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Comparative study of composition, structure and properties of *Apocynum venetum* fibers under different pretreatments

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Abstract

In this paper, the chemical degumming methods, briefly, peeled bast by hand (Fiber-H) and by machine (Fiber-M), as well as the bacterial degumming method (Fiber-B), were used to obtain the *Apocynum venetum* fibers and the fibers separately obtained by above-mentioned methods as well as the Bast of the *Apocynum venetum* had been characterized by the Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD), scanning electron microscopy (SEM) and mechanical property measurement. It was found that the Bast showed the structure of cellulose I and the cellulose I structure was well preserved in all treated *A. venetum* fibers according to the FTIR and XRD results. Meanwhile, as seen from the SEM images, the treated fibers can be directly suitable for textile industry, which was further confirmed by the mechanical property measurements. It is interesting to note that the crystallinity, the degree of orientation and the mechanical properties of Fiber-B only have slight differences with those of Fiber-H and Fiber-M. These results suggested that the bacterial degumming method had more industrial applications due to its high efficiency, low cost and especially environmentally-benign nature than the chemical degumming methods.

Keywords: *Apocynum venetum*; Cellulose fiber; Bast fiber

1. Introduction

Natural cellulose fibers have always received a great deal of attention due to their great importance of "green" and health protection properties and have been widely used in many fields, such as textile industry and daily life (Mohanty, Misra, and Drzal, 2005; Netravali and Chabba, 2003). As a wild plant fiber, *Apocynum venetum* fiber has attracted increasing interest in recent years, which can be regarded as the environmentally friendly fiber and can be degraded biologically. Since *A. venetum* fiber has many feature functions, such as antibacterial function, health protection and good mechanical properties, it is also named as 'the king of wild fiber', which can be used to produce popular antibacterial textiles and others (Xing, 2001).

In the textile industry, the research about *A. venetum* is mainly focused on two fields – the exploration of the functions and the degumming methods (Butterweck, Simbrey, Seo, Sasaki, and Nishibe, 2003; Han and Zhang, 2006; Yokozawa and Nakagawa, 2004). As *A. venetum* contains many medical ingredients, traditional Chinese medicine has used them for disease prevention and therapy and some studies showed that they had good effects on the hypertension and coronary heart disease, etc. (Lei, Yahara, and Tai, 1995; Murakami, Hishi, and Matsuda, 2001; Nishibe, 2002; Yokzawa and Kashiwada, 2002; Yokozawa, Kashiwada, and Hatori, 2002). And teas made of *A. venetum* leaves have been commercialized. Recently, to cater for the development of clothing industry with the functions of wear-comfort and health protection, the fabric made of *A. venetum* has attracted solidly increasing interest. It was found that the *A. venetum* fabric had a function of emitting far-infrared with wavelengths at 8–15 μm, which can make *A. venetum* underwear keeping warm more effectively. The

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A. venetum underwear also showed a strong character to resist ultraviolet and the permeability of ultraviolet was only 2%, which can protect the body from the ultraviolet (Hakkinen and Auriola, 1998).

Although the health functions and the pretreatment methods have been studied for a long time, to the best of our knowledge, the effect of different pretreatment methods, especially on the composition, structure and properties of *A. venetum* fibers, has not been systematically studied yet. To get the *A. venetum* fibers, generally, there are two degumming methods: chemical degumming and bacterial degumming. Previous studies showed the chemical degumming methods had many disadvantages, such as time-consuming, high cost and environmental pollution (Wu, Jiang, Li, Zhang, and XU, 2004). So many researchers sought more effective chemical degumming methods against the above questions (Han and Zhang, 2006; Wu et al., 2004). In our laboratory, we explored the fast chemical degumming method to solve these problems effectively (Han, Yuang, and Su, 2006). It was found that the *A. venetum* fibers, which were treated consecutively by hydrogen peroxide, hydrogen peroxide and urea or sodium hypochlorite, can be suitable for the textile industry. Meantime, the bacterial degumming method has been explored to get the *A. venetum* fibers, which can keep the fibers natural characteristics and reduce the pollution compared with the chemical degumming methods. In this paper, the effect of three different degumming methods, namely, peeled bast by hand (Fiber-H), peeled bast by machine (Fiber-M) and by bacteria (Fiber-B), on *A. venetum* fibers was studied. It was found that all the as-prepared fibers made by these three methods separately maintained the cellulose I structure and the structures were suitable for the fiber commercialization. These results can give better understanding of the effect of different pretreatment methods and help conduct mass production of the *A. venetum* fibers.

