Video Article

Extraction of Ramie Fiber in Alkali Hydrogen Peroxide System Supported by Controlled-release Alkali Source

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Abstract

This protocol demonstrates a method for ramie fiber extraction by scouring raw ramie in an alkali hydrogen peroxide system supported by a controlled-release alkali source. The fiber extracted from ramie is a type of textile material of great importance. In previous studies, ramie fiber was extracted in an alkali hydrogen peroxide system supported only by sodium hydroxide.However, due to the strong alkalinity of sodium hydroxide, the oxidation reaction speed of hydrogen peroxide was difficult to control and thus resulted in great damage to the treated fiber. In this protocol, a controlled-release alkali source, which is composed of sodium hydroxide and magnesium hydroxide, is used to provide an alkali condition and buffer the pH value of the alkali hydrogen peroxidesystem. The substitution rate of magnesium hydroxide can adjust the pH value of the hydrogen peroxide system and has great influence on the fiber properties. The pH value and oxidation-reduction potential (ORP) value, which represents the oxidation ability of alkali hydrogen peroxide system, were monitored using a pH meter and ORP meter, respectively. The residual hydrogen peroxide content in the alkali hydrogen peroxide system during the extraction process and the chemical oxygen demand (COD) value of wastewater after fiber extraction are tested by $KMnO_4$ titration method. The yield of fiber is measured using a precision electronic balance, and residual gums of fiber are tested by a chemical analysis method. The polymerization degree (PD value) of fiber is tested by an intrinsic viscosity method using the Ubbelohde viscometer. The tensile property of fiber, including tenacity, elongation, and rupture, is measured using a fiber strength instrument. Fourier transform infrared spectroscopy and X-ray diffraction are used to characterize the functional groups and crystal property of fiber. This protocol proves that the controlled-release alkali source can improve the properties of the fiber extracted in an alkali hydrogen peroxide system.

Video Link

The video component of this article can be found at https://www.jove.com/video/56461/

Introduction

Ramie, commonly known as 'China grass' is a perennial herb whose fiber can be used as an excellent material for the textile industry[1,2]. It is one of the main economic crops native to China; the production of ramie in China has accounted for more than 90% of the total yield in the world[1,2]. Ramie fiber is one of the strongest and longest plant fibers, lustrous with an almost silky appearance[3,4]. The long length of ramie fiber make it suitable for single fiber spinning, which is seldom seen in bast fiber. The textile made from ramie fiber possesses many excellent properties, such as coolness, antibacterial, excellent thermal conductivity, ventilation, *etc.*[3,4]

Cellulose is the main component of ramie fibers, and the other components in ramie, such as pectin, lignin, water soluble materials, are defined as gums[5,6]. Ramie fiber can be extracted by dissolving the gums in solution containing chemical reagents, in a process defined as degumming[5,6]. Mainly two approaches of ramie fiber extraction exist: chemical degumming and bio-degumming. The energy consumption, time consumption, and COD value of degumming wastewater in traditional chemical degumming is rather high, as cellulose fiber is extracted by scouring raw ramie in concentrated NaOH under high pressure for 6 to 8 h[7,8]. Alternatively, bio-degumming is an eco-friendly option for ramie fiber extraction. Nevertheless, the harsh reaction condition and sophisticated equipment inhibit its further industrial application[9,10]. Therefore, oxidation degumming with alkali hydrogen peroxide presents a valuable and alternative application to focus on, for it requires shorter degumming time and lower degumming temperature[11,12]. However, due to the strong oxidation ability of the peroxides, substantial cellulose degradation may occur during the degumming process, which can cause great damage to fiber properties[13,14]. This is the biggest drawback of alkali peroxide oxidation degumming of ramie.

In previous studies, ramie fiber was extracted in an alkali hydrogen peroxide system supported only by sodium hydroxide[15]. However, due to the strong alkalinity of sodium hydroxide, the oxidation reaction speed of hydrogen peroxide was difficult to control and thus resulted in great damage to the treated fiber[7]. To improve the properties of ramie fiber, a controlled-release alkali source, which is composed of sodium hydroxide and magnesium hydroxide, is used in this study to offer an alkali condition and buffer the pH value of alkali hydrogen peroxide system[16,17].

The rationale behind this technology can be described as follows. Magnesium hydroxide is slightly soluble in distilled water, and it can dissolve gradually into the degumming solution with the consumption of OH^- and keep the pH value and thus oxidation ability of degumming solution in an appropriate range[18]. The substitution rate (SR) of magnesium hydroxide is defined as the mole proportion of NaOH replaced by magnesium hydroxide under the total alkali dosage of 10%, and the substitution rate can be calculated by the following equation. Moreover, Mg^{2+} can prevent cellulose degradation caused by over oxidation[19,20].

$$SR = \frac{M_2 \times 40 \times 2}{M_1 \times 58}$$

Here, M_2 (g) is the weight of $Mg(OH)_2$, M_1 (g) is the weight of NaOH, 40 is the molecular weight of NaOH, 58 is the molecular weight of $Mg(OH)_2$, 2 is the number of OHs in $Mg(OH)_2$, and SR is the substitution rate.

