A facile degumming method of kenaf fibers using deep eutectic solution

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An eco-friendly and effective degumming method needed to be developed for producing kenaf bast fibers. This study devised a novel deep eutectic solvent (DES) method coupled with microwave and alkaline-ultrasonic treatment. The novel method could effectively remove the gummy matters, providing a smooth and clean fiber surface. The properties of the fibers were assessed including chemical compositions, surface structure, crystallinity index (66.68%) and thermal properties. The residual gum content (9.419%), fiber fineness (4.125tex), breaking tenacity (13.650 cn/tex) of the refined dry fibers produced by the novel treatment were comparable with the fibers produced by the traditional two-step alkali boiling method. Besides, the novel method could reduce the usage of chemical, water and time by 48.9%, by 66.7% and 66.8%, respectively. These results revealed that the novel combined DES pretreatment is a practical and feasible pretreatment method for kenaf bast degumming, demonstrating its facile, green energy-saving and fast properties in the degumming process.

Keywords: kenaf fiber; natural fibers; DES; microwave; ultrasonic

1. Introduction

The development and utilization of natural cellulose fibers has become a significant research area in the world for their economic and ecological advantages (Jiang, W et al. 2017; Kalita, S and Saikia, S. R 2019). Kenaf is an important bast fiber crop, which is drought-tolerant, barren-resistant, salt-tolerant, easy to cultivate, and high in fiber yield (Akil, H. M. and Omar, M. F 2011). Recently, extensive researches have been carried out on the development and utilization of the kenaf fibers, such as adsorption of contaminant, building materials, composites reinforcement materials, etc (Ramesh, M 2016). Besides, the high fiber yield makes the kenaf fiber a potential material for the textile industry. However, for natural bast fibers such as kenaf bast, gummy matters including pectin, hemicellulose and lignin in the raw kenaf bast must be removed before used in textiles (Shengchuan, G and Guangting, H 2015).

To remove gummy matters of the traditional plant bast fibers, the alkali boiling process is most commonly used. This process requires two boiling procedures with large amounts of sodium hydroxide, which consumes extensive costs and causes serious environmental pollution (Zheng et al, 2009). Therefore, it is necessary to develop a greener and faster process for fiber preparation.

In the last two decades, deep eutectic solvents (DESs), classified as a new generation of ionic liquids, has attracted much interest because of its lower cost and more biodegradable property (Hou Y.C et al. 2018, Loow, Y.L et al. 2019). The DES is a blend of at least two chemicals, i.e. hydrogen bond donor and hydrogen bond acceptor, in a

specific molar ratio (Hou Y.C et al, 2018). The DESs have been already utilized in many fields, including catalysis, organic synthesis, dissolution, extraction processes, electrochemistry etc (Yu, W and Song et al, 2019, Loow, Y et al, 2017). Besides, The DESs have been applied in pretreatment of biomass cellulose fibers. The DESs can extract lignin without change of cellulosic structure (Lim, W. L. et al, 2019), which is beneficial to produce cellulose fibers. On the other hand, microwave and ultrasonic treatment have been widely used in biomass energy and degumming process. These treatment methods exhibited high efficiency in modification of biomasses (Klein, Jalma, Maria, de, et al. 2018). However, there is few study combining the different methods into one integrated process to efficiently manufacture natural fibers.

Thus, the aim of this study was to promote a greener and faster method combined DES pretreatment with microwave pretreatment and ultrasonic pretreatment for manufacture of kenaf fiber. The most common DES composed of chloride and urea (molar ratio 1:2) was used in this research. The physical mechanical properties, Fourier transform infrared (FTIR) spectra, scanning electron microscopy (SEM) morphology, X-ray diffraction (XRD) spectra, and thermal gravimetric analysis (TG) of fibers samples were conducted to characterize the fiber properties. In addition, the consumption of chemicals and water, and time-saving properties of different methods were evaluated and compared.

2.Materials and Methods

[Figure 1. near here]

2.1. Materials

Raw kenaf bast samples were cultivated from the Aksu Prefecture of Xinjiang, China. The kenaf bast were manually stripped from the core, then dried and stored. The original dry kenaf bast was manually cut into segments with a suitable length of 10 cm, well mixed, and randomly selected. The drugs used in the experiment are of analytical grade.

