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Extraction and Physicochemical Characterization of Microcrystalline Cellulose from *Gigantochloa scortechinii*

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Recently, microcrystalline cellulose (MCC) has been widely utilized in numerous applications such as polymer composites, packaging materials and pharmaceutical compounds (as adsorbents or binders), owing to its exceptional properties. Thus, greener approaches and new renewable sources' raw materials have gained attention from many researchers due to the insufficient amount of non-renewable resources. This research aims to extract and characterize the MCC from bamboo fibre (*Gigantochloa scortechinii*) by going through two different methods of preparation. The first extraction of MCC (MCC-1) was carried out through the chemical alkali treatment by using 5 wt% NaOH, continued by the acidified bleaching treatment. Then, the second MCC (MCC-2) was fabricated via further acid hydrolysis method using 40 wt% sulfuric acid at 45°C for 60 min. Next, the obtained MCC-1 and MCC-2 were characterized in terms of its morphologies, structural, crystallinity, composition, and thermal stability features through scanning electron microscopy equipped with energy dispersive X-ray (SEM-EDX), fourier transform infrared spectroscopy (FTIR), X-ray powder diffraction (XRD) and thermogravimetric (TGA) analyses. The SEM analyses illustrate the shape of MCC-1 is long rod-like structure with the diameter is ranging from 14.10 to 14.21 µm. Meanwhile, MCC-2 consists of short rod-like structure, and the diameter ranging from 2.87 to 12.07 µm. The EDX analysis indicates that the carbon content in MCC-1 and MCC-2 are estimated at 54.5% and 52.2%, respectively. The crystallinity index of MCC-2 (78.68%) are calculated to be higher than MCC-1 (58.94%). In conclusion, the inexpensive MCC-2 derived from bamboo fibre (*Gigantochloa scortechinii*) is emphasized as one of the potential renewable and sustainable resources. It also has a great deal of potential to be widely adopted in a variety of industrial implementations and as a promising solution to the environmental pollution's issues.

Keywords: Extraction; bamboo fibre; *Gigantochloa scortechinii*; microcrystalline cellulose

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Countless researchers have been interested in using raw materials from renewable sources due to the insufficiency of non-renewable resources and the demand towards renewable sources, along with requirements for approaches that are compliant with environmental rules. [1]. Given this worrisome situation, cellulose is regarded as one of the most abundant and an excellent renewable natural biopolymer on the earth which can be obtained from plant biomass [1–3]. The main structural element of the plants and woods cell wall is cellulose and owing to this reason, the woods have an amazing strength as it possesses lengthy chain of interlinked sugar molecules [2]. One particular type of carbohydrate polymer is called cellulose, and it is made up of a linear chain of several hundred to several thousands of β (1→4) linked D-glucose units [1,2,4]. It is possible to modify cellulose to create a variety of useful derivative products, such as microcrystalline cellulose (MCC) and nanocrystalline cellulose (CNC).

The MCC is a white, unscented, crystalline micro powder that has a large surface area, renewability, fibrous nature, lightness, mechanical strength, and water insolubility. It is also non-toxic, biodegradable and biocompatible [4,5]. MCC has a larger specific surface area contrasted to other typical cellulose fibres, a lower degree of polymerization, and the amorphous areas are eliminated by acid hydrolysis [6]. In recent years, MCC has been widely employed in industries due to its exceptional properties. MCC is often used in numerous applications such as polymer composites, printing, automotive industries, packaging materials, pharmaceutical compounds (as adsorbents or binders),

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comprising cosmetic (binders, thickeners), food (stabilizers, anti-caking agents, fat substitutes, and emulsifiers) and bio-composites (as reinforcements) [4,7]. It has been found that cellulose derivatives may be extracted from biomass in a way that is both ecofriendly and suitable for utilization in industry [8]. MCC can be extracted from different plant biomass such as kapok fibre [1], oil palm trunk [2], rose stems [3], *Posidonia oceanica* brown algae [7], elephant grass [8], *moso* bamboo [9], oil palm empty fruit bunch [10], and jackfruit rind waste [11].

