

## TEMPO-media Oxidation Combined with Laccase for Effective Degumming Pretreatment of Hemp Fibers

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TEMPO-media oxidation and laccase have been successfully used in fiber degumming, but the combination of the two methods in the hemp degumming process has not yet been reported. In this paper, an alternative and efficient way for the pretreatment of hemp degumming was proposed as a combination of TEMPO-media oxidation with laccase and was a TEMPO-laccase system. A chemical composition analysis, Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), and scanning electron microscopy (SEM) were applied to systematically analyze and compare the degumming efficiency between the TEMPO-laccase system and other pretreatments. The results showed that after the TEMPO-laccase system degumming process, a clean and smooth surface of the degummed fibers could be observed and the non-cellulosic materials were greatly removed without any crystalline transformation. The hemp fibers after the TEMPO-laccase system degumming process had the most desirable linear density of 6.64 dtex and a tenacity value of 3.41 cN/dtex. This new pretreatment had a considerable effect on hemp degumming, which holds promise for use of high-value textiles.

*Keywords:* Hemp fiber; Degumming; TEMPO-media oxidation; Laccase

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### INTRODUCTION

Recently, fossil hydrocarbon materials have been gradually replaced by green fibers, such as flax, ramie, bamboo, and hemp due to their inferior hygienic and physiological properties and because they are uncomfortable to the touch. Hemp fibers have attracted increasing attention in textile industrial utilization (Mussig *et al.* 1998). This could be attributed to their excellent tensile strength, thermal conductivity, moisture absorption, and anti-bacterial function. It is well known that raw hemp consists of several elementary fibers held together (Bergfjord and Holst 2010) with up to 20% to 55% of noncellulosic gummy materials, where the main components are pectin, lignin, and hemicellulose, *etc.* (Kapoor *et al.* 2001; Kostic *et al.* 2008). These gummy components must be partly removed by the degumming process. Finer and softer hemp fibers can be gained after degumming for the garment application by removing the noncellulosic materials. However, part of the gummy materials should remain to meet the requirement for the spinning process, such that the hemp fibers could be used as technical fibers that consist of a small bunch of single fibers.

Various techniques for the pretreatment of hemp degumming have been explored, including the enzymatic (Liu 1999), mechanical, ultrasonic (Renouard *et al.* 2014), and chemical (Liu *et al.* 2013) methods. However, these techniques have limitations such as

the need for sophisticated conditions and time-consuming procedures for the enzymatic method, extra fiber damage in the mechanical method, and low degumming efficiency during the ultrasonic method (Riddlestone *et al.* 2006). The traditional degumming process used in the industry is a chemical method as follows: raw hemp, scutching, acid soaking, washing, alkali boiling, washing, dehydration, bleaching, oil finishing, and drying. This process leads to high environmental pollution and energy costs, which is not in accordance with the requirements of environment protection.

A new alternative degumming process has recently emerged called 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO)-mediated oxidation, which was pioneered by Isogai *et al.* (2011) and has been used for the fabrication of cellulose nanofibers. Puangsin *et al.* (2013) investigated TEMPO-mediated oxidation at various sodium hypochlorite (NaClO) addition levels, and it was revealed that most hemicelluloses originally present in the raw hemp fibers were degraded and removed from the solid oxidized products and this provided nearly pure TEMPO-oxidized celluloses with a yield of 98% to 100%. Within the research of Milanovic *et al.* (2012), TEMPO-mediated oxidation was very efficient in the removal of non-cellulosic substances with using NaClO, a catalytic amount of sodium bromide (NaBr), and TEMPO under certain conditions. Laccase, which is in a family of blue multi-copper oxidase produced by microorganisms and plants, is able to perform lignin degradation using a cocktail of oxidative enzymes. Numerous efforts have been exerted in the degumming process using laccase. Xia *et al.* (2008) investigated the effect of laccase in the linear density of jute fibers, obtaining the result that within 8 h there were no significant changes in linear density. Yan *et al.* (2009) studied the degumming efficiency of laccase-media systems in kenaf fibers and concluded that after 6 h of treatment, the lignin content of kenaf decreased greatly, along with certain improvements in mechanical properties.

Depending on the mode of action and arrangement of chemical components, laccase can be an effective method for improving the thermal and surface properties of hemp fibers (Dochia *et al.* 2013; Zhang and Yan 2013). These studies showed an excellent performance of the laccase in hemp degumming. Moreover, the TEMPO-laccase system has been proposed and is widely used in the paper industry (Aracri *et al.* 2011, 2012). However, the combination method of TEMPO-media oxidation and laccase has not been reported in the degumming process of hemp fibers.

In view of the possibility of TEMPO-mediated oxidation and laccase in removing the gummy component of the hemp fibers discussed above, the collaboration of TEMPO-mediated oxidation and laccase was used in this study as a type of pretreatment process for the removal of non-cellulosic materials, which is called a TEMPO-laccase system in this study. To evaluate its effect, three other pretreatment processes were introduced here: alkali, TEMPO-mediated oxidation, and laccase pretreatments. All of the hemp fibers after each of these pretreatments were subjected to an alkali boiling degumming process.

