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To cite this article: Huu Dat Nguyen *et al* 2013 *Adv. Nat. Sci: Nanosci. Nanotechnol.* **4** 015016

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A novel method for preparing microfibrillated cellulose from bamboo fibers

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Abstract

The bamboo fiber is a potential candidate for biomass and power source application. In this study, microfibrillated cellulose (MFC) is prepared from raw fibers of bamboo tree (*Bambusa Blumeana J A & J H Schultes*) by an alkali treatment at room temperature in association with a bleaching treatment followed by a sulfuric acid hydrolysis. Field-emission scanning electron microscopy (FESEM) images indicated that final products ranged from 20 to 40 nm in diameter. The chemical composition measurement and Fourier transform infrared (FTIR) spectroscopy showed that both hemicellulose and lignin are mostly removed in the MFC. The x-ray diffraction (XRD) results also show that MFC has crystallinity of more than 70%. The thermogravimetric analysis (TGA) curves revealed that cellulose microfibers have a two-step thermal decomposition behavior owing to the attachment of sulfated groups onto the cellulose surface in the hydrolysis process with sulfuric acid. The obtained MFCs may have potential applications in alternative power sources as biomass, in pharmaceutical and optical industries as additives, as well as in composite fields as a reinforcement phase.

Keywords: alternative power sources, bamboo, biomass, microfibrillated cellulose

Classification number: 5.16

1. Introduction

Cellulose, the primary component of green plants such as grasses, reeds, stalks and woody vegetation, is the most abundant renewable polymer resource available on earth [1]. Thanks to its attractive chemical and physical properties, cellulose is considered as a biomass resource, and has been investigated and applied for a wide range of products and materials in daily life for many decades [2–4]. In recent years, a new type of cellulose material named microfibrillated cellulose (MFC) has been developed for transparent and high gas barrier films [5], rheology modifiers, reinforcement phase in composite, cosmetics and pharmaceutical industries as well as biomass resources [1, 6, 7]. Moreover, synthesizing bio-ethanol from cellulosic biomass has been discussed as an alternative energy resource [8]. In addition, MFCs, with a high axis ratio (L/D), have attracted more attention than the traditional reinforcement phases in composite fields because of their advantages including easy availability, easy modification, high mechanical properties, biocompatibility and renewability [7, 9].

In general, cellulose is characterized as a high molecular weight carbohydrate polymer of β-1,4-linked anhydro-D-glucose units [6]. Cellulose chains are bio-synthesized and clustered into crystalline domains called microfibrils and amorphous regions. The latter are more

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vulnerable to acid attack because of their randomly oriented arrangement which results in a lower density in comparison with crystalline regions [2, 7]. The controlled acid hydrolysis of raw cellulose fibers leads to highly crystalline nanofibers through the prescribed depolymerization of amorphous regions. Due to the action of strong acids, raw fibers are broken down into crystalline microfibers or microfibrillated cellulose, having shorter diameter, ranging from a few nanometers to a few microns [10].

Acid hydrolysis depends upon the acid species, the acid concentration, the hydrolysis time and temperature. Under planned conditions, sulfuric acid hydrolysis yields microfibrillated cellulose. Moreover, when the cellulose microfibers were prepared by sulfuric acid hydrolysis, a number of negative charges could be grafted onto the surface of MFCs leading to a stable colloidal suspension [3, 11]. After the acid hydrolysis, MFCs have a rod-like shape with diameter and length ranging from 5 to 20 nm and 100 nm to several micrometers, respectively [7, 12]. Owing to their stiffness, thickness, thickness distribution, length and length distribution, these rod-shaped MFCs are also named 'whiskers' [2, 3]. The controlled hydrolysis conditions with sulfuric acid (acid concentration, temperature and time) bring about stable suspensions that are not accumulated. This effect is mainly caused by electrostatic repulsion between the negatively charged particles on their surfaces [10].

Bamboo, an abundant non-wood forest resource in Vietnam, is considered as one of the alternative resources in the wood industry to prevent the shortage of woody resources thanks to its fast growth, short renovation and easy propagation [13, 14]. Bamboos are of notable economic and cultural significance in Vietnam where there are more than 100 bamboo species. Due to their high productivity, the development of plant materials attracts more and more attention [15, 16]. Structure and mechanical properties of bamboo was well studied by Amada and Untao [14] and Amada *et al* [15]. Effects of alkali treatment of bamboo fibers on their mechanical properties have been reported by Das and Chakraborty [17].

The aim of the present study is to investigate the ability of using sulfuric acid hydrolysis route for preparing microfibrillated cellulose from bamboo fibers and to characterize the resultant products. The effect of preparation conditions including time, temperature and original size of the raw fibers, on the structure and properties of final products has also been examined.

2. Experimental

2.1. Raw materials

Bamboo trees (*Bambusa Blumeana J A & J H Schultes*) were collected from The Phu An Ecological Bamboo Museum and Botanical Reserve (*Phu An Bamboo Village*), Binh Duong, VietNam. The chemical reagents used for treating bamboo fibers and preparing microfibrillated cellulose were analytical grade purchased from Merck without further purification.

2.2. Preparation of bamboo fibers (BFs)

Internodes of bamboo trees were cut into portions 10 cm in length (the node portions and thin layer of exodermis and endodermis bark of the bamboo were removed). The cylindrical portion of culm is peeled in the longitudinal direction to make strips approximately 0.2 cm thick, 12–15 cm in length and about 3 cm in width [17]. Bamboo strips were washed by distilled water at ambient temperature to remove the dust and impurities on the strip surface. After that, they were gently rolled to extract BFs.

