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Structural characteristics and physical properties of lotus fibers obtained from *Nelumbo nucifera* petioles

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ABSTRACT

Lotus fiber is a natural cellulose fiber isolated from lotus petiole. Botanically, the fiber is the thickened secondary wall in xylem tracheary elements. In order to obtain essential information for the preparation and processing of lotus fibers, the fine structure and properties of lotus fibers were investigated by the aid of transmission electron microscopy (TEM), confocal laser scanning microscopy (CLSM), atomic force microscopy (AFM), X-ray diffraction (XRD), and so on. The results show that lotus fibers display a rough surface topography and an internal structure different from common plant fibers. The percent crystallinity and preferred orientation of crystallites in lotus fibers are 48% and 84%, respectively. Considering the average breaking tenacity and Young's modulus, lotus fibers are similar to cotton. The elongation of lotus fibers is only about 2.6% while their moisture regain is as high as 12.3%.

1. Introduction

Recently, rising competition for cropland between food and cotton, as well as increasing concerns over environment and health are creating a much more demand for exploring new types of natural fibers and greater interest in utilizing agricultural residues. Lotus (*Nelumbo nucifera*) is an aquatic perennial widely favored and planted in subtropical and temperate zone due to its values in utilitarian, aesthetics, and religion (Shen-miller, 2002). In China, there is a famous saying that "the lotus root is broken, but its fibers stay connected". Besides lotus roots, lotus petioles also contain thin fibers that can be seen as fine silky threads when the petiole is broken into pieces. At present, considerable amount of lotus petioles produced after blossom season or harvest of lotus root every year are left in the pond to decompose and wasted. As expected, these residues could generate cellulosic fibers which can be used in textiles. An example of lotus fibers for textile use is Buddhist robes which are made in Myanmar (Ogasawara, 2005; Riggs, 2004). According to the report (Hla, n.d.), the lightweight lotus-fiber fabric can give coolness in hot weather and warmth in cold weather. It also features an everlasting pleasant lotus fragrance. After Malaysian show initial interest in the lotus fibers, lotus-fiber fabric becomes well-loved, especially by Japanese (Hamilton & Milgram, 2007). However, the exploration and utilization of lotus fiber is currently very limited. Now, lotus fibers are mainly produced by hand-extraction method. No parameters are available on the structure, performance and processing technology of lotus fibers. As there is a demand for less labour intensive, more efficient methods for extracting lotus fibers, and in order to evaluate the potential of lotus fibers for fibrous application, we have initiated a research program aiming to carry out a detailed and comprehensive study of fundamental structure and properties of lotus fibers.

The fine structure and intrinsic properties are important factors in determining fiber preparation and processing technology, and end-use performance characteristics. Although ultrastructure of common fiber cell walls have been reported extensively, such structure of lotus fiber has to be further investigated using TEM because lotus fiber has a different origin compared with common plant fibers. In botany, lotus fibers are the thickened secondary cell walls of xylem tracheary elements in lotus petiole (Wang, 1950). Our previous report (Liu, Han, Huang, & Zhang, 2009) indicates that lotus fibers are essentially composed of cellulose, hemicelluloses and lignin. Lignin is a major structural component of plant fiber material, conferring mechanical strength to the cell wall (Esmeraldo et al., 2010). The presence of lignin in high concentration in the cell wall is regarded as a positive benefit, for example, in fiber-

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board industries, while it is generally regarded as undesirable by the pulp and paper industries (Fromm, Rockel, Lautner, Windeisen, & Wanner, 2003). Hemicellulose is the most hygroscopic component in fiber cell walls, although too high hemicellulose content may impair fiber quality (Rowell, 2005). So information on lignin and hemicelluloses distribution is important for evaluating fiber properties and for modifying the content of lignin and hemicellulose.

On the basis of our previous work (Liu et al., 2009), the present article mainly investigated the composition, distribution of main components, and fine structures, as well as their relations to hygroscopic and mechanical properties of lotus fibers.

2. Experimental

2.1. Material

Lotus petioles were collected from ready-to-harvest lotus fields in Weishan Lake in China. All the petioles were cleaned before extracting fibers.

2.2. Fiber extraction

Lotus fibers are obtained by hand-extraction method. Take a segment of fresh lotus petiole, and make a light score around the entire epidermis with a knife so that the petiole can rapidly split. The fibers will come out of the petiole during further stretching of the two parts. Keep in mind not to apply too much force on the petiole, or cut through it. The fibers between the two pieces of petioles can be stretched up to at least 10 cm without breaking, as shown in Fig. 1, and further stretching to 20 cm only causes a few fibers broken. The hand-extracted lotus fibers are yellowish-white in color, giving out a soft hand and pleasant lotus fragrance. Under scanning electron microscopy (Fig. 1), the number of microfibers in a lotus fiber usually ranges between 3 and 12.