After each treatment, a chemical composition analysis was used to determine the content changes of different components. A scanning electron microscope (SEM) was used to observe the surface morphology. Fourier transform infrared spectroscopy (FT-IR) and X-ray diffraction (XRD) were employed to evaluate the microscopic changes in the intensity peaks of noncellulosic materials in the degummed hemp fibers obtained from the different degumming processes. Moreover, the mechanical properties were tested to assess the fibers' quality and its possibility in high-end use.

## EXPERIMENTAL

### Materials

The raw hemp fibers used in this study were originally from Heilongjiang province of China. The main chemicals used in this study were sodium hydroxide (NaOH), sodium hypochlorite (NaClO), sodium bromide (NaBr), sodium pyrophosphate (Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub>), anhydrous sodium sulfate (Na<sub>2</sub>SO<sub>3</sub>), and TEMPO. These chemicals were of analytical reagent grade without further purification and purchased from Aladdin Biological Technology Co., Ltd. (Shanghai, China). The laccase was purchased from Ruiyang Biotechnology Co., Ltd. (Jiangsu, China). The activity of laccase was measured by monitoring the oxidation rate of 2,2-azinobis-(3-ethylbenzyl thiozoline-6-sulfonate) (ABTS) (Zhang *et al.* 2016) and the laccase activity was tested to be 600 IU/L. Deionized water was used to prepare all solutions.

### Degumming processes

Prior to all treatments, raw hemp fibers were dried at 40 °C for 24 h in an oven to ensure constant humidity content. In the alkali pretreatment, raw hemp fibers were immersed in an agent that was a water solution that consisted of 10% NaOH and left in a water bath for 2 h at 70 °C. In the laccase pretreatment, raw hemp fibers were treated for 4 h at 45 °C in a solution of 12.8% of laccase. The pH was adjusted to be between 4.5 and 5.5 with acetic acid (CH<sub>3</sub>COOH) and sodium phosphate (NaH<sub>2</sub>PO<sub>4</sub>). To quench the reaction, the solution was boiled for 5 min. In the TEMPO-mediated oxidation pretreatment, raw hemp fibers were immersed in a solution of 0.25% of TEMPO and 2.5% of NaBr. The TEMPO-mediated oxidation was initiated by adding a 13% solution of NaClO solution (9.67 mmol/g). The pH value of the slurry was maintained at 10.5 and at room temperature by adding 0.5 M (mol/L) of NaOH solution until there was no further decrease in the pH observed for 4 h (Milanovic *et al.* 2012). In the TEMPO-laccase system, raw hemp fibers were initially treated following the instruction of TEMPO-mediated oxidation pretreatment. The hemp fibers obtained from the TEMPO-mediated oxidation pretreatment experienced a laccase pretreatment process after being washed with deionized water 3 times.

The hemp fibers after different pretreatments were subjected to a further alkali boiling treatment. Pre-degummed fibers were soaked in a water solution that consisted of 10% of NaOH, 3% of Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub>, and 7.5% of Na<sub>2</sub>SO<sub>3</sub> for 2 h at 100 °C. Then, all of the hemp fibers were washed with deionized water 3 times after every degumming process and dried at 40 °C for 24 h in an oven. The degumming process routes are listed in Table 1. A number followed by ‘#’ is in regards to the type of the degummed fibers obtained from different process routes.

**Table 1.** Details of Process Routes used for Degumming Raw Hemp Fibers

Degummed Fibers	Process Route
#1	Alkali Pretreatment to Alkali Boiling Treatment
#2	TEMPO-mediated Oxidation Pretreatment to Alkali Boiling Treatment
#3	Laccase Pretreatment to Alkali Boiling Treatment
#4	TEMPO-laccase system to Alkali Boiling Treatment

## Methods

### *Weight loss ratio*

The weight loss ratio was introduced to evaluate the degumming efficiency after different degumming processes. The weight loss ratio can be calculated by using Eq. 1,

$$\text{Weight loss ratio (\%)} = \frac{w_0 - w}{w_0} \times 100 \quad (1)$$

where  $w$  (g) is the dry weight of the hemp fibers after all of the treatments and  $w_0$  (g) is the dry weight of the raw hemp fibers before each treatment.

### *Chemical composition*

The main chemical composition of hemp fibers was tested according to the Chinese standard GB/T 5889-86 (1986) by averaging the experimental results of the 6 specimens.

Five g raw hemp fibers for each sample was dried in the oven. Then, the dried fibers were processed under boiling alkali solution (150 mL, 20 g/L) for 1 h and re-boiled for 2 h with the new alkali solution. After the boiling treatment and the washing process, the fibers were dried to constant weight. The cellulose content of hemp fibers was calculated as follows.

$$\text{cellulose content (\%)} = 1 - \frac{G_0 - G}{G_0} \times 100 \quad (2)$$

where  $G_0$  is the dry weight of raw hemp sample and  $G$  is the dry weight of the sample after treatment.