2.3. Preparation of microfibrillated cellulose (MFC) from BFs

The MFC was generated from the BFs by an acid catalyzed hydrolysis method. Firstly, the BFs were ground into approximately 500 μm in size, using a cutting mill (Fritsch GmbH, Germany), with a 500 μm stainless steel trapezium-shaped sieve. The isolation of cellulose fibers required the removal of other components such as lignin, hemicellulose and pectin from the BFs. This step was achieved by applying the alkali treatment and bleaching. The alkali treatment was designed to dissolve the pectins and the hemicelluloses. BFs were treated with NaOH in different concentrations, at 30 °C, for 72 h in order to study the influence of alkali treatment in removing of hemicelluloses and pectins [17, 18]. After that, dissolved components were removed by washing with deionised water. The bleaching was done to whiten the BFs by removing phenolic compounds or chromophoric molecules in lignin. In this step, BFs are treated with 5 wt% sodium hypochlorite solution (NaOCl), at 30 °C, for 3 h [10]. After bleaching, the BFs were washed with deionised water until reaching pH of 7 and centrifuged to obtained the residue which was the raw material for acid hydrolysis. After hydrolysis, the mixture was diluted ten-fold with distilled water and neutralized with an alkaline solution until pH of 7 was achieved. The residue centrifuged was dialysed against deionised water and sonicated for 45 min in an ultrasonic bath. This suspension was freeze-dried to obtain the dried products [10].

There have been many reports related to the preparation of MFC from bagasse [18], banana [10, 19, 20], peel of prickly pear fruits [6], mulberry [3], wheat straw [21], cotton, sisal, ramie [22], etc by using 64 wt% H_2SO_4 solution at a diversity of hydrolyzing condition such as reaction temperature, reaction time and original dimension of raw materials, etc. Therefore, in the present work, the BFs were treated with 64 wt% H_2SO_4 by varying reaction times, under different controlled reaction temperatures (table 1). To study the effect of initial dimension of BFs on the hydrolysis reaction, the ground BFs were sieved using test sieves of 20, 50, 90 and 200 μm aperture sizes (Fritsch GmbH, Germany). In the following discussion, the resulting samples have been referred to as I (BFs with smaller 20 μm in size), II (BFs from 20 to 50 μm in size), III (BFs from 50 to 90 μm in size), and IV (BFs from 90 to 200 μm in size), respectively (table 2).

2.4. Characterization

2.4.1. FTIR spectroscopy. The examination of changes in the chemical composition of the cellulose fibers after chemical

Table 1. Effect of reaction conditions on the hydrolysis of BFs.

Sample code	Temperature (°C)	Reaction time (h)	Initial dimension (μm)
35II20	35	2.0	20–50
45II20	45	2.0	20–50
55II20	55	2.0	20–50
65II20	65	2.0	20–50
45II10	45	1.0	20–50
45II30	45	3.0	20–50
45II50	45	5.0	20–50
45II80	45	8.0	20–50

Table 2. Effect of initial dimension of BFs on the hydrolysis.

Sample code	Initial dimension (μm)	Temperature (°C)	Reaction time (h)
45I30	< 20	45	3.0
45II30	20–50	45	3.0
45III30	50–90	45	3.0
45IV40	90–200	45	3.0

treatment was conducted through FTIR spectroscopy. An EQUINOX 55 spectrometer (Bruker) was exploited to provide the spectrum of each sample. Spectra were taken at a resolution of $4\,cm^{-1}$, with a total of 100 scans for each sample. The FTIR spectrum was obtained in the range of $4000–400\,cm^{-1}$ in the transmission mode. The untreated BFs, the alkali treated BFs and the dried powders derived from the aqueous suspension of MFC were analyzed. The MFC samples that were prepared using the different reaction conditions were also evaluated by FTIR spectroscopy.

2.4.2. Optical microscopy. Morphology of raw BFs and alkali treated BFs was observed under a GX51 optical microscope (Olympus) controlled by computer with 50× magnification.

2.4.3. Morphology of MFC. The morphology and diameter of the various MFC samples were investigated by using field-emission scanning electron microscope (FESEM). The samples were sputter coated with platinum to prevent subsequent charging before measurement by FESEM. Images were taken on a JSM-7401F (JEOL) FESEM.

2.4.4. Chemical composition measurement. Chemical composition of fibers was measured according to the following ASTM procedures: α-cellulose (ASTM D1103-55T), lignin (ASTM D1106-56) and holocellulose (ASTM D1104-56) [19, 21]. The standard deviations were calculated by conducting three replicate measurements for each sample. The percentages of α-cellulose (A), hemicelluloses (H) and lignin (L) were calculated by following equations:

$$A = \frac{W_1}{WP} \times 100\%, \qquad (1)$$

$$H = \frac{W_2}{WP} \times 100\% - A, \qquad (2)$$

$$L = \frac{W_3}{WP} \times 100\%, \qquad (3)$$
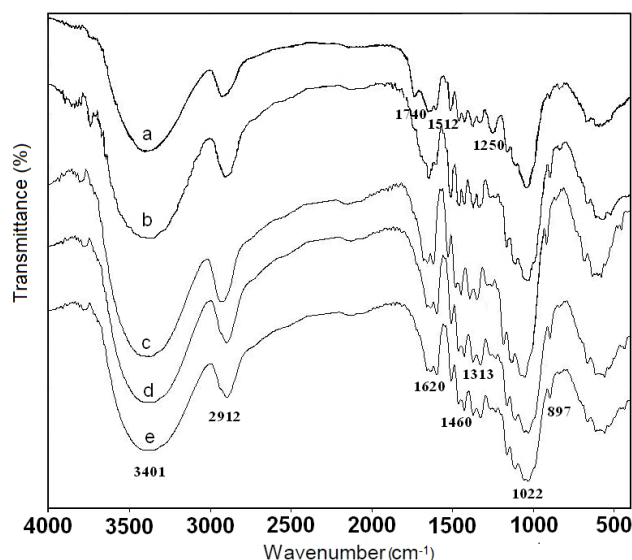
Figure 1. FTIR spectra of BFs after alkali treatment at different concentrations. (a) 0 wt%, (b) 1 wt%, (c) 2 wt%, (d) 4 wt% and (e) 6 wt% alkaline solution.

