# Steam Explosion and Alkali-Oxygen Combined Effect for Degumming of Kenaf Fiber

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Kenaf is an economically viable and ecologically friendly cellulose source. It can be used in the textile, paper, and bio-energy industries, but it has not been effectively developed and utilized because of degumming problems. To effectively take advantage of kenaf resources, to satisfy the growing demand for natural fiber, and to provide support for other fiber material degumming, steam explosion (STEX) pretreatment followed by alkali-oxygen treatment was studied. The effect of pressure on the properties of kenaf during the STEX treatment was studied, and the optimal degumming process for kenaf was selected. Results showed that STEX pretreatment removed pectin and part of the hemicellulose. Carbohydrates (cellulose and hemicellulose) could be degraded via high pressure treatment. The residual gum content and the fineness of the kenaf fiber after the alkali-oxygen treatment were good enough for textile production. High pressure was found not to be a key factor influencing the degumming process. Low pressure STEX (0.5 MPa) and alkalioxygen treatment was judged to be an efficient method for degumming kenaf fibers.

Keywords: Kenaf fiber; Steam explosion; Degumming; Cellulose

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# INTRODUCTION

Billions of tons of natural fibers are grown around the world. They are abundant, inexpensive, and readily available. Kenaf, one of these resources, is a source of cellulose that is economically viable and ecologically friendly. With the growing demand for cellulose resources (Karimi et al. 2014), kenaf has received increasing attention as an alternative fiber for the pulp and paper industry. Furthermore, kenaf fiber is of interest to the woven and non-woven materials industries (Morrison et al. 1999). Kenaf is in an advantageous position over other lignocellulosic crops since it has a short plantation cycle, can adapt to environmental conditions, requires relatively low use of pesticides and herbicides (Wang and Ramaswamy 2003), and is thus considered an economical, ecologically friendly crop (Nishino et al. 2003). Kenaf is planted largely because it is easy to grow with greater fiber yields than combined ramie and flax in China (Li and Hu 1998). According to a report of the Food and Agricultural Organisation (FAO), India, Bangladesh and China provided 90% of the world's jute and kenaf, etc. in 2010 (FAO, 2013). The growing use of plant bast fibers and insufficient ramie and flax fiber production have made kenaf a common fiber crop grown around the world to help satisfy the demand for natural fiber (Nishino et al. 2003; Yang et al. 2014).

Although kenaf resources are abundant in China, kenaf has always been used as a cordage crop to produce twine, rope, sackcloth, and other low-grade textiles because of

its coarseness, stiffness, shortness, and high lignin content (Parikh et al. 2002; Anandjiwala and Blouw 2007). However, the properties of yarn when blended with kenaf and cotton are similar to those of ramie or flax when blended with cotton (Casey 1980). The fiber degumming process is the key to taking advantage of kenaf resources effectively. Many studies report that degumming methods are important in determining fiber properties (Zheng and Yu 2002). Degumming is a process for removing noncellulosic material attached to the fibers and thereby to release the individual cellulosic fibers (Amel et al. 2013). Effective degumming involves degradation of pectin and other cementing materials, such as lignin and hemicellulose, which act as binding agents between the individual fibers and between fiber bundles. Natural degumming in biologically active water or running streams, termed retting, is the typical method of isolating bast fiber strands from plant bark. However, this process is time-consuming, pollutes the water, and yields poor reproducibility in terms of fiber properties (Zhang et al. 2005). Chemical degumming has been shown to achieve more reproducible properties, but it reduces fiber strength (Yu and Yu 2007) and produces pollutants. Microbiological degumming is preferred because of the relative controllability of the resulting fiber quality (Ramaswamy et al. 1995), but high cost is one concern for kenaf production. As a physical degumming method, steam explosion (STEX) treatment has received increasing attention and has been developed because it is less time-consuming, more environmentally friendly, offers good repeatability (Vignon et al. 1996), and, most importantly, the degradation of hemicellulose and lignin during the STEX treatment is helpful to the subsequent degumming process. As a well-known method for separating lignocellulosic material into its three main components (cellulose, lignin, and hemicelluloses) (Josefsson et al. 2002), steam explosion of lignocellulosic material has been studied at length in recent years (Excoffier et al. 1991). It has been shown to be more practical because of its high efficiency, low energy consumption, environmental friendliness, and shorter process time. Previous work by the authors investigated a degumming process of kenaf that combined STEX and ultrasonic treatment; some good results were obtained (Zhang et al. 2014), however, ultrasonic treatment is currently not able to be used in bulk production.