2.2. Experimental Methods

2.2.1. Manufacture of Kenaf Fibers

Synthesis of DES: 140 g Choline chloride and 120 g urea (molar ratio 1:2) was mixed, and magnetically stirred in a closed glass bottle for two hours at 80℃ to obtain a transparent and homogeneous liquid. Choline chloride and urea were dried under vacuum condition for 24 h at 60℃ before use.

DES treatment with microwave: The kenaf bast fiber (5 g) was treated by the DES prepared above (100 mL, Solid Liquid Ratio 1:20) for 20 mins using a microwave digestion apparatus (CEM Mars 6 Classic, USA) at a power of 600 W and 150℃ (Sirvio and Juho Antti, et al, 2017). After the reaction, the kenaf fiber was washed by deionized water, and dried in the air oven. The dried kenaf fiber was stored for following treatments and further analysis.

DES treatment with microwave and alkaline treatment with ultrasonic: The kenaf fiber (5 g) was firstly treated by the DES with microwave as previously mentioned. Then, the pretreated kenaf fiber was put in an alkali-cooking solution (100 mL, 1% (wt/wt) NaOH

and 3% (wt/wt) H_2O_2 (30%)) for 1 h in the Ultrasonic generator (Jining xinxin ultrasonic electronic equipment Co. Lid, China) at a power of 600 W and 50℃. Then the kenaf fibers were washed by deionized water and dried at 105℃ in the air oven for 6 h. The dried kenaf fiber was stored for following treatments and further analysis.

Two-step alkali boiling method: According to the described method of traditional chemical degumming process, the kenaf bast was firstly soaked by 98% concentrated sulfuric acid at 1% (wt/wt) solid loading and 50℃ for 1 h , and then washed by deionized water until neutrality was achieved. In the first stage of the two-step alkali boiling method, the 5 g sulfuric acid pretreated kenaf bast was boiled by 100 mL 1% (wt/wt) sodium hydroxide solution for 1 h. After that, the fibers were collected and washed. In the second stage, the washed kenaf fibers were subjected to alkali boiling solution for 1 h, and then washed with deionized water to neutrality. The kenaf fibers were collected and dried. The alkali boiling solution contained 1% (wt/wt) sodium hydroxide, 3% (wt/wt) hydrogen peroxide (30% wt/wt), 3% (wt/wt) sodium silicate, 2% (wt/wt) sodium sulfite, 3% (wt/wt) urea, and 3% (wt/wt) sodium tripolyphosphate, which was mixed and stirred until transparent (Song and Jiang, W. et al, 2017).

Through the above treatment methods, microwave DES pretreatment kenaf fiber, microwave and ultrasonic combined treatment kenaf fiber, traditionally chemical degumming method kenaf fiber were prepared, respectively. Because the dosage of chemical reagents and the reaction time used in the processes was in connection with the

evaluation of their cost and environment impact (Meng,C.R. Yang, J.P. et al, 2018), they were calculated and compared.

2.2.2. Morphology and SEM Images of Kenaf Fibers

The longitudinal features and surface morphology of the kenaf fibers were examined using a scanning electronic microscopy (SEM, JEM-1200EX). After coated with gold, samples were observed and photographed with the microscope operating at voltage of 15 kV.

2.2.3. Analysis of Chemical Composition of Kenaf Fibers

The samples were processed by following the China National Standard (GB5889--86) and the American Laboratory Analytical Procedure (NREL/TP-510-42618) to determine major chemical compositions, including lignin, hemicellulose, and cellulose (Song,Y. and Han,G et al, 2017). Prior to testing, each specimen was conditioned in a constant temperature and humidity environment (20 ± 2℃ and 65 ± 2% relative humidity) for 24 h. All experiments were performed independently in triplicate and the results given are the means of the results.

2.2.4. Fourier Transform Infrared Spectroscopy Imaging Analysis

The FTIR spectra of the kenaf fibers samples were collected to analyze the changes of chemical structure with a Bruker Spectrophotometer (NICOLET 5700). The transmission method with KBr discs was used in the test. The spectra were recorded in the wavenumber range of 500–4000 cm^{-1} and a resolution of 2 cm^{-1}.

