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Cellulose whiskers extracted from mulberry: A novel biomass production

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ABSTRACT

A kind of cellulose whiskers were extracted from the branch-barks of mulberry (*Morus alba* L.) by an alkali treatment at 130 °C and subsequently a sulfuric acid hydrolysis. AFM image showed that the diameter of obtained whiskers was ranged from 20 to 40 nm. The chemical compositions analysis, FT-IR, XRD results indicated that the hemicellulose and lignin were removed extensively in the cellulose whiskers, with a crystallinity of 73.4%. The TGA curves implied a two-stage thermal decomposition behavior of cellulose whisker due to the introduction of sulfated groups into the crystals in the sulfuric acid hydrolysis process. The obtained whiskers may have the potential applications in the fields of composites as a reinforcing phase, as well as in pharmaceutical and optical industries as additives.

1. Introduction

Cellulose, an organic component from the primary cell wall of green plants with the formula $(C_6H_{10}O_5)_n$, is the most common organic compound on earth. Its attractive chemical and physical properties make itself a biomass resource that has been investigated and exploited for many decades and will still be an indispensable raw material for paper-making, food, and additives in optical and pharmaceutical industries (de Souze Lima & Borsali, 2004 Stephen, 1995; Whistler & BeMiller, 1997). Furthermore, cellulosic biomass for ethanol production (Sassner, Galbe, & Zacchi, 2008; Sun & Cheng, 2002) has been put forward as an alternative energy strategy in many countries to meet the demands of energy shortage. On the other hand, compared with the metallic or inorganic reinforcement nanoparticles, the cellulose whiskers, with a high axis ratio (L/d), have attracted a great attention for their easy availability, easy modification chemically and mechanically, as well as their biocompatibility and renewability (Azizi-Samir, Alloin, & Dufresne, 2005; Wang, Cao, & Zhang, 2006). So that, plenty of thermoplastic matrixes reinforced by the cellulose whiskers had been produced (Capadona, Shanmuganathan, Tyler, Rowan, & Weder, 2008; Petersson, Kvien, & Oksman, 2007; van den Berg, Schroeter, Capadona, & Weder, 2007), which have the desirable properties and can be used for various applications.

In nature, the cellulose molecular chains are biosynthesized and self-assembled into microfibrils, which are composed of crystalline domains and amorphous. The latter are susceptible to acid attack because cellulose chains in these regions are randomly oriented in a spaghetti-like arrangement leading to a lower density in these noncrystalline regions (de Souze Lima & Borsali, 2004. So that, the acid hydrolysis could be possibly used to obtain the nanocelluloses, and the dimensions of nanocelluloses are basically dependent not only on the acid species, acid concentration, time, and temperature of hydrolysis reaction, but also on the different origins of cellulose (Azizi-Samir et al., 2005). It was reported that the rod-like cellulose particles from different sources have a diameter and length ranging from 5 to 20 nm and 100 nm to several micrometers, respectively, after the acid hydrolysis (Candanedo, Roman, & Gray, 2005). Moreover, when the nanocelluloses were prepared by sulfuric acid hydrolysis, a number of negative charges could be introduced onto the surface of microcrystals contributing to a stable colloidal suspension (Dong, Revol, & Gray, 1998).

Mulberry trees are extensively grown (e.g., China, India) for their leaves as food for silkworms. The bark of mulberry tree is fibrous and used for paper-making. In addition, the mulberry bark has been used as a herb for removing heat from the lung, relieving asthma and inducing diuresis, and it has anti-HIV, anti-oxidative, anti-hypotensive and cytotoxic activities (Du et al., 2003). However, mass utilization of mulberry bark is not carried out yet. Thousands tons of mulberry branches consisting of bast and stalk are harvested for firewood or agro-wastes every year. In this work, we obtained cellulose fibers firstly from mulberry branch-barks by high temperature alkali treatment, which were then hydrolyzed

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by sulfuric acid to produce cellulose nano-whiskers successfully. Since the energy shortage is currently becoming a tough issue for the sustainable development project throughout the world, it seems urgent to find the environmental acceptable and renewable substitutes.

