

## A green degumming process of ramie

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### A B S T R A C T

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Ramie provides the longest and strongest natural fiber in textile industry, but its traditional degumming process is costly and requires a large amount of alkali, which causes serious environmental concerns. In the current work, a steam explosion (STEX) treatment followed by sodium percarbonate (SP) soak degumming process was investigated. Microstructure, chemical composition and mechanical properties of the refined ramie fibers were comprehensively characterized. The residual gum content was below 5%, the fineness was higher than 1600 Nm (6.25 dtex), the breaking tenacity was 5.4 cN/dex, and the whiteness was above 50%. All of the properties met the requirements of Chinese national standard, and the breaking tenacity and whiteness were notably better than those of the fibers degummed traditionally. In addition, environmental impacts of the new degumming process were evaluated. Only 50% chemicals were needed for the new process, and chemical oxygen demand (COD) of the waste reduced to 35% of the traditional method. Therefore, the new method was more environment-friendly and economically feasible. It has great potential for industry applications.

## 1. Introduction

Ramie is widely used in clothing fabrics, twines, industrial packaging, cordages and fiber reinforcements (Li et al., 2015; Liu et al., 2012; Luan et al., 2017; Ni et al., 2018). However, before ramie fiber could be used in textile, gummy matters in the raw bast have to be removed through a degumming process (Angelini et al., 2015; Yang et al., 2016; Zheng et al., 1988). The traditional degumming process requires two steps of treatments with large amounts of NaOH and other hazardous chemicals. The process not only has high cost, but also causes serious environment concerns (Zheng et al., 2001). Therefore, new environment-friendly degumming method with lower cost is desired.

Steam explosion (STEX) treatment is an efficient and environment-friendly method for degumming of various natural fibers (Gao et al., 2015; Zhang et al., 2014). Previous studies suggest that STEX is also effective on plant bast degumming. It can remove part of gummy matter from the raw materials, degrade some polysaccharides, and break bast to fiber bundles (Zhang et al., 2016). But STEX treatment alone couldn't complete the degumming process, and only fiber bundles could be obtained from this process (Jiang et al., 2017a,b). Therefore, a

subsequent chemical degumming treatment is still necessary. As an environment-friendly and economically feasible reagent, sodium percarbonate (SP) has been traditionally used as beaching agent in textile industry, (Hage and Lienke, 2006; JC, 1964). Recently, its effects on ramie degumming were reported (Liu et al., 2011). Although the quality of ramie fiber extracted with SP alone is not good enough for textile production, the reagent showed positive effects on the degumming.

Therefore, the combination of STEX and SP treatment was hypothesized to be a promising method to degum ramie with low pollution and high fiber quality. This method was investigated in detail in this study.

## 2. Experimental details

### 2.1. Material

The ramie bast samples were supplied by the Research Center of Bast-fiber Plant in Hunan Province, China. All the ramie material was tiled under the ambient conditions (20–30 °C and < 50% humidity) for

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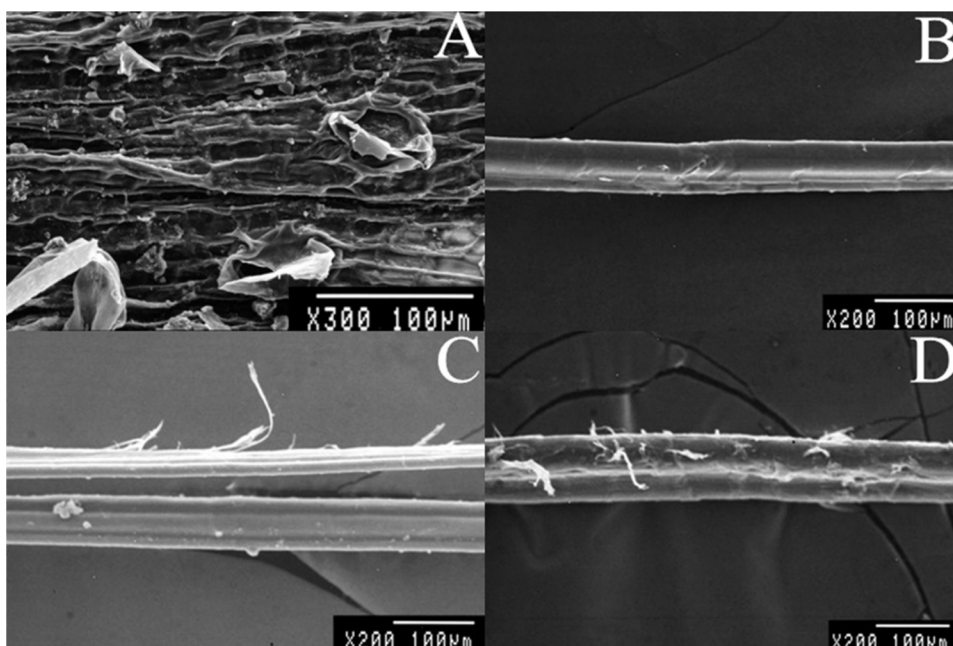


Fig. 1. SEM images of (A) raw ramie bast; (B) ramie fibers treated with traditional alkali degumming; (C) ramie fibers treated with STEX only; (D) ramie fibers treated with STEX-SP degumming.

