Utilization of deep eutectic solvent as a degumming protocol for *Apocynum venetum* bast

Yan Song · Nie Kai · Wei Jiang · Yuanming Zhang · Haoxi Ben · Guangting Han · Arthur J. Ragauskas

Abstract Deep eutectic solvent (DES) pretreatment coupled with alkaline degumming on *Apocynum venetum* bast was studied for developing an eco-friendly and effective degumming method. A 1:2 mixture of choline chloride and urea was used in a microwave-assisted DES pretreatment optimized to 60 min and 110 °C. The impact of the deep eutectic solvent pretreatment on the properties of produced semi-degummed and refined dry fibers were assessed by examining the fiber chemical composition, surface microstructure, cellulose crystallinity, degree of polymerization, and fiber thermal properties. The combined DES and alkaline degumming treatment effectively removed the gummy matter including hemicellulose and lignin from the *A. venetum* bast, providing a smooth and clean fiber surface. This combined DES and alkaline degumming treatment also shows the ability to extract amorphous cellulose, leading to an increase of fiber crystallinity and a decrease of the average cellulose molecular weight. The thermal stability of the fibers was improved significantly after the degumming treatment. The refined dry fibers produced by this combined DES-alkaline treatment exhibited comparable chemical and physical properties (e.g. residual gum content, fiber length, fiber fineness, breaking tenacity) compared with traditionally alkali degumming method. These results revealed that DES is a practical and feasible pretreatment protocol for *A. venetum* bast degumming.

Keywords *Apocynum venetum* · Fiber · Deep eutectic solution · Alkaline · Degumming · Lignin

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Introduction

The textile industry has been shifting away from synthetic chemical fibers to natural cellulosic fibers because of environmental concerns and the depletion of the petroleum resource reserves (Wang et al. 2007). Natural cellulosic fibers including cotton, ramie, and linen offer several advantages including renewability, biocompatibility, and environmental friendliness (Dong et al. 2014; Liu et al. 2018). The consumer demand for these conventional natural cellulosic fibers keeps increasing, thus research and industrial interests for alternative natural fibers such as *Apocynum*

venetum, kenaf or hemp fibers keep growing (Wang et al. 2007; Ramesh 2016).

The growing area of *A. venetum* amounts to 1,330,000 ha with an average yield of 5–6 t/ha in China according to a published report (Liu et al. 2018). This fiber is attracting increased textile interest as it exhibits an attractive aspect ratio of 1200 and antibacterial properties (Li et al. 2012; Liu et al. 2018), which provides promising advantages for textile fiber production. *Apocynum venetum* bast needs to be degummed to remove non-cellulosic components such as hemicellulose and lignin prior to high value textile fiber processing (Hun and Obendorf 2016). Furthermore, compared to the conventional natural fibers including ramie, flax and hemp etc., *A. venetum* bast contains more hemicellulose and lignin proportion that needs to be removed.

Several degumming or pre-degumming techniques have been extensively investigated and widely applied in the last few decades, which can be classified as physical, chemical, and biological methods. Physical degumming methods such as mechanic, ultrasound, and steam explosion are typically inefficient at removing non-cellulosic materials from bast (Rabemanolontsoa and Saka 2016). Biological methods including bacterial and enzymatic methods are often uneconomical (Hutterer et al. 2017). Among all the available degumming processes, chemical treatments including alkali, acid, and oxidizing agents appear to be the most efficient and economical means to produce cellulosic natural fibers. However, the traditional chemical degumming method requires two boiling stages employing a large amount of chemical reagents which causes environmental concerns (Zhang et al. 2016). Thus, more environmentally friendly chemical degumming technologies need to be developed.