2.3. Morphological studies

A JEOL JSM-6390LV scanning electron microscope (SEM) was used to observe morphologies of xylem tracheary elements and lotus fibers. Lotus petiole macerations were made with Jeffrey's solution (Sass, 1940). SEM sample was chemically fixed and critical point-dried as previously described (Pan, Mao, Han, & Zhang, 2008). Permanent paraffin section was used to observe transversal features of lotus petioles under light microscope.

2.4. Fiber composition

The amount of cellulose and hemicelluloses in lotus fibers were determined using the method described by Sun, Fang, Goodwin, Lawther, & Bolton (1998). Lignin in lotus fibers was determined as Klason lignin according to ASTM standard method D1106-96. Three replications were made for each compositional analysis and the average with ± one standard deviation is reported.

2.5. Sample pretreatment

Hand-extracted raw lotus fibers were extracted by phenethyl/alcohol (2:1, v/v) for 6 h to remove waxy and fatty substances. Delignification of lotus fiber was performed by chlorite-acetic acid method (Rowell, Pettersen, Han, Rowell, & Tshabalala, 2005), so the resulting fiber with a residue lignin content of less than 4% was called holocellulose. The holocellulose was further refluxed in an aqueous solution of 17.5% sodium hydroxide at 20 °C for 2 h to remove the hemicellulose fraction generating a solid residue basically composed of cellulose. Before the preparation of ultrathin sections, all the samples were swelled in 18% sodium hydroxide at room temperature for 3 h.

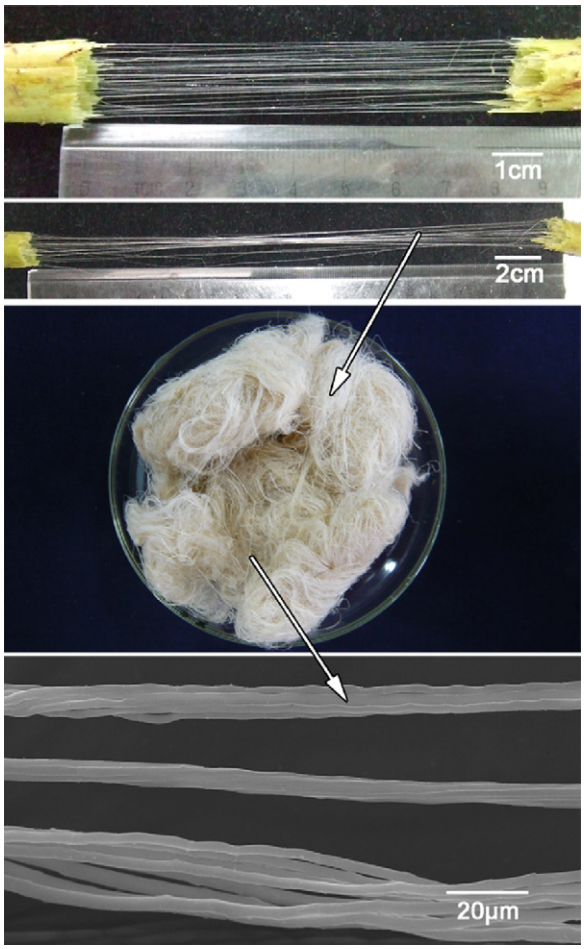

Fig. 1. Hand-extracted lotus fibers.

2.6. Atomic force microscopy (AFM) characterization

The surface morphology of lotus fibers before and after delignification was investigated using a MultiMode SPM from Digital Instruments with a NanoScope IV controller. AFM images were recorded in a tapping mode.

2.7. Transmission electron microscopy (TEM) sample preparation

The swollen lotus fibers were fixed in 2.5% glutaraldehye prepared in 0.1 M phosphate buffer (pH 7.2) at 4 °C. After fixation overnight, they were rinsed in 0.1 M phosphate buffer for 2 h and post-fixed in 0.2 M phosphate-buffered 1% osmium tetroxide (OsO_4) for 6 h at the same temperature. Following 2 h rinse in the same buffer, the fixed samples were dehydrated gradient ethanol solutions (30%, 50%, 70%, 80%, 100% × 2) before being transferred to propylene epoxide and gradually introduced to Spurr's epoxy resin in increments. After the samples in 100% resin, the resin was polymerized at 70 °C for 24 h. Ultrathin sections (approximately 700 nm in thickness) were prepared on a LKB-5 ultramicrotome with a diamond knife and collected onto carbon coated copper grids. Ultrathin sections were either double stained with 2% uranyl acetate (20 min) and lead citrate (20 min) or poststained with a 1% solution of potassium permanganate ($KMnO_4$) prepared in 0.1% sodium citrate for 3 min (Donaldson, 1992). All the specimens were examined with a JEOL-1200 EX electron microscope.