Bamboo, which comes from both the forestry and agricultural industries, is a promise filler material for thermoplastic composites. Bamboo is a member of the *Bambusoideae* subfamily of the grass family (*Gramineae*) [12,13]. It is extensively available in Asia and a rapidly growing plant with a short maturation time. Besides, it is also believed to be inexpensive than other forest resources [14]. However, the main issue with natural materials is that they are hydrophilic, which makes it hard to adhere to a hydrophobic polymer matrix. Furthermore, natural fibre shows low moisture resistance which results in significant water absorption, poor mechanical features, and dimensional stability [15]. Alkali treatment is a typical fibre treatment that several researchers utilize to give single fibre and fibre/polymer matrix composites a high fibre matrix adhesion and excellent mechanical performance. As a result of the alkaline treatment, the external surface of natural fibre is stripped of some of the lignin, hemi-cellulose, wax, and oil that covered it. The remaining lignin, wax, and hemicellulose improve the matrix-fibre interface and maintain excellent adhesion between the matrix and the natural fibre [14].

Thus, in the present work, the extraction of MCC-1 and MCC-2 from bamboo fibre (*Gigantochloa scortechinii)* via a simple and inexpensive methods were investigated. By utilizing the bamboo fibre (*Gigantochloa scortechinii)* as the raw materials, not only it is renewable, easily accessible, inexpensive, relatively simple to extract, biocompatible, and non-toxic, but it is also one of the potential alternate ways to solve the issues regarding environmental pollution and have wide application in industry. As MCC surfaces have a fair amount of -OH groups, they promote surface modification and increase their applications in industry without having any harmful impacts. Moreover, bamboo is an inexpensive material that is now being regarded as a replacement for wood because of its rapid rate of growth and great mechanical characteristics [16]. The preparation of MCC-1 and MCC-2 involved chemical alkali, acidified bleaching treatment, and acidic hydrolysis. The physical and morphological properties of MCC-1 and MCC-2 were characterized and compared.

EXPERIMENTAL

Chemicals and Materials

Raw bamboo fibre (*Gigantochloa scortechinii*) was collected from Kuala Keniam, Pahang National Park, Malaysia. Sulphuric acid (H_2SO_4, 95–97 %), sodium hydroxide (NaOH) pellets, nitric acid (HNO_3, 65 %) and sodium chlorite ($NaClO_2$) pellets were used to extract MCC from bamboo fibre. All reagents were purchased from R&M Chemicals and they have been used just as they were supplied with no purification process. Throughout the research, deionized water was utilized.