2. Materials and methods

2.1. Raw materials

The branch-barks of mulberry (*Morus alba* L.) were from Huzhou Academy of Agricultural Sciences (Huzhou, China). The chemical reagents used for degumming of mulberry fibers and preparation of cellulose nano-whisker were analytical grade without further purification.

2.2. Preparation of cellulose fibers

The mulberry branch-barks were cut into ca. 5 cm in length and washed by distilled water at ambient temperature to remove the dust and impurities on the bark surface. The barks were pre-treated by soaking into 1 w/v% NaOH solution at 80 °C for 2 h, followed by washing with adequate distilled water to remove the mulberry epidermis. The barks without epidermis were then treated by 1 w/v% NaOH solution with the addition of 1 w/v% Na_2S and a bath ratio of 1:30 at 80 and 130 °C, respectively, for 1.5 h to get the cellulose fibers. Here, the treatment at 130 °C was carried out in a chamber (ES-315, Tomy Seiko, Japan), while that at 80 °C was carried out in a beaker. The cellulose fibers were bleached by 0.7 v/v% sodium chlorite at 80 °C for 1.5 h with an acetate buffer, a mix solution of 2.7 g NaOH and 7.5 mL glacial acetate acid in 100 mL distilled water, to remove the lignin residues (de Rodriguez, Thielemans, & Dufresne, 2006; Wang, Sain, & Oksman, 2007). The bleached fibers were washed repeatedly by distilled water and subsequently air-dried.

2.3. Preparation of cellulose whisker suspension

Colloidal suspension of cellulose whiskers in water was prepared as described in detail elsewhere (Azizi-Samir et al., 2005; Candanedo et al., 2005; Dong et al., 1998; Wang et al., 2006). Specifically, 10 g above bleached fibers were hydrolyzed in 100 mL sulfuric acid solution (64 w/w%) at 60 °C for 30 min under strong agitation, which was stopped by adding 100 mL cold distilled water. The diluted suspension was centrifugated at 10,000 rpm for 10 min to get the precipitates. The precipitate was suspended in other 200 mL cold distilled water again with strong agitation, followed by a centrifugation. This process was repeated until the supernatant became turbid, i.e., becoming a colloidal suspension. The colloidal suspension was collected and dialyzed against distilled water for 3 d. After 10 min of sonication treatment, the suspension was stored in a refrigerator at 4 °C with addition of chloroform to avoid the bacterial growth. In the suspension, the cellulose whisker content was around 1 w/w% from the weight method.

2.4. Chemical composition measurement

The chemical composition of mulberry branch-barks at different stages was measured according to the method reported by Zobel, Stonecypher, Browne and Kellison (1966). The α-cellulose content was calculated by further treating the cellulose fibers with NaOH after the holocellulose content determination. The lignin content was measured according to Technical Association of Pulp and Paper Industry (TAPPI) standard T222 om-83 and TAPPI 250UM-85.

2.5. Morphology of mulberry fibers and cellulose whiskers

The original mulberry bark and its products at different stages were coated with gold by a vacuum sputter coater and the surface morphologies were observed by a scanning electron microscopy (JSM-5610, JEOL, Japan) with an accelerating voltage of 15 KV. The dimensional image of cellulose whiskers was determined by an atomic force microscopy (XE-100E, PSIA, Korea) in non-contact mode. A drop of diluted suspension (0.1 w/w %) was dispersed on the surface of a clean slide and dried at ambient temperature. The diameters of mulberry fibers and whiskers were calculated by DigitalMicrograph™ (Gatan Inc.) and XEI (PSIA Inc., ver. 1.6.1 Alpha) imaging softwares from the SEM and AFM images, respectively, which were used to illustrate the size distributions.