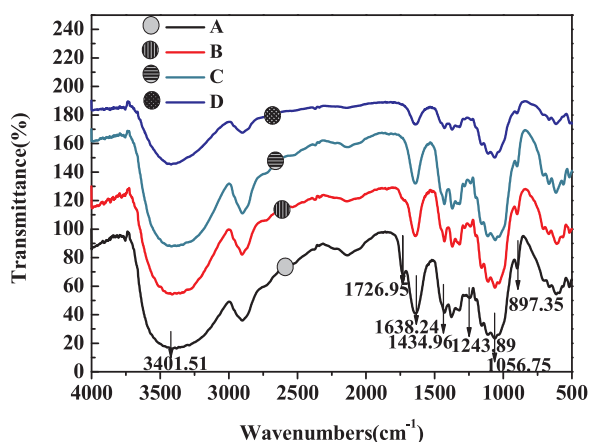


Fig. 2. FT-IR spectra of (A) raw ramie bast; (B) ramie fibers treated with traditional alkali degumming; (C) ramie fibers treated with STEX only; (D) ramie fibers treated with STEX-SP degumming.

more than one week before degumming. All chemicals used in the treatments were analytical pure and used without further purification. All the experiments were conducted in triplicates in this study.

## 2.2. Traditional alkali degumming

Ramie (30 g) and NaOH (concentration 1%, liquid-to-solid ratio (LSR) 20:1) were boiled with additives for 1 h twice. The additives include 3% hydrogen peroxide, 3% NaSiO<sub>3</sub>, 2% anhydrous Na<sub>2</sub>SO<sub>4</sub>, 3% carbamide and 3% Sodium polyphosphate.

## 2.3. STEX followed by SP soak (STEX-SP) treatment

The STEX-SP treatment includes the following three steps:

Peracetic acid (PAA) presoak: Ramie (30 g) and PAA (concentration 2%, LSR 12:1, pH 5.0) were soaked at 55 °C for 10 min. The PAA solution could be reused for 6–8 batches.

STEX treatment: The STEX treatment on presoaked ramie was conducted at 0.5 MPa for 5 min.

SP soak: Pretreated ramie, SP (concentration 2%, LSR 12:1) and additives reacted at 90 ± 5 °C for 1 h. The additives include 3% hydrogen peroxide, 3% NaSiO<sub>3</sub> and 2% Sodium polyphosphate.