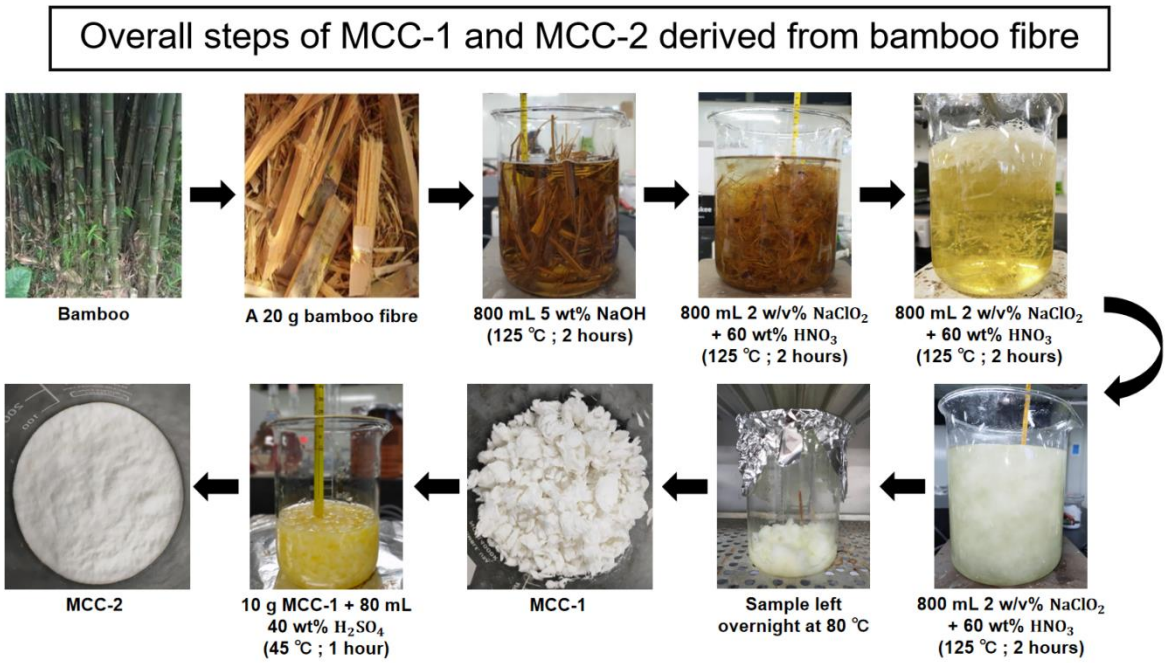

Figure 1. Overview of MCC-1 and MCC-2 experimental procedure.

Extraction of MCC-1 from Bamboo Fibre

A straightforward alkali treatment employing 5 wt% NaOH was used to extract MCC-1 from bamboo fibre (*Gigantochloa scortechinii*), which was then subjected to the typical acidified bleaching procedure [1]. First, 20 g of bamboo fibre were boiled for 2 hours at 125 °C with constant mechanical stirring in 800 mL of 5 wt% NaOH. The sample was then thoroughly washed with distilled water until the pH was neutral. The material was then subjected to the $NaClO_2$ treatment. The sample was heated to 125 °C for two hours while being vigorously mechanically stirred in 800 mL of a 2 w/v% $NaClO_2$ solution with a few drops of 60 wt% HNO_3. A white solid sample was obtained after two iterations of the $NaClO_2$ treatment.

Preparation of MCC-2

The synthesized MCC-1 underwent further acid hydrolysis treatment to form MCC-2. With the ratio of 1:8 (wt%) MCC-1 over liquor, the MCC-1 was hydrolyzed using a 40 wt% H_2SO_4 at 45 °C for 60 minutes while being mechanically stirred. The sample's hydrolysis was then stopped by adding distilled water that was five times as large as the original amount. The white diluted suspension was then rinsed repeatedly until a consistent pH was achieved. Figure 1 depicts an overview of the experimental procedures for the preparation of MCC-1 and MCC-2.

Characterization Methods

The morphology, elemental composition and mapping of MCC-1 and MCC-2 were determined by scanning electron microscope equipped with energy dispersive X-ray analyzer (SEM- EDX, TESCAN VEGA3). The functional groups of the prepared MCC-1 and MCC-2 were analyzed by Fourier transform infrared (FTIR) between 400 to 4000 cm^{-1} on a Perkin Elmer infrared spectrometer using attenuated total reflection (ATR) accessory. Thermal stabilities of MCC-1 and MCC-2 were characterized using a thermogravimetric analyzer (TGA, Perkin Elmer). The analysis was performed under a nitrogen atmosphere with a nitrogen flow rate of 20 $mL \cdot min^{-1}$ at a heating rate of 10 $°C \cdot min^{-1}$ over a temperature range of 25–800 °C. The phase and crystalline characteristic of prepared MCC-1 and MCC-2 were characterized using X-ray diffraction (XRD) (PAN analytical with Cu-Kα radiation probe beam of 1.54056 Å wavelength) with 25-70° range. The size of the particle was estimated by the Scherrer equation on the width half maximum (FWHM) of the samples (200) peak. The crystallinity of the samples was calculated from diffraction intensity data using the empirical method for native cellulose. The crystalline-to-amorphous ratio material was determined using Eq. (1):

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

where Cr.I is the crystallinity index, I_{200} is the maximum intensity of the diffraction from 200 plane at $2\theta = 22.7°$ and I_{am} is the minimum intensity corresponding measured at $2\theta = 18°$ [17].