Completely degumming kenaf fiber is difficult because, unlike flax, it contains lignin as a major constituent of its fibers. Kenaf primary fibers are short; therefor it is important to apply only partial degumming and to avoid removing too much of the noncellulosic material. Some of the binding agents must be left in the material to sustain the assembly individual primary fibers into larger process fibers. In this study, STEX pretreatment coupled with an alkaline oxygen degumming process was used to separate kenaf into fiber bundles.

## EXPERIMENTAL

## Sample Preparation

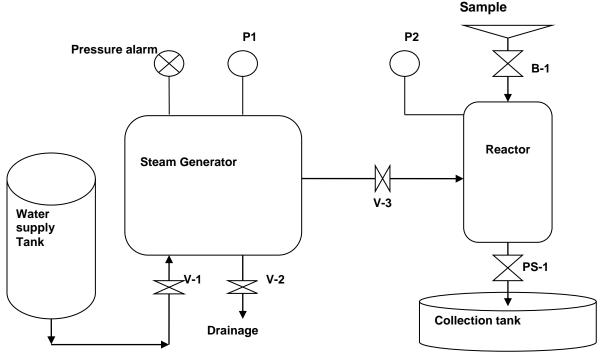
Kenaf fibers were collected from the Aksu Prefecture of Xinjiang, China. First, kenaf fibers were chopped to a uniform size of approximately 10 cm, and then the kenaf fiber segments were uniformly mixed and dried in an air oven at 75 °C for 3 h. The chopped fiber segments were divided into four groups (100 g per group) and impregnated with distilled water (fiber-to-liquor weight ratio of 1:20) at room temperature for 24 h.

## **STEX Pretreatment**

The steam explosion equipment (BGDR-4.5H) used in this experiment was made independently by Qingdao University (Shandong, China). Figure 1 below shows the process flow scheme of the steam explosion device. It was found that the best treatment parameters for STEX were pressure between 1.5 and 6.5 MPa and residence time from 2 to 10 min (Singh *et al.* 2015). However, when higher pressure is applied, the process is more dangerous, consumes more energy, and the requirements of the STEX equipment are higher, increasing its price.

The STEX intensity is mainly related to the pressure and residence time. Extending the residence time could reduce the pressure requirement. In this study, low pressure, long residence time (15 min) treatment was used. The prepared samples were exploded with varied steam pressures (0.5 to 1.5 MPa) for a fixed time (15 min). After treatment, the pressure was immediately released.

The steam-exploded fibers were collected and dried in an air oven at 75  $^{\circ}$ C for 10 h. Thirty grams of sample were used for characterization, and the rest was used for follow-up studies.



**Fig. 1.** Process flow scheme of the steam explosion equipment (B: ball valve, PS: pressure relief valve, V: valve, P: digital pressure transducer)

## Alkaline Oxygen Degumming Process

The steam-exploded fibers were bleached using a solution (fiber-to-liquor ratio of 1:20) of 5% sodium hydroxide, 3% hydrogen peroxide, 3% sodium silicate, 2% anhydrous sodium sulfite, 3% urea, 3% sodium tripolyphosphate, 2% JFC, and 2% defoaming agent (owf) at 75 °C for 2 h. All of the solutions were prepared on a weight basis (Parikh *et al.* 2002; Wang and Ramaswamy 2003; Qu *et al.* 2005). After bleaching, the fibers were thoroughly washed in distilled water until there was no solution remaining on them (by using phenolphthalien to test the pH value) and then oven-dried at 105 °C for 3 h.

Figure 2 shows the experimental design for the study.