The technology of this protocol can be extended to the extracting, bleaching, and modifying of plant materials in an alkali hydrogen peroxide system. However, it must be noted that the selection of pH value and reaction temperature of the alkali hydrogen peroxide system is key for this technology[21]. The pH value of the alkali hydrogen peroxide system can be adjusted by changing the substitution rate[17]. The pH value and thus oxidation ability of the alkali hydrogen peroxide system decrease with the increasing of substitution rate. When the reaction temperature is set at 85 °C, the free radical reaction plays the main role in the system and the strong oxidation of the system is suitable for dissolving materials; when the reaction temperature is set at 125 °C, the free radical reaction is inhibited and a large amount of HOO exists in the system, which makes the system suitable for bleaching[19].

Protocol

1. Oxidation Degumming of Ramie

1. **Preparing the oxidation degumming solution**
   1. Dissolve 2 g H_2O_2, 1 g alkali (a mixture of $Mg(OH)_2$ and NaOH), 0.4 g $Na_5P_3O_{10}$, 0.1 g anthraquinone, and 0.2 g HEDP in 100 mL distilled water to make the degumming solution.

2. **Oxidation degumming of ramie**
   1. Immerse 10 g raw ramie in the degumming solution and scour it under 85 °C for 60 min.
   2. Raise the temperature to 125 °C (with pressure of 0.6 kg) and scour for another 60 min.
      NOTE: See the Discussion for explanation on raising the temperature.

3. **Reducing the ramie fiber**
   1. Dissolve 0.4 g $NaHSO_3$ in 100 mL distilled water to prepare the reducing solution. Then, treat the degummed fiber in the reducing solution at 90 °C for 60 min.
      NOTE: The carboxyl groups and aldehyde groups in cellulose generated in the oxidation reaction cause the reduction of hydrogen bonds and thus damage to the fiber property. Reducing can improve the property of the fiber by converting the carboxyl groups and aldehyde groups back to hydroxyl groups.

4. **Follow-up treatment**
   1. Wash the degummed ramie fiber thoroughly with deionized water.
   2. Immerse the fiber in degumming oil at 90 °C for 15 min and then dry the fiber in an oven (125 °C) for 4 h.

2. Testing of the Degumming Solution Property

1. **Solubility test of $Mg(OH)_2$**
   1. Dissolve 2 g $Mg(OH)_2$ in 100 mL distilled water.
   2. Separately, dissolve 2 g $Mg(OH)_2$ in 100 mL solution with completely soluble degumming additives, including 0.4 g $Na_5P_3O_{10}$ and 0.2 g HEDP.
   3. Raise the temperature of the solutions described in steps 2.2.1 and 2.2.2 to 85 °C.
   4. Extract the undissolved $Mg(OH)_2$ with sintered discs.
   5. Calculate the solubility of $Mg(OH)_2$ by the following formula:
      $$Solubility = 2 - m$$
      NOTE: Here *m* (g) is the weight of the undissolved $Mg(OH)_2$.

2. **Effect of the $Mg(OH)_2$ substitution rate on pH value, ORP value, and residual H_2O_2 content of the degumming solution**
   NOTE: ORP[12,19] is an important water chemistry parameter and it provides a measurement tool for the oxidizing or reducing capacity of the ambient water. Solutions with stronger oxidation property have a higher ORP value. The $Mg(OH)_2$ substitution rate refers to the mole proportion of NaOH replaced by $Mg(OH)_2$ under the total alkali dosage of 10% (on weight of fabric).
   1. Prepare the degumming solutions with $Mg(OH)_2$ substitution rate of 0%, 20%, 40%, 60%, 80%, and 100% according to step 1.1, respectively.
   2. Immerse the raw ramie in the degumming solutions described in step 2.2.1.
   3. Start the degumming process according to step 1.2.

4. Wash the combined ORP electrode with distilled water and dry it in the air. Then immerse the combined ORP electrode meter in the degumming solutions to read the ORP value every 10 min. Immerse the pH electrode meter in the degumming solutions to read the pH value every 10 min.
5. Test the H_2O_2 content of the degumming solutions every 10 min by the $KMnO_4$ titration method according to Chinese standard GB 22216-2008[7].

3. Testing the Ramie Fiber Property

1. **Yield of degumming**
   1. Calculate the yield of degumming using the following equation:
   $$yield = \frac{w}{W} \times 100\%$$
   NOTE: Here *w* (g) is dry weight of fiber after degumming; *W* (g) is dry weight of ramie before degumming.