RESULTS AND DISCUSSION

Surface Morphology and Elemental Composition Analysis

The surface morphological characteristics of the prepared MCC-1 and MCC-2 were characterized via scanning electron microscopy (SEM) analysis at a magnification of 100x and 400x, respectively (Figure 2). It can be observed from the SEM image that the shape of MCC-1 is long rod-like structure, and the diameter is ranging from 14.10 to 14.21 µm (Figure 2a and b). Meanwhile, MCC-2 consists of short rod-like structure with the diameter ranging from 2.87 to 12.07 µm (Figure 2c and d). The size of MCC-2 is smaller than MCC-1, probably due to the hydrolysis method in preparing MCC-2 as the acid breaks the MCC-1 long structure (Figure 2a) into shorter parts. The elemental composition of samples was conducted using energy-dispersive X-ray analysis (EDX) together with chemical mapping analysis (Figure 3) and (Figure 4). The EDX analysis indicates that both MCC-1 and MCC-2 mainly contain carbon (C) and oxygen (O) as observed in (Figure 3d) and (Figure 4d), and no other peak detected for MCC-1 spectra, confirming the purity of the samples. Meanwhile, the detected gold (Au) peaks in MCC-2 spectra are from the sputter coating of SEM (Figure 4d). The carbon content in MCC-1 and MCC-2 are estimated at 54.5 % and 52.2 % accordingly, indicates MCC-2 has lower carbon percentage than MCC-1. The mapping images show good dispersion of the cellulose particles. Elements are represented by red and green for C and O, respectively and the weight percentage of C in the EDX analysis of MCC-2 is observed to decrease as compared to MCC-1.

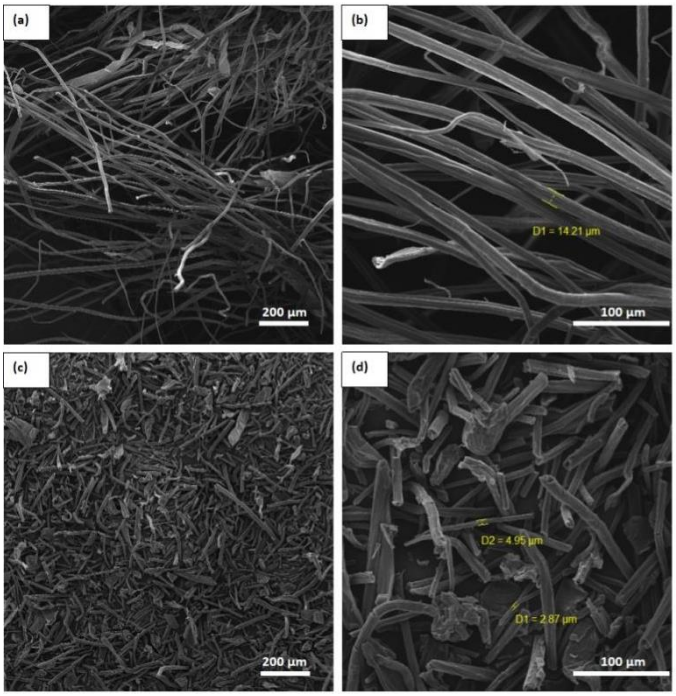

Figure 2. SEM images of MCC-1 (a-b) and MCC-2 (c-d).

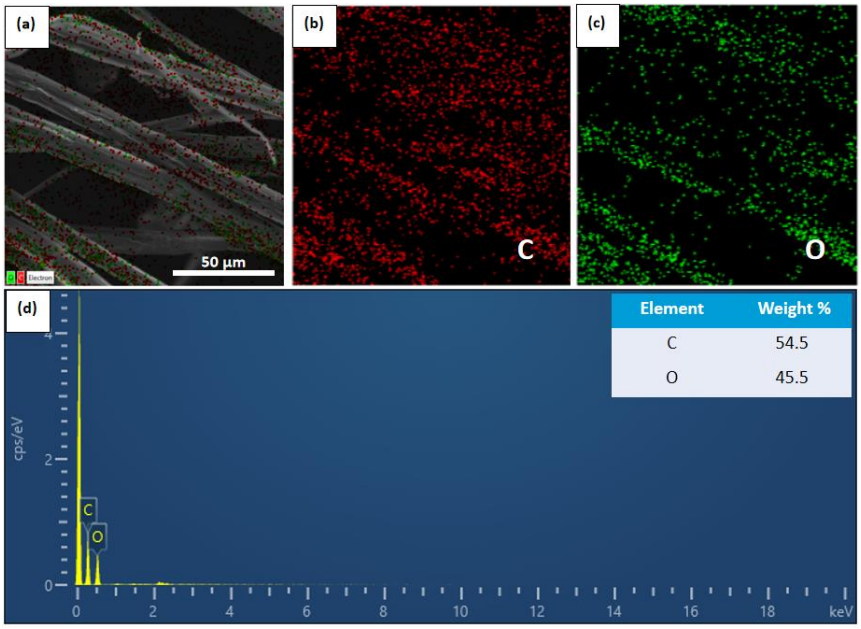

Figure 3. EDX analysis of (a) selected image of MCC-1 with corresponding elemental mapping images for C and O are presented in (b) and (c), respectively and (d) the elemental spectra and composition of the sample.