## 2.4. Degumming evaluation

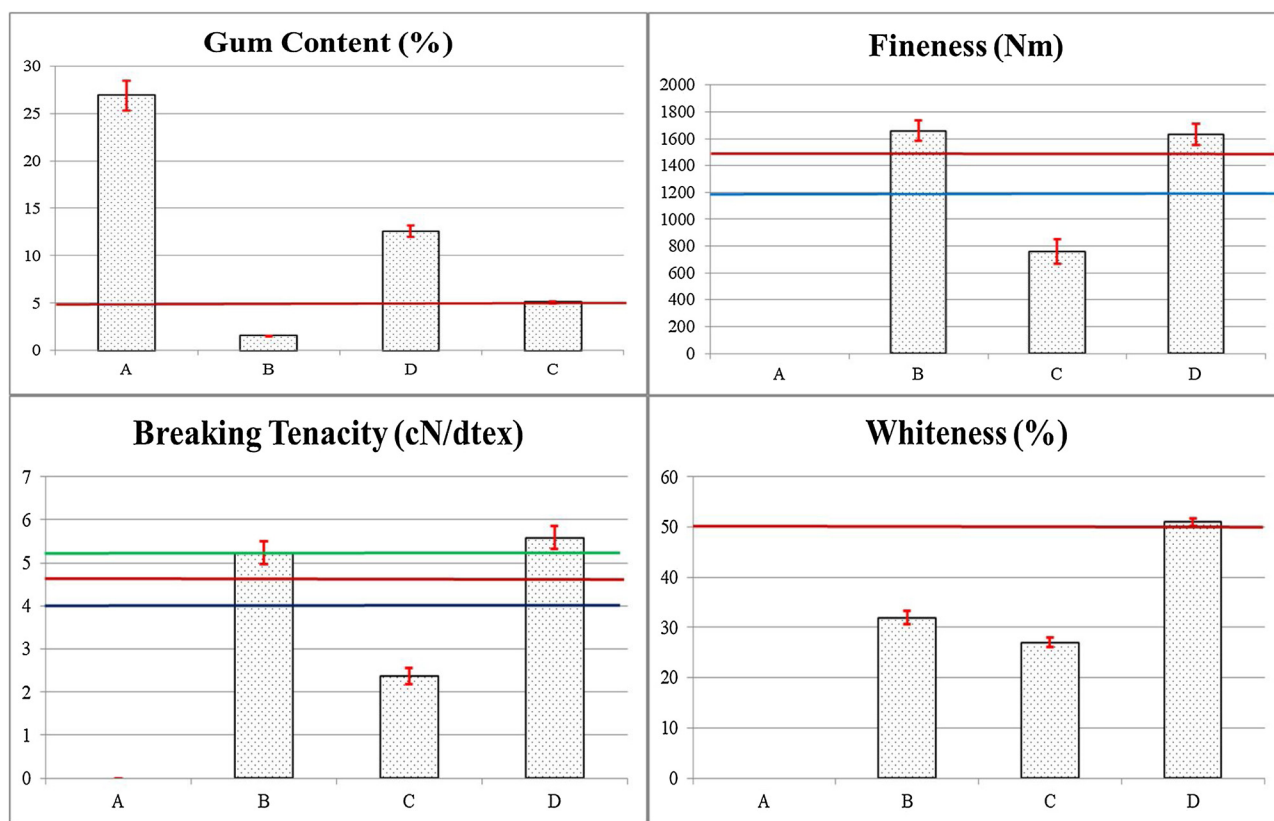
The ramie fibers at different stages of degumming processes were characterized with SEM (JSM-840) and FT-IR (Nicolet iS50). The gum content (GB 5889-86, Jiang and Shao, 1986b), fiber fineness (GB 5884-86, Jiang and Shao, 1986a), breaking strength (GB 5886-86, Jiang and Shao, 1986c) and the whiteness (GB/T 5885-1986, Jiang and Shao, 1986d) were measured based on Chinese national standards.

The COD of the degumming waste was analyzed based on HJ 828-2017 (China National Environmental Monitoring Centre, 2017), the pH value was measured with a pH meter (Leici PHS-25), and the colority was determined by the dilute method, which diluted the degummed water to the level that color was invisible, and recorded the dilute ratio.

## 3. Results and discussion

### 3.1. Surface morphology of ramie fiber

SEM images of ramie fibers presented a clear view on degumming capability. The raw ramie bast has a rough and coarse surface with no fiber exposed (Fig. 1A). After different degumming process, the gummy materials in ramie bast were dramatically destroyed and separated, which resulted in several types of fibers. It was found that the traditional alkali treatment showed the strongest degumming ability, single fibers with clean and smooth surface could be produced using the method (Fig. 1B). However, the steam explosion treatment only had the strength to yield fiber bundles (two or more single fibers together); there are still some gummy materials on and inside the fiber bundles (Fig. 1C). Then, after the SP soaking, gummy materials were significantly reduced and single fibers were revealed, only a little gummy materials (white pieces) can be seen on the surface of the fiber (Fig. 1D). In summary, the new method had comparable degumming efficiency to the traditional treatment.



**Fig. 3.** Physical properties of (A) raw ramie bast; (B) ramie fibers treated with traditional alkali degumming; (C) ramie fibers treated with STEX only; (D) ramie fibers treated with STEX-SP degumming.

(Note: The green lines, red lines and blue lines are the third-class, second-class and first class requirements by Chinese national standard “GB/T 20793-2006” for refined dried-ramie fiber, respectively)

**Table 1**  
Chemical usage for treating 1 kg of raw ramie.

Regents	Traditional degumming (SLR1:20)		STEX-SP(SLR1:12)	
	Concentration	Chemical usage	Concentration	Chemical usage
NaOH	1% × 2	0.40 kg	0	0
H <sub>2</sub> O <sub>2</sub>	3%	0.60 kg	3%	0.36 kg
Na <sub>2</sub> SiO <sub>3</sub>	3%	0.60 kg	2%	0.24 kg
Na <sub>2</sub> SO <sub>3</sub>	2%	0.40 kg	0	0
Carbamide	3%	0.60 kg	0	0
Sodium polyphosphate	3%	0.60 kg	2%	0.24 kg
Sodium percarbonate	0	0	6%	0.72 kg
Peracetic acid	0	0	2%/(6-8)	0.03
Total		3.20 kg		1.59 kg

Note: Estimated chemical cost.