2.2.5. Physical Properties of Fibers Analysis

Fiber fineness affects yarn properties such as yarn strength and strand uniformity (El Achaby, M. Kassab, Z et al. 2018). This experiment was referred to the middle section cutting weighing method of Chinese standard GB/T 5884-1986, The fineness of the kenaf fiber was calculated using the fixed weight and the fiber length. The equation was listed following:

$$N_t = \frac{G}{10nL_c} \quad (1)$$

Nt: the fineness of fibers (The mass of 1 km long fiber at the specified moisture regain rate) (tex); G: the cutting middle section of fibers weight (g); n: the numbers of fibers; Lc: the cutting length (cm)

According to the Chinese standard GB/T 5882-1986, the breaking strength and elongation at break of kenaf fiber were tested by a single fiber strength tester (Favimat-Airobot, Germany).

2.2.6. Crystalline Analysis

X-ray diffraction (X-RD) test was carried out to measure the crystallinity index (CrI) of cellulose samples. The samples were scanned by a Bruker D8 Advanced X-ray diffractometer under a voltage of 40 kV and a current of 50 mA with a scanning 2°/min in the range of 5° to 50°. The crystallinity degree of the kenaf fiber samples was calculated according to the following equation of traditional Segel method (Nagaraja, B. Ganesha, P.

et al, 2019). The maximum intensity of the natural cellulose crystallinity lattice (002) diffraction was taken at about 2θ = 22.6° and the maximum intensity of the amorphous phase was taken at about 2θ = 18°.

$$CrI = [\frac{I_{002} - I_{amorph}}{I_{002}}] \times 100\% \quad (2)$$

I_{002}: the maximum peak intensity at lattice diffraction (200); Iamorph: the intensity collected at 2θ=18°, representing the amorphous area in native cellulose.

2.2.7. TG Analysis

In order to investigate the change of thermodynamic properties of the kenaf fibers, thermogravimetric analysis was carried out using a TGA (Pyris, Perkin Elmer, USA) from room temperature to 600°C under nitrogen atmosphere with a constant heating rate of 10 °C/min. TGA analysis was conducted using approximate 10 mg samples.

3. Results and discussion

3.1. Morphology and SEM Images of Kenaf Fibers Analysis

As shown in Figure 1, the kenaf fibers were gradually separated from the raw bast after degumming treatment. The morphology of kenaf fibers treated with different methods was analyzed by SEM to gain greater insight into the surface structure. As presented in Figure 2, the surface morphology was evidently changed after the degumming treatments. The raw kenaf bast showed a rough surface with many gummy matters which cemented the single fibers together (Figure 2(a)), while the gummy matters between single fiber were partially removed after DES treatment with microwave (Figure 2(b)). Most of the impurities were

clearly removed by DES treatment combined with microwave and alkali-ultrasound (Figure 2(c)). The fibers prepared by this method were similar to the single fiber prepared with the traditional chemical two-step alkali boiling method (Figure 2(d)). The two methods not only efficiently removed gummy matters, but also preserved the inherent morphological structure of kenaf. The single fibers exhibited a smooth and clean surface, which was consistent with the chemical composition analysis and previous research (Jiang Wei, Song Yan et al. 2018). The result indicated that the ultrasonic technology could improve affinity between the fiber surface and the alkali solution.

[Figure 2. near here]

3.2. Chemical Composition Analysis

Cellulose, lignin, and hemicellulose are the three primary components of kenaf bast and native plants, which accounts for about 85%-90% of the raw bast samples (Yan, S. Guangting, H. et al, 2018). The residual gum rate (mainly lignin and hemicellulose) reflected the quality of the prepared fiber to some extent. Figure 3 summarized the residual gum rate and chemical composition (cellulose, lignin, hemicellulose) of the raw kenaf bast, microwave-DES pre-degummed fibers, and microwave-ultrasonic-DES pre-degummed kenaf fibers, chemical degummed kenaf fibers. As shown in Table 1, although microwave assistant DES treatment can significantly reduce the residual gum rate in kenaf bast (from 28.5% to 18.1%), a large amount (18.1%) of gummy matters was still existed in the bundle fibers, which hindered application of the fibers in textile production (Xiaojing, B. and Lijun, Q et al, 2014). In order to further remove the residual gum, ultrasonic alkaline treatment

was conducted, which reduced the gum content from 18.1% to 9.4% with removal of 2.9% hemicellulose and 8.9% lignin. This result was consistent with previous work that lignin was readily removed by alkali (Reddy, N and Yang, Y.Q et al, 2018). At the same time, the cellulose content was increased from 76.4% to 88.2%. Although the gum content of the combined DES with microwaves and ultrasonic method (9.4%) was still higher than that of the"two-step" chemical alkali boiling method (7.4%), it may not affect the textile fiber quality (Zhang, X et al, 2014).