Fourier Transform Infrared Spectroscopy (FTIR) Analysis

The chemical compositions of the samples are further elucidated via the Fourier-transform infrared spectroscopy (FTIR) analysis. The FTIR spectrum of MCC-1 and MCC-2 present peaks at 3300, 2895, 1428, 1316, 1030 and 897 cm^{-1}, which MCC-1 peaks are slightly more intense than MCC-2 (Figure 5). Meanwhile, these bands are also identified in the FTIR spectrum of MCC obtained from kapok fibre [1], *Posidonia oceanica* brown algae [7], elephant grass [8], oil palm empty fruit bunch [10], and jackfruit rind waste [11]. The MCC-1 and MCC-2 spectra shown in Figure 5 that show peaks at 3330 cm^{-1} and 2895 cm^{-1} are C-H stretching vibration and O-H stretching vibration, respectively associated to the cellulose component [18]. This is an indication that the cellulose content is preserved throughout the chemical treatments performed [6,7]. The strong peaks of O-H vibration indicate

strong intermolecular and intramolecular bond [2]. Furthermore, the bands at 1631 cm^{-1} and broad peak at 1030–1051 cm^{-1} are due to the carboxylate group C=O stretching and the C–O stretching vibration mode of cellulose chemical structure, respectively [10]. The CH_2 asymmetric bending and CH_2 wagging motion in MCC-1 and MCC-2 can be observed at absorption spectra of 1428 cm^{-1} and 1316 cm^{-1} accordingly. At 1160 cm^{-1}, the C-O-C group's asymmetric bridge stretching vibration is detected [1]. The absorption band at 1103 cm^{-1} was assigned to the hydroxy-association spectrum, while the absorption band at 897 cm^{-1} reveals the usual cellulose structure connected to the cellulosic β-glycosidic linkages that comprise of C_1-H and O-H bending [7]. No discernible change is seen between the spectra of MCC-1 and MCC-2. The outcomes show that after acid hydrolysis, the cellulose molecular structure does not change.

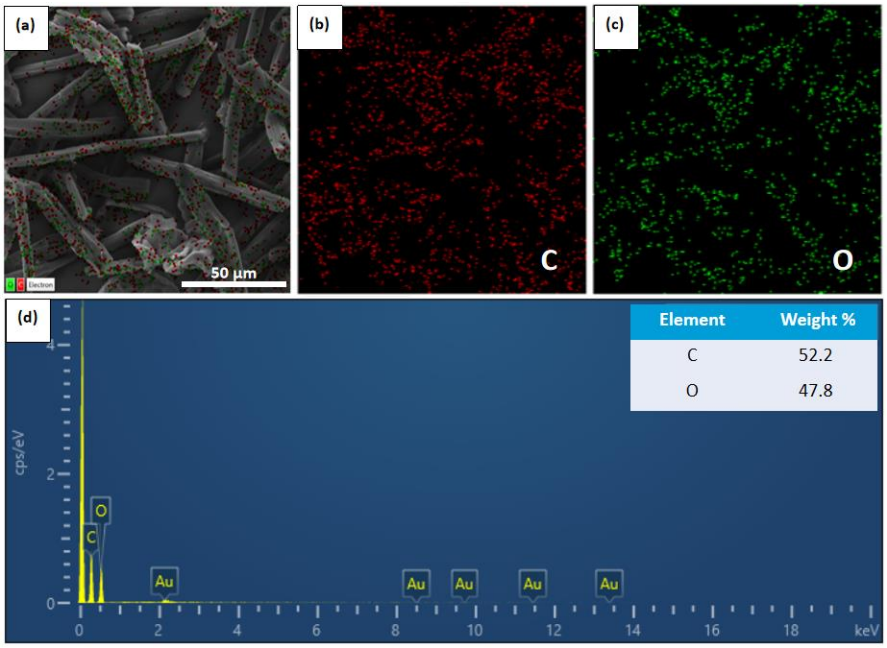

Figure 4. EDX analysis of (a) selected image of MCC-2 with corresponding elemental mapping images for C and O are presented in (b) and (c), respectively and (d) the elemental spectra and composition of the sample.