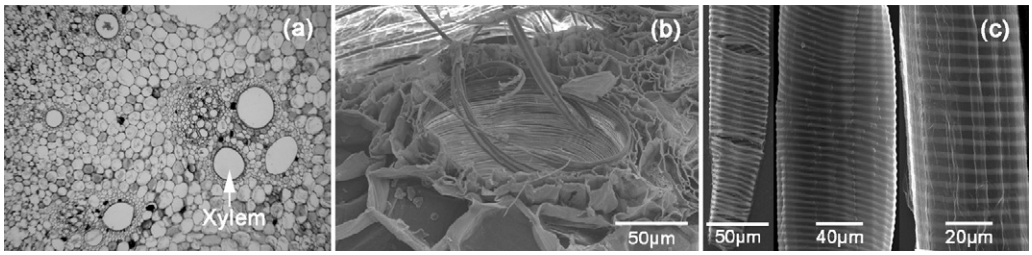

Fig. 2. (a) Light microscopic image of cross section of a lotus petiole. (b) SEM image of cross section of a tracheary element. (c) Portions of macerated tracheary elements.

2.8. Confocal laser scanning microscopy (CLSM)

Lotus fibers were mounted in distilled water and directly examined with a LSM 510 (Zeiss) CLSM taking argon ion laser as excitation source. The excitation wavelength of 488 nm was applied to detect the natural fluorescence of lignin (Donaldson, Singh, Yoshinaga, & Takable, 1999).CLSM images were processed by LSM image browser (version 3.0, Zeiss).

2.9. Fourier transform infrared (FT-IR) spectroscopy analysis

Infrared spectra were obtained in a wavenumber range of 400–4000 cm^{-1} on a Nicolet Centaurus IR Microscope. Fibers were ground and mixed with KBr in the concentration of about 1 mg/100 mg. They were then pressed into transparent thin pellets. Spectra outputs were recorded in the absorption mode as a function of wave number.

2.10. Crystal structure and molecular weight measurement

The crystal structure of lotus fibers was studied in terms of the % crystallinity, crystallinity index, crystal size and the orientation of the cellulose crystals employing X-ray diffraction. The wide-angle X-ray diffractograms were recorded on a Japan Rigaku D/max 2550 PC X-ray diffractometer equipped with Ni-filtered Cu-K_α radiation of wave length 0.154 nm, and operated at 40 kV, 250 mA with a scanning speed at 10°/min. The Bragg angle was scanned from 5° to 60°.

Cellulose of delignified lotus fiber was converted into its nitrate derivative by the methods described by Alexander and Mitchell (1949) for the determination of weight average molecular weight (M_w) via combined analytical system (BI-MwA) of a gel permeation chromatography (GPC) (Waters) and light scattering (LS) (Brookhaven). A 0.2% (w/w) cellulose nitrate/tetrahydrofuran solution for analysis was prepared in a freshly distilled chromatographically pure tetrahydrofuran, and analyzed at 35 °C using tetrahydrofuran as the eluent at a flow rate of 1.0 mL/min and injection volume of 50.0 μL.

2.11. Fiber properties

The fineness, tensile strength, tensile elongation, and Young's modulus of the fibers were determined by a Favimat-Airobot (Textechno Herbert Stein) tensile tester. A gauge length of 10 mm, a given pretension of 0.05 cN and a test speed of 2 mm/min were used for the test. 100 fibers were tested to determine the fiber properties. The average, minimum, and maximum values as well as percent coefficient of variation (% CV) were reported. Moisture regain of the fibers was investigated by drying the fibers at 105 °C in a hot air oven and later allowing the fibers to regain moisture under standard testing conditions of 65% relative humidity and 21 °C.

3. Results and discussion

3.1. Morphological structures

Fig. 2 shows the morphology of lotus petiole and secondarily thickened tracheary elements in xylem of vascular bundles. Vascular bundles of varying sizes scattered throughout lotus petiole ground tissue (Fig. 2a). Secondary wall thickenings in xylem tracheary elements are clearly visible (Fig. 2b). The helical secondary wall thickenings are called lotus fibers (the term "fiber" as used herein refers to a slender and greatly elongated substance capable of being spun into yarn but not the thick-walled fiber cells) in the present study. The original morphological features of lotus fibers can be observed from macerated tracheary elements portions (Fig. 2c). Tracheary elements vary markedly in size and in pattern of helical secondary thickenings. Because each of these highly scattered vascular bundles is surrounded by large amount of ground tissue, it is difficult to extract lotus fibers using traditional chemical methods. Fortunately, hand-extraction method can easily isolate lotus fibers, causing almost no damage to the fibers. The morphological structure of lotus fibers after being isolated from the petiole have been described in detail by Liu et al. (2009).