Over the last decade, research work into ionic liquids (ILs) have illustrated their promising properties as a green pretreatment for bioresources and their application as a degumming agent (Yang et al. 2015). Despite these promising aspects, the cost of most ILs and detrimental changes caused by ILs in cellulose have limited the use of ILs as a viable degumming agent (Yu et al. 2019). The use of deep eutectic solvents (DES) which share many physicochemical properties with ILs and they have been classified as a new generation of ionic liquids (Satlewal et al. 2018). DES is defined as a homogeneous mixture consisting of a hydrogen bond donor (HBD) and hydrogen bond acceptor (HBA) forming a hydrogen bond network with a lowered melting point (Abbott et al. 2004). In addition, DES offers several benefits including lower reagent costs in comparison to most ILs, convenient recycling processes, and non or limited toxicity (Lim et al. 2019). What's more, the components of DES such as urea, choline chroride, lactic acid etc. are mostly bio-degradable and environment-friendly (Abbott et al. 2004). The recyclable and reusable performance made DES greener to the environment. And it has been reported that a typically DES cost is only approximately 20% of an IL (Loow et al. 2017). Equally important, it was suggested that the primary effect of DES in biomass pretreatment lies in the ability to extract lignin while the cellulosic structure remains unaltered (Loow et al. 2017; Chen et al. 2018), which is favorable for a practical degumming method. Published applications of DES in biomass process includes pretreatment, delignification, sugar recovery, and biodiesel production (Loow et al. 2017). The application of DES in degumming is very limited in comparison with the development of DES in other areas. Since the DES was shown to be capable of extracting lignin and hemicellulose while protecting cellulose, it is clearly an attractive pretreatment solvent to degum *A. venetum* bast (Lynam et al. 2017). The ability of DES degumming is supposed to result from the action of breaking hydrogen bonds of lignin and hemicellulose. To examine the degumming capacity of DES, the most common DES composed of choline chloride and urea with the assistance of microwave was selected for this study. Given the limited literatures on DES degumming, the effect of microwave operating time, duration time, and reagent molar ratio on the microwave-aided DES treatment was investigated initially. Then, the fiber chemical composition, Fourier Transform infrared (FTIR) spectra, X-ray diffraction (XRD) spectra, Gel Permeation Chromatography (GPC) curves, Thermogravimetric (TG) analysis, Scanning Electron Microscope (SEM) morphology were characterized to evaluate the degumming capacity of DES pretreatment. Finally, the refined dry fibers produced by combined DES-alkaline treatment and traditional alkali treatment were compared for basic chemical and physical textile fiber properties.

Experimental methods

Materials

Raw *A. venetum* bast was cultivated from the Aksu Prefecture of Xinjiang, China. The original bast samples were obtained after being mechanically de-shelled. All chemicals purchased were of reagent grade except the spectrum pure KBr and all of them were used as received without further purification.

Degumming of *Apocynum venetum* fibers

The raw *A. venetum* bast samples were stored at ambient conditions (*i.e.*, 20–30 °C and \ 50% humidity) for 2 weeks before processed.

Apocynum venetum fibers degummed by combined DES-alkaline treatment

DES pre-degumming treatment

At first, choline chloride and carbamide were mixed and heated at 80 °C (oil bath) for 1 h to form a transparent solution. Then the raw *A. venetum* bast samples, 10 cm length, were immersed in the DES solution and treated with a MARS-6 laboratory microwave oven (CEM, USA, 1000 W) equipped with 50 mL sealed Teflon vessels. All the DES treatment experiments had a liquid-to-solid ratio (LSR) of 20:1 (v/w), the reagents molar ratio, the microwave operating temperature and duration time are summarized in Table 1 for the process optimization with a single factor experimental method. The influence of microwave retention time, operating temperature and reagent molar ratio were investigated and optimized step by step. The variable parameters were determined for different levels at every time while keeping the other factors constant. Moreover, the residual gum content was chosen to evaluate the degumming ability of DES treatment.

Alkaline degumming treatment

After the optimized DES pretreatment, the semi-degummed fibers were washed and then cooked with 1% NaOH solution (w/w) for 1 h. Then the refined *A. venetum* fibers were washed thoroughly and oven-dried (GZX-9240 MBE) for evaluation. Figure 1 below illustrates the experimental design of this study. All the degumming experiments were operated for three times and the results given are averaged.

Apocynum venetum fibers degummed by traditional alkali treatment

As described in the former literature, raw *A. venetum* bast samples, 10 cm length, were refluxed twice to degum the bast fibers (Liu et al. 2018). First, the samples were refluxed in 1% NaOH solution (w/w) for 1 h and then washed with deionized water. Next, the washed samples were transferred into a refluxing solution of 1% NaOH, 3% H_2O_2 (30%), 3% $NaSiO_3$, 2% Na_2SO_4, 3% CN_2H_4O and 3% $(NaPO_3)_n$ for 1 h. Both treatments employed a liquid-to-solid ratio (LSR) of 20:1 (v/w). After cooking, the fibers were washed repeatedly until the pH of effluent was neutral. Then the traditionally degummed *A. venetum* fibers were oven dried and stored for further analysis.