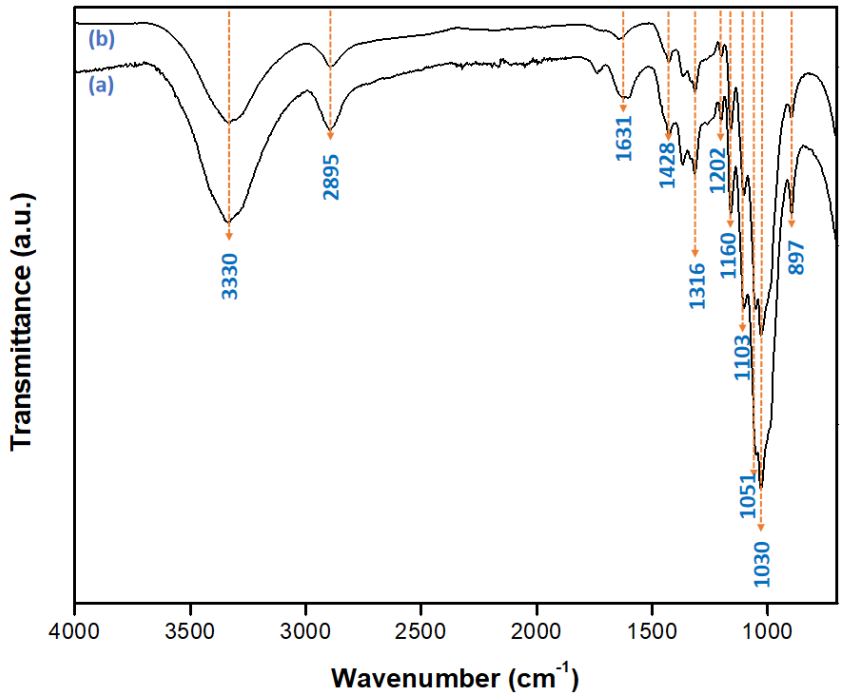

Figure 5. FTIR spectra of (a) MCC-1 and (b) MCC-2.

Crystallinity Analysis

The crystallinity index and the crystalline structure of MCC-1 and MCC-2 are investigated by using XRD analysis. Figure 6 represents the X- ray diffraction patterns. It can be observed that both MCC-1 and MCC-2 exhibit two similar peaks around 16.0º and 22.7º, corresponding to the crystal planes of (110) and (200). Owing to the crystal lattice's perfection in the (200) plane, the intensity peak at 22.7º is increased for MCC-2 [1]. In which peak intensity at 22.7º increases, it indicates more cellulose exposure, while when peak intensity at 16.0º slightly increases, it reflects the existence of amorphous cellulose [5]. The crystallinity index (Cr I) percentages calculated from the XRD patterns for MCC-1 and MCC-2 are 58.94 % and 78.68 %, respectively. The crystallinity index of MCC-2 is higher than MCC-1, is probably attributed to the fibre material as well as the further hydrolysis method for preparation of MCC-2. Similar results were reported in a study by Tarchoun et al., [7]. Furthermore, the MCC-1 crystallinity is enhanced by the acid concentration addition, due to the cellulose's crystalline part that was intensified while the amorphous part was reduced [19]. It is significant to mention that the toughness of the structure of cellulose is anticipated to enhance with the increase of crystallinity percentage [7]. The benefits of crystallinity higher than 70 %, include a notable improvement in thermally and chemically stability, also the resistance towards bacteria [1]. As seen in Figure 6, the preferred plane of the MCC-1 and MCC-2 are along the (200) direction and the particles size have been computed from the width of (200) plane using the Debye Scherrer formula as following equation Eq. (2) [20]:

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

Where K is a constant, λ is the wavelength of X-rays employed radiation (1.54056 Å), β is corrected full width at half maximum and θ is Bragg angle. The calculated crystallite size of the MCC-1 and MCC-2 are 4.32 nm and 4.44 nm, accordingly.