3.2. Content and distribution of main components in lotus fiber

The FT-IR analysis showed that lotus fiber consisted mainly of cellulose, hemicellulose, and lignin (Liu et al., 2009). The content of cellulose, hemicellulose, and lignin are 41.4 ± 0.29%, 25.87 ± 0.64%, and 19.56 ± 0.32%, respectively. In comparison with common plant fibers, the natural lotus fibers contain a relatively high amount of lignin and hemicellulose.

Lignin distribution was examined by comparing the staining differences with potassium permanganate (Hepler & Fosket, 1970; Maurer & Fengel, 1990) between the raw lotus fibers and delignified lotus fibers. Transmission electron micrographs of cross-sections of raw and delignified lotus fibers are shown in Fig. 3a and b, respectively. From Fig. 3, it can be seen that the periphery of the control fiber was intensively stained (Fig. 3a) but the staining at the same position became very weak after delignification (Fig. 3b), an indication that lignin was highly concentrated in the outmost layer of lotus fiber. Fibers were further examined by CLSM and FTIR spectroscopy to confirm the absence of lignin. Bright fluorescence (Fig. 3c) can be clearly observed on the control sample, however, the intensity of fluorescence declined notably after delignification (Fig. 3d and d′). Autofluorescence with blue light (488 nm) excitation is primarily due to the existence of lignin and the increased fluorescence intensity means higher lignin concentration according to Donaldson et al. (1999). In FTIR spectra (Fig. 4), the characteristic absorption band of aromatic rings around 1510 cm^{-1} and 1595 cm^{-1} (Gierlinger, Goswami, & Schmidt, 2008; Salamone, 1996) of the control sample (Fig. 4a) disappeared after delignification (Fig. 4b); the small broad band at about 1460 cm^{-1} due to $-CH_3$ deformation in both lignin and hemicellulose (Fig. 4a) (Salamone, 1996) was also greatly weakened when the fiber is free