2. **Residual gums of the fiber**
   NOTE: Residual gums of fiber were tested according to Chinese standard 5889 - 86.
   1. Measure the dry weight of the fiber (about 5 g) in a weighing bottle and immerse it in a flask (with reflux condensing tube) containing 150 mL NaOH solution (20 g/L).
   2. Raise the temperature to 100 °C and keep at this temperature for 1 h.
   3. Refresh the NaOH solution.
   4. Raise the temperature to 100 °C and maintain for 2 h.
   5. Wash the fiber in a sample sieve.
   6. Measure the dry weight of the fiber in weighing bottle.
   7. Calculate the residual gums of fiber using the following equation:
   $$Residual\ gums = \frac{m - M}{m} \times 100\%$$
   NOTE: Here m (g) is the dry weight of fiber; M (g) is the dry weight of ramie after NaOH scouring.

3. **PD test**
   NOTE: Test the PD value of ramie fiber according to Chinese standard GB 5888-86[15].
   1. Degrease the ramie fiber by submerging in 2:1 (v/v) benzene and ethyl alcohol mixture.
   2. Evaporate the solvent in air at room temperature.
   3. Cut the samples into short pieces (1-2 mm, about ~20-23 mg for each sample) using scissors.
   4. Keep the samples in a controlled-humidity atmosphere (20 ± 2 °C, relative humidity = 65 ± 2%) in a weighing container until it reaches equilibrium water content before removing the materials required for test purposes.
   5. Immerse a copper wire (0.5 mm diameter) in concentrated nitric acid, followed by 98% anhydrous ethylenediamine. Then, wash the copper granule thoroughly with distilled water.
   6. Put the fiber sample and copper granule in a plastic bottle (with top).
   7. Add 10 mL distilled water and 10 mL 1 mol/L cupriethylenediamine solution into the plastic bottle, and stir using a magnetic stir bar to prepare 0.2 g/100 mL (approximately) ramie fiber cupriethylenediamine solution.
   8. Transfer 6.5 mL ramie fiber cupriethylenediamine solution to a Ubbelohde viscometer to measure its intrinsic viscosity. Calculate the relative viscosity by the following equation:
   $$\eta_r = \frac{t}{t_0} \times K$$
   NOTE: Here η_r is the relative viscosity, t (s) is the average time of the ramie fiber cupriethylenediamine solution flowing through the Ubbelohde viscometer, and t0 (s) is the average time of the 0.5 mol/L cupriethylenediamine solution flowing through the Ubbelohde viscometer.
   9. Calculate the PD value of the ramie fiber by the following equation:
   $$PD = 156[\eta] \times \frac{C}{C'}$$
   NOTE: Here [η]' is the intrinsic viscosity, the [η]×C value can be obtained from a table in Chinese standard GB 5888-86, and C' is the concentration of ramie fiber cupriethylenediamine solution.

4. **Linear density of the fiber**
   1. Calculate linear density of fiber using the following equation:
   $$N_{dt} = \frac{10G}{nL_c}$$
   NOTE: Here L_c is the cutting length (40 mm), *n* is the numbers of fibers, and G (g) is weight of the fiber. Linear density of the fiber refers to the weight of a 1,000 m long fiber under official regain of ramie (12%).

5. **Mechanical property test**
   1. Equilibrate the fiber samples in standard atmospheric condition (T = 20 ± 2 °C, RH = 65% ± 2%) for 24 h.

2. Test the tenacity, breaking elongation, and work of rupture of the fiber using the fiber strength instrument under the following setting of 20 °C, RH 65%, and pre-tension of 0.3 cN/dtex. Set the clamping distance at 20 mm and the descending speed of the bottom clamp at 20 mm/min[7,15].

6. **COD value of degumming wastewater**
   1. Test the COD of degumming wastewater according to Chinese standard GB/T 15456-2008 'Industrial circulating cooling water-Determination of the COD-Potassium permanganate method'[7,15].

7. **XRD test**
   1. Obtain crystallinity of the fiber using X-ray diffraction. Record XRD patterns 2θ = 5-60 ° with a diffractometer equipped with a graphite monochromator and Cu Kα radiation at λ = 0.154 nm (40 kV, 200 mA).

8. **FTIR analysis**
   1. Obtain the FTIR pattern of fiber using spectrometer. Set the scan times at 30, the range at 4,000-400 cm^{-1}, and the resolution at 8 cm^{-1}. Determine the chemical functional groups in treated fiber using FT-IR analysis.

Representative Results

The solubility of $Mg(OH)_2$ in distilled water and degumming solution was studied (**Figure 1**). The effect of $Mg(OH)_2$ substitution rate on pH value and ORP value (**Figure 2**) of the degumming solution was tested. The degumming yield and residual gums of fiber degummed under various $Mg(OH)_2$ substitution rate were calculated (**Figure 3**). DP value, crystallinity, tensile properties of fiber (**Figure 4**), and COD value of wastewater (**Figure 5**) were used to evaluate the effect of $Mg(OH)_2$ on degumming. The FTIR pattern of fiber was obtained (**Figure 6**). The residual H_2O_2 content of the degumming solution during the fiber extraction process was tested (**Figure 7**) and the effect of the degumming temperature in the second stage is shown in **Table 1**. The comparison of oxidation degumming (using sustainable alkali source and NaOH) and traditional degumming is shown in **Table 2**.