[Table 1. near here]

3.3. Fourier Transform Infrared Spectroscopy Analysis

The FTIR spectra were conducted to analyze the main chemical compositions changes (residual gum, hemicellulose, lignin, cellulose) of the samples. 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 β-1,4 glucosidic linkages. The bands at 1425 cm^{-1} were assigned to an ester bond in hemicellulose (Iman Dindarloo Inaloo et al, 2019). As shown in the Figure 3, the peaks at 1425 cm^{-1} were weakened after the combined DES with microwave and alkaline-ultrasonic method and the "two-step" chemical alkali boiling method, which was consistent with the gradual elimination of hemicellulose and the increase in cellulose content as described in Figure 3. On the other hand, the peaks related to hemicellulose were weakened or vanished after degumming treatment, verifying hemicellulose removal during treatment. The bands at 1638 cm^{-1}, 1543 cm^{-1} and 1022 cm^{-1} were all reduced, demonstrating the loss of lignin content during the degumming processes (Hu, S et al, 2008). These results were in accordance with the previous reports (Song, Y, 2019, Kallel, F,

2016). These results indicated that the combined DES with microwave and alkaline-ultrasonic method was an excellent degumming process.

[Figure 3. near here]

3.4. Physical Properties Analysis of Fibers

Physical properties of the fibers were analyzed, including elongation, average fineness, breaking tenacity. As shown in the Figure 5, the elongation of kenaf fibers subjected to the DES treatments (2.760%~2.795%) didn't change much compared with the kenaf fiber produced by the two-step chemical process (2.750%), indicating the elongation of the fibers was not greatly influenced by the removal of the hemicellulose and lignin. However, the fineness of the kenaf fibers of the microwave-DES pretreatment (8.264 tex) was higher than the fibers of the combined DES with microwave and alkaline-ultrasonic pretreatment (4.125 tex) and the fibers of the two-step chemical alkali boiling method (3.215 tex) (Figure 4). This was because the residual gummy matters in the fibers of the microwave-DES treatment could agglomerate the fibers together (Ying-Hua, T et al, 2010). Furthermore, the breaking tenacity of the fibers prepared by the microwave-DES pretreatment (10.303 cN/tex) were weaker than the fibers produced by the combined DES combined DES with microwave and alkaline-ultrasonic pretreatment (13.650 cN/tex) and the fibers of the "two-step" chemical alkali boiling method (18.893 cN/tex). The above results combined with the results in Figure 2 and Figure 4 confirmed that treatment of plant bast fiber by microwave-DES could disperse the single kenaf fibers at a certain extent, while the alkali with the ultrasonic could further disperse the single fibers. Thus, the combined DES with microwave and alkaline-ultrasonic pretreatment was effective in producing fibers with high

qualities, which was comparable to the traditional two-step alkali degumming method.

[Figure 4. near here]

3.5. Crystalline Analysis

The X-ray diffraction patterns of the samples were presented in Figure 5. 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 (Yu, W et al, 2019). The peak intensity of the 200 lattice planes increased after degumming treatment, indicating crystallinity was increased. Based on the calculation method of Segal, the crystallinity index of the raw kenaf bast, the kenaf fibers of the microwave-DES pretreatment, the kenaf fibers of the combined DES with microwave and alkaline-ultrasonic pretreatment, the kenaf fibers of the chemical degumming method increased from 60.52% to 64.06%, 66.68% and 72.93%, respectively. As shown in the chemical component analysis, the non-cellulosic components such as hemicellulose and lignin were extracted by DES and further removed by alkaline-ultrasonic treatment. The dissolution of the amorphous cellulose and removal of some hemicelluloses should contribute to the increased crystallinity (Wei, J et al, 2017).