where W is the weight of the original oven dry fibers, W_1 is weight of the oven dry α-cellulose residue, W_2 is weight of the oven dry holocellulose residue, W_3 is weight of the oven dry lignin residue and P is the proportion of moisture-free content.

2.4.5. Thermogravimetric analysis. The thermal properties of the MFCs were conducted by means of thermogravimetric analysis. The thermal behavior of original BFs, bleached BFs and the MFC ($\sim$10 mg) were determined by a TGA Q500 series thermogravimetric analyzer (TA Instruments), across a temperature range of 30–700 °C, at a heating rate of 10 °C min, in a nitrogen environment (purge rate details: balance chamber flow rate $= 30\,cm^3\,min^{-1}$, furnace flow rate $= 150\,cm^3\,min^{-1}$).

2.4.6. XRD analysis. XRD examinations were performed on a D8 Advance system (Bruker). The diffracted intensity of Cu-Kα radiation (0.154 nm, 40 kV and 30 mA) was measured in a 2θ range between $5°$ and $40°$. The BFs powder and the hydrolyzed BF samples were subjected to crystallinity analysis. Crystallinity index (Ci) of MFC samples were calculated by the following equation:

$$Ci = \frac{I_{002} - I_{Amorph}}{I_{002}} \times \%, \qquad (4)$$

where I_{002} is the maximum intensity of the 002 peak which represents both crystalline and amorphous material and I_{Amorph} is the lowest height between the 200 and 110 peaks ($2\theta \sim 18°$), which represents amorphous material only [9, 21].

3. Results and discussion
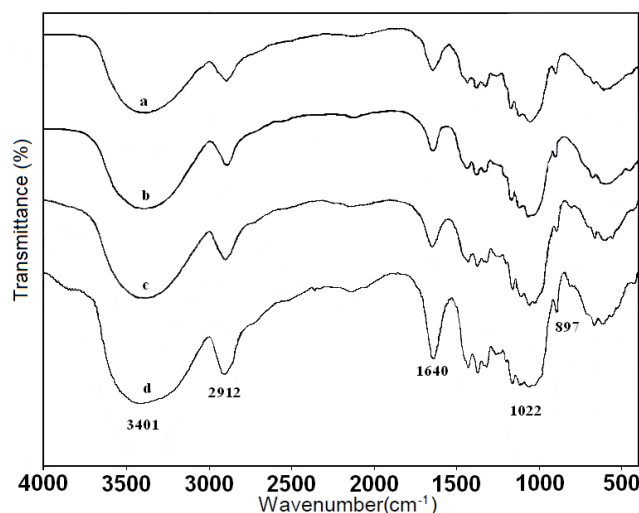
3.1. FTIR spectroscopic analysis

The changes in the chemical composition of BFs due to the hydrolysis were analyzed through FTIR spectroscopy. Figure 1 displays the FTIR spectra of the raw BFs and

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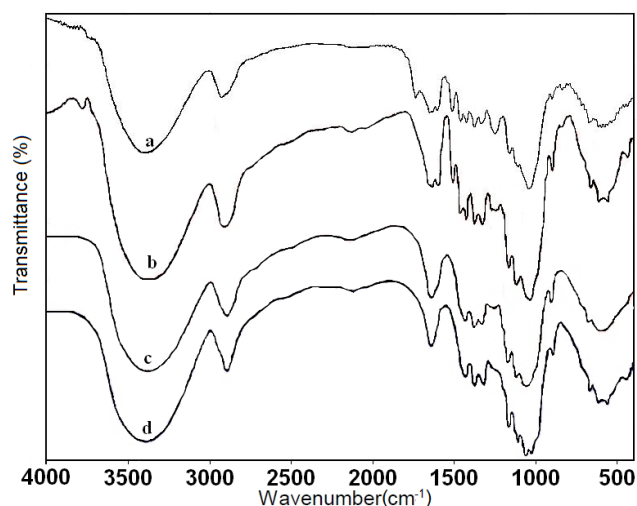
Figure 2. FTIR spectra of BFs after different treatments: (a) raw BFs, (b) 2 wt% alkali treated BFs, (c) bleached BFs, (d) hydrolyzed BFs (sample 45II30).

Figure 3. FTIR spectra of BFs after different treatments: (a) 35II30, (b) 45II30, (c) 45II50, (d) 45I30 samples.

alkali treated BFs with different concentration. The FTIR spectroscopic analyses of the untreated BFs, the alkali treated BFs, the bleached bamboo fibers and the hydrolyzed BFs (figure 2) indicate that composition changes occur in the fibers during hydrolysis. The hydrophilic nature of raw BFs and treated BFs is characterized by the broad band in the 3800–3000 cm^{-1} region, which is related to the –OH groups present in their main constituent. The peak at 2912 cm^{-1} is connected with the aliphatic saturated C–H stretching vibration in lignin polysaccharides including cellulose and hemicellulose. The peak at 1740 cm^{-1} that is observed in the spectrum of the untreated BFs is involved either to the acetyl and uronic ester groups of the hemicellulose or to the carboxylic group of the ferulic and p-coumeric acids of lignin and hemicellulose [3, 10]. The absence of this peak is observed in the FTIR spectrum of the alkali treated BFs with 2 wt% alkaline solution (figure 1), indicating the removal of hemicellulose and lignin from the sample. As the concentration of alkali solutions increases, the FTIR spectra are unchanged. Therefore, BFs were treated with 2 wt% alkaline solution in the following experiments.