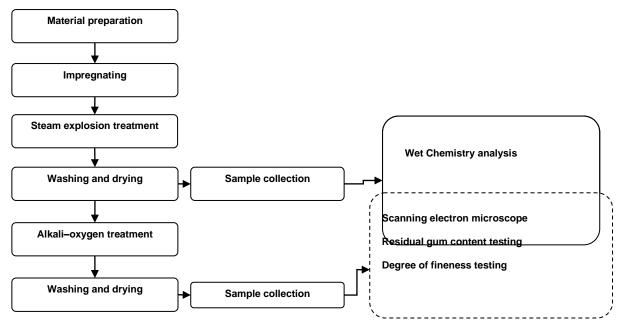


Fig. 2. Schematic diagram for the degumming and characterization process

# **Chemical Composition Analysis**

The chemical composition analysis was performed following the National Renewable Energy Laboratory (NREL) standard (Sluiter et al. 2008a, b) and the Chinese National Standard GB 5889-86 (Jiang and Shao 1986). The wax content of the kenaf fibers was determined by applying a mixture of benzene and ethanol (volumetric ratio 2:1) and boiling for 4 h. Distilled water was used (boiling for 3 h) to determine the watersoluble matter content. The residue was transferred into an ammonium oxalate solution and boiled for 3 h to determine the pectin content. The holocellulose (cellulose and hemicellulose) content of the kenaf fibers was determined by treating the residue with a mixture of acetic acid and sodium chlorite solutions at 75 °C for 3 h. The cellulose content of the fibers was then determined by further treating the holocellulose with boiling, 20% sodium hydroxide to remove the hemicellulose. The lignin content of the kenaf fibers was determined by treating them with a sulfuric acid solution: 72% H<sub>2</sub>SO<sub>4</sub> was used on the dewaxed sample at 20 °C for 2 h. The solution was then diluted to 4% and transferred into an autoclave at 121 °C, and, finally, a 1-h treatment was applied in the autoclave to completely hydrolyze the carbohydrates, yielding acid-insoluble lignin. The acid-insoluble lignin (AIL) is the residue (remaining solids) captured from the hydrolysis suspension. The acid-soluble lignin (ASL) was measured using a UV spectrophotometer. The total lignin content is the sum of the AIL and the ASL. All the experiments were repeated three times, and all of the solutions were prepared on a weight basis.

## **Scanning Electron Microscope Analysis**

A Hitachi S-4300SE scanning electron microscope (SEM) was used to examine the morphology of the kenaf fibers. The acceleration voltage was 1.5 kV, and the magnification was 20 to 5,000,000. Samples were stuck to the slides and coated with gold, and finally, they were viewed and photographed with the SEM.

## **Residual Gum Content**

The residual gum content in the kenaf fibers was determined to evaluate the degree of kenaf fiber degumming achieved. According to the Chinese National Standard (GB 5889-86), the residual gum content of the fibers was calculated using the following equation (Eq. 1),

$$W_{j} = \left[\frac{G_{0} - G_{1}}{G_{0}}\right] \times 100 \%$$
<sup>(1)</sup>

where  $W_j$  is the residual gum content,  $G_0$  is the dry weight of the sample before testing, and  $G_1$  is the dry weight of the sample after testing.

## **Degree of Fineness**

Fiber fineness is an important indicator for the ability to use fibers in certain applications. According to the Chinese national standard (GB 5884-86), the fibers were carded into bunches and cut to 10 cm in length. The cut-off fibers were weighed and counted. The fineness of the fibers was calculated using the following equation (Eq. 2),

$$N_{\rm m} = \frac{L \times n}{G} \tag{2}$$

where  $N_{\rm m}$  is the metric count, *n* is the number of fibers, *L* is the fiber length (mm), and *G* is the weight of the fiber (g).

# **RESULTS AND DISCUSSION**

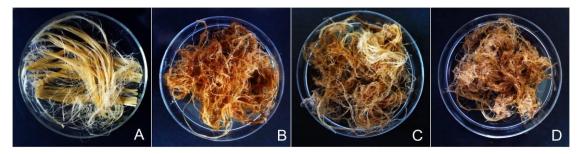
## The Morphology of Kenaf Fibers

Steam explosion treatment effectively changed the morphology of the kenaf fibers. Figure 3 shows the morphology of the kenaf fibers treated with STEX under different pressures. With increasing STEX pressure (from B to D), the reaction became more intense, the color of the kenaf fibers changed (gradually deepened from light yellow to tan), the shape of the kenaf fibers changed (from a sheet to a bundle), and the fibers gradually spread out and became more separated and shorter. When the STEX pressure rose to 1.5 MPa, the fiber suspension seemed like a pasty mess. This implies that STEX has strong effects on the deconstruction of biomass, which can facilitate degumming.