To remove the resins, oils, fats, and waxes, raw hemp fibers were firstly degreased with alcohol-benzene. Then 1 g of degreased raw hemp fibers for each sample was placed in weighing bottles and dried at 105 °C for 3 h in the oven. The oven-dried specimens were weighed and transferred to a 50 mL beaker with a glass cover, followed by slow addition of 30 mL concentrated H<sub>2</sub>SO<sub>4</sub> (72%). The specimen was mixed well with the acid by constantly stirring for at least 1 min and allowed to stand for 24 h. The materials were transferred into a 500 mL beaker, 270 mL of deionized water were added, and the mixture was boiled for 1 h. After allowing the insoluble material to settle, it was filtered into a weighted sand core funnel that had been dried. The residue was washed free of acid with hot water, then the sand core funnel with the lignin was dried at 105 °C for 3 h, and weighed. The lignin content of raw hemp fibers was calculated as follows,

$$\text{lignin content (\%)} = \frac{G_L - G_F}{G_D} \times 100 \quad (3)$$

where  $G_D$  is the dry weight of degreased raw hemp fibers,  $G_L$  is the dry weight of sand core funnel with lignin, and  $G_F$  is the dry weight of empty sand core funnel.

### *Scanning electron microscopy*

The SEM measurements of the degummed fibers were performed using an SEM (JSM-5600LV, JEOL Ltd., Tokyo, Japan) at 10 kV after gold coating (Yixin Technology, Beijing, China) under a temperature of 20 °C and a relative humidity (RH) of 65%.

### *Fourier transform infrared spectroscopy*

The FT-IR spectra was examined by a PerkinElmer spectrometer (Spectrum Two, Buckinghamshire, UK). A total of 30 scans were taken over a range from 4000 cm<sup>-1</sup> to 400

$\text{cm}^{-1}$  with a resolution of  $4 \text{ cm}^{-1}$ . The baseline correction and smoothing were accomplished prior to further analysis.

#### *X-ray diffraction*

The XRD analysis was completed by using a Rigaku diffractometer (D/max- 2550 PC, Tokyo, Japan) equipped with a Cu tube of 40 kV and 200 mA. The patterns were recorded in a  $2\theta$  range of  $5^\circ$  to  $60^\circ$ .

#### *Degree of polymerization*

The degree of polymerization (DP) of degummed hemp fiber was determined in accordance with the Chinese standard GB/T 5888-86 (1986) and by using an Ubbelohde capillary viscometer (Zvisco, Hangzhou, China), which measured the relative viscosity of the hemp fibers solution in the copper ethylene-dimined solvent at first and then calculated the intrinsic viscosity by the specific coefficient method. Before the tests, all of the samples were degreased using a Soxhlet extractor (Xiyang, Shanghai, China) with a benzene and ethyl alcohol mixture in a 2:1 (v/v) ratio as the solution.

#### *Mechanical treatment and properties tests*

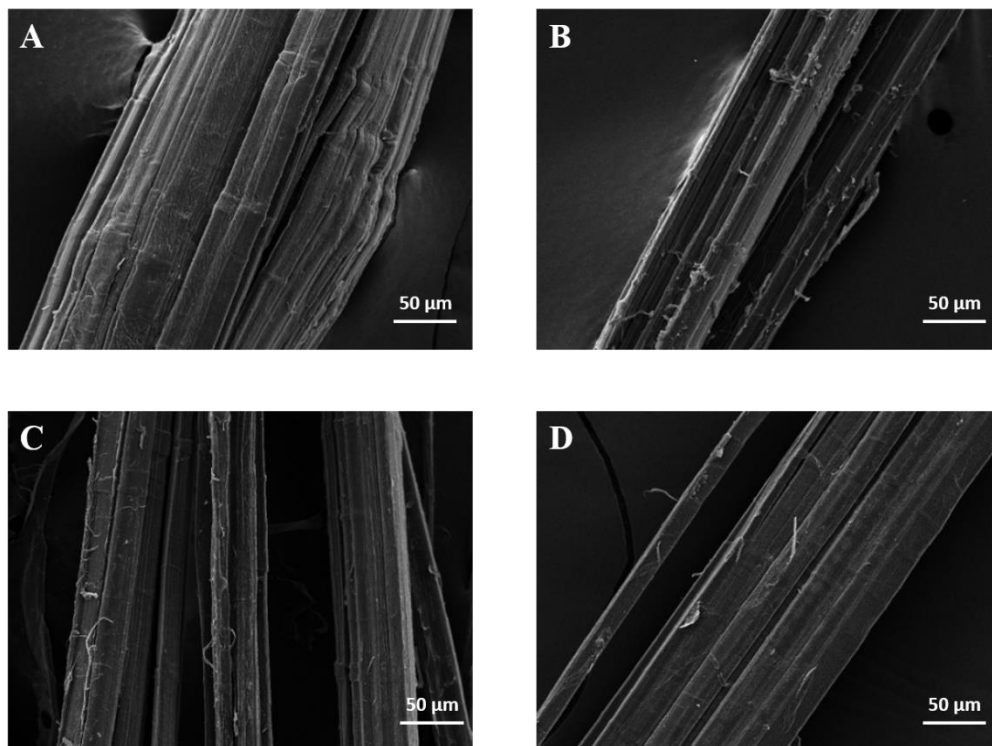
Before starting the mechanical properties tests, the degummed hemp fibers were firstly treated by a carding process, which was essential in the spinning process. Their primary function was to loosen the degummed fiber and convert them into uniform, straight, parallel, and separate fibers in the yarn (Harwood *et al.* 2008). The carding process was performed on FA210B Flat Card (Hongda, Qingdao, China) with the speed of the cylinder, stripper, and doffer of 330 r/min, 720 r/min, and 20 r/min, respectively. Before the mechanical properties tests, all of the samples were balanced in standard atmospheric condition with a temperature of  $20 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$  and relative humidity of  $65\% \pm 3\%$  for 24 h. The linear density, tenacity, and elongation at break were tested in accordance to the Chinese standards GB/T 18147.4 (2000) and GB/T 18147.5 (2000). The average values were obtained using results from the 30 specimens.

