improved processing technologies but also consideration of economic and sustainability factors.

Hence, this study investigates the potential of bamboo, particularly *D. asper*, as a sustainable source for MCC extraction. The process involves alkali treatment with NaOH, bleaching with acidified NaClO₂ with HNO₃, and acid hydrolysis using H₂SO₄. The resulting MCC is characterized for morphology, elemental composition, functional groups, crystallinity, and thermal stability using SEM-EDX, FTIR, XRD, and TGA. The findings are expected to address environmental issues associated with conventional MCC production while promoting bamboo as a viable material for broader industrial applications such as construction materials and bio-based plastics. This supports the advancement of green industries aligned with the United Nations Sustainable Development Goals (SDGs).

2. MATERIALS AND METHODS

2.1 Chemicals and Materials

Bamboo (*D. asper*) was collected from a local bamboo processing facility in Lipis, Pahang, Malaysia. Bamboo culms aged approximately 3–4 years, representing the mature stage with high cellulose content were selected. Only the middle internodal sections of the culm were utilized to ensure uniform fiber quality. The collected bamboo was cut into small pieces and air-dried for 7 days to reduce

surface moisture, followed by oven drying at 60 °C for 24 hours to achieve constant weight. Sodium hydroxide (NaOH, 99 %) pellets, sodium chlorite (NaClO₂, 80 %) powder, nitric acid (HNO₃, 65 %), and sulfuric acid (H₂SO₄, 98 %) were purchased from R&M Chemicals, Malaysia. Isopropanol was purchased from HmBG Chemicals. Deionized water was used throughout the experimental procedures.

2.2 Extraction of MCC

MCC was extracted from *D. asper* via sequential extraction process and was adapted from literature with a little modification [9]. The extraction process is presented in Figure 1. Bamboo was chopped, washed with distilled water, and air-dried to remove dirt on the surface. The dried bamboo fibers were subsequently treated with 800 mL of 5 % (w/v) NaOH solution at 105 °C in fume hood for 2 hours by continuous stirring. This step was intended to eliminate hemicelluloses, lignin and other extractives from bamboo fibers. The residue was filtered and washed with distilled water until a final pH of neutral filtrate achieved. The neutralized sample was subsequently dried in an oven at 80 °C for further treatment.