**Fig. 3.** Morphology of kenaf fibers after STEX. (A) raw kenaf bast; (B) kenaf fiber after STEX (0.5 MPa, 15 min); (C) kenaf fiber after STEX (1.0 MPa, 15 min); (D) kenaf fiber after STEX (1.5 MPa, 15 min)

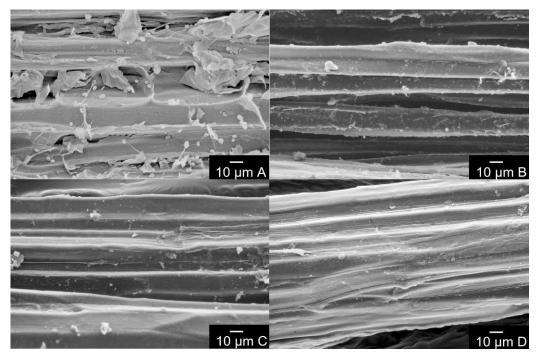
According to the experimental design, the kenaf fibers were treated with alkalioxygen degumming after the steam explosion treatment. Figure 4 shows that alkalioxygen treatment effectively changed the morphology of the kenaf fibers. After alkalioxygen treatment, the STEX-treated kenaf fibers (shown in Figs. 4B, 4C, and 4D) were more dispersed and finer than untreated fibers (shown in Fig. 4A). Alkali-oxygen treatment bleached the kenaf fibers significantly and the colour of all kenaf fibers turned lighter. The kenaf fibers became fluffy and soft. The above results demonstrate that the alkali-oxygen treatment further degummed the kenaf bast and fibers and isolated hemicellulose and lignin, making the final fiber softer and lighter in color.



**Fig. 4.** Morphology of kenaf fibers after STEX treatment and alkali-oxygen treatment. (A) raw kenaf bast treated with alkali-oxygen; (B) raw kenaf bast treated with alkali-oxygen after STEX (0.5 MPa); (C) raw kenaf bast treated with alkali-oxygen after STEX (1.0 MPa); (D) raw kenaf bast treated with alkali-oxygen after STEX (1.5 MPa)

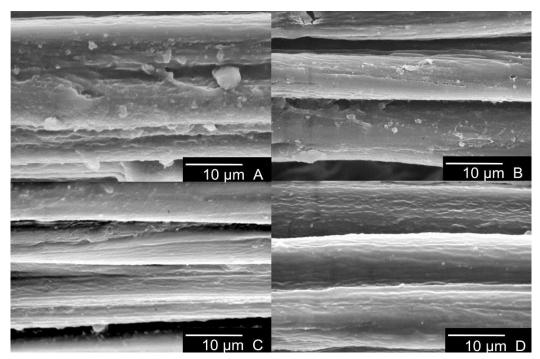
# **SEM Analysis**

The STEX treatment resulted in structural and chemical changes to the fiber surfaces (Fig. 5).



**Fig. 5.** SEM micrographs (the magnification is 600) of original and STEX-treated kenaf fiber. (A) raw kenaf bast; (B) kenaf fiber after STEX (0.5 MPa, 15 min); (C) kenaf fiber after STEX (1.0 MPa, 15 min); (D) kenaf fiber after STEX (1.5 MPa, 15 min) The SEM micrographs of the original kenaf fiber and the STEX-treated kenaf fiber were taken to investigate the structural changes in these fibers. It was expected that the surface morphology of the untreated fiber would be different than that of the treated fiber, particularly in terms of smoothness and roughness. Figure 5A shows that the original kenaf fibers were in bundles with rough surfaces due to the presence of pectin, hemicellulose, lignin, and other impurities. Figures 5B, 5C, and 5D clearly show individual fibers after the removal of hemicellulose, lignin, and pectin. With increasing STEX pressure (from B to D), the pectin, lignin, and other impurities were removed to greater degrees, and the surfaces of the fibers became smoother, gradually improving fiber splitting.

According to the experimental design, the kenaf fibers were next treated with alkali-oxygen degumming after the steam explosion treatment. Figure 6 shows that alkali-oxygen treatment effectively removed the pectin, lignin, and other impurities from the fiber surface.



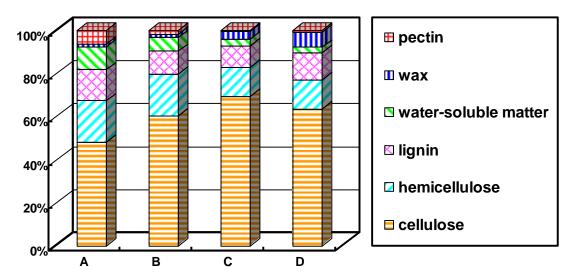
**Fig. 6.** SEM micrographs (the magnification is 2500) of STEX- and alkali-oxygen-treated kenaf fiber. (A) raw kenaf bast treated with alkali-oxygen; (B) kenaf fiber treated with alkali-oxygen after STEX (0.5 MPa, 15 min); (C) kenaf fiber treated with alkali-oxygen after STEX (1.0 MPa, 15 min); (D) kenaf fiber treated with alkali-oxygen after STEX (1.0 MPa, 15 min);