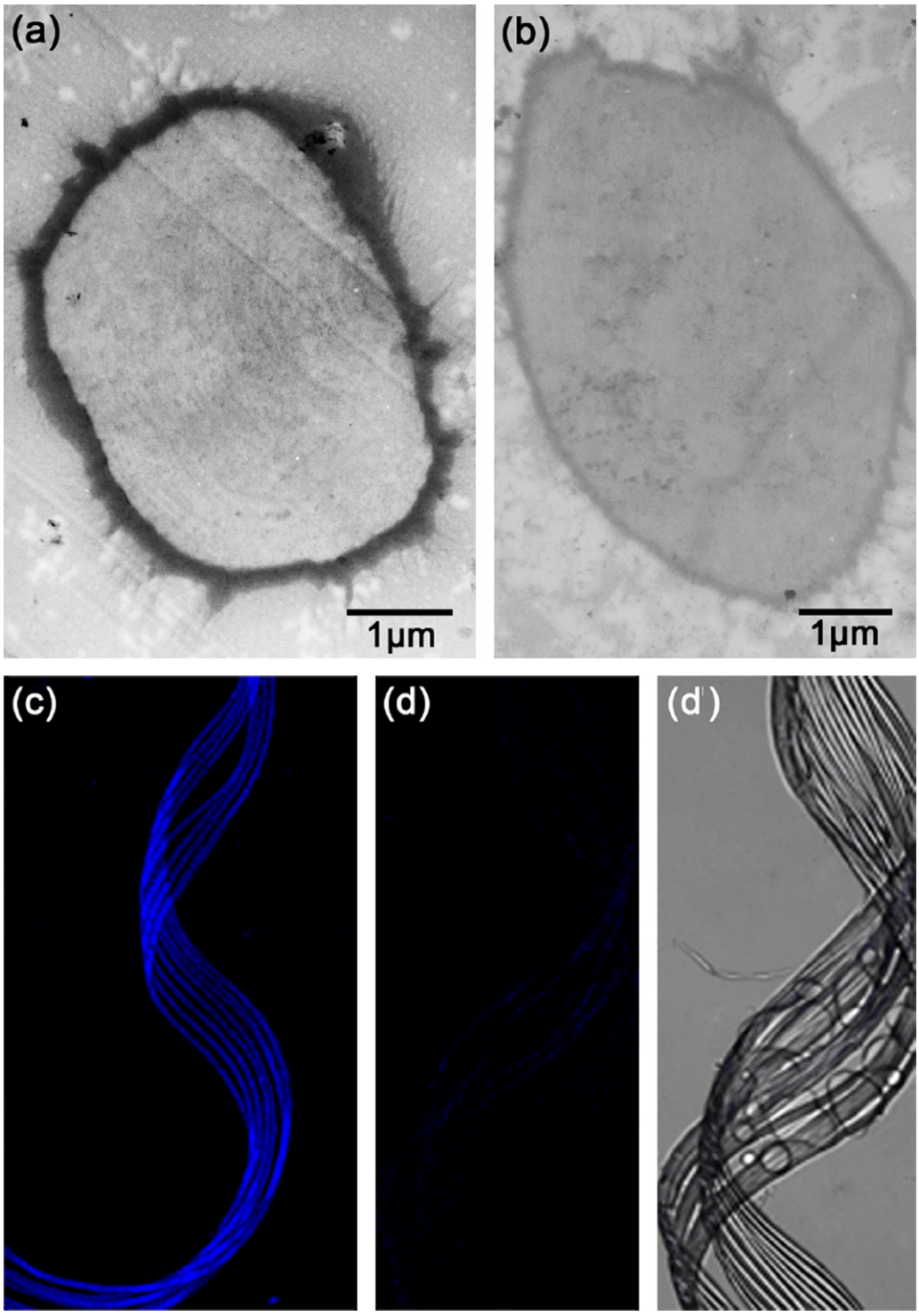

Fig. 3. TEM and CLSM images of lotus fibers before and after delignification: (a) TEM image of raw lotus fiber, stained with $KMnO_4$; (b) TEM image of delignified lotus fiber, stained with $KMnO_4$; (c) CLSM fluorescence images of raw lotus fiber; (d) CLSM fluorescence images of delignified lotus fiber and its corresponding CLSM bright-field image (d′).

of lignin (Fig. 4b). Therefore, based on TEM, CLSM and FTIR results, it is logical to deduce that lignin is primarily distributed on the periphery of lotus fiber. Only a small amount of lignin scattered in the S2 layer is indicated by the weak staining in Fig. 3a.

Similarly, distribution of hemicellulose within lotus fiber was qualitatively determined on the basis of ultrastructural comparisons of fibers with and without a hemicellulose component. Before removal of hemicellulose (Fig. 5a), the fiber had a loose S1 layer and compact S2 layer in which no visible micropores were observed. The hemicellulose constituent was clearly reflected on FTIR spectroscopy (Fig. 4a) by a carbonyl band at 1735 cm^{-1} and C–O symmetric bridge stretching of uronic ester groups at 1247 cm^{-1} (Gierlinger et al., 2008). However, no hemicellulose was detected by FTIR when the fiber was treated with NaOH causing loss of hemicellulose, for the absorption band at 1735 cm^{-1} and 1247 cm^{-1} completely disappeared (Fig. 4c) and the whole spectrum became

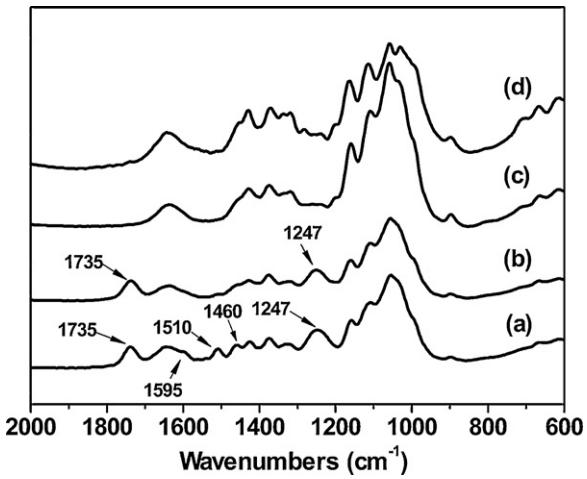

Fig. 4. FTIR spectra of raw lotus fibers (a), lotus fiber holocellulose (b), lotus fiber cellulose (c), and cotton fibers (d).

the same as that of cotton (Fig. 4d). From the TEM image (Fig. 5b), it can be seen that most of the S1 layer was absent and many interstices and micropores showed up on the S2 layer. It is quite possible that the ultrastructural changes are due to the elimination of hemicellulose. Therefore, the analysis above demonstrates that hemicellulose is rich in the outmost layer and also dispersedly located between cellulose microfibrils throughout the S2 layer.

3.3. Surface microstructure

The characterization of surface microstructure was carried out with AFM. Multiple images were taken at different locations on each sample to ensure that the results were indeed typical.