## RESULTS AND DISCUSSION

### Surface Morphologies Analysis

The surface morphologies of the degummed fibers that used different treatments are shown in Fig. 1. After the degumming process with the alkali pretreatment (Fig. 1a), a certain amount of non-cellulosic materials was observed on the surface of the #1 degummed fibers. Moreover, the fibers were still adhered to each other and single fibers were not observed, which suggests that the alkali pretreatment degumming process was inefficient in removing gummy materials. When the hemp fibers were pretreated with TEMPO-oxidation (Fig. 1b) and laccase (Fig. 1c), the gummy substances on the surface of the #2 and #3 degummed fibers were less than the #1 degummed fibers. However, quite a few gummy substances still remained in the #2 and #3 degummed fibers. The surface of the #4 degummed fibers after the TEMPO-laccase system and alkali treatment is shown in Fig. 1d. It was observed that the fiber surface became clear and smooth, such that individual fibers could clearly be seen and the adhesive gummy materials between the fibers were considerably removed. The entire phenomenon discussed above suggests that the TEMPO-laccase system was more effective than the alkali, TEMPO-mediated oxidation and laccase

pretreatments. The surface morphologies of the hemp fiber could have a lot of influence on the mechanical properties, which will be discussed in a later section.



**Fig. 1.** SEM micrographs of degummed fibers (magnification of 1000x): (A) alkali pretreatment; (B) TEMPO-oxidation pretreatment; (C) laccase pretreatment; (D) TEMPO-laccase system pretreatment