Figure 6A shows the SEM micrograph of kenaf fiber treated using alkali-oxygen degumming without STEX pretreatment. Impurities were observed on the surface of this fiber. On the other hand, Figs. 6B, 6C, and 6D show similar fibers subjected to alkali-oxygen degumming after STEX treatment at different pressures. As shown in Fig. 6B, few impurities still remained on the fiber surface after the mildest STEX pretreatment. This indicates that STEX at 0.5 MPa for 15 min was not intense enough to effectively remove the impurities from the kenaf fibers. Figure 6C shows the SEM micrograph of kenaf fiber treated by STEX at 1.0 MPa for 15 min. Nearly all impurities were removed from the fiber surface by this pretreatment. The SEM micrograph of the 1.5-MPa STEX-

treated kenaf fiber was similar in appearance to that of the kenaf fiber treated with 1.0-MPa STEX. As compared to Fig. 6C, Fig. 6D shows jagged, rougher fiber with a cleaner surface. The steam explosion pretreatment deconstructed the fibers and increased the reactive area, enhancing cellulose accessibility. All SEM micrographs of the kenaf fiber show that STEX-alkali-oxygen treatment more effectively degums kenaf fiber than individual treatment with STEX or alkali-oxygen degumming.

# **Chemical Composition Analysis**

According to the NREL standard (Sluiter *et al.* 2008a,b) and the Chinese National Standard GB 5889-86 (Jiang and Shao 1986), the chemical composition of the kenaf fibers was determined and is shown in Fig. 7, after steam explosion treatments at different pressures.



**Fig. 7.** Main chemical component contents in kenaf. (A) raw kenaf bast; (B) kenaf fiber after STEX (0.5 MPa, 15 min); (C) kenaf fiber after STEX (1.0 MPa, 15 min); (D) kenaf fiber after STEX (1.5 MPa, 15 min)

The main chemical component content of the kenaf fiber changed significantly following steam explosion treatment. The treated fibers contained higher percentages of cellulose and lower percentages of hemicellulose, lignin, and residual gum compared to the untreated fibers. With increased steam explosion pressure, the kenaf fiber degumming became more complete. Sample C exhibited the greatest degree of degumming: the pectin, hemicellulose, and lignin removal extents were 94.0, 34.3, and 36.5%, respectively, and the cellulose content increased by 33.4%, approaching 70%. When the pressure was increased to 1.5 MPa (Fig. 7D), the cellulose content decreased and the lignin and wax contents greatly increased.

Steam explosion causes chemical changes within fibers because water itself acts as an acid at high temperatures. During steam explosion, hemicellulose and pectin are hydrolyzed by acids, and the hemicellulose eventually becomes water-soluble (Toussaint *et al.* 1991; Hautala *et al.* 2004; Jiang *et al.* 2014). Cellulose is slightly depolymerized and lignin softens and is eventually depolymerized (Mosier *et al.* 2005). However, under extreme conditions (high temperatures and pressures), xylose (a hemicellulose) is degraded, forming furfural, and glucose (a cellulose monomer) is degraded to 5-

hydroxymethyl furfural (Iroba *et al.* 2014). The cellulose structure was subjected to some degree of damage, as part of it was depolymerized and removed, resulting in a partial reduction of the cellulose content. Some researchers (Heitz *et al.* 1987), using the index log  $R_0$ , have described the explosion as a logarithmic function of pressure and residence time (Eq. 3),

$$R_{0} = t \times \exp\left[\frac{(T - 100)}{14.75}\right]$$
(3)

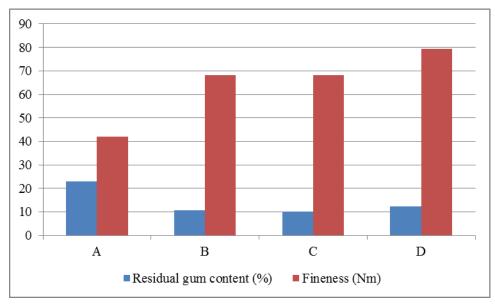
where *t* is the residence time (min) and *T* is the reaction temperature (°C).

The more severe the reaction, the greater was the degree of fiber splitting that was observed. This makes the fiber separation effect more obvious, but the conditions must be controlled to within a certain range because extreme reaction conditions damage the cellulose.