2. Experimental

2.1. Material

All the samples of *A. venetum* were chosen from the same group of one year's white *A. venetum* living in Xinjiang province, PR China. The gum content was 54.26% (Han, Ju, Liu, and Zheng, 2004). The pith tissue of *A. venetum* cannot be used to obtain fibers and was, therefore, mechanically or manually separated from the Bast parts. The Bast of the *A. venetum* was used as raw material.

The Bacteria were purchased from China Center of Industrial Culture Collection (CICC) with number of 036 (Han, Wang, Su, Zheng, and Zhang, 2004).

2.2. Fiber production

The fiber pretreatment conditions mentioned below were the optimized conditions. Many trials of fiber pretreatment were conducted by varying pretreatment parameters, such as, the concentration, time, temperature and the liquor-to-bast ratio (Zhang and Han, 2005). The optimized conditions were decided based on the quality of the fibers produced.

2.2.1. The process using chemical degumming and peeled bast by machine (Fiber-M)

Process. Peeled bast by machine → pre-impregnation → washing → first cooking → washing → second cooking → washing → acid rinsing → washing → dewatering → shaking → drying.

Pre-impregnation. H_2O_2 solution (6 g/L), normal temperature and pressure, liquor ratio 1:15, pH value 6.5, time 20 min.

First cooking. NaOH solution (10 g/L), $NaSiO_3$ solution (2%), sodium tripolyphosphate (2%), normal pressure, temperature 100 °C, liquor ratio 1:15, time 1 h.

Second cooking. Similar to the first cooking step except for the treated time elongated to 2.5 h.

Acid rinsing. Sulfuric acid (1 g/L), normal temperature and pressure, liquor ratio 1:15, time 2 min.

Washing. Washing with the hot water above 70 °C, then hand washing and spray rinsing with tap water (Han et al., 2004).

2.2.2. The process using chemical degumming and peeled bast by hand (Fiber-H)

Process. Peeled bast by hand → pre-impregnation → washing → first cooking → washing → second cooking → washing → acid rinsing → washing → dewatering → shaking →drying.

The parameters are the same with those in Section 2.2.1 (Han et al., 2004).

2.2.3. The process using bacterial degumming and peeled bast by machine (Fiber-B)

Process. Peeled bast by machine → soften → degumming with bacteria → killing bacteria under high pressure → washing → dewatering → drying.

Culture medium. Albumen peptone (2%); glucose (1%); yeast (0.5%); NaCl (0.5%); agar (2%), temperature 121 °C, pH 7.0, time 20 min.

Preparation of bacteria. Drew lines intensively on solid culture platforms with the bacterium freezing in refrigeratory, and put the platforms in the constant temperature box for 24 h at 37 °C.

Degumming with bacteria. $K_2HPO_4 \cdot 3H_2O$ 8 g/L, temperature 42 °C, liquor ratio 1:25, pH 9.1, time 24 h, rotational speed of oscillator 150 r/min (Han et al., 2004).

2.3. X-ray diffraction

The three different *A. venetum* fibers, Fiber-M, Fiber-H and Fiber-B as well as the Bast of the *A. venetum* were used for X-ray diffraction measurements. The four samples were ground in a Wiley mill and all the powders were then screened by 60 meshes prior to pressing. Pressing of sample powders was carried out under axial force about 7.5 tons and for 30 s to fill a circular hole with diameter of 13 mm.