2.6. Fourier Transform Infrared (FTIR) spectroscopy

The FTIR spectra were recorded on an attenuated total reflection fourier transform infrared (ATR-FTIR) instrument (Nicolet 5700, Thermo Electron Corp., USA) in the range of 400–4000 cm^{-1} with a resolution of 4 cm^{-1}. The samples were ground into powder by a fiber microtome and then blended with KBr followed by pressing the mixture into ultra-thin pellets.

2.7. Powder X-ray diffraction and crystallinity measurements

In order to investigate the crystallinity of mulberry fibers and cellulose whiskers, the milled sample powders were analyzed at ambient temperature by step scanning on a X-ray powder diffractometer (ARL X'TRA, Thermo Electron Corp., USA) using a monochromatic CuKα radiation (λ = 1.54 Å) in the range of 2θ = 10°–50° with a step size of 0.04° and a scanning rate of 5.0°/min. MDI JADE 5.0 software was used to calculate the crystallinity of each sample.

2.8. Thermal stability

The thermal stabilities of untreated mulberry barks, mulberry fibers and cellulose whiskers were characterized using a Pyris I thermogravimetric analyzer (Pyris Diamond TGA, Perkin-Elmer, USA). The amount of sample for each measurement was about 1 mg. All of the measurements were performed under a nitrogen atmosphere with a gas flow of 20 mL/min and heated up to 700 °C at a heating rate of 20 °C/min.

3. Results and discussion

3.1. Chemical composition

The chemical composition of mulberry bark at different stages is shown in Table 1. The original mulberry bark consists of 37.38% of α-cellulose, 25.32% of hemicellulose, 9.99% of lignin. After the pre-treatment of barks with the NaOH solution, the α-cellulose content was doubled, while both hemicelluloses and lignin reduced to no half of originals. Comparing with the alkali treat-

Table 1
Chemical composition of mulberry barks at different stages.

Samples	α-Cellulose (%)	Hemicelluloses (%)	Total lignin (%)
Original mulberry barks	37.38 ± 2.31	25.32 ± 2.45	9.99 ± 0.82
Pre-treated mulberry barks	76.08 ± 1.95	11.80 ± 1.48	4.34 ± 0.14
Cellulose fibers obtained at 80 °C	82.01 ± 1.32	8.26 ± 0.84	1.77 ± 0.22
Cellulose fibers obtained at 130 °C	88.54 ± 1.72	4.23 ± 0.44	0.86 ± 0.35

ment of pre-treated mulberry barks at 80 °C, the treatment at 130 °C could obtain the cellulose fibers with more α-cellulose, less hemicellulose and lignin, in which the contents of α-cellulose, hemicellulose and lignin in the cellulose fibers were 88.54%, 4.23% and 0.86%, respectively.

3.2. Morphology of mulberry fibers and cellulose whiskers

Fig. 1 shows the surface morphology of mulberry barks at different stages, indicating that the fibers in the original mulberry barks (Fig. 1a) were bonded together by massive cement materials, which were obviously diminished after the pre-treatment of barks with the NaOH solution (Fig. 1b) and the fiber-bundles were dispersed into individual fibers with a diameter of ca. 25 μm. Comparing with the smooth cellulose fibers obtained at 130 °C by alkali treatment of pre-treated mulberry barks, the fibers obtained at 80 °C has some lignin and hemicellulose remained, which agrees well with the chemical composition analysis.

In the cellulose fibers, the sulfuric acid hydrolysis usually could cleave the amorphous region of microfibrils transversely, resulting in a diameter reduction of fibers from micron to nanometers (Azizi-Samir et al., 2005). Fig. 2 shows the AFM image of cellulose whiskers prepared by the treatment of cellulose fibers with sulfuric acid solution, indicating that the dominant size of whiskers was ca. 30 nm. The diameters of both mulberry fibers obtained at 130 °C by alkali treatment of pre-treatment mulberry barks and whiskers were calculated and their size distribution were illustrated in Fig. 3. It shows that 90% of mulberry fibers had the diameter ranged from 20 to 36 μm and 33.3% of fibers centered at 28–30 μm. On the other hand, after the sulfuric acid hydrolysis, 42.4% of obtained whiskers had the diameter ranged from 25 to 30 nm, which is similar to those obtained from cotton, wood pulp and tunicin, possibly due to the aggregation of monocrystals (Candanedo et al., 2005). The AFM image also showed that the length of mulberry cellulose whiskers was around 400–500 nm.