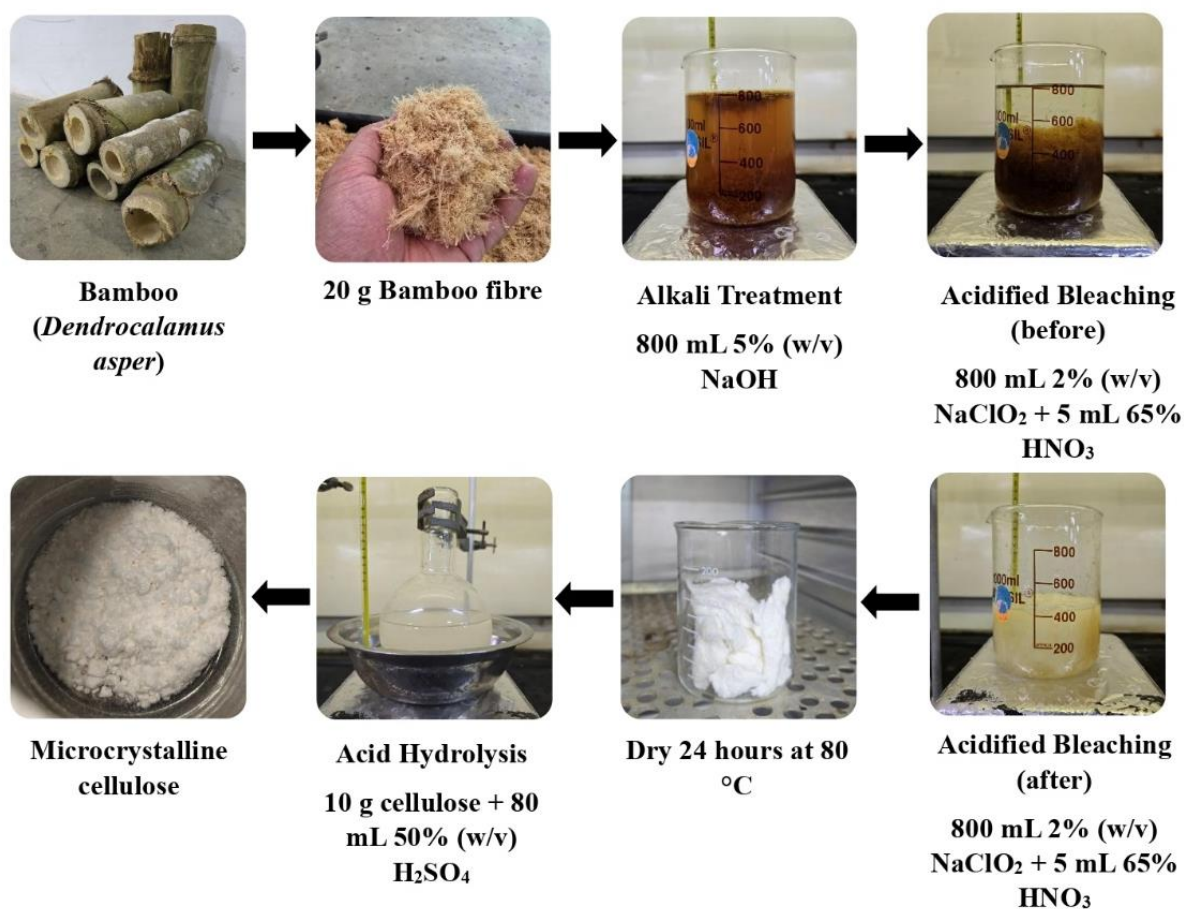


Figure 1: Overall MCC extraction process

The alkali-treated bamboo was subjected to an acidified bleaching process to further remove residual lignin and other impurities. This was achieved by using 800 mL 2 % (w/v) NaClO₂ solution, acidified with 5 mL of 65 % (w/v) HNO₃. The solution was stirred at 105 °C for 2 hours. This bleaching was carried out twice more making up a total of 6 hours, to obtain highest concentration of lignin removal. After each bleaching cycle, it was filtered, washed one time with isopropanol and distilled water, followed by drying the sample and keeping it at 80 °C. Cellulose-rich sample was put aside for acid hydrolysis. Only half the original weight of the fiber was obtained at this stage, showing 50 % yield.

The purified cellulose was acid hydrolyzed to decompose amorphous regions and isolate MCC. Hydrolysis of cellulose with 80 mL 50 % (w/v) H₂SO₄ (ratio of cellulose to acid is 1:8) at 45 °C for 60 min with constant stirring was done to avoid uneven reaction. To prevent any cellulose degradation, further the reaction was stopped by diluting the mixture with 400 mL distilled water (5 times the acid dose). The MCC was washed a single time with isopropanol and multiple times with distilled water until the pH of the washing water was neutral. Lastly, the MCC was dried in an oven at 40 °C for 24 hours before being crushed to obtain the final product as a powder. For every 20 g of bamboo fiber, the MCC yield was 10 g.

All chemical treatments were performed in a fume hood with suitable Personal Protective Equipment (PPE) (lab coat, nitrile gloves, safety goggles, and face shield). Alkaline and acidic waste solutions were pH 7 neutralized before disposal, following the institutional guidelines on chemical waste disposal while used NaClO₂ solutions were treated with sodium thiosulfate, ensuring a complete destruction of residual oxidizing agents, and disposal.

2.3 Characterization of MCC

MCC morphology and elemental composition were determined by SEM/EDX (TESCAN VEGA3). Before starting the imaging, the MCC was coated with a thin layer of gold using sputter coater for 3 minutes to further improved surface conductivity. The SEM image was taken at 400 magnifications around 15kV acceleration voltage. EDX analysis was conducted simultaneously to identify its elemental composition focusing on the distribution of carbon and oxygen. FTIR analysis was performed to determine functional groups present in the MCC. Spectra in the range from 4000 to 500 cm⁻¹ were recorded using the FTIR spectrophotometer (Perkin Elmer Spectrum One). Various peaks correlated with cellulose structure were analyzed to verify the synthesized MCC. XRD was performed using the PANalytical Xpert Pro diffractometer [10], with CuK_α radiation, 40 kV voltage, and 35 mA current and the 2θ angle range 5-90° for studying the crystalline structure of MCC. The crystallinity index (Cr. I) was calculated with the aid of the Segal equation [11], Eq. 1, and the particle size was calculated by Debye Scherrer formula [12], Eq. 2:

$$Cr. I (\%) = \frac{I_{200} - I_{am}}{I_{200}} \times 100 \quad (1)$$

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (2)$$

with I₂₀₀ is the intensity of crystalline peaks measured at 2θ = 22.7° and I_{am} is the intensity of amorphous peaks measured at 2θ=18°. K is Scherrer constant (0.9), λ is the radiation wavelength (0.15406 nm), β is the full width at half maximum (rad), and θ is Bragg angle (rad). The thermal stability of MCC was studied using a Thermogravimetric Analysis (TGA), TGA analyzer (Mettler Toledo TGA/DSC 1) under a nitrogen atmosphere. A heating rate of 10 °C/min was applied for the analysis (25 °C to 800 °C), while the thermal decomposition and the thermal stability of the MCC were evaluated by weight loss curve analysis.

3. RESULTS AND DISCUSSION

3.1 Surface Morphological and Elemental Composition Analysis

The SEM image (Figure 2(a)) shows MCC at 400x magnification. The extracted MCC reveals distinct small rod-like structures, typical of MCC. Alkali and acid treatment efficiently break down lignocellulosic structure into pure cellulose strand. Figures 2(b-d) show the elemental mapping for MCC. The scattering of elements showing homogeneous distributions for carbon (green) and oxygen (red), are depicted in Figures 2(c) and 2(d), respectively. The EDX analysis (Figure 2(e)) shows that

the MCC has carbon (C) content of 43 % and oxygen (O) content of 57 %. There are no other elements detected ensuring that the synthesized MCC is pure.