The theory above indicates that sample D underwent significant degradation of cellulose, producing some low degree of polymerization carbohydrates extractable by benzene and ethanol. This could explain the observed increase in the wax content, decrease in cellulose content, and increase in lignin content.

## **Residual Gum Content and Degree of Fineness**

The residual gum content and the degree of fineness of the kenaf fibers were determined and are shown in Fig. 8 following alkali-oxygen treatment.



**Fig. 8.** Residual gum content and fineness of degummed kenaf fiber. (A) raw kenaf bast treated with alkali-oxygen; (B) kenaf fiber treated with alkali-oxygen after STEX (0.5 MPa, 15 min); (C) kenaf fiber treated with alkali-oxygen after STEX (1.0 MPa, 15 min); (D) kenaf fiber treated with alkali-oxygen after STEX (1.5 MPa, 15 min)

The residual gum content and fineness significantly improved following alkalioxygen treatment. Compared with the raw kenaf bast, the kenaf fibers subjected to steam explosion pretreatment had lower residual gum content and greater fineness. After the alkali-oxygen treatment under the same conditions, the kenaf fiber degumming became more extensive with increasing STEX pressure. Figure 8D shows the maximum fineness but did not have the lowest residual gum content. As was analyzed in the previous section, extreme reaction conditions (high pressures) resulted in cellulose damage; further, the oxidative alkali-oxygen degumming process can disrupt the cellulose structure more. Part of the cellulose was depolymerized and removed, resulting in a reduction of the cellulose content.

For textile production, most of the bast fibers are in the form of bundles that are adhered together with lignin and hemicellulose. As the gum content is gradually decreased, the fibers became much finer. Although the kenaf fibers were still in the form of bundles, the residual gum content was decreased and the fibers had been converted into a significantly finer form following STEX pretreatment and alkali-oxygen treatment.

Successful degumming should yield kenaf fiber with low residual gum content and high fineness (Nm). In this study, sample D had the highest fineness but was subject to cellulose degradation, as discussed above. Sample B, which had the lowest gum content and relatively high fineness, was considered the best kenaf fiber achieved in this research, demonstrating that the best degumming process in this study was STEX at 0.5 MPa for 15 min with subsequent alkali-oxygen treatment. This also shows that highpressure STEX was not the best when STEX was associated with other degumming processes. STEX at low pressure was more effective and economical.

Compared with the authors' previous study involving the use of STEX and ultrasonic vibrations to degum kenaf (Zhang *et al.* 2014); similar results (residual gum content were both around 10%) were obtained by using STEX combined with alkali-oxygen treatment. But less alkali was required (5% NaOH) in comparison with 7% NaOH (5% in presoak process and 2% in ultrasonic treatment). Moreover, STEX combined with alkali-oxygen treatment is more suitable for bulk production than STEX combined with ultrasonic treatment under the current technique.

In conclusion, the STEX-alkali-oxygen treatment effectively removed pectin, lignin, and other impurities from the kenaf fibers, increased their relative cellulose content, increased the fiber splitting degree, and made the fiber potentially useful in textile manufacturing processes. The experimental conditions must be controlled to within an appropriate range.

## CONCLUSIONS

- 1. STEX pretreatment provided significant, effective degumming of kenaf fibers. Appropriately increasing the pressure helped to remove gum substances (pectin, hemicellulose, and lignin) and improved the relative content of cellulose.
- 2. Excessively high-pressure STEX treatment could degrade carbohydrates (cellulose and hemicellulose).
- 3. STEX pretreatment followed by alkali-oxygen treatment yielded high-quality kenaf fiber with a clean surface, high cellulose content (low residual gum content), and high fineness.
- 4. The best degumming process in this study was STEX pretreatment at 0.5 MPa for 15 min with subsequent alkali-oxygen treatment. This process could also be applied to other materials in future studies.

## ACKNOWLEDGMENTS

This work was supported by the Award Funds for Outstanding Middle-Aged and Young Scientists of the Shandong Province (BS2014CL044 and BS2013CL008), Taishan Scholars Construction Engineering of Shandong Province, and the Program for Scientific Research Innovation Team in the Colleges and Universities of the Shandong Province.

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Article submitted: February 16, 2015; Peer review completed: May 7, 2015; Revised version received: June 15, 2015; Accepted: June 24, 2015; Published: July 17, 2015. DOI: 10.15376/biores.10.3.5476-5488