**Table 2**  
Characterization of degummed waste water.

Index	Traditional degumming	STEX-SP
COD value (mg/L)	7400 ± 117	4348 ± 71
pH	13.69 ± 0.4	10.91 ± 0.3
Colority (dilute ratio)	350 ± 10	200 ± 10

### 3.2. FT-IR analysis of ramie fiber

As shown in Fig. 2, the absorptions of the three cellulose

characteristic peaks such as 3402 cm<sup>-1</sup> (OH stretching), 1057 cm<sup>-1</sup> (C–O stretching) and 897 cm<sup>-1</sup> (β-(1, 4) glycosidic linkage) kept strong after the treatments, indicating that the cellulose in the bast was retained after the degumming processes. In contrast, the characteristic peaks at 1727 cm<sup>-1</sup> (C=O bending) and 1436 cm<sup>-1</sup> (CH<sub>2</sub> bending) for hemicellulose and 1244 cm<sup>-1</sup> (C–O bending) and 1636 cm<sup>-1</sup> (C=O stretching) for lignin were significantly reduced or even eliminated after the treatments, confirming that they were almost completely removed through the degumming processes. The FT-IR spectra demonstrate that both of the traditional and the new degumming methods were able to remove gummy materials from ramie bast and retain clean cellulose fibers.

### 3.3. Chemical and physical properties of ramie fiber

The gum content, fiber fineness, fiber breaking tenacity and whiteness are major indexes to evaluate the degumming quality in textile area. Comparable same or better values of the above indexes are essential for the new degumming method.

The gum content of the raw ramie bast was 25.9%. It reduced to 11.6% after the STEX treatment and below 5% after the SP soaking. The result met the requirement of Chinese national standard, and was close to that of the traditional alkali degumming process (Fig. 3A).

The average fiber fineness reached 780 Nm (equals to 12.82 dtex) after the STEX treatment and increased to above 1600 Nm (6.25 dtex) after the SP soak, which was very similar to that of the traditional method (Fig. 3B). Both of them met the second-class requirement of 1500 Nm by the Chinese national standard (Jiang and Shao, 1986a).

The initial breaking tenacity of STEX treated fibers was 2.4 cN/dex (Fig. 3C). After the SP soaking, the tenacity increased to 5.4 cN/dex. The result was better than that of the traditional method and notably

exceeded the first-class requirement of 4.5 cN/dex by the Chinese national standard.

Since there is a bleaching step after the traditional alkali degumming, the whiteness of ramie after degumming is not required by the Chinese national standard. However, the ramie fibers treated by the STEX-SP process were much whiter because the bleaching effects of SP and PAA used in the new method (Fig. 3D). The resulted fibers reached the second-class whiteness requirement of the Chinese national standard for refined dried-ramie fiber. The result suggests that no or much less bleaching is required for the fibers obtained from the new degumming process, resulting less environmental impact.

### 3.4. Economic feasibility and environmental impact

The STEX-SP method significantly reduced the chemical usage and avoided to use strong alkali in the degumming process. To degum 1 kg ramie, the chemical usage decreased from 3.2 kg for the traditional method to 1.59 kg for the new method (Table 1). Moreover, the COD of the degumming waste water was reduced from 7400 mg/L for the traditional method to 4348 mg/L for the new one (Table 2). When taking the decrease of the SLR (from 1:20 to 1:12) into account, the COD ratio of the waste water of the new and traditional methods was calculated to be  $\frac{4348 \times 12}{7400 \times 20} = 0.35$ . The pH of the waste water was 13.69 and 10.91 for the traditional and new methods, respectively. The waste water of the new method was much less basic. Moreover, the colority of the waste water reduced from 350 for the traditional method to 200 for the new one. The results strongly suggest that the new method is much cleaner and more environment-friendly than the traditional method.

## 4. Conclusion

The STEX-SP degumming process successfully removed most gum materials in ramie bast and resulted high quality ramie fiber. Compared to the traditional alkali degumming, the newly developed process was more economically feasible and eco-friendly. Less than 50% chemicals were used and only 35% of waste water COD was generated for the new method. The properties of the fiber from the new method were comparable to or better than those of the traditional method. The STEX-SP degumming process for ramie has bright potential for industry applications.

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## Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at <https://doi.org/10.1016/j.indcrop.2018.04.045>.