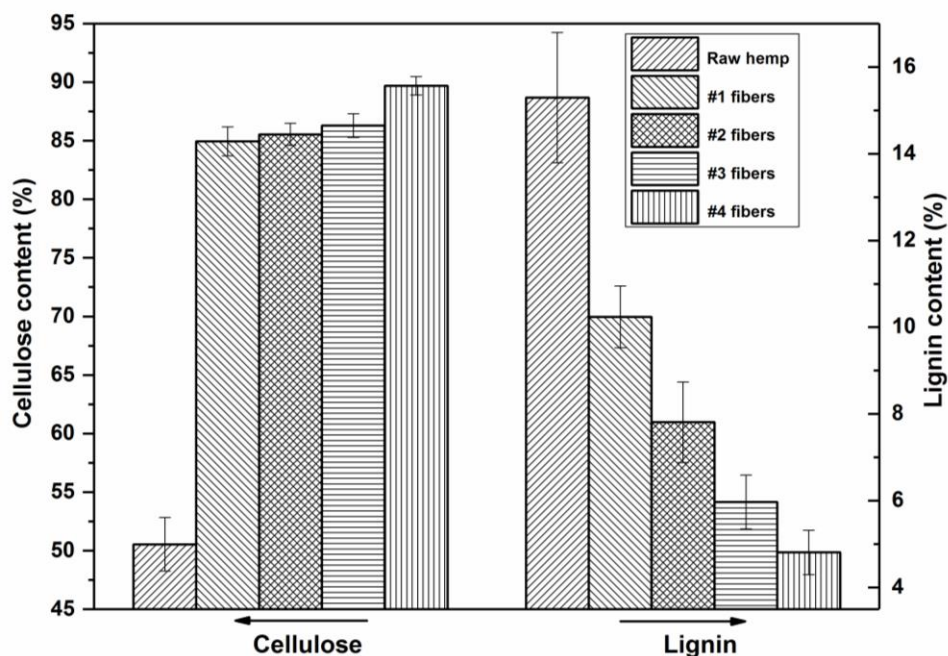
### Chemical Composition Analysis

The chemical composition of the raw hemp and the degummed fibers is summarized in Table 2. The results showed, in comparison to the raw hemp fibers, that all of the degummed hemp fibers increased in cellulose content and decreased in lignin content. Moreover, other noncellulosic materials, such as hemicellulose, pectin, water-soluble matter, fats, and wax, were removed to a great extent, which was due to gummy materials that were easily reacted with the degumming solutions and became soluble in water. Table 2 shows that the weight loss ratio was in good agreement with the content of cellulose and had a negative correlation with the lignin content. This was because a high weight loss ratio indicated a large removal of gummy materials, which led to a high content of cellulose. This phenomenon indicated that each degumming process had noticeable effects on the removal of noncellulosic materials, and the TEMPO-laccase system, in particular, was the most efficient process.

Moreover, as can be seen in Fig. 2, the cellulose content of degummed fibers obtained from the alkali pretreatment process was the lowest. After the TEMPO-oxidation and the laccase pretreatment, the cellulose content was increased to some extent, but the #4 degummed fibers obtained from the TEMPO-laccase system had the largest cellulose content.

**Table 2.** Weight Loss Ratio and Chemical Composition of Raw Hemp and Degummed Fibers

	Weight Loss Ratio (%)	Cellulose (%)	Lignin (%)	Hemicellulose (%)	Pectin (%)	Water-soluble Matter (%)	Fats and wax (%)
Raw Hemp	---	50.55 ± 2.30	15.29 ± 1.50	19.45 ± 1.33	4.92 ± 0.16	7.94 ± 0.76	1.85 ± 0.32
#1	40.12 ± 2.19	84.95 ± 1.23	10.24 ± 0.71	3.86 ± 0.13	0.16 ± 0.11	0.25 ± 0.08	0.54 ± 0.18
#2	41.67 ± 2.01	85.54 ± 0.94	7.81 ± 0.93	4.85 ± 0.28	0.20 ± 0.09	1.12 ± 0.11	0.48 ± 0.19
#3	42.09 ± 1.98	86.30 ± 1.01	5.97 ± 0.62	4.65 ± 0.18	0.07 ± 0.21	2.29 ± 0.17	0.72 ± 0.27
#4	45.66 ± 1.73	89.69 ± 0.78	4.81 ± 0.51	4.31 ± 0.25	0.08 ± 0.12	0.72 ± 0.07	0.39 ± 0.04

**Fig. 2.** The contents of cellulose and lignin of raw hemp and degummed fibers

Meanwhile, the lignin content decreased remarkably after the laccase pretreatment and the TEMPO-laccase system pretreatment processes due to the effect of laccase. That was because lignin is a polymer that is biologically synthesized from three monomers: p-coumaryl, coniferyl, and sinapyl alcohol (Abreu *et al.* 1999), and it was hard to separate the lignin from the cellulose. Laccase can be used for bonding lignocellulosic materials by oxidation of phenol compounds (Batog *et al.* 2008). The results of the lignin content shown

in Table 2 confirmed the outstanding performance of laccase in lignin degradation. Altogether, the highest content of cellulose and the lowest content of lignin confirmed the results that showed the gummy materials were mostly removed after the TEMPO-laccase system degumming process.

### Chemical Structure Analysis

FT-IR analysis of the degummed fibers was performed to identify the variations of hemp fiber composition after the degumming treatments.