Although the solubility of $Mg(OH)_2$ in degumming solution was higher than that in distilled water due to the slat effect of degumming auxiliaries, it was still insufficiently soluble, and thus a controlled-released property was applied (**Figure 1**). When a controlled-released alkali source was used, the pH value of the degumming solution was stable and decreased with the rising of substitution rate (**Figure 2A**). The decreasing of the ORP value was slower under higher substitution rate (**Figure 2B**). Residual gum analysis revealed that the yield of degumming and residual gums of fiber increased with the substitution rate; the substitution rate should be above 60% to prevent fiber adhesion. (**Figure 3**). The DP value, crystallinity, and tensile properties of fiber increased with the substitution rate from 0% to 20%, but decreased upon further rising of the substitution rate (**Figure 4**): this is explained by the excessive amount of gums that were retained in the fiber when the substitution rate was over 20%. When the substitution rate was set at 20%, the pH value of the degumming solution was 11.8; and the tenacity, elongation, rupture, DP value, and hemicellulose content yield of fiber increased by 39.82%, 12.13%, 46.15%, 14.89%, and 5%, respectively (**Figure 2**, **Figure 3**, **Figure 4**). Moreover, the COD value of the degumming waste water decreased by 20% (**Figure 5**). In FTIR patterns of the fiber, the signals in the region of 3,400-2,800 cm^{-1} and the peak at 2,900 cm^{-1} were due to the stretching vibration of -CH and -OH in cellulose, and these signals existed in all samples. The carbonyl peak at 1,730-1,750 cm^{-1} was attributed to the C=O stretching of C-OH bending in hemicellulose, and this signal was stronger when the substitution rate was lower, which indicated that hemicellulose can be removed more effectively under a lower substitution rate (**Figure 6**). Residual H_2O_2 contents increased by 3 g/L when using the controlled-released alkali source; however, the substitution rate did not influence the residual H_2O_2 content (**Figure 7**). When the controlled-release alkali source was used for degumming, the decomposition speed of H_2O_2 was controlled by the degumming temperature. In the initial period of degumming (0 to 60 min), cellulose degradation seldom occurred, for it was covered by gums. Therefore, a large amount of free radicals was needed and the temperature should be set at 85 °C. After 60 min, most of the gums were removed and the cellulose was exposed to the degumming solution: the temperature should be raised to 125 °C to slow down the free radical reaction speed and therefore prevent the degradation of cellulose (**Table 1**). The comparison of oxidation degumming (using a sustainable alkali source and NaOH) and traditional degumming revealed that fiber degummed in alkali hydrogen peroxide system supported by controlled-release alkali source achieved the best properties (**Table 2**).

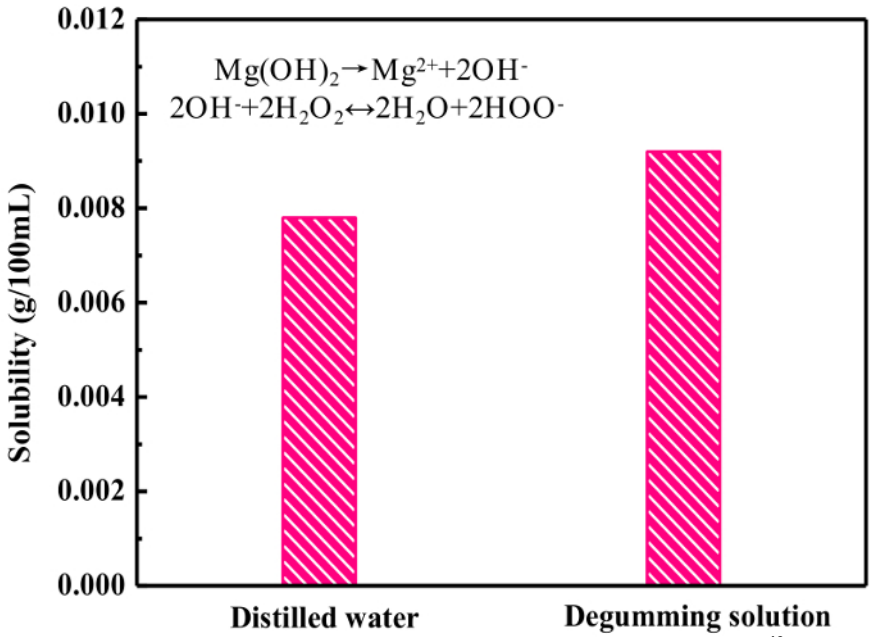

Figure 1. The solubility of $Mg(OH)_2$ in distilled water and degumming solution[19]. $Mg(OH)_2$ showed higher solubility in degumming solution compared with that in distilled water, because of the salt effect of degumming auxiliaries. $Mg(OH)_2$ dissolves into the degumming solution slowly according to the inset chemical equations. Please click here to view a larger version of this figure.