2.3.1. Determination of crystallinity and crystallite size

The wide-angle X-ray diffraction (XRD) was carried on a Rigaku D/MAX-RC X-ray diffractometer recorded using CuKα_1 ($\lambda = 1.540598$ Å) radiation. The X-ray unit operated at 40 KV and 30 mA. Angular scanning was continued 3°–60° at 1°/min and data were collected using 2-step scan mode with angular intervals of 0.05°.

Crystallinity of cellulose in *A. venetum* samples was calculated from diffraction intensity data using empirical method proposed by Segal, Creely, Martin, and Conrad for native cellulose (Segal, Creely, Martin, and Conrad, 1959):

$$\mathrm{CI} = (I_{002} - I_{\mathrm{am}})/I_{002} \times 100\% \tag{1}$$

where the CI is the crystalline index, I_{002} is the maximum intensity of the (002) lattice diffraction and I_{am} is the intensity diffraction at $18°2\theta$ degrees. The counter reading at peak intensity of the (002) lattice diffraction is said to represent the crystalline material and the peak intensity at 18° corresponds to the amorphous material in cellulose material (Ganjyal, Reddy, Yang, and Hanna, 2004; Weimick, LaForge, and Jacobs, 2002).

Dimensions of the unit cell of cellulose crystal in the fiber and the lattice distances were determined assuming the monoclinic crystal system and using the equations reported by Hindeleh and Johnson (1971) and Hindeleh (1980). The average size of crystallite was calculated from the Scherrer equation (Hindeleh, 1980). This method is based on the width of the diffraction patterns occurring in the X-ray reflected crystalline region. In this study, the crystallite sizes of the *A. venetum*samples were determined by using the diffraction pattern obtained from the (101) and (002) lattice planes.

$$D_{(hkl)} = k\lambda/p\cos\theta \tag{2}$$

where $D_{(hkl)}$ is the size of crystallite, k is the Scherrer constant (0.89), λ is the X-ray wavelength, and p is the half-width of the (002) peak in reflection.

2.3.2. X-ray diffraction pattern

X-ray diffraction patterns were recorded on A Bruker D8 Discover model diffractometer. In order to compare the degree of orientation, the four different samples of *A. venetum* were calculated using the following equation.

$$\Pi = (180° - H)/180° \times 100\% \tag{3}$$

Where Π is the orientation degree, H is the width of the Debye's intensity curve on the half height on equator, and is expressed by degree (Cave, 1997; Hu and Hsieh, 1996).

2.4. FT-IR spectroscopy

Fourier Transform-Infrared spectra were acquired using a Bruker Equinox 55, and the spectra were obtained with an accumulation of 32 scans and with a resolution of 2 cm^{-1}. In order to obtain good resolution spectra it was necessary to mill the *A. venetum* to an average length of 0.2 mm. The milled fibers were then mixed with an analytical grade KBr and the mixture was pressed into a disk for the FT-IR measurements.

2.5. Scanning electron microscopy (SEM)

The SEM images of the four samples were examined using a Hitachi S-4300 SE microscope. Prior to the analysis, the samples were coated with gold (layer thickness ~30 nm) to avoid sample charging under the electron beam.

2.6. Fiber properties

The tensile properties of the fibers were measured using an YG001 tensile testing machine. A gauge length of 10 mm with a crosshead speed of 2 mm/min for *A. venetum* fibers and a crosshead speed of 7 mm/min for cotton and ramie were used for testing. 50 fibers of each sample were tested for breaking elongation, modulus and work of rupture.