In the spectrum of the raw BFs, the peak at 1512 cm^{-1} is related to the aromatic —C═C— stretch of the aromatic rings of lignin. The shortage of this peak in the hydrolyzed BFs is ascribed to the removal of lignin by acid hydrolysis. The lack of peaks at 1740, 1512 and 1250 cm^{-1} (figure 2) expresses the effective elimination of lignin, pectin and hemicelluloses in the hydrolyzed BFs. The peak at 1460 cm^{-1} is linked to the —CH$_2$— bending. The sharp peak observed at 1313 cm^{-1} is in regard to —C—H asymmetric deformations. The peaks in the region from 1200–950 cm^{-1} are owing to —C—O— stretching. The —C—O—C— pyranose ring skeletal vibration leads to a prominent peak at 1022 cm^{-1}. The intensity of this peak rises, which means that the cellulose content increases [10, 21]. The peak at 897 cm^{-1} is connected with glycosidic —C$_1$—H deformation, a ring vibration and —O—H bending. These characters imply the β-glycosidic linkages between the anhydroglucose units in cellulose [10, 21]. Figure 3 shows the FTIR spectra of the MFC that were prepared under different conditions. All the samples

prepared under varying conditions show spectral patterns similar to that of cellulose.

3.2. Morphology analysis

Figure 4 shows the microscopic images of original BFs and alkali treated BFs. The original BFs (figure 4(a)) were covered by massive cement materials, which were obviously diminished after the treatment of BFs with the akaline solution (figure 4(b)). It is clear that hemicellulose, lignin on the surface of BFs has been eliminated, which is confirmed in the FTIR results.

Figure 5 shows the FESEM images of MFCs generated by the hydrolysis route, using 64 wt% H$_2$SO$_4$ solution at various reaction temperatures (35, 45, 55, 65 °C), with reaction time being 2 h. The results reveal that the hydrolysis temperature strongly affects the size decrease of BFs. As the reaction temperature increases, under otherwise identical conditions, the considerable reduction in size is achieved (figures 5(a)–(c)). At 65 °C, the hydrolyzed fibers convert to particle-shape microfibers from fiber-shape ones which is presented in figure 5(d) [10]. Figures 5(e) and (f) show the FESEM images of the two samples hydrolyzed at 45 and 50 °C (sample 45II20 and 55II20) with higher magnification, which indicates the presence of MFC of 20–40 nm in diameter.

Figure 6 shows FESEM images of MFC prepared by the acid hydrolysis of BFs at the temperature of 45 °C for a period of 1, 3, 5 and 8 h, respectively. From the images, it is obvious that when the duration of hydrolysis rises, the dimension of the resulting cellulose microfibers is reduced. Figures 6(e) and (f) show the FESEM images of the two samples hydrolyzed in 3 and 5 h (sample 45II30 and 45II50) with higher magnification, which also indicates the presence of MFC of 20–40 nm in diameter.