Thermal Stability Analysis

Thermogravimetric analysis (TGA) was employed to examine the thermal stability of the MCC-1 and MCC-2. Figure 7 displays the results from the TGA curves which clearly show the relationship between temperature (°C) and weight percentage (%) for both samples. The findings demonstrate that both samples show an initial weight loss in the region of 100 to 220 ºC and thermally stable when the temperature increases up to 560 ºC. The initial weight loss ranges from 25-120 °C probably attributed by moisture evaporation and other volatile compounds within the samples [3, 5]. While the cellulosic components lost more weight at 220 °C until 400 °C as a result of the cellulose's degrading processes (such as dehydration, decomposition of glycosyl units, depolymerization and decarboxylation), charred remnant is formed afterward [7].

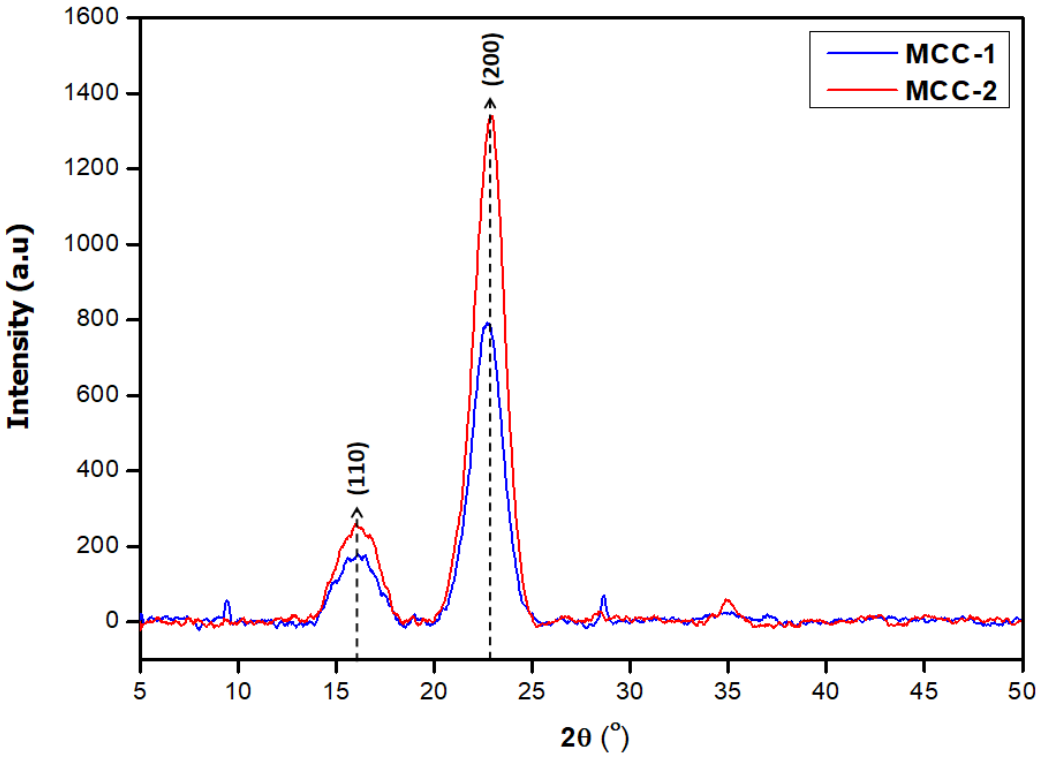

Figure 6. XRD pattern of MCC-1 and MCC-2

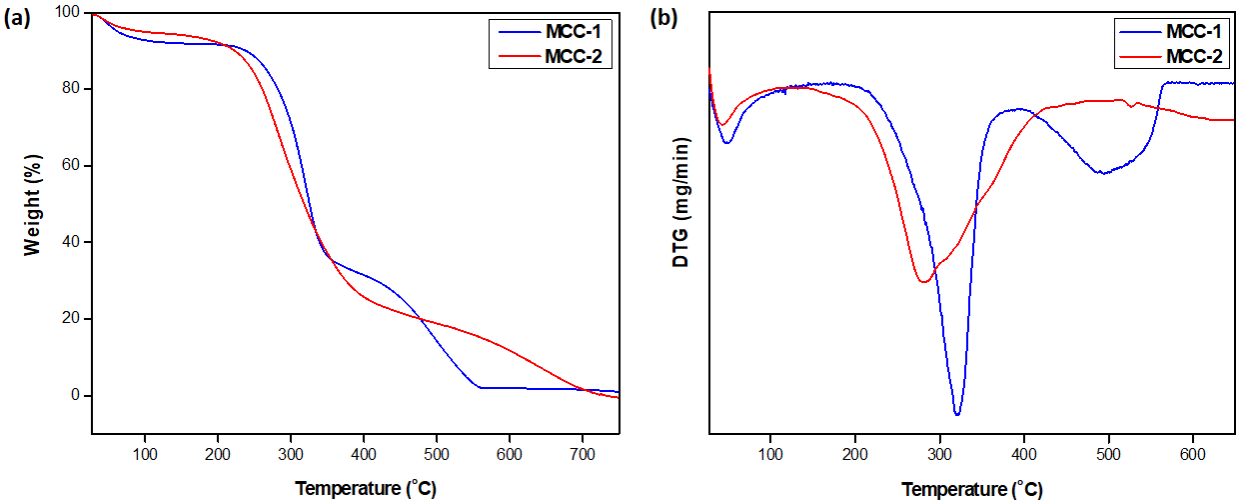

Figure 7. (a) TGA and (b) DTG curves of MCC-1 and MCC-2.

The TGA curve also illustrates that MCC-1 and MCC-2 undergo a different degradation step. The DTG analysis of the samples indicates the thermal stability of MCC-2 (290 °C) are lower than MCC-1 (321 °C). This is probably due to the deposition of sulphate groups during the acid hydrolysis process. The use of H_2SO_4 during the extraction process resulted in the presence of sulphate groups inside the MCC-2 matrices. Hence, the thermal stability of MCC-2 is affected, mainly due to a dehydration reaction caused by the sulphate groups [1]. Meanwhile, the decomposition peaks of MCC-1 are mostly associated with the glycosidic bonds' scission, levoglucosan and formation of char [7]. The char residual weight at 600 °C of MCC-1 (1.58 %) is higher than that of MCC-2 (0.35 %) respectively, which indicates almost complete thermal decomposition. These results are in agreement with the statement reported