3. Results and discussions

3.1. X-ray diffraction (XRD) study

3.1.1. Wide-angle X-ray diffraction

To investigate the crystal structures of the as-prepared *A. venetum* fibers, the XRD patterns were measured for these samples. Fig. 1 shows the wide-angle XRD patterns of the four samples, the Bast, Fiber-H, Fiber-M and Fiber-B. It can be seen that the Bast showed cellulose I structure (Dyer and Daul, 1985) with the diffraction peaks of the 2θ angles at 15°, 16.3°, 22°, 34.5°, which were assigned to (101), ($10\bar{1}$), (002) and (040) planes, respectively. And it can be further confirmed that the different

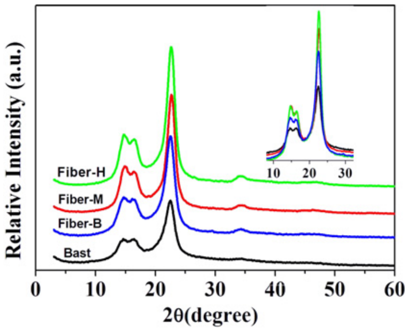

Fig. 1. XRD diffraction data of all the fiber samples: the Bast of the *A. venetum* (black line); Fiber-B (using the bacterial degumming method) (blue line); Fiber-M (red line) and Fiber-H (green line) (using the chemical degumming method, peeled bast by hand and by machine, respectively). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

pretreatment methods did not change the *A. venetum*'s crystal structure, namely, all the as-prepared *A. venetum* fibers still maintained the structure of cellulose I.

In order to compare the four samples more clearly, we normalize the XRD curves on the peak of $2\theta = 18°$ (inset of Fig. 1). It can be found that the strength of the diffraction peaks at (002), (040) and (101) planes in the samples of Fiber-H, Fiber-M, and Fiber-B was much stronger than that in the Bast sample, indicating that the crystal structure of the treated *A. venetum* fibers is better than that of the Bast. The crystallinity index (CI) which can influence the strength and stiffness of fibers (Keekes et al., 2003; Meredith, 1953; Morton and Hearle, 1993) was calculated according to the Segal empirical method (Segal et al., 1959) and it can give a quantitative measure of the crystallinity in fibers. For the bast, the CI was 0.728. However, the CIs were 0.830, 0.823 and 0.782 for Fiber-H, Fiber-M and Fiber-B, respectively, which were higher than that of cotton at 0.60 but smaller than that of flax at 0.80 (Cave, 1997). And also the CIs showed in the order of Fiber-H > Fiber-M > Fiber-B > Bast. Furthermore, the crystal size and dimensions of a unit cell of the samples were calculated, which were in good accordance with that of cellulose I (Dyer and Daul, 1985). All these results indicated that the crystallinity in the treated *A. venetum* fibers was much better than that of the Bast.

3.1.2. X-ray diffraction patterns

Generally, the degree of fibrous orientation can be compared from the Debye rings on the equatorial direction (Reddy and Yang, 2005). If the spot is brighter, it means that the fiber has higher orientation degree. Fig. 2 shows the diffraction patterns of the four samples and these results of XRD images indicated the existence of oriented crystals in the samples (Cao and Billows, 1999; Morton and Hearle, 1993). It can be seen that the diffraction pattern of the Bast showed longer diffracting arcs than those of other samples, suggesting that the orientation degree of the Bast was the lowest one with the orientation degree of 0.822 similar to that of stalk fibers. The diffraction images of all the pretreated *A. venetum* fibers showed narrower and brighter patterns and the orientation degrees were 0.844, 0.883 and 0.833, for Fiber-M, Fiber-H and Fiber-B, respectively. So the orientation degree showed in the order of Fiber-H > Fiber-M > Fiber-B > Bast, which was the same as the CI results.