For the raw lotus fiber, the non-smooth surface appears, with granular, fibrous, or block microstructure (Fig. 6a). Grooves of varying depth on the fiber surface are also apparent. The grains or particles have a diameter of 30–300 nm and some of them are seen to overlap each other. Such a surface topography would have a beneficial effect on hygroscopic and quick drying properties of the fibers. When lotus fibers are free of lignin and pectin on the surface, they exhibit mainly fibrous topography (Fig. 6b and c). The morphology and arrangement of fibril aggregates can also be observed. Fibrils aggregate into bundles with a diameter of 30–100 nm. Fibril bundles can be arranged in a parallel or criss-cross fashion as shown in Fig. 6b and c, respectively. The criss-cross pattern is generally present on the outermost surface. In the disordered region, fibril aggregates often appear to branch, coalesce, and bend upward or inward, forming grooves or cracks on the fiber surface. Such area is characterized by the irregular raised surface and the relatively bigger roughness (Rms = 77.495). The region in which fibril aggregates parallel to each other has small undulation on the surface and relatively smaller roughness (Rms = 29.122). In comparison, the disordered region is more accessible to moisture and chemicals.

3.4. Ultrastructure of lotus fiber

TEM is most likely to be used to investigate the internal structure of the fibers. Dewaxing (defatting), delignification and swelling pretreatment of lotus fibers were performed before preparing ultrathin sections, in order to enhance the penetration and diffusion of chemicals into the sample. The ultrastructure of lotus fibers after pretreatment is shown in Fig. 7. Fig. 7a is a typical TEM image of the transverse section, in which no distinct polylamellate structure seen in other plant fibers (Parameswaran & Lises, 1976) was observed. The fiber is composed of only two layers, namely the very thin S1 layer (about 100 nm) and the broader S2 layer. The P layer (primary wall) is absent from the S1 layer because lotus fiber is just the thickened secondary wall material of tracheary element but not a whole cell wall (Wang, 1950). The outermost layer (S1) is loosely attached to the rest part of lotus fiber (S2 layer). The inner layer (S2) is homogeneous and compact in terms of texture. After most of the hemicellulose content was removed, the arrangement of cellulose microfibrils in lotus fibers can be more clearly observed in TEM images (Fig. 7b). Fig. 7b reveals that microfibrils are oriented almost parallel to longitudinal axis of the fiber and the microfibril orientation in the whole S2 layer is very uniform. Highly parallel microfibrils are arranged close to each other, except for the interstices originated from removal of hemicellulose as seen in Figs. 7b and 5b. So, the ultrastructure of lotus fibers is quite different from that of other plant fibers, such as bamboo fibers. Bamboo fibers have more complicated polylamellate structure with alternating and irregular narrow and broader lamellas and the microfibrils also have different orientations in different lamellas (Parameswaran & Lises, 1976). In comparison, the lotus fibers are much simpler in structures and microfibril orientation. It is believed that the different structure characteristics between the bamboo and lotus fibers might be responsible for their respective mechanical properties such as tenacity and elongation.

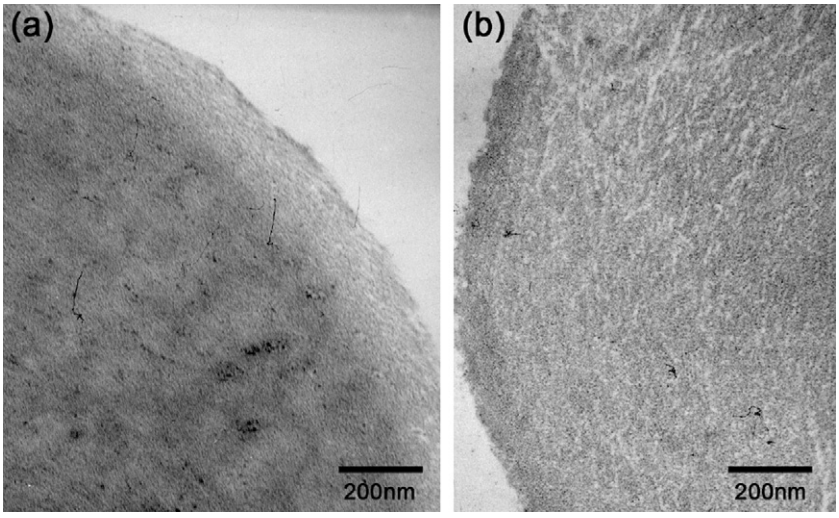

Fig. 5. Transverse sections of lotus fibers before (a) and after (b) removal of hemicellulose, stained with uranyl acetate and lead citrate.