[Figure 5. near here]

3.6. TG Analysis

The TG analysis was carried out to determine the thermal degradation characteristics of fibers. As seen from the TG (Figure 6a) and Differential thermal gravity (DTG, Figure 6b) curves, peaks at about 80℃ and 330–360℃ were attributed to the evaporation of water and cellulose degradation, respectively. Compared with the raw kenaf bast (335°C), the

degradation temperatures of the kenaf fibers of microwave-DES pretreatment (361°C), and the kenaf fibers of the combined DES with microwave and alkaline-ultrasonic pretreatment (352°C) were increased, which was attributed to the removal of non-cellulosic materials. The degradation temperature of the kenaf fibers of the microwave-DES pretreatment (361°C) was slightly higher than kenaf fibers of the chemical degumming method (352°C), which was attributed to the partial decomposition of non-cellulosic materials and amorphous cellulose during the alkaline treatment. However, the subtle decrease in thermal stability would not affect the quality of textile fibers.

[Figure 6. near here]

3.7. The Consumption of Time and Chemical Reagents for Preparation Kenaf Fiber Analysis

The chemical usage, and time cost and water consumption for the preparation of 1 kg raw kenaf bast were shown in Table 2 and Table 3, respectively. The chemical usage was significantly reduced by the combined DES with microwave and alkaline-ultrasonic pretreatment. The chemical usage decreased from 3.6 kg for the traditional method to 1.84 kg for the new method (Table 2). Therefore, the serious pollution caused by the alkali would be dramatically reduced. Moreover, because the components of DES reagent could be recycled and reused (Li A.L et al, 2018, Guo Z et al, 2019), contamination burden could be further reduced. Furthermore, the times cost was reduced from 4.0 h to 1.33 h, which greatly improved the production efficiency. The results strongly suggested that the novel method was much effective and greener than the traditional method.

[Table 2. near here]

[Table 3. near here]

4. Conclusion

The kenaf fiber was prepared by a more rapid and greener method using DES combined with microwave and alkaline-ultrasonic. The characteristics of the fibers were analyzed by SEM, chemical composition analysis, FTIR, XRD, TGA. After the novel treatment of the kenaf bast, the components such as gummy part and hemicellulose, lignin were largely removed, which led to cleaner micro-surface of the kenaf fibers, and increased cellulose crystallinity and thermal stability. These properties were comparable with the fibers produced by the traditional degumming method. Besides, both the consumption of water and the chemicals were decreased compared with traditional chemical degumming. Thus, this work provided a novel green degumming method for processing the plant bast fibers, and provided a new way for the utilization of DES.

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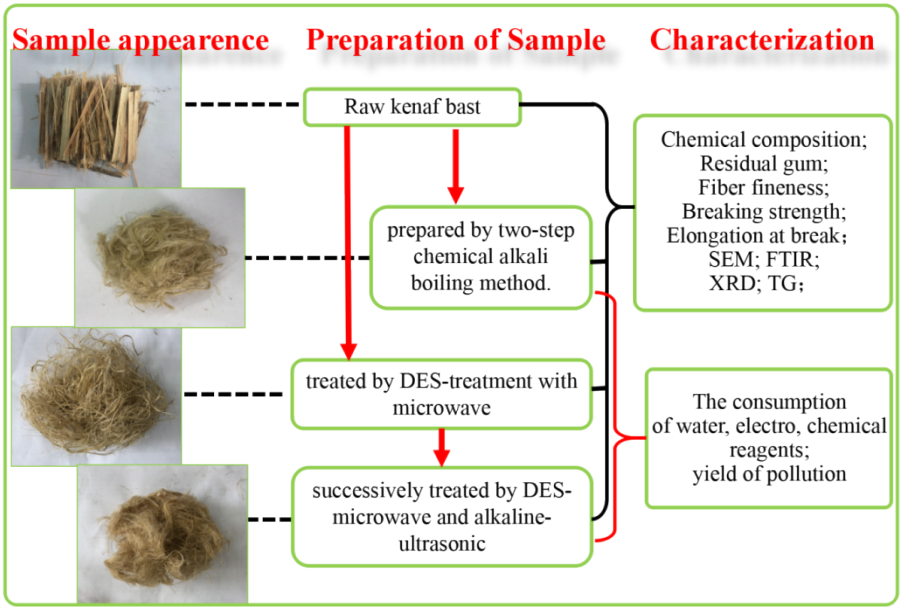

Figure 1. Schematic diagram for these experiment.