Evaluation of *Apocynum venetum* fibers

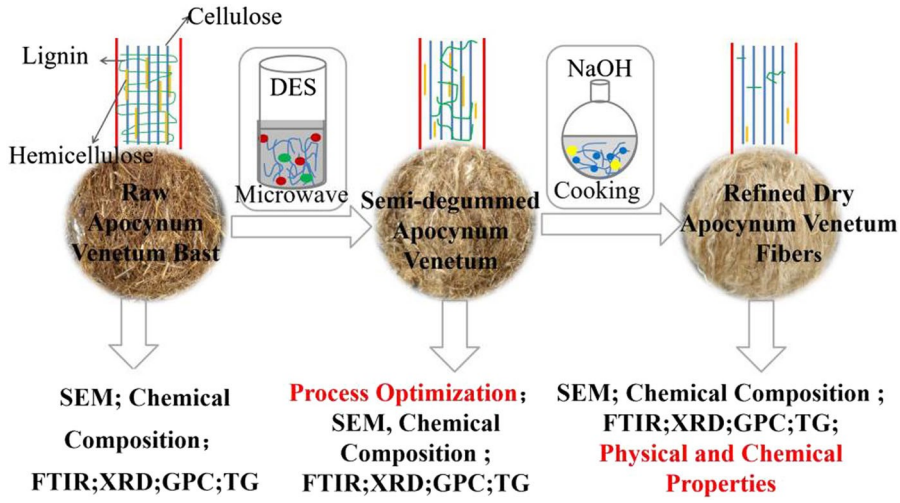
Chemical composition analysis

Chemical composition analysis of the fibers was determined based on the Chinese National Standard (GB 5889-1986) and the American Laboratory Analytical Procedure (NREL/TP-510-42618), which was reported in detail in the literature (Song et al. 2018).

Table 1 DES process parameter optimization experiment design

Parameters/factor level	1	2	3	4	5
Time (min)	20	40	60	80	1
Temperature (°C)	80	90	100	110	1
Molar ratio [choline chloride: urea (w/w)]	1:1	1:1.5	1:2	1:2.5	1

Fig. 1 Schematic diagram for the experiment



The water-soluble material, pectin, lignin, hemicellulose and cellulose content of the raw *A. venetum* bast, semi-degummed fibers, and refined dry fibers were determined following the standard procedures.

FTIR of Apocynum venetum fiber

The FTIR spectra of the *A. venetum* fiber samples were acquired with a Bruker spectrophotometer NICOLET 5700 (Bruker, Germany). The samples were milled to 40 mesh (1 mg), mixed with KBr (100 mg) and pressed into discs. The spectral data was acquired over the wavelength range of 500–4000 cm^{-1} with a resolution of 2 cm^{-1} for 32 scans per spectrum.

Surface morphology of Apocynum venetum fiber

The fiber surface morphology was observed with a scanning electron microscope (JSM-840). The samples were sprayed with platinum and photographed under an accelerating voltage of 15 kV and magnification of 500.

XRD of Apocynum venetum fiber

XRD spectra were measured with a D8 Advanced X-ray diffractometer (Bruker, Germany). The spectra were collected in a 2h range of 5° to 40° at 40 kV and 50 mA, with a scanning rate of 2°/min. The crystalline index (CrI) of *A. venetum* fiber was calculated according to the Segal method equation previously reported for native cellulose (Song et al. 2018). The equation was listed as follows:

$$CrI = \frac{I_{200} - I_{amorph}}{I_{200}} \times 100$$

where I_{200} represents the maximum peak intensity of the 200 cellulose crystalline lattice diffraction (around 22.6° for native cellulose). While, I_{amorph} is the intensity taken at 2h = 18°, associating the amorphous area in native cellulose.

Molecular weight analysis of cellulose

Cellulose from the fiber samples was extracted according to the chemical composition analysis procedure (Song et al. 2018). Ethylenediamine was employed to activate the dried cellulose. After activation, the dried cellulose was dissolved in a mixture of polyacetyl diamine (DMAc) and lithium chloride (LiCl) for 48 h to obtain a cellulose solution. The solution was filtered through a 0.22 lm filter membrane before the gel permeation chromatography (GPC) analysis. The solution volume was 100 lL and injected in the GPC at a flow rate of 0.7 mL/min at 50 °C of Viscotek TDA305max (Malvern, UK). Standard polystyrene from Malvern was used to calibrate the GPC curves with a mobile phase of DMAc ? 0.5%LiCl.

Thermal stability characterization

Thermogravimetric analysis of the fiber samples was conducted on Pyris TGA, (PerkinElmer, USA). Before the TGA test, the samples were cut and milled to 40 mesh and stored at room temperature for 24 h. TGA analysis was conducted using approximate 10 mg samples. The analysis temperature was conducted from room temperature to 600 °C under nitrogen atmosphere. The temperature was increased up with a constant heating rate of 20 °C/min.

Physicochemical properties of refined dry Apocynum venetum fiber

The residual gum content, fiber fineness, breaking tenacity and fiber length are four of the most important indexes for textile fibers. These fiber indexes influence yarn quality directly. Prior to testing, each specimen was conditioned in the constant temperature and humidity environment (23 ± 2 °C and 50 ± 5% RH) for 2 days. Also, they were measured according to the Chinese National Standard GB 5889-86, GB 5884-86, GB 5886-86, GB 5887-86, respectively.