Figure 7 displays FESEM images of MFC that were prepared by the acid hydrolysis of BFs having different initial dimensions. Figures 7(a)–(d) show the SEM images of MFC prepared by the hydrolysis of BFs obtained after sieving in sieves of 20, 50, 90, 200 μm aperture size, respectively.
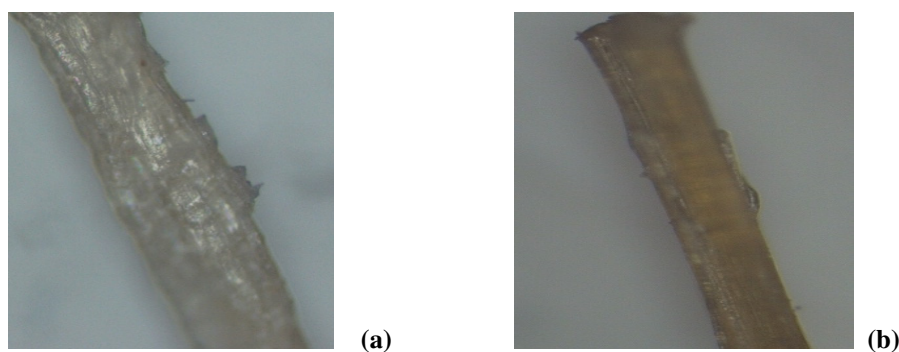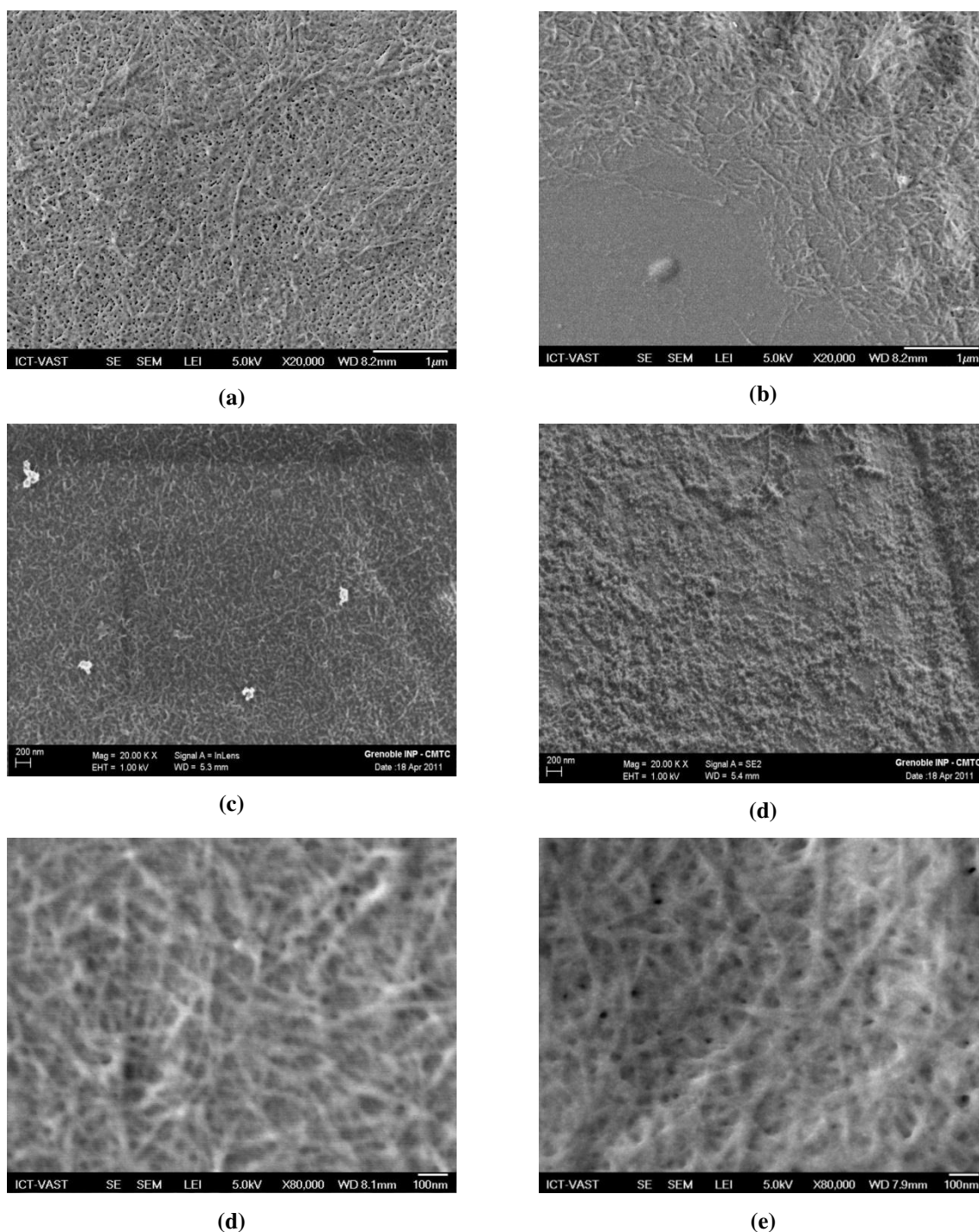
Figure 4. Optical microscopy images of (a) raw BFs and (b) alkali treated BFs with the 2 wt% NaOH solution.
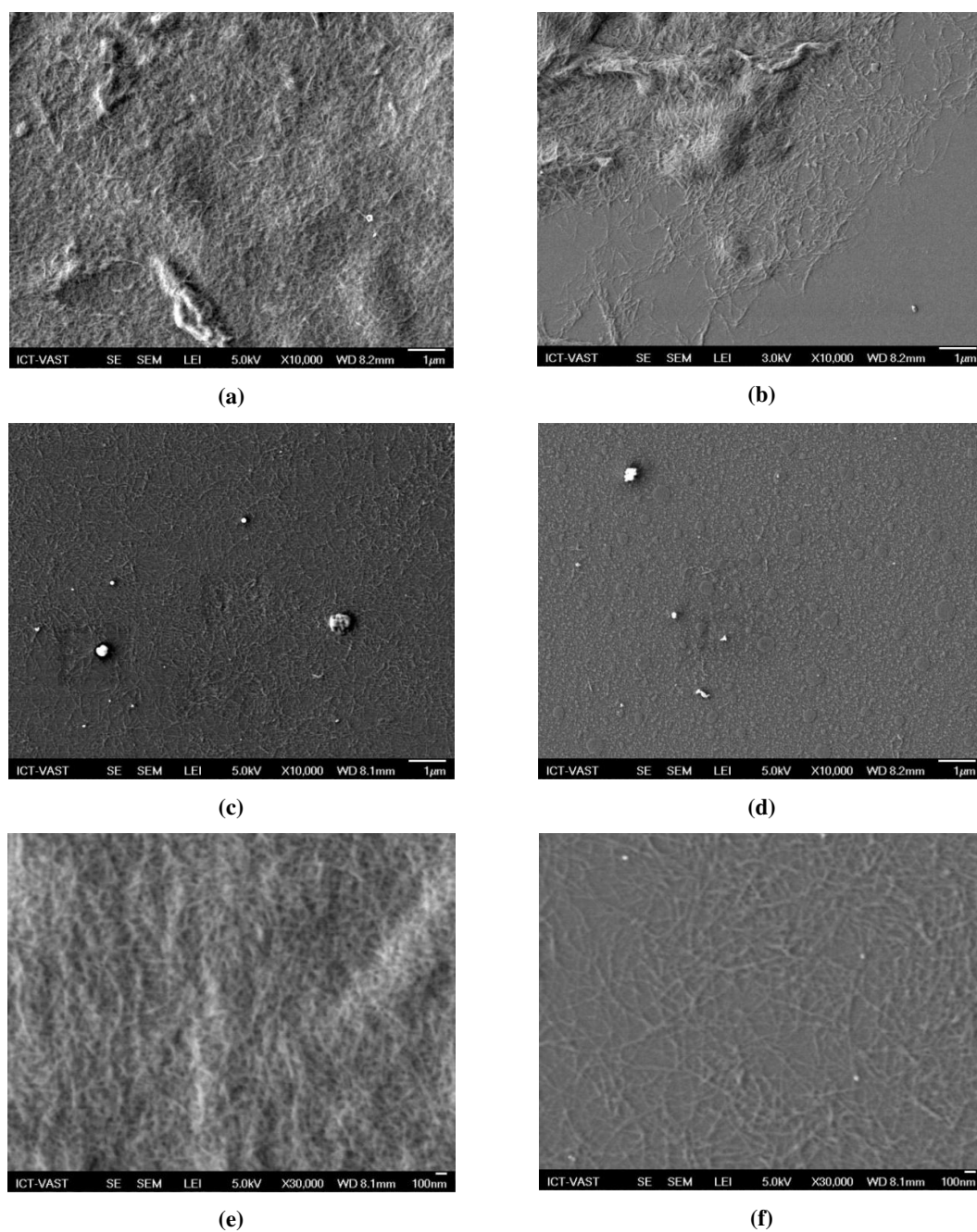
Figure 5. BFs hydrolyzed at various reaction temperatures: FESEM image of sample (a) 35II20, (b) 45II20, (c) 55II20, (d) 65II20. FESEM image with higher magnification of sample (e) 45II20, (f) 55II20.