## References

- Angelini, L.G., Scalabrelli, M., Tavarini, S., Cinelli, P., Anguillesi, I., Lazzeri, A., 2015. Ramie fibers in a comparison between chemical and microbiological retting proposed for application in biocomposites. *Ind. Crop. Prod.* 75, 178–184.
- China National Environmental Monitoring Centre, 2017. Water Quality Determination of Chemical Oxygen Demand Dichromate Method, HJ 828-2017. Ministry and environmental protection of the People's Republic of China, Beijing.
- Gao, S., Han, G., Jiang, W., Zhang, Y., Zhang, X., 2015. Steam explosion and alkali-Oxygen combined effect for degumming of kenaf fiber. *BioResources* 10, 5476–5488.
- Hage, R., Lienke, A., 2006. Applications of transition-metal catalysts to textile and wood-pulp bleaching. *Angew. Chem. Int. Ed.* 45, 206–222.
- JC, V.W., 1964. Textile bleaching composition. Google Patents.
- Jiang, F., Shao, K., 1986a. Count Testing of Ramie Fiber, GB 5884-86. China Standard Press, Beijing, China.
- Jiang, F., Shao, K., 1986b. Quantative Analysis of Ramie Chemical Composition, GB-5889-86. China Standard Press, Beijing, China.
- Jiang, F., Shao, K., 1986c. Testing Method of Breaking Strength of Ramie Fiber GB 5886-86. China Standard Press, Beijing, China.
- Jiang, F., Shao, K., 1986d. Testing Method of Whiteness of Ramie Fiber GB 5885-86. China Standard Press, Beijing, China.
- Jiang, W., Han, G., Zhang, Y., Liu, S., Zhou, C., Song, Y., Zhang, X., Xia, Y., 2017a. Monitoring chemical changes on the surface of kenaf fiber during degumming process using infrared microspectroscopy. *Sci. Rep.* 7, 1240.
- Jiang, W., Han, G., Zhou, C., Gao, S., Zhang, Y., Li, M., Gong, Y., Via, B., 2017b. The degradation of lignin, cellulose, and hemicellulose in kenaf bast under different pressures using steam explosion treatment. *J. Wood Chem. Technol.* 1–10.
- Li, Z., Meng, C., Yu, C., 2015. Analysis of oxidized cellulose introduced into ramie fiber by oxidation degumming. *Text. Res. J.* 85, 2125–2135.
- Liu, G.L., Cui, Q.L., Yu, C.W., 2011. The application of peracetic acid and sodium percarbonate in green production processing of ramie. *Adv. Mater. Res.* 1423–1427 Trans Tech Publ.
- Liu, Z., Duan, S., Sun, Q., Peng, Y., Feng, X., Zheng, K., Hu, Z., Zhang, Y., 2012. A rapid process of ramie bio-degumming by *Pectobacterium* sp. CXJZU-120. *Text. Res. J.* 82, 1553–1559.
- Luan, M.-B., Liu, C.-C., Wang, X.-F., Xu, Y., Sun, Z.-M., Chen, J.-H., 2017. SSR markers associated with fiber yield traits in ramie (*Boehmeria nivea* L. Gaudich). *Ind. Crop. Prod.* 107, 439–445.
- Ni, J.-L., Zhu, A.-G., Wang, X.-F., Xu, Y., Sun, Z.-M., Chen, J.-H., Luan, M.-B., 2018. Genetic diversity and population structure of ramie (*Boehmeria nivea* L.). *Ind. Crop. Prod.* 115, 340–347.
- Yang, Z., Tan, Z., Li, F., Li, X., 2016. An effective method for the extraction and purification of chlorogenic acid from ramie (*Boehmeria nivea* L.) leaves using acidic ionic liquids. *Ind. Crop. Prod.* 89, 78–86.
- Zhang, X., Han, G., Zhang, Y., Wang, Q., Jiang, W., Gao, S., 2014. Degumming of kenaf fibers by combining steam explosion with ultrasonic treatment. *Sheng wu gong cheng xue bao = Chin. J. Biotechnol.* 30, 734–742.
- Zhang, X., Han, G., Jiang, W., Zhang, Y., Li, X., Li, M., 2016. Effect of steam pressure on chemical and structural properties of kenaf fibers during steam explosion process. *Bioresources* 11, 6590–6599.
- Zheng, Y., Zhang, Z., Luo, Z.-W., 1988. Optimizing the complex formulation of boiling-off liquor for ramie chemical degumming. *Text. Res. J.* 58, 663–666.
- Zheng, L., Du, Y., Zhang, J., 2001. Degumming of ramie fibers by alkalophilic bacteria and their polysaccharide-degrading enzymes. *Bioresour. Technol.* 78, 89–94.