Results and discussion

Parameter optimization of DES pre-degumming treatment analysis

The temperature, reaction time and DES molar ratio are important parameters for DES degumming capacity and were optimized for their degumming ability to release the burden of subsequent degumming procedure. Figure 2 summarizes the results of the parameter optimization studies. The residual gum content of the raw *A. venetum* bast was calculated to be 45.6%.

The effect of microwave retention time on residual gum content was conducted at 90 °C with a 1:2 (choline chloride: urea) molar ratio. As is shown in Fig. 2a, the residual gum content suffered a dramatic decrease with an increase of duration time from 20 to 60 min and then underwent an increase with further prolonged operation time. The minimum residual gum content of the fibers under 60 min retention time was only half of the raw *A. venetum* bast samples. Consequently, 60 min was chosen as the optimal microwave treating time for these studies. The extraction of residual gum was attributed to the hydrolysis of reactive chemical linkages including lignin-carbohydrate ester linkages and hemicellulose linkages as well as the dissolution of gummy matters into the DES solution (Lim et al. 2019). However, after the removal of gummy material, cellulose was exposed and then reacted with the DES solution. The microwave-assisted DES may lead to subtle solubility of amorphous cellulose, leading to an increase of the relative amount of gummy matters. For example, amorphous cellulose in rice straw was shown to dissolve in choline chloride/urea (Parviainen et al. 2015).

The effect of microwave temperature on residual gum content was conducted for a 60 min retention time and 1:2 (choline chloride: urea) molar ratio. Figure 2b exhibits that the residual gum content was reduced to 14% (around 1/3 of the original sample) and then hit a plateau before increasing incrementally with reaction temperature. The reason may be

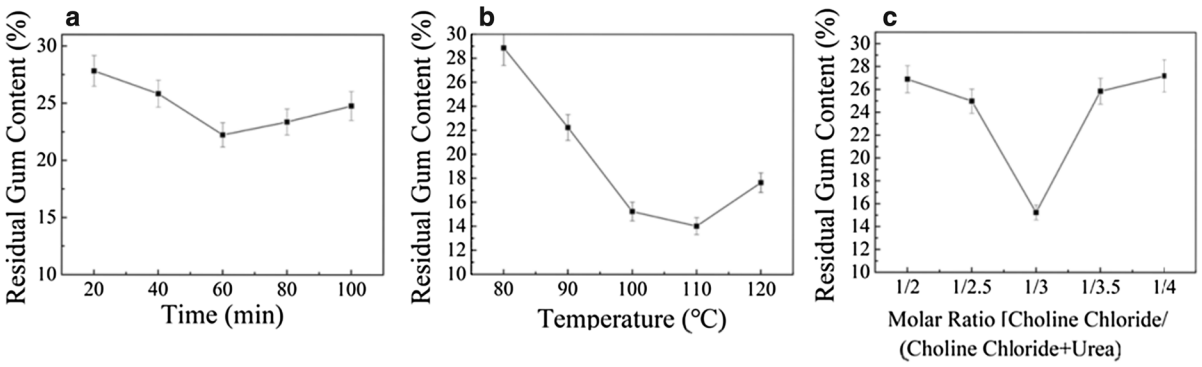


Fig. 2 Effect of time (a), temperature (b) and molar ratio (c) on residual gum content

attributed to the reduction of viscosity of DES. Shahbaz et al. (2011) discovered that an increased temperature would lower the DES density because of the accelerated motion of DES particles. The higher temperature contributed to the better flowability of DES solution. Thus, the DES would penetrate into the samples and then degrade or dissolve the gummy matters more readily. While too high a temperature may reduce the cellulose yield. The operating temperature of 110 °C was chosen in the following experiment.

Under the operating temperature of 110 °C and a retention time of 60 min, Fig. 2c showed that the residual gum content was lowest under the molar ratio of 1:2 for choline chloride and urea. The residual gum content increased marginally with a lower or higher molar ratio, which is attributed to the fact that hydrogen bonding interactions were strongest under a 1:2 of choline chloride and urea, also, the preferred viscosity of the solution under the molar ratio of 1:2 contributed to the reaction (Sirviö et al. 2019). Based on the optimization experiments, an operating temperature of 110 °C, 60 min retention time and a 1:2 molar ratio for choline chloride and urea were selected as ultimately fixed parameters for the evaluation experiment.

Chemical composition analysis and FTIR analysis

Figure 3 summarizes the chemical composition of the raw *A. venetum* samples, semi-degummed fibers and refined dry fibers. Cellulose, lignin, and hemicellulose are the three primary components of *A. venetum* bast, which accounts for 92.94% of the original bast samples.

It is clear from Fig. 3 that non-cellulosic materials including pectin, hemicellulose, and lignin were partially removed with the DES pretreatment and further extracted with alkaline treatment. The water-soluble matter and pectin was almost eliminated in the final refined dry fibers. Hemicellulose was reduced from 14.14 to 11.21% for the DES treatment and lowered further to 3.12% for the subsequent alkaline treatment. This trend was attributed to the fact that the hemicellulose was not degraded sufficiently for the DES treatment, but the DES helped its removal in the subsequent alkaline treatment. Therefore, the combined DES-alkaline degumming worked together to remove hemicellulose.