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(a)

(b)

(c)

(d)

(d)

(e)

Figure 6. BFs hydrolyzed at various reaction times: FESEM image of sample (a) 45II10, (b) 45II30, (c) 45II50, (d) 45II80. FESEM images with higher magnification of sample (e) 45II30, (f) 45II50.

Figure 7 definitely shows that the less the dimensions of the raw BFs, the easier it is to prepare MFC with smaller dimensions [10].

3.3. Chemical composition measurement

Table 3 shows the chemical composition of raw BFs, alkali treated BFs, bleached BFs and hydrolyzed BFs. The result indicates that the original BFs possess the lowest percentage of α-cellulose, the highest percentage of hemicellulose and lignin. After treating BFs with the 2 wt% alkaline solution, the content of α-cellulose increased from 45.24 to 64.08%.

On the contrary, hemicellulose content decreases from 24.16 to 15.87% and lignin decreases from 22.32 to 16.12%. This shows that when the raw fibers were treated with alkaline solution, the small part of hemicellulose and lignin in the surface of BFs was removed. In comparison with the alkali treated BFs, the bleaching raised α-cellulose content in the cellulose fibers to 82.23%. It also reduced the percentage of hemicelluloses and lignin to 7.52 and 5.97%, respectively. This indicates that the further dissolution of the hemicellulose and the depolymerization of the lignin components has occurred during this step [21]. During the bleaching treatment, the hemicellulose is partially broken down and the lignin

(a)

(b)

(c)

(d)
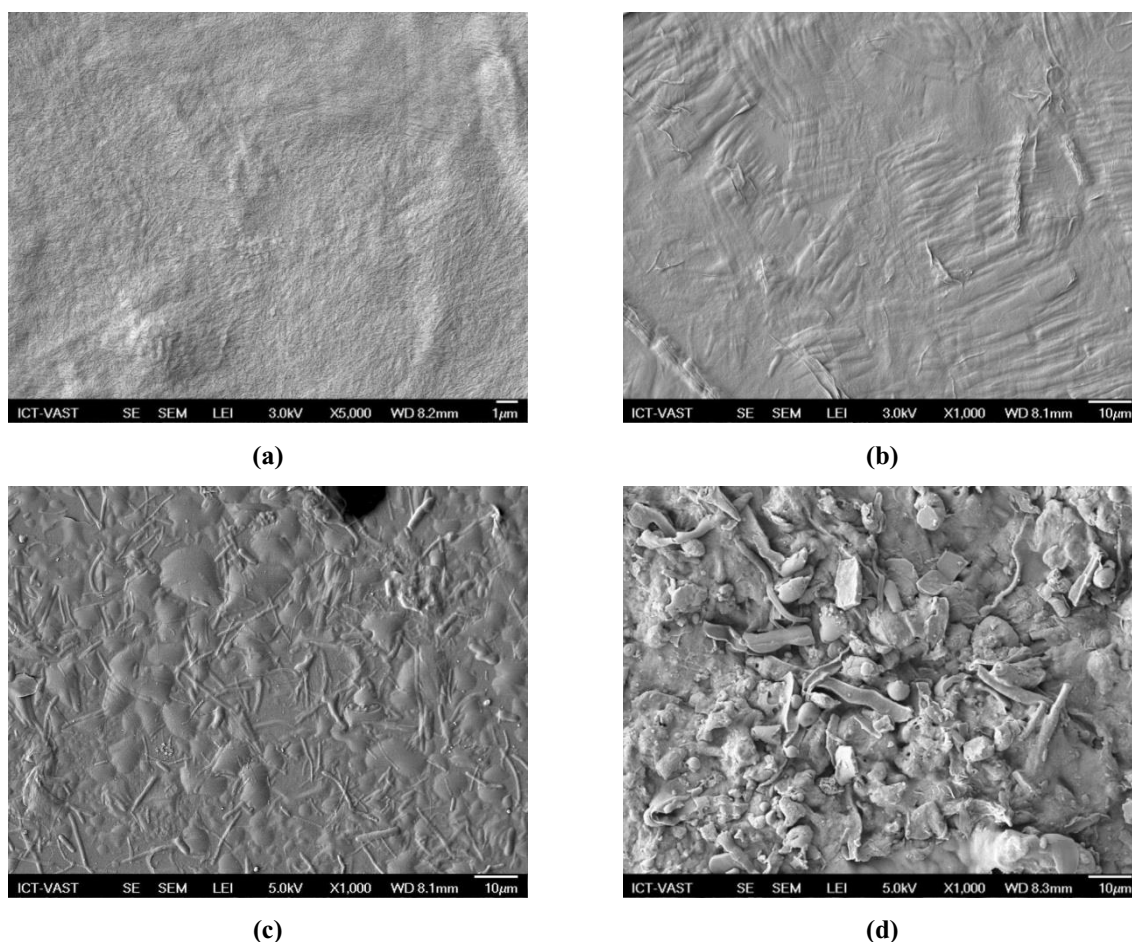
Figure 7. MFC from raw BFs with different dimensions: FESEM image of sample (a) 45I30, (b) 45II30, (c) 45III30, (d) 45IV30.