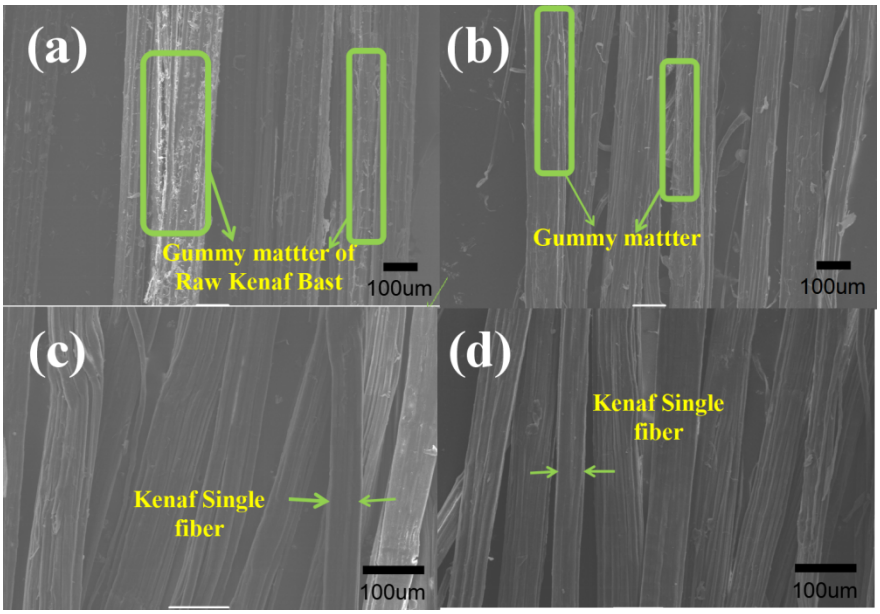

Figure 2. SEM images of the kenaf fibers samples. (a): raw kenaf bast; (b): microwave-DES pre-degummed kenaf fibers; (c): microwave-DES-ultrasonic degummed kenaf fibers; (d): chemical degummed kenaf fibers.

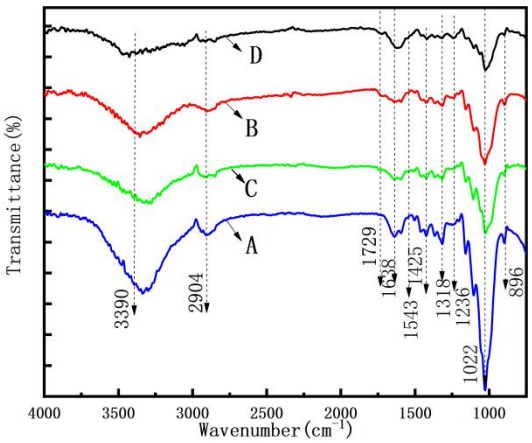

Figure 3. The FTIR spectra curves of A: raw kenaf bast; A: raw kenaf bast; B: microwave-DES pre-degummed kenaf fibers; C: microwave-DES-ultrasonic degummed kenaf fibers; D: chemical degummed kenaf fibers.

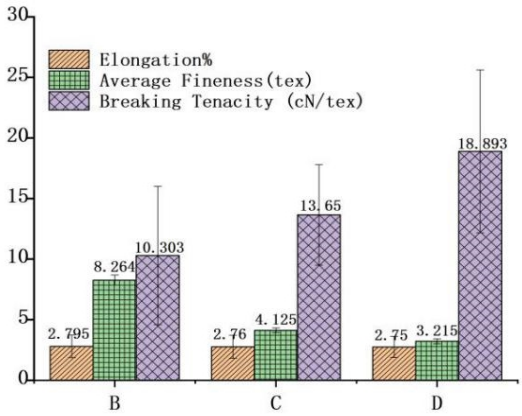

Figure 4. The breaking elongation, average fineness, breaking tenacity (cN/tex) of A: raw kenaf bast; B: microwave-DES pre-degummed kenaf fibers; C: microwave-DES-ultrasonic degummed kenaf fibers; D: chemical degummed kenaf fibers.