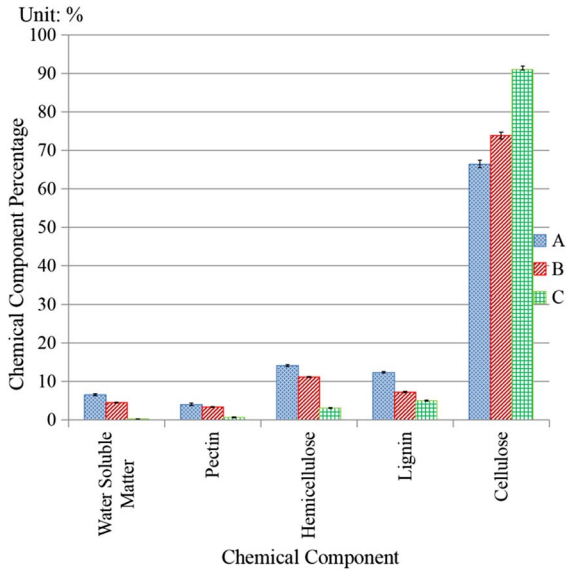


Fig. 3 Chemical composition of the *A. venetum* samples. A: raw *A. venetum* bast, B: semi-degummed *A. venetum* fibers treated with DES, C: refined dry *A. venetum* fibers treated with combined DES-alkaline treatment

When it comes to lignin, it is known that lignin is complex and hard to remove from the bast. However, the results proved the fact that the microwave aided DES had advantages in delignification. The mechanism is devoted that during the process, the rupture of ester and ether bonds in lignin, extraction of lignin phenolic compounds as well as partial hydrolysis of a-ether bonds in lignin caused the lignin to be fragmented and became more accessible and dissolved in DES (Chen et al. 2019). Many researchers have confirmed the delignification capacity of DES in other bioresources and found that it resulted in the disruption of inter- and intra- hydrogen bonds in lignocellulosic biomass, leading to the subsequent breakdown of chemical linkages (Loow et al. 2017). With the removal of the non-cellulose materials, the relative cellulose percentage was increased to over 90% for the resultant refined dry fibers, which was close to the ramie fibers (Fan et al. 2010) and flax fibers (Sharma et al. 2011).

The FTIR spectra were recorded to analyze the changes in chemical structure further and are summarized in Fig. 4. The band at 3400 cm^{-1} was attributed to polysaccharide/lignin hydroxyl groups, and the band at 896 cm^{-1} confirmed the presence of cellulose b-1,4 glucosidic linkages. While, the bands at

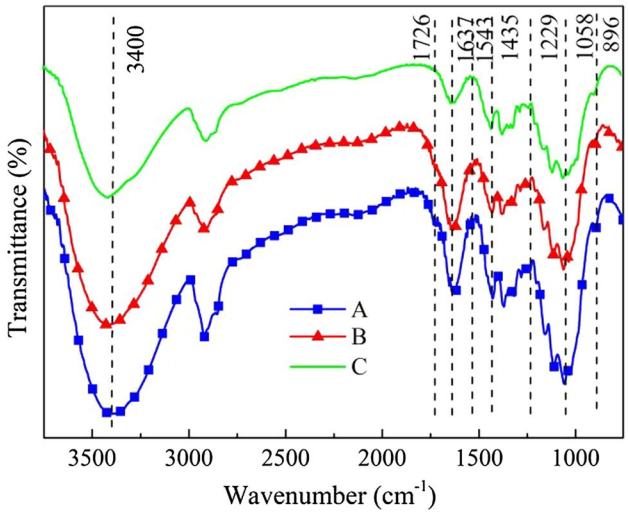


Fig. 4 FTIR spectra of the *A. venetum* samples. A: raw *A. venetum* bast, B: semi-degummed *A. venetum* fibers treated with DES, C: refined dry *A. venetum* fibers treated with combined DES-alkaline treatment

1726 cm^{-1} and 1435 cm^{-1} were assigned to carboxylic acid and an ester bond in hemicellulose (Liew et al. 2018). The hemicellulose characteristic peaks were weakened and even vanished after degumming treatment, revealing hemicellulose removal during treatment. Lignin characteristic absorption bands at 1637 cm^{-1}, 1543 cm^{-1} and 1058 cm^{-1} were all reduced in intensity, indicating the loss of lignin content during the degumming process (Li et al. 2012). It can also be observed the signal at 1229 cm^{-1} correlated with pectin which was lowered in intensity after DES pretreatment and became a trace signal after a subsequent alkaline treatment (Song et al. 2017). These results were in agreement with the above chemical composition analysis. Also, the same results were found by Yu et al. (2019). By comparing the infrared spectra, it was suggestive that a DES pretreatment has excellent degumming potential, especially when combined with a subsequent alkali degumming.

