Properties of Natural Cotton Stalk Bark Fiber under Alkali Treating

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ABSTRACT: The natural cotton stalk (CS) bark fiber was obtained through degumming of CS bark, and then treated by sodium hydroxide solution. To investigate the influence of sodium hydroxide treatment on the properties of the natural CS bark fiber, the fineness, length, breaking strength, X-ray diffraction, and morphology were tested. The results showed that the fineness, length, and crystallinity of the fiber decreased when the fiber was treated by sodium hydroxide solution. When the sodium hydroxide content increased from 10 to 50 g/L, the strengths of the fiber increased. The surface of the fiber after treating was bright compared with before treating, and the single cell had convolutions after alkali treating. © 2012 Wiley Periodicals, Inc. J Appl Polym Sci 000: 000–000, 2012

Key words: cotton stalk bark fiber; sodium hydroxide treatment; morphology; crystallinity

INTRODUCTION

Natural fibers present important advantages such as low density, appropriate stiffness and mechanical properties and high disposability and renewability. Moreover, they are recyclable and biodegradable. There has been lot of research on the use of natural fibers in reinforcements and textile.[1,2] Ramie, flax, hemp, and some other bast fibers have been used as textile materials. Ashori reported that nonwood, such as bamboo, kenaf, hemp, jute, and sisal, was used for papermaking to meet the possible shortfall of wood fiber.[3–5] Agro-residues, produced from commercial processing of crop plants, are usually considered to be of little inherent value and represent a disposal problem.[6] However, these material could in many cases represent an abundant, inexpensive, and readily available source of renewable lignocellulosic biomass for different purposes.[7] The chemical compositions and fiber morphology of agro-residues namely lemon balm stalk, bagasse stalk, cotton stalk (CS), and tobacco stalk were compared.[6] Cordeiro et al.[8] reported the effects of chemical modification using 1% NaOH on the properties of Iranian cultivated eucalyptus, spruce, bagasse, and wheat straw. The crystallinity of fibers and the specific interaction was improved by the alkaline treatment, with more relevance to the agro-fibers. As agricultural waste, both CS and CS bark usually are used for papermaking, composites, and as regenerated cellulose for rayon.[9–11] Beside, some CS is burned. The cultivation of cotton generates plant residue equivalent to three to five times the weight of the fiber produced.[9] The cotton plant residue left after harvest is mostly comprised of stalks and it has been estimated that nearly 2.5–3.5 tons of stalks are generated per acre of cotton grown depending on the type of harvester used to harvest cotton fibers.[12] The CS bark has a proportion of 26% of CS weight.[13] Liu and Liu[14] reported that cotton-producing area in India was 10.9 million hectares (about 26.9339 million acres) in the year of 2010. The cotton-producing area in USA was over 3.7 million hectares (about 9.1427 million acres) in the year of 2009/2010.[15] Therefore, there are great amounts of CS bark in the world each year because a lot of cotton is produced annually. There is about 4 million tons of CS bark annual in China.[13] CS bark, from the outside bast of CS, has cellulose and noncellulose substances, such as hemicellulose, pectin, lignin, and wax. The natural CS bark fiber, obtained from CS bark through degumming, is a bast fiber. The coarseness, stiffness, low cohesive performance, and some other disadvantages seriously restrict the CS bark fiber from spinning.

Bast fibers are complex in structure. Natural CS bark fiber is formed by a bundle of single cells.[16] Intersingle cell was bonded together by noncellulose substance. The dimensions and properties of the

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TABLE I
The Effect of Sodium Hydroxide on the Properties of the Fiber

Sodium hydroxide content (g L^{-1})	Temperature (°C)	Time (min)	Fineness (tex)	Length (mm)	Breaking strength (cN $dtex^{-1}$)
10	60	40	3.05	37.6	4.25
20			2.80	35.8	4.08
30			2.65	32.0	5.48
40			2.47	29.2	6.1
50			2.28	27.6	6.25
60			2.25	25.2	5.0

fibers formed are determined by the number and dimensions of single cells which in turn is influenced by the variety of stalks and extraction conditions used to obtain the fibers. Generally, the CS bark fiber is obtained through degumming under the condition of alkali solution. To investigate the fineness and length change of CS bark fiber under the condition of alkali treating because of single cells separating further, and mercerization of CS bark through alkali treating, the post-sodium hydroxide treating of natural CS bark fiber was employed in this work.