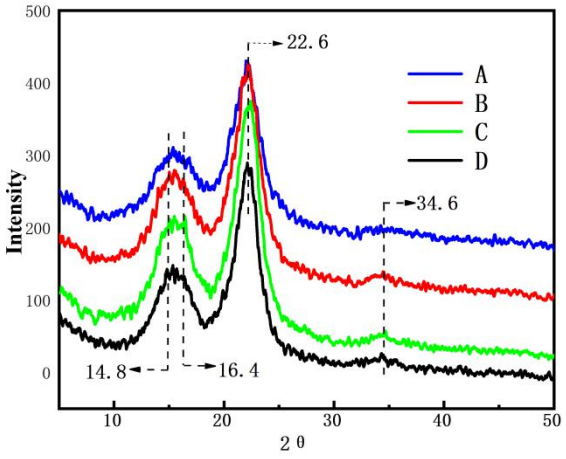

Figure 5. the XRD curves of A: the raw kenaf bast; A: raw kenaf bast; B: microwave-DES pre-degummed kenaf fibers; C: microwave-DES-ultrasonic degummed kenaf fibers; D: chemical degummed kenaf fibers.

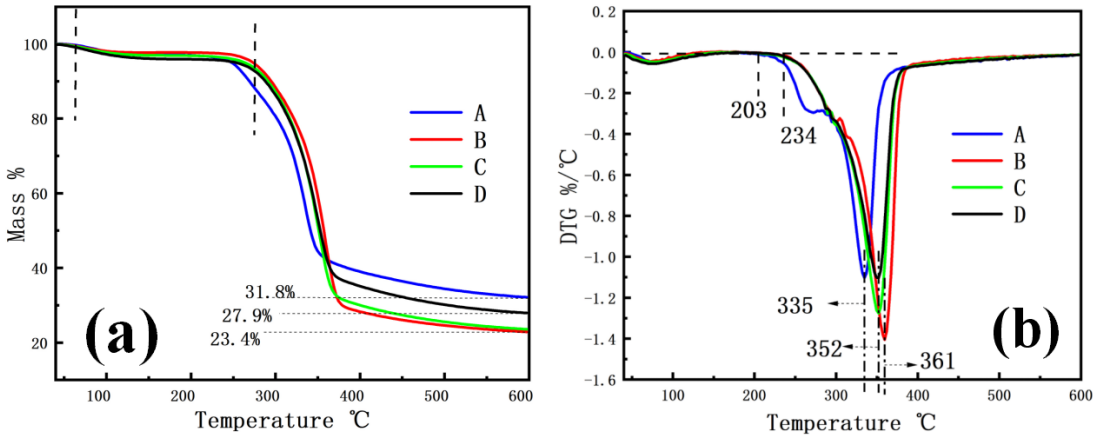

Figure 6. The TG (a) and DTG (b) curves of A: the raw kenaf bast; A: raw kenaf bast; B: microwave-DES pre-degummed kenaf fibers; C: microwave-DES-ultrasonic degummed kenaf fibers; D: chemical degummed kenaf fibers.

Table 1. The residual gum rate and main chemical compositions contents (hemicellulose, lignin, cellulose) of raw kenaf bast;microwave-DES pre-degummed kenaf fibers; microwave-DES-ultrasonic degummed kenaf fibers; chemical degummed kenaf fibers.

Treatments	Residual gum (%)	Hemicellulose (%)	Lignin (%)	Cellulose (%)
raw kenaf bast	28.47	15.75	15.7	50.65
microwave-DES pre-degummed kenaf fibers	18.11	12.18	11.46	76.40
microwave-DES -ultrasonic degummed kenaf fibers	9.42	9.27	2.57	88.17
chemical degummed kenaf fibers.	7.38	7.38	2.87	89.45

Table 2. Chemical reagent usage for the preparation of 1kg raw kenaf bast.

Regents	Two-step alkali boiling		DES(Microwave)+ lye(ultrasonic)	
	Concen-tration	Chemical usage(kg)	Concen-tration	Chemical usage(kg)
NaOH	1%+1%	0.40	1%	0.20
H_2SO_4	1%	0.20	-	-
H_2O_2	3%	0.60	3%	0.60
Na_2SiO_3	3%	0.60	-	-
Sodium sulfite	2%	0.60	-	-
Sodium tripolyphosphate	3%	0.60	-	-
urea	3%	0.60	46%	0.48
Choline chloride	-	-	54%	0.56
Total	-	3.6	-	1.84

Table 3. The consumption of time and water for the preparation of 1kg raw kenaf bast.

Methods	Two-step alkali boiling	DES(Microwave)+

		lye(ultrasonic)
The consumption of times(h)	1+1+2=4.0h	1/3+1=1.33h
The consumption of water(kg)	20*3=60kg	0+20=20kg
Yield of pollution	0.2kg gummy matter in 60kg water	0.09kg gummy matter in 20kg water