SEM analysis

The morphology of treated cellulose fibers was investigated by SEM to gain greater insight into the surface structure, as presented in Fig. 5. It is evident that the surface morphology was significantly changed after degumming treatment. The raw *A. venetum* bast (Fig. 5a) showed a rough surface with many gummy matters that cemented the single fibers together. The gummy matters are largely removed after DES treatment, the SEM images clearly illustrated loosen bundle fibers which was attributed to the removal of lignin and hemicellulose. Furthermore, the surface changes also led to more effective contact with the alkali solution, resulting in an overall benefit to the degumming process. Consequently, the fiber bundles were separated further by the following alkaline treatment. The single fibers exhibited a smooth and clean surface. Moreover, the width of *A. venetum* fiber is approximately 10 lm, which is similar to the results published by Liu et al. (2018). The phenomenon is consistent with the chemical composition analysis and also other researchers (Kallel et al. 2016).

XRD analysis

The X-ray diffraction patterns of the original *A. venetum* bast, semi-degummed fibers, and refined dry fibers are presented in Fig. 6. The spectra with the typical (1–10), (110), (200), as well as (004) lattice of cellulose I were observed, revealing that both the DES and alkaline treatment kept the original cellulose I crystal structure (French 2013). As known from the chemical component analysis, the non-cellulosic components such as hemicellulose and lignin were extracted by DES and further removed by alkaline treatment. Thus, the peak intensity of the 200 lattice planes increased after degumming treatment, indicating an increase of crystallinity. The dissolution of the amorphous cellulose and removal of some hemicelluloses presumably contribute to this increase crystallinity (Loow et al. 2017). Based on the calculation method of Segal, the crystallinity index of the samples

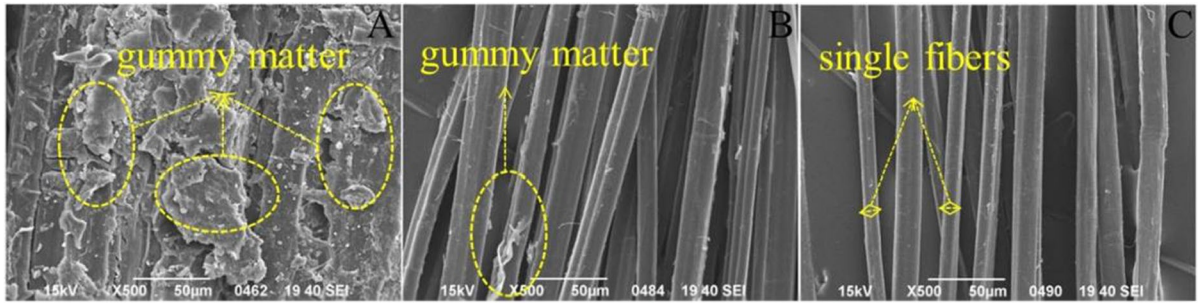


Fig. 5 SEM pictures of the *A. venetum* samples. a raw *A. venetum* bast, b semi-degummed *A. venetum* fibers treated with DES, c refined dry *A. venetum* fibers treated with combined DES-alkaline treatment

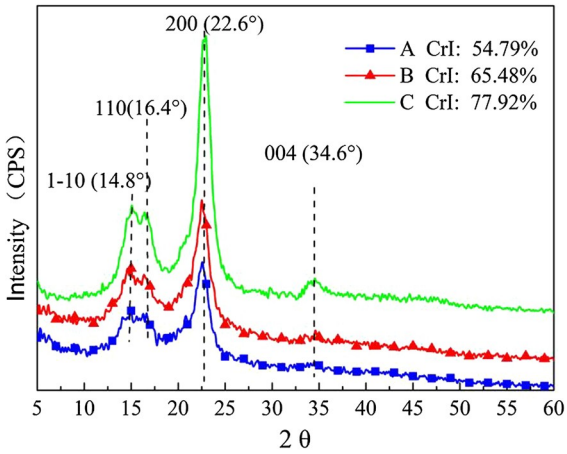


Fig. 6 XRD patterns of the *A. venetum* samples. A: raw *A. venetum* bast, B: semi-degummed *A. venetum* fibers treated with DES, C: refined dry *A. venetum* fibers treated with combined DES-alkaline treatment

increased from 54.79% (original *A. venetum* bast) to 65.48% (semi-degummed fibers) and 77.92% (refined dry fibers), respectively. The crystallinity trends were similar to the DES, and alkali degumming treatment reported previously (Dong et al. 2014; Yu et al. 2019). However, the increasing range of crystallinity was lower than that of the cellulose component. Also, when compared with our former research, the crystallinity of the refined dry fibers was not as high as reported (Wang et al. 2007). This was attributed to the fact that DES treatment made it easier to remove cellulose chains on crystallites in the subsequent alkaline treatment. Moreover, Vigier et al. (2015) claimed that DES resulted in a reduction in cellulose crystallinity when used to pretreat biomass.