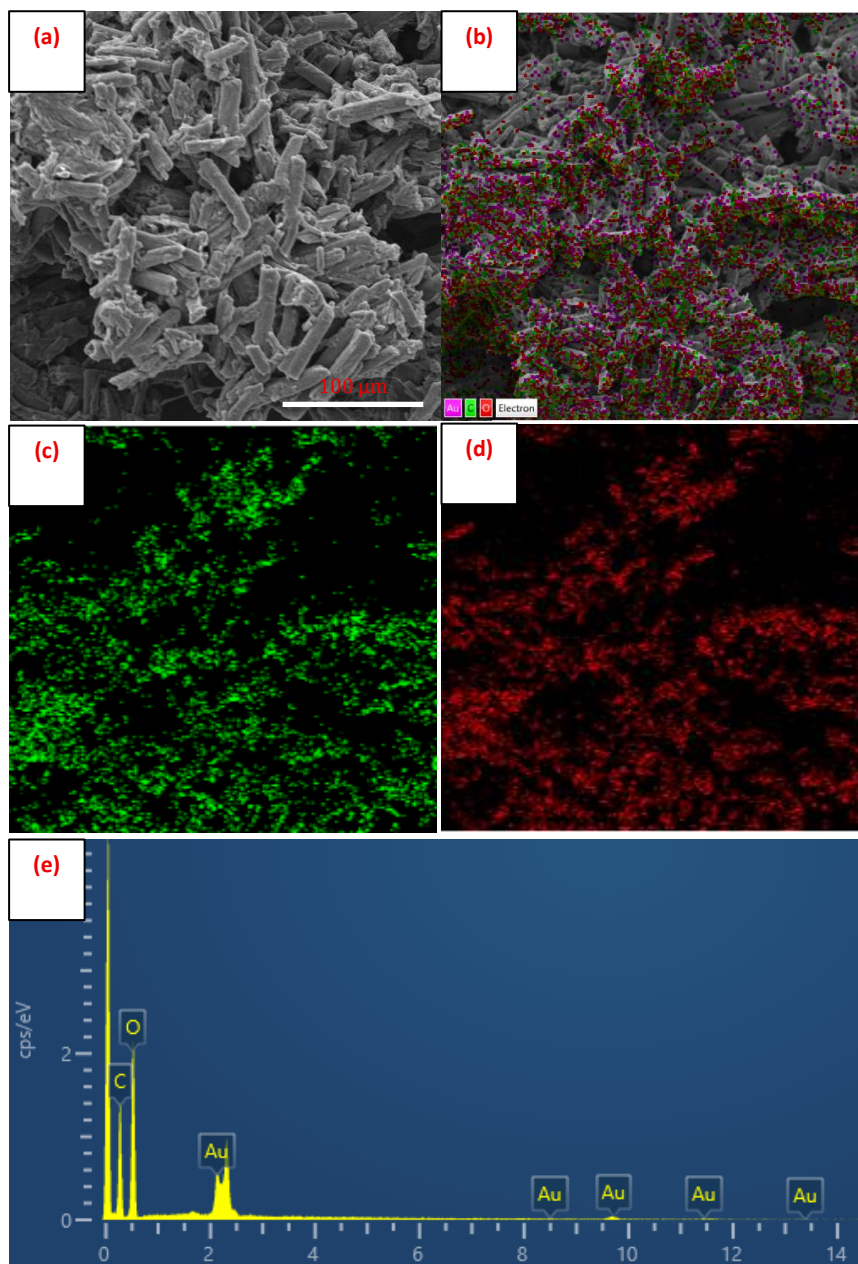


Figure 2: (a) SEM image of MCC at 400x magnification, (b) Elemental mapping for MCC at 400x magnification, (c) Elemental mapping for carbon, (d) Elemental mapping for oxygen and (e) EDX spectrum for MCC

3.2 Fourier Transform Infrared (FTIR) Spectroscopy Analysis

The FTIR spectrum of the MCC (Figure 3) obtained from *D. asper* exhibits peak at 3335 cm^{-1} which attributes to O-H stretching vibration, showing the inclination to hydrophilic properties of the sample and cellulose components is preserved [9,13]. Peak at 2896 cm^{-1} is assigned as C-H stretching vibration. There is a peak at 1635 cm^{-1} (H-O-H bending) because of the absorbed water molecules by MCC. There are some other works which reported that the strong peak at 1635 cm^{-1} was due to C=O carbonyl stretching vibration [14]. This is because of the different state of sample. Usually, peak at 1635 cm^{-1} of pure MCC is due to water absorbed, but for modified or less pure MCC, the peak is due to carbonyl stretching. Peak at 1027 cm^{-1} corresponds to C-O-C stretching vibration of pyranoside ring,

1314 cm^{-1} to CH_2 wagging vibration and at 894 cm^{-1} to rocking vibration of β -glycosidic linkage which are consistent with the presence of cellulose. These functional group bands are similar to those reported for MCC extracted from other plant sources, such as *Lagenaria siceraria* Fruit Pedicles, and *Posidonia oceanica* brown algae indicating the presence of typical cellulose features [13,15]. The absence of peaks at 1500-1600 cm^{-1} and at 1727 cm^{-1} indicates that large amount of lignin and hemicellulose have been removed during extraction process [16].