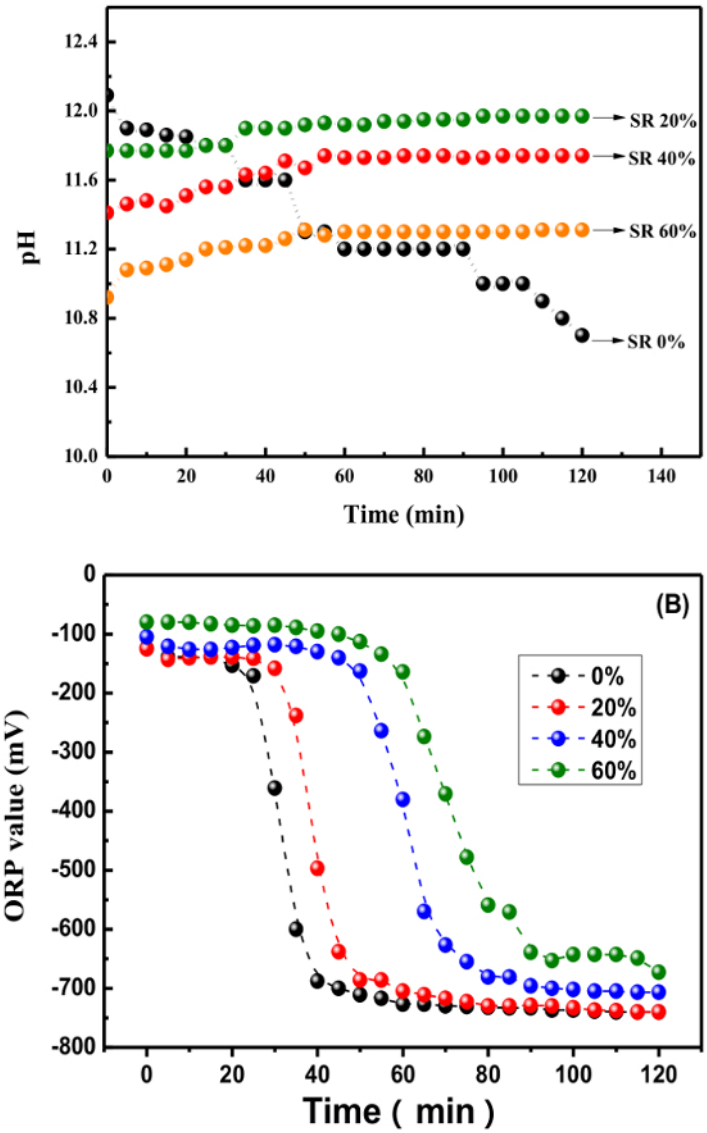

Figure 2. The effect of $Mg(OH)_2$ substitution rate on degumming solution properties. (**A**) The pH value of degumming solution. When $Mg(OH)_2$ was used, the pH value of the degumming solution was stable and decreased with the rising of substitution rate. (**B**) The ORP value of degumming solution[19]. The decreasing speed of ORP value was slower under higher SR value. SR = substitution rate. Please click here to view a larger version of this figure.

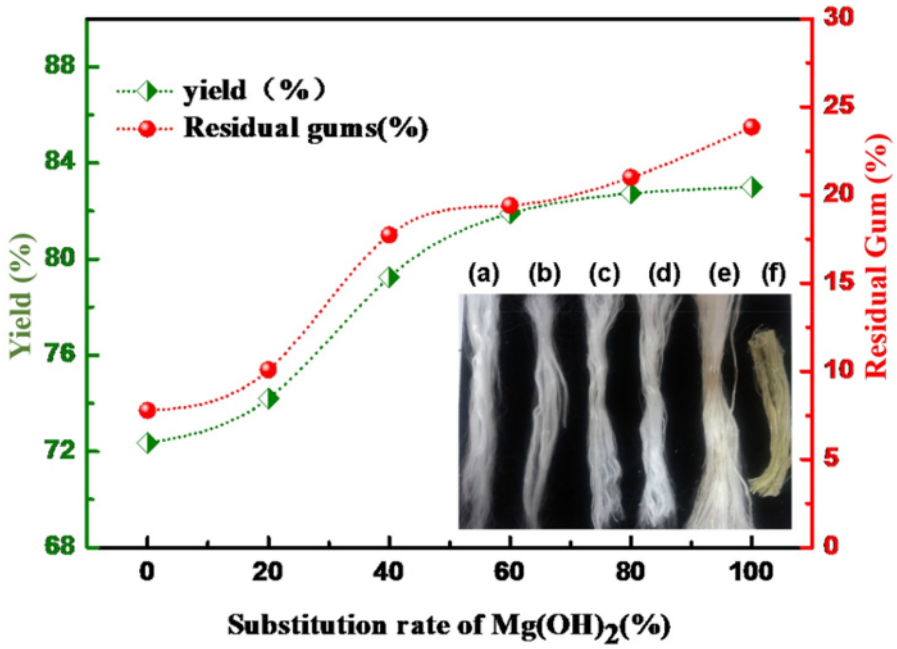

Figure 3. The effect of $Mg(OH)_2$ substitution rate on degumming yield and residual gums of fiber. The inset image shows the topographies of ramie fiber degummed under $Mg(OH)_2$ substitution rates of: (**a**) 0%, (**b**) 20%, (**c**) 40%, (**d**) 60%, (**e**) 80%, (**f**) 100%[19]. The yield of degumming and residual gums of fiber increased with substitution rate and substitution rate should be above 60% to prevent fiber adhesion. Please click here to view a larger version of this figure.