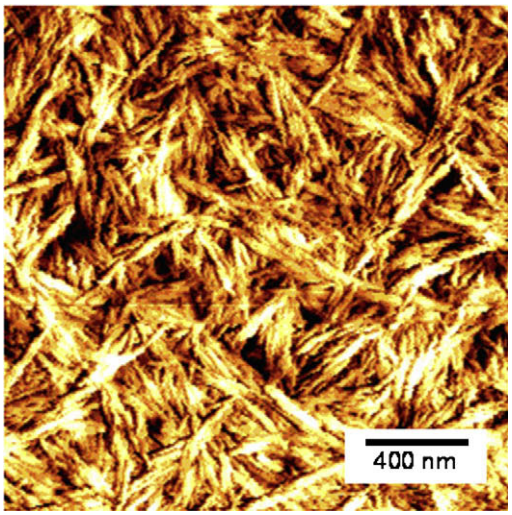

Fig. 2. AFM image of cellulose whiskers prepared by the treatment of sulfuric acid solution.

3.3. FTIR spectroscopy analysis

Fig. 4 shows the FTIR spectra of original mulberry barks, pre-treated mulberry barks, cellulose fibers obtained at 130 °C by alkali treatment and cellulose whiskers obtained after the sulfuric acid hydrolysis. The peak centered at 1736 cm^{-1} in the spectrum of original mulberry barks (Fig. 4a) is attributed to the acetyl and uronic ester groups of hemicellulose or the ester linkage of carboxylic group of ferulic and *p*-coumaric acids of lignin and/or hemicellulose (Sain & Panthapulakkal, 2006; Sun, Xu, Sun, Fowler, & Baird, 2005), which decreased in the spectrum of pre-treated barks (Fig. 4b) and finally disappeared in the spectra of cellulose fibers obtained at 130 °C by alkali treatment (Fig. 4c) and cellulose whiskers obtained after the sulfuric acid hydrolysis (Fig. 4d) due to the