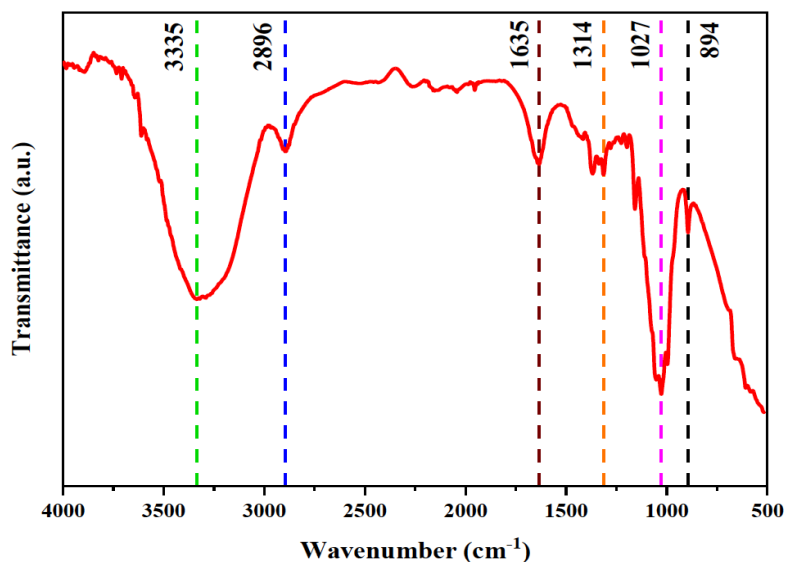


Figure 3: FTIR spectra of the MCC

3.3 Crystallinity Analysis

Figure 4 depicts the XRD pattern for MCC, showing three prominent peaks at 2θ angle of 12.38° , 20.24° and 22.06° which correspond to the $(\bar{1}10)$, (110) , and (200) planes of cellulose I β , respectively. These match the standard pattern of cellulose I β in the ICDD/JCPDS database: Card No. 00-056-1718. The highest peak observed at $2\theta = 22.06^\circ$ corresponds to the characteristic (200) planes of cellulose crystalline structure [15,17]. To assess the degree of crystalline index (Cr.I) of the extracted MCC was calculated to be 58 %, indicating a moderate level of crystalline structure in the sample. This crystalline percentage is lower than that of pure cellulose but remains within the typical range (55-80 %) for MCC reported in previous literature [15]. The calculated crystallite size of MCC at 22.06° is 561.77 nm, which is relatively large compared to MCC from other sources such as cotton linter (40–120 nm, Cr.I \approx 70–80 %) [18] and bamboo (*Phyllostachys edulis* (Carrière) J. Houz.) fiber (2.1 nm, Cr.I \approx 77.2 %) [19]. Morphite crystallites tend to have lower overall crystallinity because an amorphous region separates them from less amorphous material. Bigger crystallites tend to have more amorphous regions or less lattice packing in its internal matrix which can lead to lower overall Cr.I values. From the mechanical perspective, higher amorphous content of large crystallites can improve the ability of material to absorb impact resulting in superior toughness of the material. Toughness and impact resistance often linked with rigidity of structure, as the rigidity produced by the increased crystalline content of the material. The moderate Cr.I of MCC from *D. asper* can be useful for applications such as reinforcing photocatalyst composites, providing a good compromise between stiffness (for structural integrity) and some flexibility (to reduce brittleness). This is shown in minimum value of Cr.I obtained, translating into moderate toughness of the MCC [13].

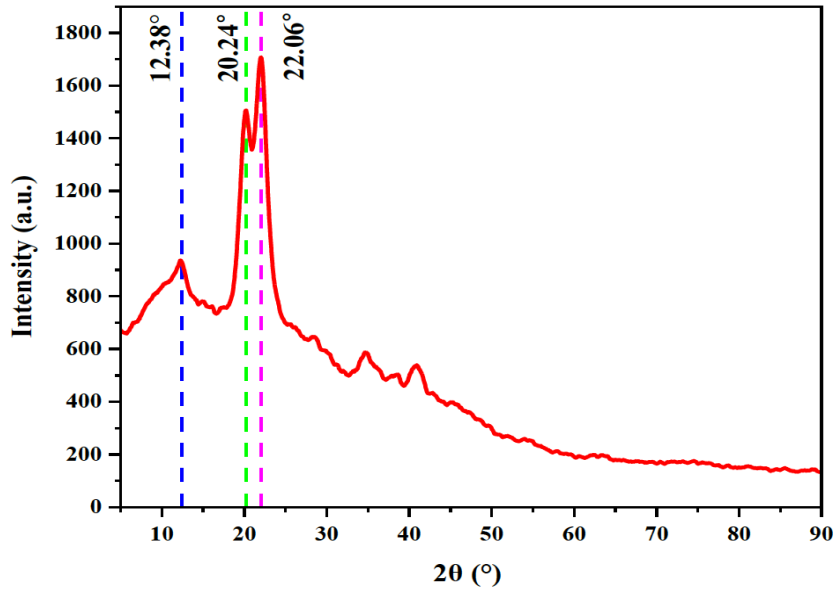


Figure 4: XRD pattern for MCC

3.4 Thermal Stability Analysis

Figure 5 shows TGA and DTG curve for MCC. TGA analysis from 25 °C to 800 °C is presented in Figure 5(a), and its analysis shows a marked initial weight reduction caused by moisture and volatile substances.