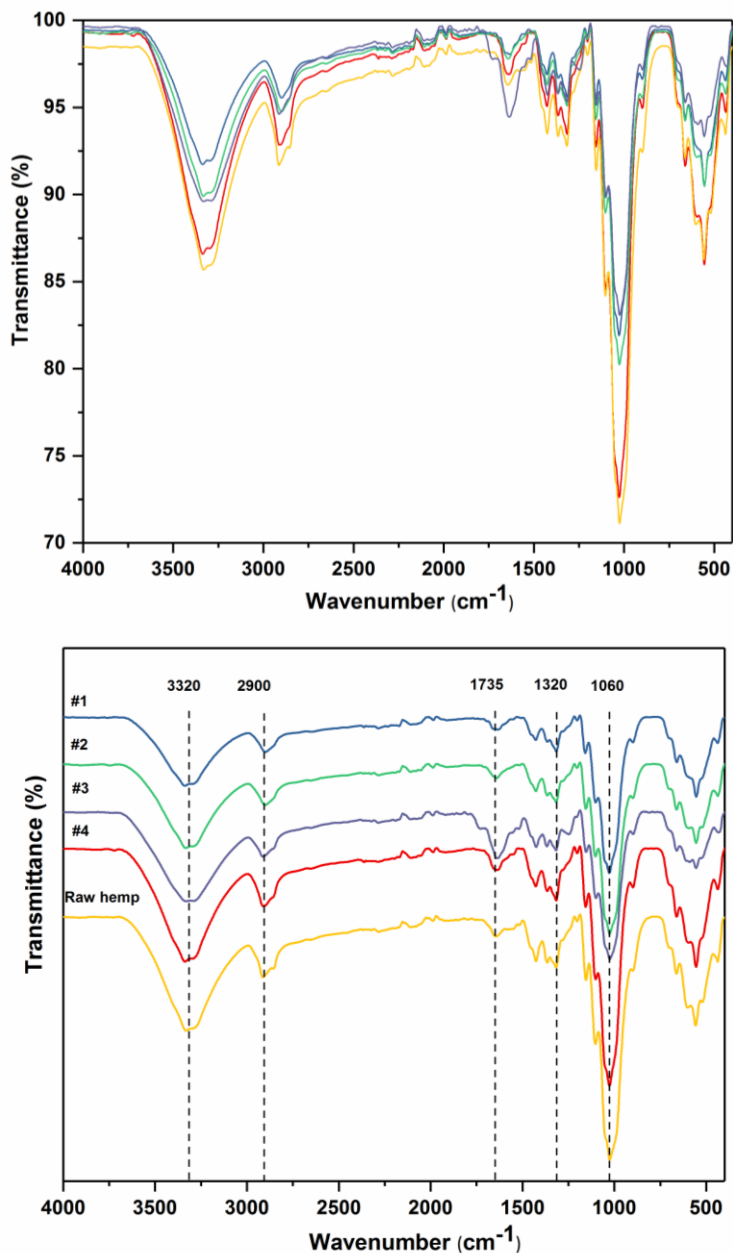


Fig. 3. FT-IR spectra of degummed fibers

As shown in Fig. 3, there was a peak at  $3320\text{ cm}^{-1}$  ( $-\text{OH}$  group stretching vibration),  $2900\text{ cm}^{-1}$  ( $\text{C-H}$  bond vibrations),  $1320\text{ cm}^{-1}$  ( $\text{C=O}$  stretching vibration), and  $1060\text{ cm}^{-1}$



(C=O stretching vibration) in the spectra curves of all of the degummed fibers, which was mainly attributed to the elemental functional groups in lignocellulosic materials that was much smoother than the raw hemp fibers. This indicated that the four degumming processes were efficient in removing lignin. Moreover, after the different pretreatments and the same alkali treatments, the intensities of these four lignin characteristic peaks decreased gradually from the #1 degummed fibers to the #4 degummed fibers, which was in high accordance with the reduction of the lignin content demonstrated in Table 2. This suggested that the TEMPO-laccase system was the most effective method to remove lignin when compared with the three other processes.

However, when compared with the sharp peak of raw hemp, the peak at  $1735\text{ cm}^{-1}$  that corresponded to C=O stretching from the ketones and/or esters of hemicelluloses almost disappeared. This change was related to the heavy removal of hemicellulosic materials, as shown in Table 2.

### Crystal Structure Analysis

The crystalline formation of hemp fibers after the degumming treatment was investigated *via* XRD, as shown in Fig. 4. The peaks around  $14.8^\circ$  and  $16.4^\circ$  were characteristic of cellulose crystal assignments (Okano and Sarko 2010). Moreover, major diffraction peaks for  $2\theta$  ranging between  $22^\circ$  and  $23^\circ$  that corresponded to the crystallographic plans of cellulose I were observed (Le Troedec *et al.* 2008). After the 4 treatments, the positions of characteristic cellulose peaks did not change and the overall trend of the curves were in resemblance, which indicated that the degumming processes did not cause the crystalline transformation of hemp fibers.