Table 3. Chemical composition of BFs at different stages.

Sample name	α-cellulose (%)	Hemicellulose (%)	Lignin (%)
BFs	45.24 ± 3.78	24.16 ± 2.30	22.32 ± 2.54
Alkali treated BFs	64.08 ± 2.33	15.87 ± 2.12	16.12 ± 1.90
Bleached BFs	82.23 ± 2.11	7.52 ± 1.87	5.97 ± 0.96
MFC (45II30)	95.53 ± 2.67	2.21 ± 0.54	1.67 ± 0.33

is depolymerized, forming sugars and phenolic compounds that are soluble in water. However, the result reveals that the highest content of α-cellulose and the lowest content of hemicellulose and lignin were reached by acid hydrolysis, in which the contents of α-cellulose, hemicellulose and lignin in the final product were 95.53, 2.21 and 1.67%, respectively. The hydrolysis of glycosidic linkages in hemicellulose and the ether linkages in lignin are catalyzed by sulfuric acid. At the end of the hydrolysis, the cellulose is de-polymerized and defibrillated resulting in microfibrillated cellulose [19]. The removal of hemicellulose and lignin in chemically treated BFs is also verified in FTIR results.

3.4. Thermogravimetric analysis

Figure 8 shows the thermogravimetric analysis (TGA) and differential thermogravimetry (DTG) curves of original BFs, bleached BFs and MFC (sample 45II30). The initial weight loss initiated at 70 °C is attributed to the evaporation of free water in the samples. Due to the low decomposition temperature of hemicellulose, lignin and pectin [3, 10], the curve of original BFs indicates an earlier weight loss started at approximately 210 °C, and then reached a dominant peak at 348 °C on DTG curve related to the pyrolysis of cellulose. On the other hand, the TGA curve of the bleached BFs shows a higher decomposition temperature which starts at 230 °C before reaching dominant peak at 351 °C. The TGA curve of the MFC sample, however, shows significantly different degradation behavior from raw BFs. The lower temperature decomposition starts at around 220 °C and then reaches the dominant peak at 343 °C. In addition, a shoulder peak begins at around 220 °C followed by the main decomposition peak at 343 °C, suggesting there are two degradation stages. The lower temperature stage may correspond to the degradation of more accessible, and therefore more highly sulfated amorphous regions, whereas the higher temperature stage is related to the breakdown of unsulfated crystal interior. Actually, the introduction of sulfated groups into the crystals in the sulfuric acid hydrolysis process could reduce the thermostability of MFC. This result is similar to those reported by Li *et al* [3].

On the other hand, less weight residue of bleached BFs than that of original BFs is due to the fact that the hemicellulose and lignin were mostly removed from the cellulose fibers, while the increased weight residue of cellulose whisker is because of the sulfate groups acting as the flame retardants [3].