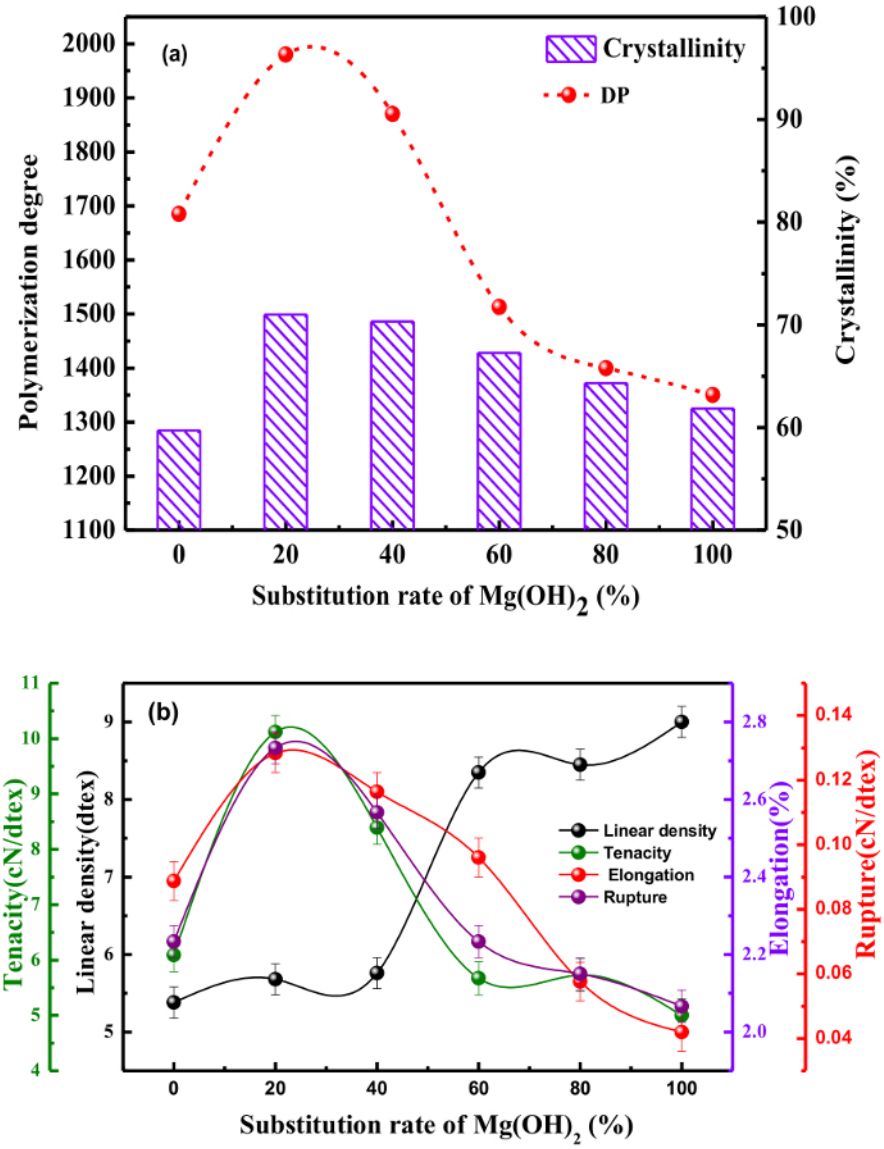

Figure 4. The effect of $Mg(OH)_2$ substitution rate on: (**A**) the DP value and crystallinity of fiber; and (**B**) the tensile properties of fiber[19]. DP value, crystallinity, and tensile properties of fiber increased with SR from 0% to 20%, but decreased with further rising of substitution rate. Error bars represent the standard deviation of data from 30 duplicate tests. Please click here to view a larger version of this figure.