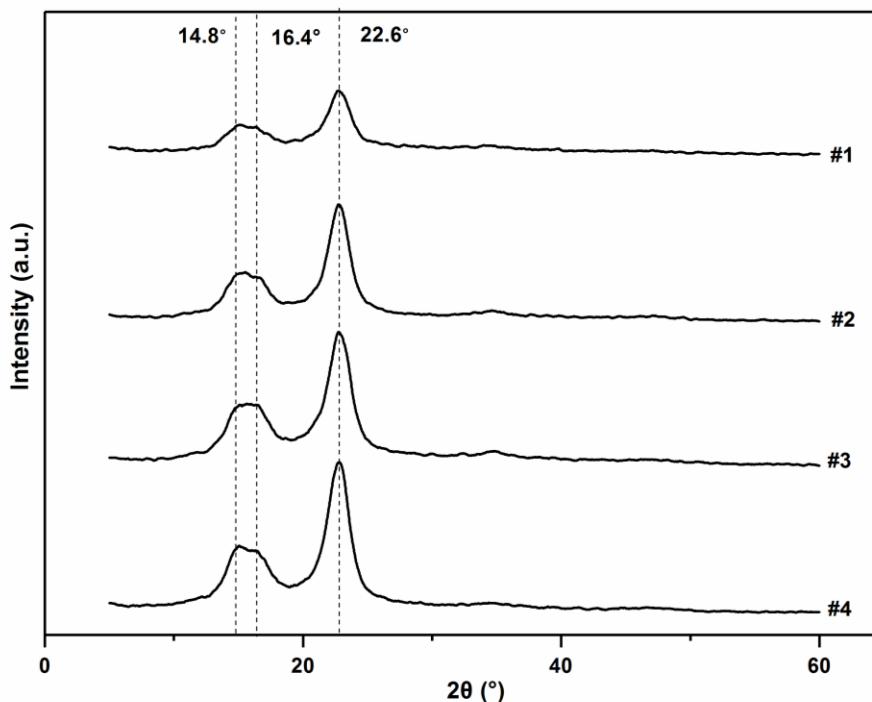


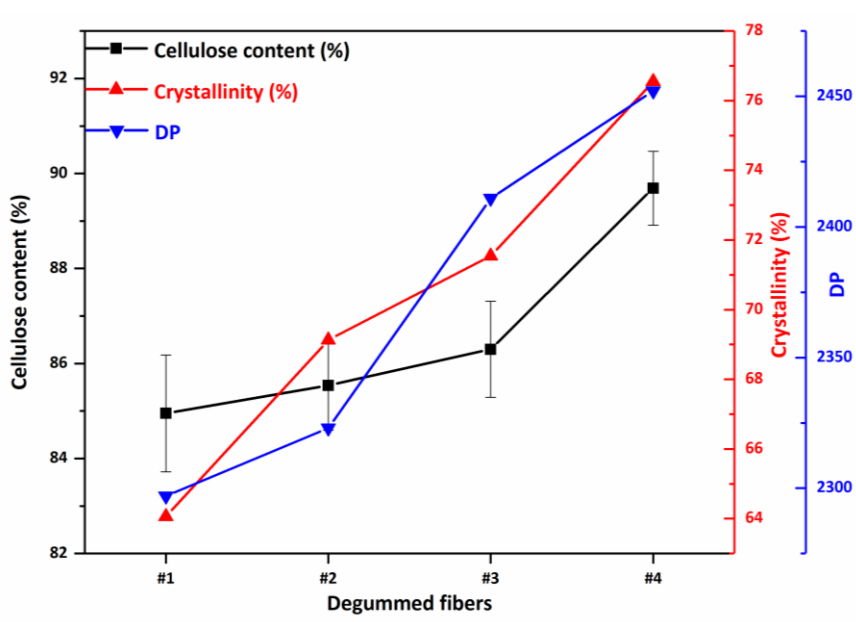
Fig. 4. X-ray diffraction patterns of degummed fibers

The crystallinity index of the degummed fibers is shown in Table 3. It was observed that the crystallinity index gradually increased, which shared the same trend with the cellulose content. After the TEMPO-laccase system, the #4 degummed fibers had the biggest crystallinity index. This is because when there was a higher content of cellulose, more non-cellulosic materials that contained a high amount of amorphous materials and impurities were removed (Ebringerová *et al.* 2005), resulting in a bigger crystallinity index.

**Table 3.** Crystallinity Index and DP of Degummed Fibers

Degummed Fibers	#1	#2	#3	#4
Crystallinity (%)	64.06	69.13	71.54	76.55
DP	2297	2323	2411	2452

The degree of polymerization (DP) of the fibers after degumming is shown in Table 3 and Fig. 5, which to some extent reflects the degumming efficiency. It was observed that the DP of the #2 degummed fibers was larger than that of the #1 degummed fibers. When the TEMPO was added into a solution, the fibers were subjected to an oxidative condition, during which the cellulose macromolecule chains decomposed into smaller ones, which could induce a declining trend of the degree of polymerization. However, the removal of gummy materials with a low molecule count in fibers led to a considerable increase of DP. Moreover, the DP of the #4 degummed fibers was the largest, which was due to the highest cellulose content and the highest removal of gummy materials. The same tendency of the three curves in Fig. 5 also shown a close relationship between cellulose content and crystallinity, as well as DP value. Therefore, the removal of gummy materials played a dominant role in increasing the degree of polymerization of the residual solid material.



**Fig. 5.** The cellulose content, crystallinity and DP of degummed fibers

## Mechanical Properties

**Table 4.** Mechanical Properties of Degummed Fibers

Degummed Fibers	Length (mm)	Linear Density (dtex)	Tenacity (cN/dtex)	Breaking Elongation (%)
#1	65	10.71 ± 0.44	2.78 ± 0.23	2.81 ± 0.17
#2	57	9.55 ± 0.28	2.88 ± 0.19	3.05 ± 0.31
#3	52	8.19 ± 0.54	2.93 ± 0.11	3.21 ± 0.24
#4	43	6.64 ± 0.25	3.41 ± 0.15	3.53 ± 0.13

To meet the requirement of downstream processing, a certain tenacity and breaking elongation was desired after the degumming process (Kang and Epps 2009). Moreover, better and smaller linear density makes high-value textile end use possible.