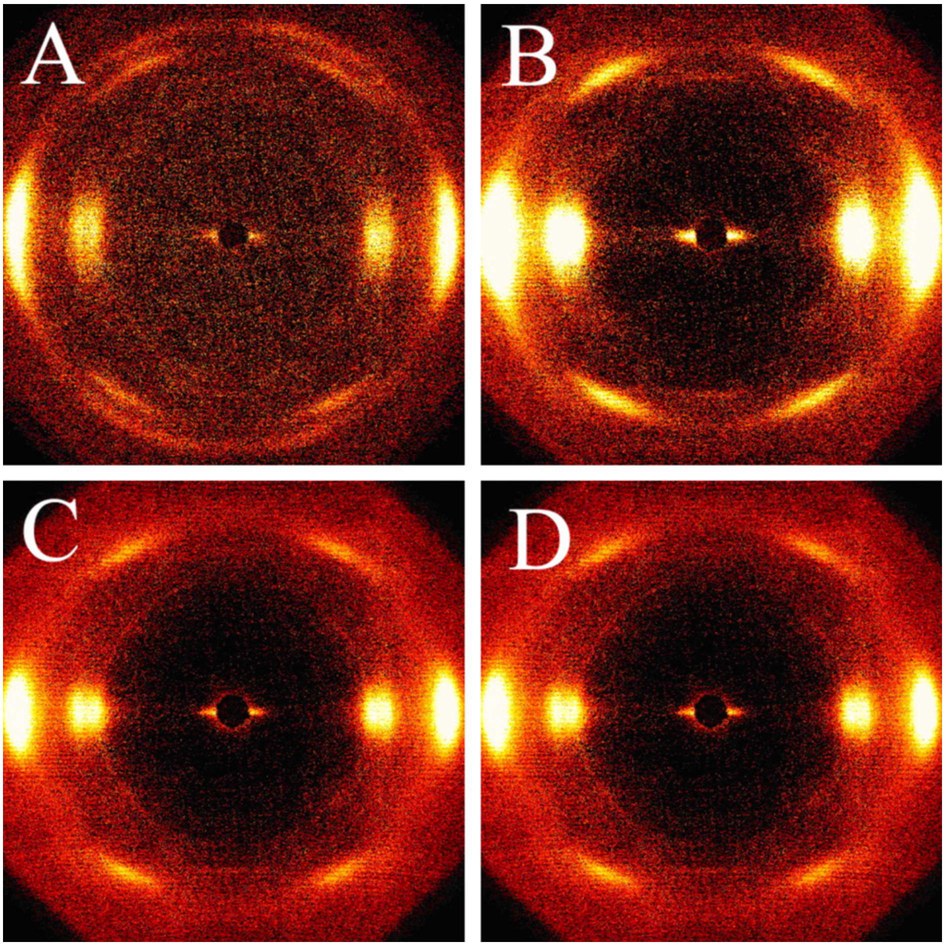

Fig. 2. X-ray diffraction pattern of the four samples: (A) the Bast of the *Apocynum venetum*; (B) Fiber-M; (C) Fiber-H; (D) Fiber-B. The bright diffraction spots in the picture are due to the orientation of cellulose crystals in the four samples.