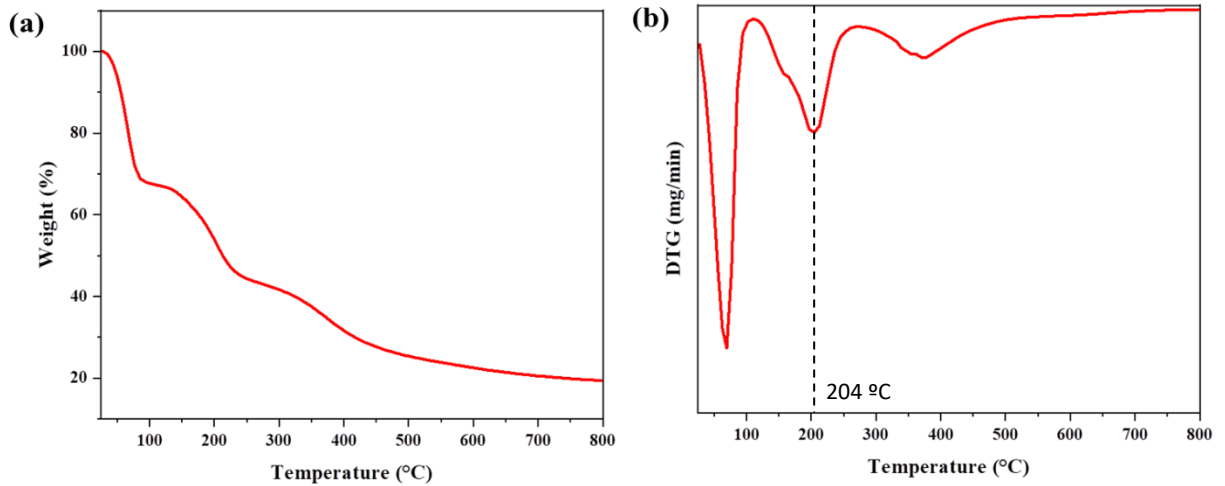


Figure 5: (a) TGA and (b) DTG curve for MCC

The second significant degradation step happens at 150 °C–385 °C, which involves the degradation of hemicellulose (normally 200-260 °C), thermal degradation of cellulose (generally 275-350 °C), and the gradual breakdown of lignin over a wider temperature range [17]. DTG curve shown in Figure 5(b) reveals that the MCC exhibits good thermal stability up to around 180-210 °C, most likely due to sulfate group during acid hydrolysis [1]. Thermal stability by acid hydrolysis of bamboo

is enhanced through the removal of amorphous materials, similar to lignin and hemicellulose, by the acid hydrolysis. Addition of sulfate groups may alter degradation kinetics, by serving as catalysts for dehydration and char formation at high temperatures. According to literature reports, MCC containing sulfate groups typically have slightly lower maximum degradation temperatures than desulfated MCC due to acid-catalyzed depolymerisation, but possibly higher char yield which could benefit flame-retardant applications [20]. The thermal stability of the extracted MCC allows for the possibility of exploitation at higher processing temperatures in various processes.

4. CONCLUSIONS

In this work, *D. asper* bamboo fiber could be a promising and sustainable resource for MCC extraction. The recovered MCC has rod-like structures and contains 43 % of carbon and 57 % of oxygen. The FTIR spectrum obtained is consistent with other MCC sources. The crystallinity index calculated for the MCC is 58 %. The MCC reveals initial weight loss and is thermally stable as temperature increases to 500 °C. DTG analysis confirms MCC has thermal stability around 180-210 °C. As bamboo, especially *D. asper*, was found to be an efficient alternative to traditional cellulose sources like wood as well as cotton. The findings would contribute to sustainable application of this material. Moreover, the utilization of bamboo in MCC can contribute to the fulfilment of the United Nations' Sustainable Development Goals (SDG), advocating an environmentally friendly production system by decreasing the dependency on non-renewable resources.

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Author Contributions

All authors contributed toward data analysis, drafting and critically revising the paper and agree to be accountable for all aspects of the work.

Disclosure of Conflict of Interest

The authors declare there are no conflict of interest in this work.

Compliance with Ethical Standards

The work is compliant with ethical standards.

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