GPC analysis

A series of *A. venetum* samples were subjected to a cellulose extraction procedure to obtain greater insight into possible degradation of the cellulose chains during treatment. The molecular weight distribution curves for the cellulose samples are shown in Fig. 7. The location of the primary cellulose peak had shifted towards lower weights, indicating partial degradation of the cellulose chains. The weight-average molecular weight for the fiber cellulose was reduced from 47 to 42 kg/mol after DES treatment. It was previously reported in the literature that cellulose was partially soluble in the DES treatment (Loow et al. 2017). Moreover, the microwave irradiation treatment could contribute to its solubility. Thus, the relative cellulose molecular weight was reduced a bit compared to the

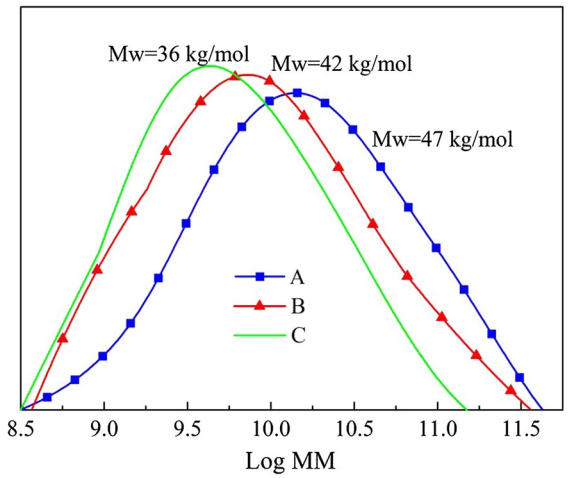


Fig. 7 GPC spectra of the *A. venetum* samples. A: raw *A. venetum* bast, B: semi-degummed *A. venetum* fibers treated with DES, C: refined dry *A. venetum* fibers treated with combined DES-alkaline treatment

original cellulose. After alkaline treatment, the cellulose peak had shifted even farther to a lower value of 36 kg/mol. This was attributed to alkaline-induced depolymerization of cellulose (*i.e.*, peeling reactions) which has also been observed for the alkaline treatment (Song et al. 2018). These results confirmed the partial damage of cellulose during degumming treatment and also explained the decrease of fiber crystallinity. While the final molecular weight of the refined dry fiber was still in the same order of magnitude with the original bast samples, hence it may have little effect on fiber strength properties.

TG analysis

The TG analysis of the original raw *A. venetum* bast, semi-degummed fibers, and refined dry fibers was carried out to determine the thermal fiber degradation characteristics which are shown in Fig. 8. As seen from the TG (Fig. 8a) and Differential thermal gravity (DTG, Fig. 8b) curves, two degradation peaks at 77 °C and 325–365 °C were attributed to the evaporation of water and cellulose degradation, respectively (Yu et al. 2019). The degradation temperature increased for the semi-degummed *A. venetum* fibers (365 °C) treated with DES compared to the raw *A. venetum* bast (325 °C), which was attributed to the removal of non-cellulosic materials. While the slight degradation temperature decrease of refined dry *A. venetum* fibers treated with combined DES-alkaline treatment (358 °C) compared with semi-degummed fibers corresponded to the partial decomposition of amorphous cellulose during the alkaline treatment. The subtle decrease in thermal stability will not affect the though quality of textile fibers. Similar results have been reported in cellulose degummed fibers by Zhou et al. (2012).

Chemical and physical properties analysis

Fibers obtained from the combined DES-alkaline treatment and traditional two-stage alkali treatment were compared and presented in Fig. 9. The refined dry fibers of combined DES-alkaline treated fibers showed slightly higher residual gum content (6.54% and 4.49%, respectively). However, a slightly higher residual gum content may not affect the textile fiber quality but can contribute to the production yield. The residual gum corresponded to longer fibers (3.15 cm compared with 2.01 cm), which was beneficial to the spinning process. Due to the increased residual gum content, the value of average fiber fineness was also a bit higher (4.05 dtex and 3.35 dtex, respectively). Furthermore, the breaking tenacity of fibers produced by combined DES-alkaline treatment was stronger than the conventionally twice-boiling alkaline fibers (14.14 cN/dtex compared with 11.21 cN/dtex). Based on the above data, in conclusion, the combined DES-alkaline treatment is effective in producing fibers with