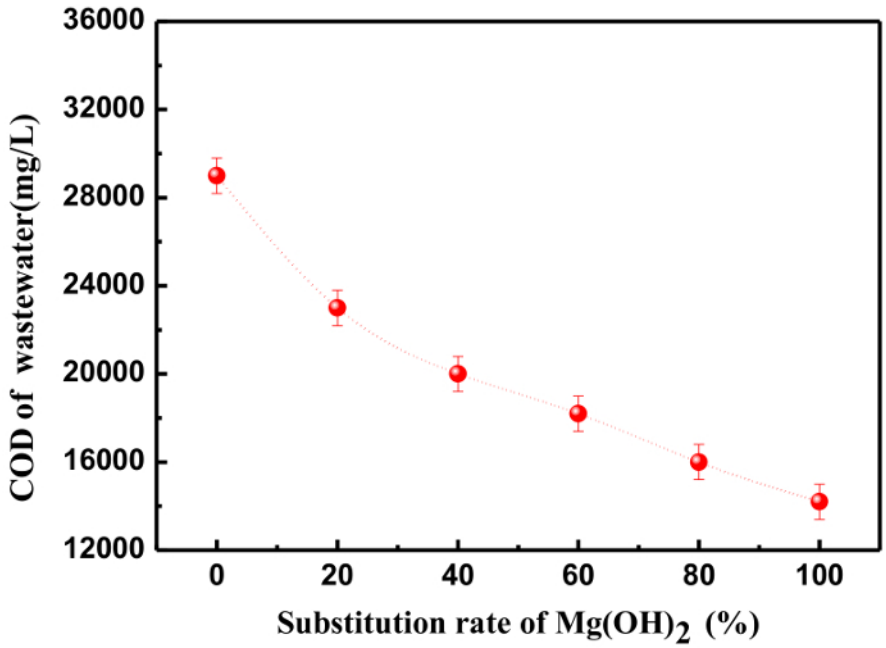

Figure 5. The effect of $Mg(OH)_2$ substitution rate on COD value of degumming wastewater[19]. The COD value of degumming wastewater decreased with the rising of substitution rate. Error bars represent the standard deviation of data from 30 duplicate tests. Please click here to view a larger version of this figure.

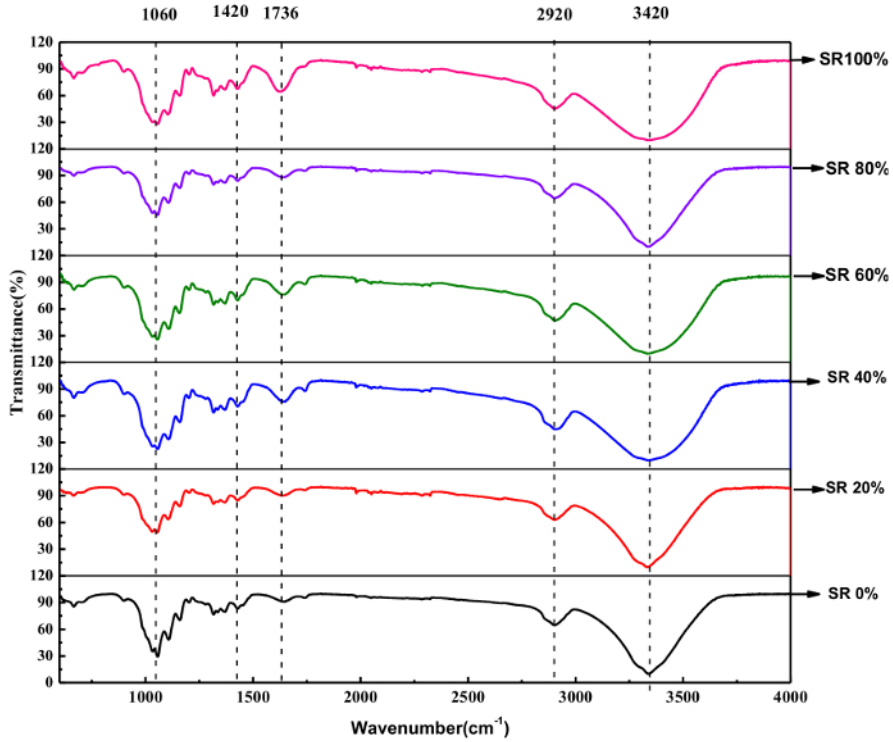

Figure 6. FTIR of fiber degummed with various substitution rates of $Mg(OH)_2$[19]. The signals in the region of 3,400-2,800 cm^{-1} and the peak at 2,900 cm^{-1} were due to the stretching vibration of -CH and -OH in cellulose; these signals were present in all samples. The carbonyl peak at 1,730-1,750 cm^{-1} was attributed to the C=O stretching of C-OH bending in hemicellulose; these signals were stronger when SR was lower, which indicated that hemicellulose can be removed more effectively under lower substitution rate. Please click here to view a larger version of this figure.