in study by Tarchoun and co-workers. The elimination of non-cellulosic chemicals, inorganic compounds, and amorphous regions by the alkali, bleaching, and further step of acid hydrolysis treatments on MCC-2 might eventually increase the thermal stability of the manufactured MCC, according to these findings [7]. The change in crystallinity could be the reason of the difference in thermal stability. Therefore, a MCC with high crystallinity will be benefited with excellent thermal stability. Additionally, the extraction technique and the raw types of woods also has an impact on the thermal stability of the MCC that has been extracted [1].

CONCLUSION

This study revealed the viability of extracting micro-crystalline cellulose from bamboo fibre (*Gigantochloa scortechinii*) via simple and inexpensive extraction method. Its physicochemical and structural properties were significantly altered via two different chemical treatment procedures for cellulose extraction. With the additional acid hydrolysis method using 40 wt% H_2SO_4 to prepare MCC-2, the cellulose amorphous region from MCC-1 was effectively eliminated, resulting in the fibres' diameters being reduced to the range of 2.87 till 12.07 µm. The crystallinity index of MCC-2 was 78.68 %, which was calculated to be higher than MCC-1 (58.94 %). The results of the morphological, chemical composition, TGA, FTIR, and XRD tests demonstrated that the lignin and hemicellulose in MCC-2 had been removed. The bamboo fibre that had been hydrolysed, particularly MCC-2, had certain improvements including enhanced crystallinity and improved thermal stability. In MCC-2, the cellulose content remains around 52.2 %. The extracted MCC-2 exhibit enormous potential as one of the prospective alternatives that could address the issue of environmental contamination and find wide-spread implementation in various industrial applications.

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