More specifically, as seen in Table 4, the #1 degummed fibers had relatively undesirable linear density, tenacity, and breaking elongation because the alkali pretreatment soaking in a low temperature removed a small amount of noncellulosic materials and the excessive gummy materials had a negative effect on the fiber properties. The mechanical properties in the #2 degummed fibers was superior to the #1 degummed fibers. This could possibly be explained by the conversion of hydroxyl groups into aldehyde and carboxyl groups, which was evoked by the effort of the TEMPO oxidation. The fibers' tenacity and breaking elongation under a certain range increased due to the increased amounts of aldehyde and carboxyl (Li *et al.* 2016). Because the laccase pretreatment was principally aimed at removing lignin that was the main obstruction of hemp fibers' mechanical properties, the effective removal of lignin content promoted a substantial rise in the mechanical properties in the #3 degummed fibers. However, when compared with the most desirable mechanical properties in the #4 degummed fibers, other noncellulosic materials that remained in the #3 degummed fibers caused a loss of the mechanical properties. The largest tenacity and elongation at break in the #4 degummed fibers was due to the aggressive removal of the gummy substances during the TEMPO-laccase system.

As shown in Table 4, the #4 degummed fibers had the smallest linear density because of the largest removal of gummy materials. In the meantime, after this TEMPO-laccase system, the raw hemp fibers were divided into several small bundles, which was referred to as technical fiber. This confirmed the theory that a more severe degumming condition led to a large improvement in the fineness of the degummed fiber.

The mechanical properties were greatly influenced by the cellulose content. This is attributed to the ability of the cellulose to serve as a skeleton in the degummed fibers. As can be seen in Fig. 6, the curves of tenacity and breaking elongation had the same upward trend with the curve of cellulose content. However, the curves of length and linear density had the opposite downward trend with that of cellulose content. A high cellulose content appeared to be essential to achieve superior properties of degummed hemp fibers, such that a proper degumming process is required.

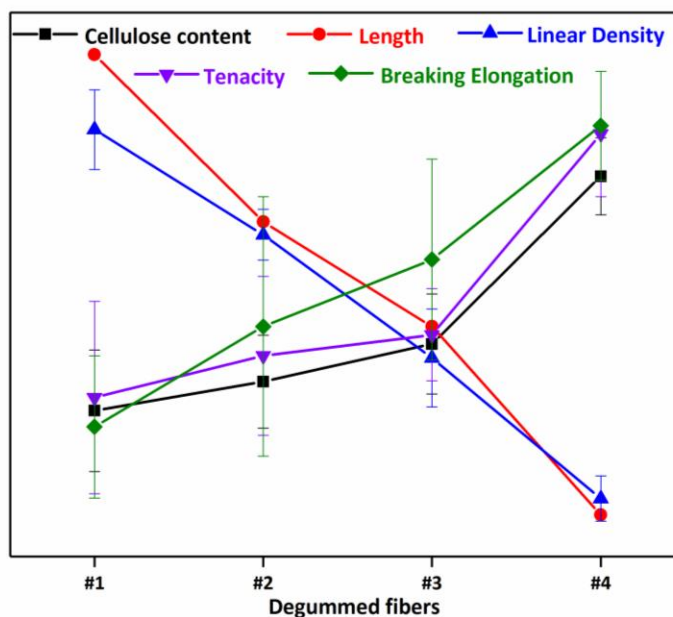


Fig. 6. The schema of mechanical properties of degummed fibers

## CONCLUSIONS

A combined treatment with TEMPO and laccase was used in the hemp degumming process in this study. The results were systematically characterized in terms of chemical composition, SEM, FT-IR, and XRD.

1. The degummed fibers obtained from the TEMPO-laccase system had the smallest lignin and other noncellulosic materials contents, which were substantially removed by the TEMPO-laccase system degumming process. The SEM micrographs showed the cleanest and smooth surface of the degummed fibers obtained from the TEMPO-laccase system, which indicated an excellent degumming effect. The FT-IR analysis confirmed that the noncellulosic materials were greatly removed, especially in the removal of lignin and hemicellulose by the TEMPO-laccase system degumming process. Based on XRD, in spite of the high removal of noncellulosic materials, the TEMPO-laccase system degumming processes did not cause crystalline transformation of hemp fibers. The highest crystallinity also confirmed the highest cellulose content.
2. Further efforts were committed to investigate the mechanical properties of degummed fibers. These efforts showed that the degummed fibers after the TEMPO-laccase system had the most desirable linear density of 6.64 dtex and tenacity of 3.41cN/dtex, which was of vital importance in further processing.
3. During the TEMPO-laccase degumming. The gummy materials were first reacted with the oxidant (NaClO) before the cellulose because they covered over the cellulose in raw hemp fibers. Whether there exists an oxidative reaction in cellulose was determined by the dosage of NaClO. A higher dosage of NaClO in the solution might induce the oxidative reaction not only in the gummy materials, but also in cellulose, which could lead to extra damage in the cellulose. To protect the cellulose and degrade non-

cellulosic materials in the meantime, it is important to control the dosage of NaClO, which needs to be further considered in future work.

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