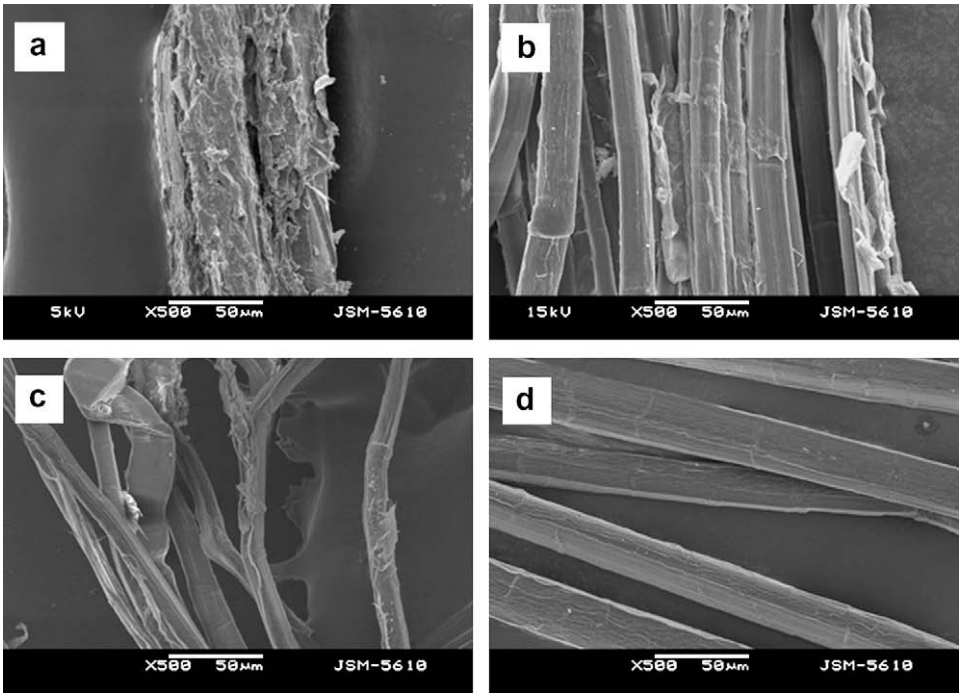

Fig. 1. SEM images of (a) original mulberry barks, (b) pre-treated barks with the NaOH solution, and cellulose fibers obtained at (c) 80 °C and (d) 130 °C, respectively, by alkali treatment of pre-treated mulberry barks. Scale bars, 50 μm.

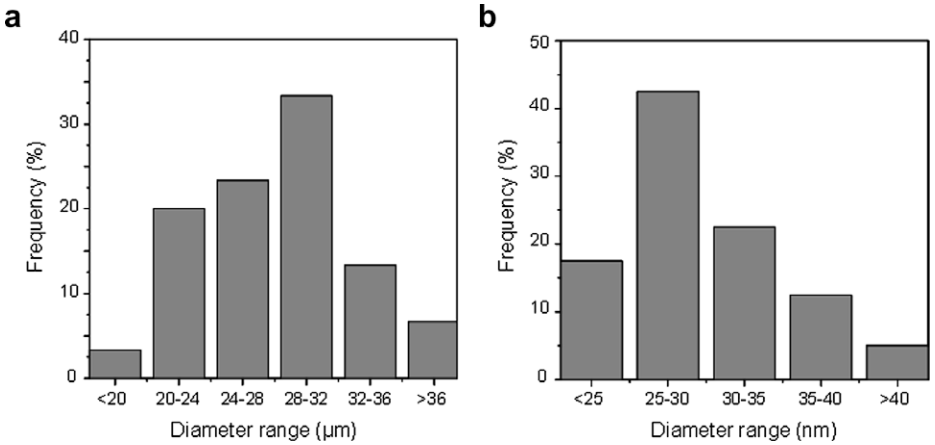

Fig. 3. Size distributions of (a) cellulose fibers obtained at 130 °C and (b) cellulose whiskers.

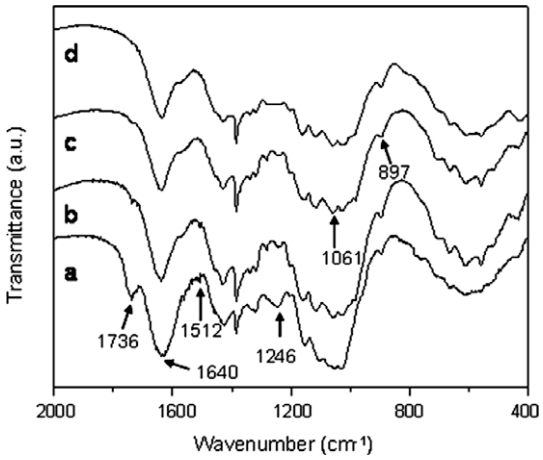

Fig. 4. FT-IR spectra of (a) original mulberry barks, (b) pre-treated barks with the NaOH solution, (c) cellulose fibers obtained at 130 °C by alkali treatment of pre-treated mulberry barks and (d) cellulose whiskers.