3.2. FTIR study

As a relatively easy method, FTIR spectroscopy has been widely used in cellulose research, from which the direct structural information and changes can be obtained during various chemical treatments. Fig. 3 show the FTIR spectra of the *A. venetum* fibers after pretreatments as well as the Bast of *A. venetum* to further disclose the structural change during the different degumming process. It can be seen that the absorption peaks of 3420 cm^{-1}, ascribed to OH group, and the absorption peaks in the fingerprint regions at 1432, 1164 and 1058 cm^{-1} attributed to cellulose structure almost did not change for all the fiber samples (During, 1991; Silverstein and Webster, 1998). Meantime, the FTIR spectra of the *A. venetum* fibers after pretreatments were almost the same with very slight different in the absorption wave number maybe due to the apparatus error. However, the FTIR spectrum of the Bast was distinctly different from those of treated fibers. First, the Bast sample showed the CH$_2$ asymmetric and symmetric vibrations at 2918 and 2852 cm^{-1}, respectively, assigned to the less-ordered band of the alkyl chain (Guo, Liu, Nakahara, and Ushida, 2006; Snyder, 1967), indicating the existence of the wax or wax like substances, while this absorption band was disappeared in the treated fibers, indicating the effective remove of the waxes during the degumming process. However, the absorption band at 2918 cm^{-1} was disappeared and the band at 2900 cm^{-1}, ascribed to CH and CH$_2$ stretching vibrations, was clearly observed from the spectra of the treated fiber samples (Silverstein and Webster, 1998). Second, it can be obviously found that the vibration peak at 1735 cm^{-1} in the spectrum of the Bast, which was ascribed to the C═O stretching of methyl ester and carboxylic acid in pectin or the acetyl group in hemicelluloses (Silverstein and Webster, 1998), was disappeared from those of the treated fibers. This indicated pectin and hemicelluloses can be successfully extracted by the degumming method. Third, the absorption band at 1626 cm^{-1} of the Bast was drastically weakened and shifted to 1637 cm^{-1} for Fiber-H, Fiber-M and Fiber-B, ascribed to antisymmetric COO- stretching, was observed(During, 1991), indicating the used degumming methods can remove the lignin effectively. Fourth, the absorption band at 1435 and 1377 cm^{-1} assigned to the CH$_2$ and CH symmetric bending mode (Silverstein and Webster, 1998), respectively, shifted to 1431 and ~1372 cm^{-1}. And the band at 832 cm^{-1} attributed to an aromatic C—H out-of-plane vibration in the lignin was obviously decreased in intensity after the pretreatment (Sun, Lawther, and Banks, 1996). This further indicated that the treatment reduced lignin content. Therefore, it can be concluded that the absorption bands of all the non-cellulose substances, such as hemicellulose, pectin, lignin, and wax like substances, were completely disappeared or drastically reduced. Furthermore, all the as-prepared *A. venetum* fibers can be confirmed belonging to the cellulose I structure. All these results proved the effective pretreatment of these three degumming methods.

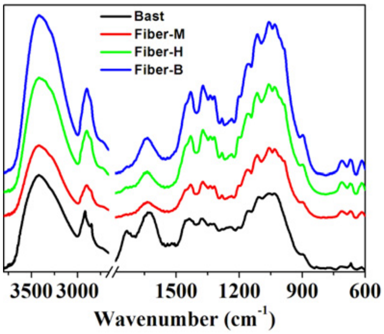

Fig. 3. The FTIR spectra of the four *A. venetum* samples.

3.3. SEM examination of the fibers

Similar to the other natural fibers, the *A. venetum* fibers in the bast were covered with a layer consisting of hemicellulose, pectin, lignin, and wax like substances as shown in Fig. 4A, which can help to bind the single cells of the *A. venetum* fibers and form optimum organizations to protect the *A. venetum* from being damaged by the ambient environment (Zafeiropoulos, Williams, Baillie, and Matthews, 2002). However, the out-layer of the fibers was successfully removed after the degumming processes and the surface of the treated fibers (Fig. 4B–D) became smoother as compared to that of untreated materials. The treated fibers have width of 10–25 μm, which can be directly used to textile industry and other applications. Furthermore, the fibers have convolutions along their length with inner cavum and tapered ends and the knots can be seen in some fibers through optical microscopy image.

3.4. Fiber properties

The fiber properties of the pretreated *A. venetum* fibers as well as the cotton and ramie are summarized in Table 1. The data were not including those of the Bast due to the experimental limitation of the Bast nature. It can be found that the moduli of Fiber-H, Fiber-M and Fiber-B were much higher than that of cotton but lower than that of ramie. Furthermore, the modulus of the Fiber-B was the highest in the three *A. venetum* fibers, which was mainly because of the residue of the lignin, and this was in good accordance with the FTIR spectra, namely, the more residue of the lignin in the Fiber-B made the fiber has higher modulus. On the other hand, the lower modulus of Fiber-H, Fiber-M and Fiber-B than that of ramie indicated that the products made from the treated *A. venetum* fibers will be more flexible and softer. However, the treated fibers have lower work of rupture than that of ramie and cotton, indicating that the *A. venetum* fibers maybe less durable (Reddy and Yang, 2005). Nevertheless, *A. venetum* fibers through different degumming methods are suitable for direct processing on the conventional textile machinery and also for blending with the other common fibers.