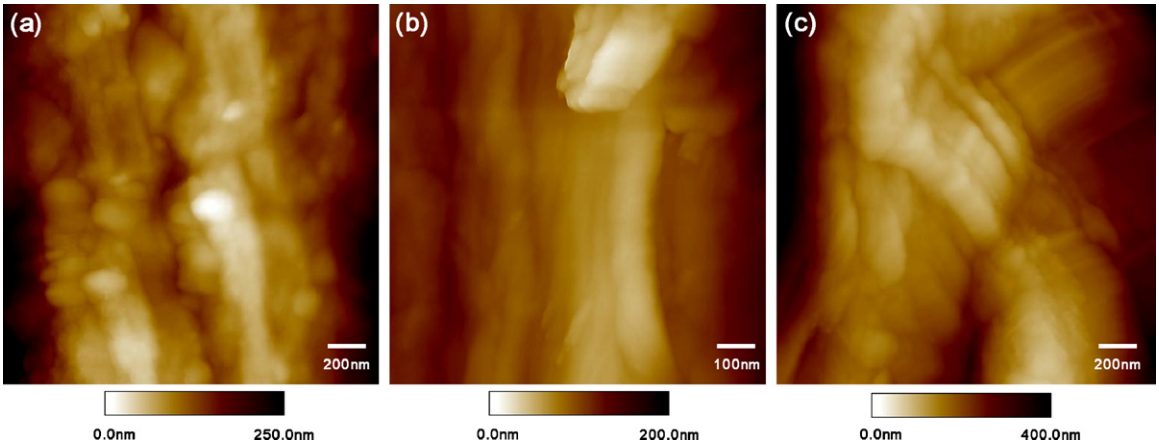

Fig. 6. AFM images of lotus fibers. (a) Surface topography of raw lotus fiber, (b) fibril aggregates aligned in a parallel fashion and (c) fibril aggregates arranged in a criss-cross fashion.

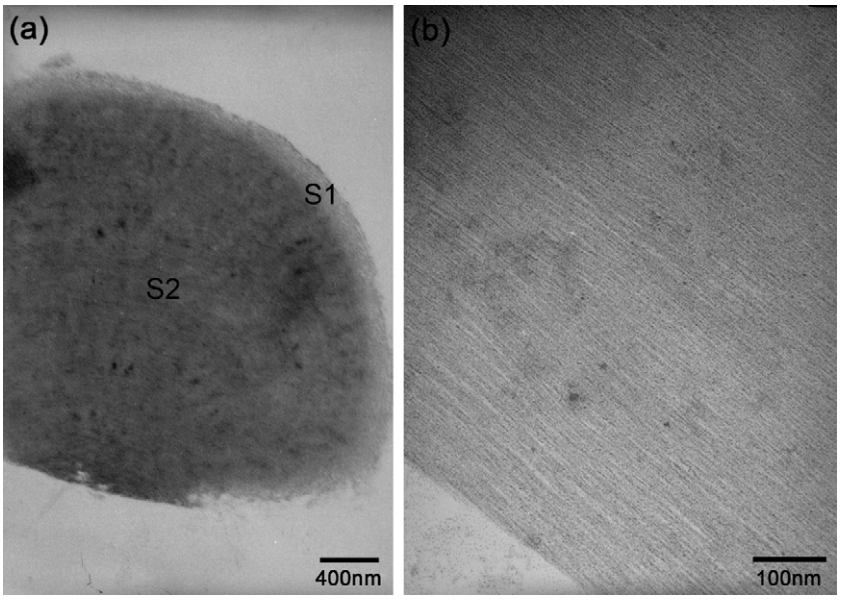

Fig. 7. TEM image of lotus fiber: (a) transverse section and (b) longitudinal section, stained with uranyl acetate and lead citrate.

3.5. Crystallinity and crystal orientation

Preliminary results on crystal structure of lotus fiber have been reported in our previous article (Liu et al., 2009). In this work, we calculate the crystallinity, preferred orientation of crystallites, and crystal size in lotus fiber using the methods of Segal, Creely, Martin, and Conrad (1959), Bohn, Fink, Ganster, and Pinnow (2000), and Hindeleh (1980), respectively. The percent crystallinity of mature lotus fibers is 48%, lower than that of cotton fibers (about 65%) (Reddy & Yang, 2008). The crystallinity index of lotus fibers is 52, indicating that lotus fibers have a slightly lower order of crystallites (Mwaikambo & Ansell, 2002) than cotton (60) (Reddy & Yang, 2008). As expected from the ultrastructure, lotus fibers have a high preferred orientation of crystallites (about 84%), much higher than cotton but similar to flax (He, Tang, & Wang, 2007). Fibers with a low degree of crystallinity have lower strength but greater hygroscopicity and chemical reactivity because of high amorphous regions (Ward, 1950). High crystallite orientation and crystallinity index give the fibers higher strength but lower elongation.