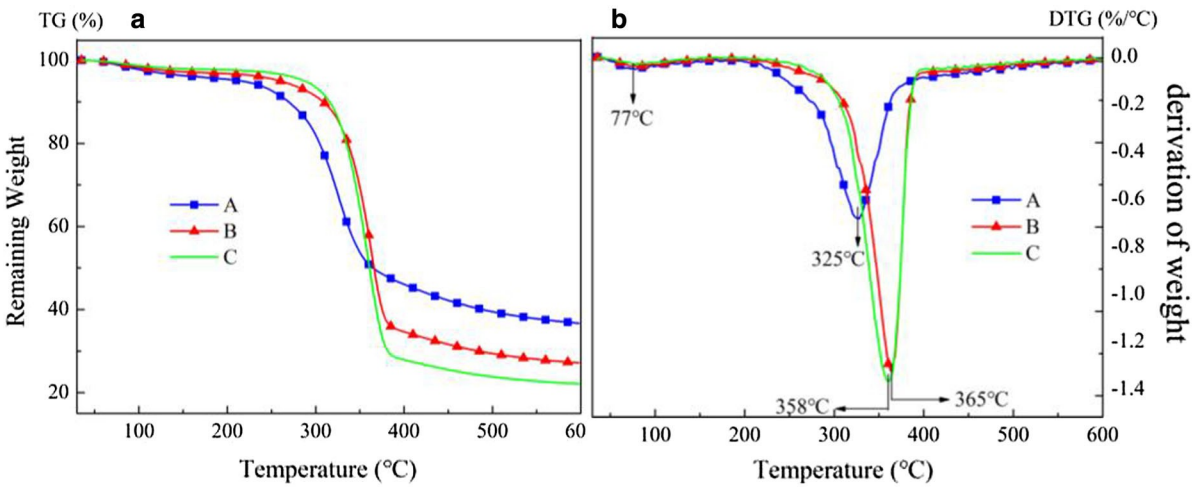


Fig. 8 TG (a) and DTG (b) patterns of the *A. venetum* samples. A: raw *A. venetum* bast, B:semi-degummed *A. venetum* fibers treated with DES, C: refined dry *A. venetum* fibers treated with combined DES-alkaline treatment

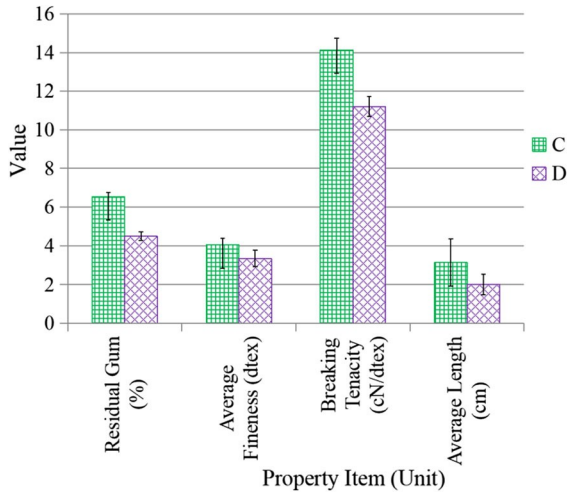


Fig. 9 Chemical and physical properties of the *A. venetum* samples. C: refined dry *A. venetum* fibers treated with combined DES-alkaline treatment; D: refined dry *A. venetum* fibers treated with traditionally twice-boiling alkaline treatment

qualities that are comparable to the traditional alkali degumming method.

It should be further noted that besides the environmental-friendly characteristics of DES components, researchers have claimed only a subtle loss of extraction strength in DES after being used for three pretreatment cycles using DES as a bioresource pretreatment (Hu et al. 2008). The reusable ability made DES degumming greener. Thus, the microwave assisted DES treatment is a promising green protocol for the degumming of natural bast fibers. However, the recycling and reuse of DES needs to be evaluated in future studies.

Conclusion

To develop a greener degumming method for bast-fibers, a two-stage DES, alkaline treatment was conducted to degum *A. venetum* bast. It was found that the DES pretreatment possesses a marked contribution to bast-fiber degumming. The chemical composition analysis revealed that the DES pretreatment effectively extracted hemicellulose, lignin and also contributed to the following alkaline treatments, which was further validated by FTIR. Meanwhile, the gummy matter on the surface of the *A. venetum* bast was found to be gradually extracted and revealed by SEM analysis. Consequently, fiber crystallinity and thermal stability increased after degumming treatment. Furthermore, cellulose degradation was observed by GPC and TG analysis. In conclusion, the combined DES-alkaline treatment produced fibers with qualities that are comparable to a conventional alkaline degumming method. These observations could be instrumental in improving degumming technologies. However, the recovery and reuse of DES waste liquid as well as the techno-economic analysis should be investigated in future studies.

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