(a)　　　　　　　　　　　　　　　　　　　　　　(b)
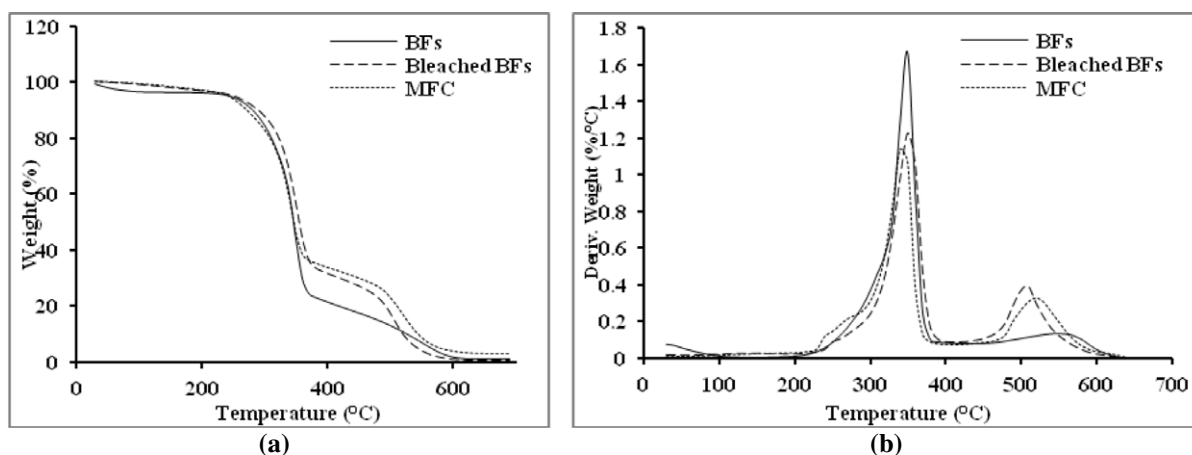
Figure 8. TGA and DTG curves of original BFs, bleached BFs and hydrolyzed BFs (sample 45II30).
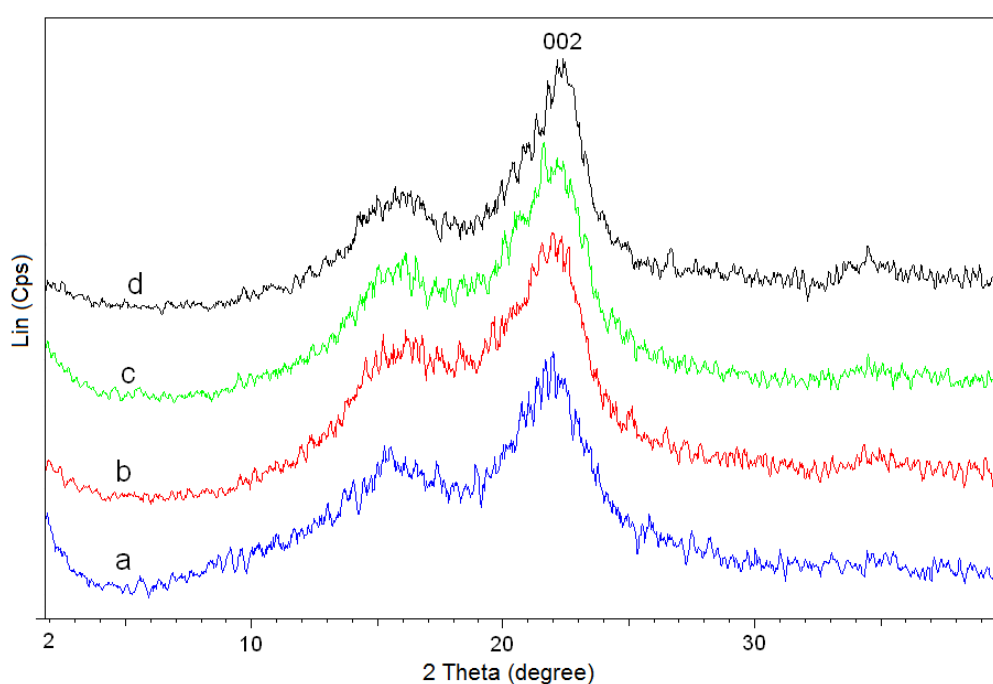
Figure 9. XRD spectra of different fiber samples: (a) untreated BFs, (b) 65II30, (c) 45II80 and (d) 45II30.

Table 4. Crystallinity index of raw BFs and MFCs obtained at different conditions.

Samples	Crystallinity index (%) (equation (4))
Original BFs	56.72
65II30	49.59
45II80	57.14
45II30	70.50

3.5. X-ray diffraction analysis

The crystalline behaviors of raw BFs and the influences of hydrolysis condition on crystallinity of MFC are studied by wide angle XRD. Figure 9 shows the XRD diagrams of the initial BFs, hydrolyzed BFs in different conditions. From the results, it is obviously that cellulose has crystalline nature with an intensive peak at $2\theta = 22.6°$ corresponding to 0 0 2 lattice plane and a shoulder peak in the region

$2\theta = 14$–$17°$ [10, 21]. Table 4 presents crystallinity index (Ci) for raw BFs, hydrolyzed BFs at 65 °C for 3 h (65II30), hydrolyzed BFs at 45 °C for 8 h (45II80), and hydrolysed BFs at 45 °C for 3 h (45II30). The results in figure 9 show that crystallinity increases from 56.72% for untreated BFs (curve a) to 70.50% for MFC in curve d (sample 45II30). The elimination of noncellulosic polysaccharides and the dissolution of amorphous regions lead to higher crystallinity of MFC. Hydrolysis takes place in the amorphous regions prior to the crystalline domains in that the former are more vulnerable to acids. This increase of crystallinity after acid treatment has been discussed by several authors [6, 19, 21]. The results also reveal that reaction conditions have substantial effects on crystallinity of final products. When the reaction temperature reaches 65 °C, under otherwise identical conditions, the crystallinity index substantially decreases to 49.59% (curve b in figure 9 and table 4), indicating that the hydrolysis of crystalline domains takes place at high temperature. Crystalline domains, which are broken

down by hydrolysis, convert to amorphous regions and then dissolve out. As the reaction time endures to 8 h instead of 3 h, and other conditions are unchanged, the crystallinity index considerably reduces to 57.14% (curve c in figure 9). This reveals that after occurring in amorphous regions, the hydrolysis takes place in crystalline domains. The hydrolysis of crystalline domains by raising reaction temperature or enduring reaction time are also confirmed in FESEM results.

4. Conclusions

In this study, microfibrillated cellulose was prepared from BFs by controlled hydrolysis in acidic medium. The results show that final products' properties depend on the hydrolysis conditions such as the temperature of the hydrolysis and reaction time. The major constituent of the final products was found to be cellulose. FESEM images show that fiber diameter ranges from 20 to 40 nm. FTIR and XRD studies verify the removal of lignin and hemicellulose through chemical treatment of the raw BFs, which resulted in improved thermal stability via TGA and DTG results. Significant size reductions were obtained when BFs of initially smaller dimensions were used. The materials that have been developed show potential for application in power bio-source, for use in a variety of polymer composite systems as well as a range of biomedical applications.

Acknowledgments

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