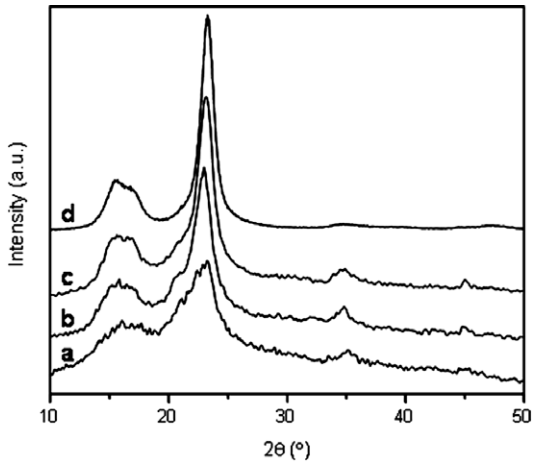

Fig. 5. X-ray diffraction patterns of (a) original mulberry barks, (b) pre-treated barks with the NaOH solution, (c) cellulose fibers obtained at 130 °C by alkali treatment of pre-treated mulberry barks and (d) cellulose whiskers.

removal of hemicellulose and lignin from the mulberry fibers during the step-wise chemical extraction. The band at 1640 cm^{-1} is assigned to the absorbed water. Although the tiny peak at 1512 cm^{-1} in Fig. 4a may be from the water, we believe that it was predominantly contributed from the aromatic C=C stretch of aromatic ring in the lignin since this peak disappeared in other three spectra due to the removal of lignin (Sun et al., 2005; Xiao, Sun, & Sun, 2001). Other two peaks at 1061 and 897 cm^{-1} are associated with the C–O stretching and C–H rock vibrations of cellulose (Alemdar & Sain, 2008), which appeared in all of the spectra. No difference was found in the spectrum of cellulose whiskers compared with that of cellulose fibers, suggesting that in the case of hydrolysis, the molecular structure of cellulose was not changed.

3.4. X-ray diffraction

Fig. 5 shows the XRD patterns of original mulberry barks, pre-treated mulberry barks, cellulose fibers obtained at 130 °C by alkali treatment and cellulose whiskers obtained after the sulfuric acid hydrolysis. The patterns indicated that the celluloses obtained from mulberry barks presented a typical form of cellulose I since there was no doublet in the main peak at $2\theta = 23°$ (Klemm, Heublein, Fink, & Bohn, 2005). From the original barks, cellulose fibers to the whiskers, the diffraction peak at 23° for cellulose I form became sharper and sharper, indicating an increase of crystallinity. Due to the significant increase of relative peak intensity at 23°, other two peaks at 35° and 45° were almost disappeared in Fig. 5d. The crystallinity of each sample were calculated and listed in Table 2. A gradually increase of crystallinity from 46.9% for the original barks, 58.8% for the pre-treated mulberry barks, 63.7% for the cellulose fibers obtained at 130 °C by alkali treatment to 73.4% for the cellulose whiskers was observed. From the original barks to the cellulose fibers, the increase of crystallinity was undoubtedly due to the removal of hemicellulose and lignin, which are existed in the amorphous regions, leading to the realignment of cellulose molecules. Moreover, in the process of hydrolysis, the hydronium ions could penetrate into the amorphous regions of cellulose promoting the hydrolytic cleavage of glycosidic bonds and finally releasing the individual crystallites (de Souze Lima & Borsali, 2004. During the aggregation forming the nano-whiskers,

Table 2
Crystallinities of mulberry barks at different stages.

Samples	Crystallinity (%)
Original mulberry barks	46.9
Pre-treated mulberry barks	58.8
Cellulose fibers obtained at 130 °C	63.7
Cellulose whiskers	73.4