Compared with other cellulose fibers, lotus fiber has the smallest crystal size, which is about 2.5 nm. The crystal size of cellulose obtained from cotton and flax fibers is about 6.1 nm (Gumuskaya,

Table 1
Physical properties of lotus fibers compared with cotton and flax.

Parameter	Lotus fiber				Cotton	Flax
	Mean	Max	Min	%CV		
Fineness (dtex)	0.91	1.81	0.56	32.59	1.5–2.0	1.7–3.3
Strength (cN/dtex)	2.23	5.25	1.07	36.59	2.4–3.1	4.1–5.5
Modulus (cN/dtex)	78.5	144.1	12.9	34.70	50–80	175–184
Elongation (%)	2.60	4.07	1.88	22.38	6.0–9.0	1.6–3.3
Moisture regain (%)		12.3		7.16		–

Usta, & Kirci, 2003) and 2.8 nm (Reddy & Yang, 2005) corresponding to (0 0 2) lattice plane respectively. According to Reddy and Yang (2005), small crystal size means large surface area. Higher surface area increases moisture and chemical absorptions of the fibers.

3.6. Molecular weight

Lotus fibers contain considerable amount of lignin which would cause incomplete dissolution in tetrahydrofuran. In order to obtain a more accurate molecular weight value, lotus fibers were delignified before GPC test (Hubbell & Ragauskas, 2010). The average molecular weight of fiber-forming polymers is an important factor and it determines the mechanical properties of the fibers which they form. Increase in the average degree of polymerization (DP) causes increase in strength of the fiber up to a certain limit (Geller et al., 1971). The GPC result showed that the M_w values of nitrates for lotus fiber is 855,660 g/mol, corresponding to average DP of 5281. These values were the same as ramie cellulose (Timell, 1955) and similar to those found by Timell (1957) for native hemp (4800) and jute (4700) cellulose. The DP value is 800–10,000 for plant cellulose (Klemm, Heublein, Fink, & Bohn, 2005). Lotus fibers have a DP value just inside this range. A satisfactory molecular weight makes it possible for lotus fibers to obtain the mechanical strength necessary for use as textile fibers.

3.7. Fiber properties

Fineness and tensile properties of raw lotus fibers are listed in Table 1. In our previous work (Liu et al., 2009), it was pointed out that the number of microfibers in a lotus fiber usually ranges between 3 and 12. Lotus fibers have different amount of interconnections and different diameter uniformity in different growing periods of the petioles. Hence, the high % CV values in Table 1 were a consequence of these variations. The fineness of hand-extracted lotus fiber is much smaller than that of common plant fibers (Yao, Zhou, & Huang, 1990). The average tensile strength is 2.23 cN/dtex, Young's modulus is 78.5 cN/dtex and the breaking elongation is 2.6%. As expected from the relatively high lignin and hemicellulose content, simple fiber architecture, highly parallel but unvaried microfibril orientation, and low crystallinity, the overall breaking tenacity of lotus fiber is similar to that of cotton but lower than that of flax (Reddy & Yang, 2009a; Yao et al., 1990). Inadequate binding substances between parallel microfibers is also regarded as unfavorable for fiber tensile strength, although each microfiber can be longer than 20 cm. Lotus fiber has an elongation equivalent to that of flax and a Young's modulus similar to that of cotton fiber (Reddy & Yang, 2009b).

Moisture regain of lotus fibers is much higher than that of cotton, which was tested under the same circumstances. Low crystallinity, small fineness, and rough microstructures on the surface may contribute to moisture absorption properties of the fibers. In addition, the presence of relatively high amount of hemicellulose is responsible for a great deal of the moisture regain (Rowell, 2005).

4. Conclusions

Lotus fibers are natural cellulosic fibers extracted from lotus petioles by hand. The fibers contain relatively high amount of lignin and hemicellulose. Lignin is predominantly distributed in the outer layer of the fiber. Hemicelluloses between microfibrils are distributed throughout the whole fiber. Microfibrils are arranged in both parallel and criss-cross fashion on the surface of the fiber but are monotonously orientated inside the fiber. Lotus fibers have a low crystallinity but highly preferred orientation of crystallites. The average DP of cellulose in lotus fibers is equivalent to that in ramie fibers. In comparison with cotton, the very long and thin lotus fibers have greater moisture absorption properties, similar tensile strength but lower elongation. The results in the present study are expected to deepen the knowledge of lotus fibers, and provide essential information for preparation and processing of these fibers.

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