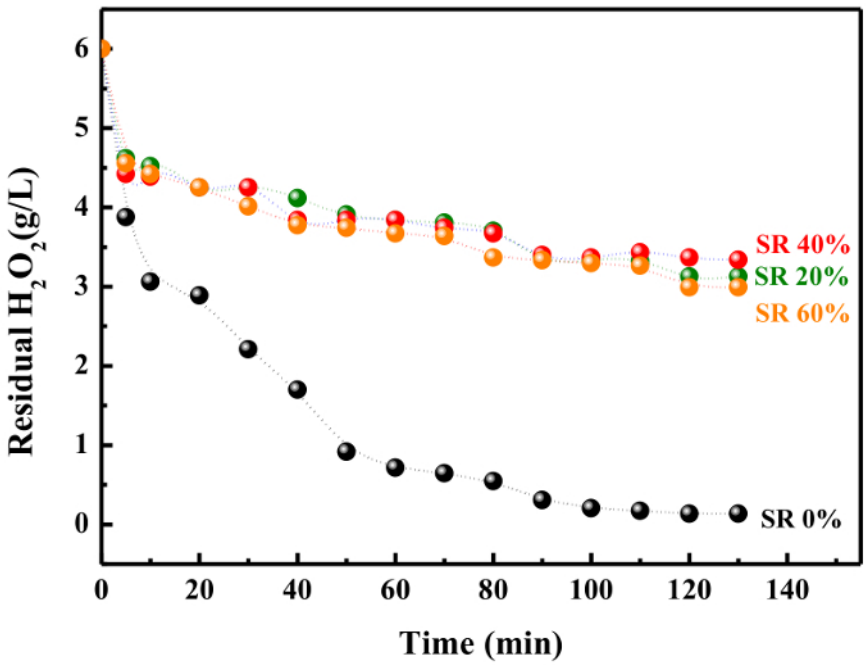

Figure 7. Residual H_2O_2 contents in degumming solution with various substitution rate of $Mg(OH)_2$[19]. Residual H_2O_2 contents increased when the controlled-released alkali source was used; however, the substitution rate did not have influence on the residual H_2O_2 content. SR = substitution rate. Please click here to view a larger version of this figure.

Temperature	Linear density (dtex)	Tenacity (cN/dtex)	Elongation (%)	Rupture (cN/dtex)
100 °C	6.1	6.69	2.33	0.08
125 °C	5.6	8.3	2.75	0.14

Table 1. Tensile properties of fiber scouring under various temperature in the second stage[19]. The fiber exhibited better tensile properties under higher scouring temperature.

	Oxidation degumming		Traditional degumming
	SR 20%	SR 0%	
Yield (%)	74.2	72.34	65
Tenacity (cN/dtex)	10.12	6.09	7.8
Elongation (%)	2.72	2.39	2.43
Rupture (cN/dtex)	0.13	0.07	0.1
PD value	1980	1685	1732
COD value (mg/L)	23000	29000	29800

Table 2. Comparison of oxidation degumming. Comparison of oxidation degumming (using sustainable alkali source and NaOH) and traditional degumming[19] ramie fiber. Fiber degummed in an alkali hydrogen peroxide system supported by a controlled-release alkali source achieved the best properties. SR = substitution rate.

Discussion

The setting of $Mg(OH)_2$ substitution rate and reaction temperature was the key point of this protocol. $Mg(OH)_2$ substitution rate can influence the pH value and thus oxidation ability of degumming solution. The best $Mg(OH)_2$ substitution rate for ramie degumming was 20%, because cellulose cannot receive enough protection under a substitution rate below 20%, and an excessive amount of residual gums (low DP value and crystallinity) would be retained in fiber under a substitution rate above 20% (**Figure 4A**).

The reaction temperature can influence the reaction pathway of hydrogen peroxide. There were two parallel reactions in the oxidation degumming of ramie: the first was the reaction between H_2O_2 and gums; the second was the reaction of H_2O_2 and cellulose, which can cause damage to cellulose and thus decrease the tensile properties of degummed fiber. The rise of temperature can induce the acceleration of the two reactions (the reaction speed increased by 2 or 4 times, with temperature rise per 10 °C). The growth of the reaction speed for H_2O_2 and gums was much higher than H_2O_2 and cellulose, because its activation energy is higher, which made it more sensitive to temperature change. In the initial period of degumming (0 to 60 min), cellulose degradation seldom occurred, because it was covered by gums. Therefore, a large amount of free radicals was needed and the temperature should be set at 85 °C. After 60 min, most of gums were removed and the cellulose was exposed

to the degumming solution; the temperature should be raised to 125 °C to slow down the free radical reaction speed and therefore prevent the degradation of cellulose (**Table 1**).

The technology of this protocol can be extended to other areas, such as the extracting, bleaching, and modifying of plant material in alkali hydrogen peroxide system. The $Mg(OH)_2$ substitution rate and reaction temperature should be set according to the specific conditions. Normally, the pH value and thus oxidation ability of alkali hydrogen peroxide system decreases with the increasing of substitution rate. When the reaction temperature is set at 85°C, the free radical reaction plays the main role in the system and the strong oxidation ability makes the system suitable for dissolving materials; when the reaction temperature was set at 125 °C, the free radical reaction was inhibited and a large amount of HOO existed in the system, which makes the system suitable for bleaching[19]. The limitation of this technology is that the pH value of hydrogen peroxide system can only be set at values between 10.0 to 12.0 when the controlled-released alkali source is used.

We have demonstrated a method of improving the property of oxidation degummed ramie fiber by using $Mg(OH)_2$ as the sustainable alkali resource (**Table 2**). This technology is now being applied in the pilot stage, and we expect that this technology will continue to develop.

Disclosures

The authors have nothing to disclose.

Acknowledgements

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