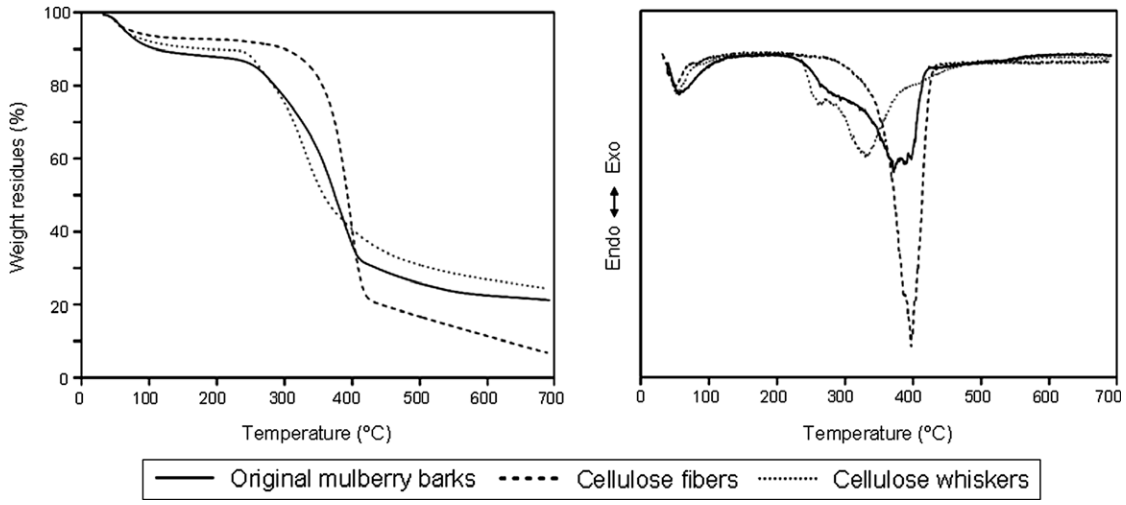

Fig. 6. TG and DTG curves of original mulberry barks, cellulose fibers obtained at 130 °C by alkali treatment of pre-treated mulberry barks and cellulose whiskers.

realignment of monocrystals may occur, leading to the further increase of crystallinity of whiskers obtained in this work.

3.5. Thermostability analysis

Fig. 6 shows TG and DTG curves of original mulberry barks, cellulose fibers obtained at 130 °C by alkali treatment and cellulose whiskers obtained after the sulfuric acid hydrolysis. The initial weight loss started at 60 °C is due to the evaporation of water in these samples. Due to the low decomposition temperature of hemicellulose, lignin and pectin (Morán, Alvarez, Cyras, & Vázquez, 2008), the curve of original mulberry barks shows an earlier weight loss started at around 210 °C, which reached a dominant peak at 350 °C on the DTG curve accounted for the pyrolysis of cellulose. On the other hand, the cellulose fibers obtained at 130 °C by alkali treatment shows a higher decomposition temperature at 397 °C, which is much higher than those reported by Yang et al (Yang, Yan, Chen, Lee, & Zheng, 2007). However, the cellulose whiskers showed significantly different degradation behavior from mulberry fibers. Lower start decomposition temperature at 220 °C and broader degradation temperature were detected. There is a shoulder peak at 228 °C before the main decomposition peak at 335 °C, suggesting there are two degradation stages similar to that reported by Maren and William (Maren & William, 2004). 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 whiskers as reported elsewhere (Julien, Chornet, & Overend, 1993; Kim, Nishiyama, Wada, & Kuga, 2001).

On the other hand, less weight residue of cellulose fibers than that of original mulberry barks is due to that fact that the hemicellulose and lignin were mostly removed from the cellulose fibers (Nguyen, Zavarin, & Barrall, 1981), while the increased weight residue of cellulose whisker is because of the sulfate groups acting as the flame retardants (Maren & William, 2004).

4. Conclusions

In this work, we successfully extracted the cellulose fibers and whiskers from the branch-barks of mulberry (*Morus alba* L.), which is an abundant biomass planted in China. Generally, the cellulose fibers obtained at 130 °C by alkali treatment had higher cellulose content and lower hemicellulose and lignin contents than those obtain at 80 °C, which had the size predominantly ranged from 20 to 36 μm and high thermostability. On the other hand, the cellulose whiskers produced by sulfuric acid hydrolysis had high crystallinity with a size ranged from 20 to 40 nm and a length of 400–500 nm. The cellulose whiskers revealed two-stage decomposition behavior due to the introduction of sulfated groups into the crystals in the sulfuric acid hydrolysis process.

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