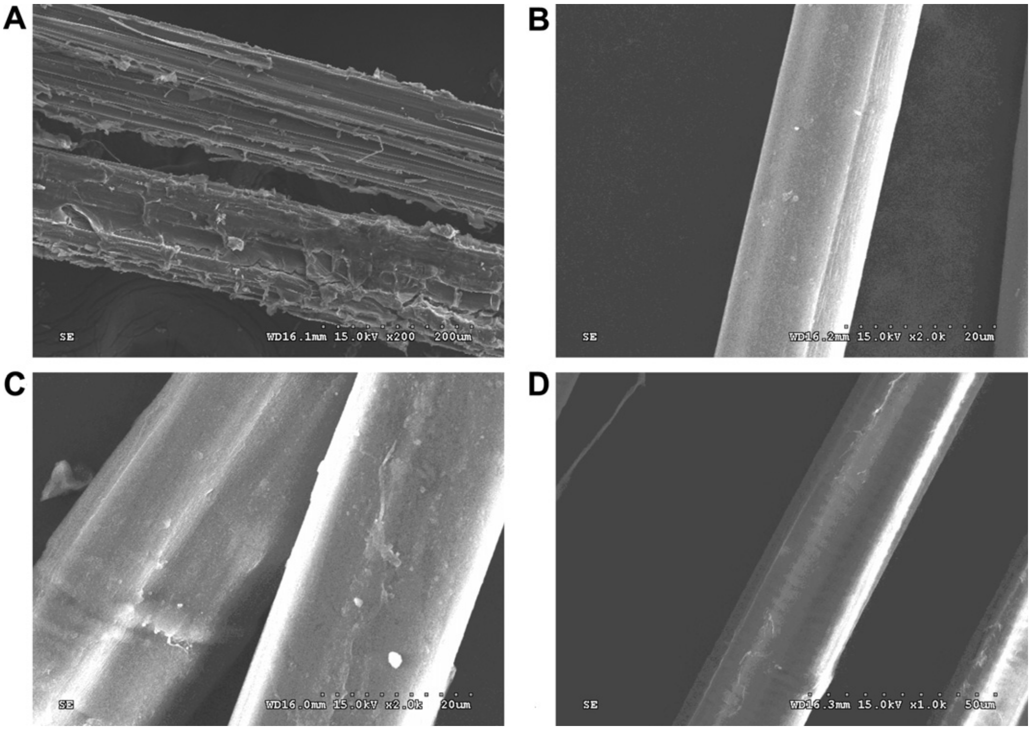

Fig. 4. SEM micrographs of the four *A. venetum* samples: (A) The bast of *A. venetum*; (B) Fiber-M; (C) Fiber-H; (D) Fiber-B.

Table 1
The mechanical properties of the treated *A. venetum* fibers as well as those of cotton and ramie

	Ramie	Fiber-M	Fiber-H	Fiber-B	Cotton
Modulus (cN/dtex)	405.5	318.9	383.7	394.5	96.6
Elongation (%)	3.22	4.06	3.58	3.26	10.69
Work of rupture (uJ/dtex)	26.47	12.36	13.72	11.82	16.77

4. Conclusion

Natural *A. venetum* cellulose fibers were obtained from different degumming methods and were characterized by various techniques. The experimental results showed that the Fiber-B, Fiber-M and Fiber-H as well as the Bast of *A. venetum* have the typical cellulose I structure and the treated *A. venetum* fibers have the structure and properties suitable for direct textile and other applications due to the well-defined fiber structure and the successful removal of the non-cellulose substances. Furthermore, the research results also disclosed that the bacterial degumming method has superior features for industrial applications than those of chemical degumming methods due to the high efficiency, low cost and environmental-benign nature. These results can give better understanding of the effect of different pretreatment methods and help conduct mass production of the *A. venetum* fibers.

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