MATERIALS AND METHOD

Materials

Natural CS bark fiber was obtained through degumming of CS bark, and then lightly carded.

Sodium hydroxide was purchased from Xi'an Chemical Industry, China.

Method

The natural CS bark fiber was treated with sodium hydroxide 10–60 g/L, and kept at 40–100°C for 20–100 min with fiber to liquor ratio 1 : 50. At the end of the desired treatment, the fibers were thoroughly washed with distilled water.

Testing

Breaking strength was tested using an YG001N fiber breaking strength tester, and test length of the fiber was 10 mm. For each result, 50 fibers were tested.

The natural CS bark fibers were conditioned in a standard testing atmosphere of 21°C and 65% relative humidity for at least 24 h before testing the fibers. Fineness of the fibers was measured in terms of Tex by weighing a known length of the fibers. Tex is defined as the weight of the fibers in grams per 1000 m of the fibers. The fineness was tested according to GB/T12411.3. The single fibers length was tested using a ruler, and then the length of fiber was the average of 30 fibers.

The morphology of the fiber was observed with a KYKY-2800B scanning electronic microscope, and the fibers were coated with gold, and then the testing was done.

The crystallinity of the fiber was tested with a D/MAX-2400 X-ray diffraction analyzer. The test conditions were as follows: voltage 46 kV, current 100 mA, Cu Kα radiation, scanning scope $2\theta = 10°–70°$, and scanning speed 10°/min.

The flexibility of fibers was tested with Y321B handle twister. The fibers are clamped, and then twirl the handle clockwise or counterclockwise up to the all fibers broken. The fibers with 40 mm in length were cut by a cuter and about 3 mg weight for each test. The flexibility of fibers was calculated according to eq. (1).[17]

$$D = \frac{n \times 40 \times 0.1}{L \times G} \quad (1)$$

In which, D is flexibility, twist/m tex; n is twist number of fibers broken; L is the length of fibers, mm; G is weight of fibers clamped, mg.

The natural CS bark fibers were dried, respectively, before and after treating in an oven at 110°C

TABLE II
The Effect of Treating Time on the Properties of the Fiber

Treating time (min)	Sodium hydroxide content (g L^{-1})	Temperature (°C)	Fineness (tex)	Length (mm)	Breaking strength (cN $dtex^{-1}$)
20	50	60	3.24	32.5	4.41
40			2.87	30.1	5.63
60			2.62	29.6	6.23
80			2.41	25.3	5.24
100			2.37	24.8	4.51

TABLE III
The Effect of Temperature on the Properties of the Fiber

Treating temperature (°C)	Sodium hydroxide content (g L^{-1})	Treating time (min)	Fineness (tex)	Length (mm)	Breaking strength (cN $dtex^{-1}$)
40	50	60	30.8	30.3	4.87
60			26.7	27.0	6.23
80			23.8	25.4	6.56
100			23.6	24.1	5.61

until a constant weight, W_1 and W_2, was reached, respectively. The weight loss rate of natural CS bark fibers was calculated according to eq. (2).

$$Y = \frac{W_1 - W_2}{W_1} \times 100\% \quad (2)$$

In which, Y is weight loss rate, %; W_1 is weight of natural CS bark fibers before treating, g; W_2 is weight of natural CS bark fibers after treating, g.

RESULTS AND DISCUSSION

Effect of sodium hydroxide

The fiber from CS bark is put into the alkali solution. The sodium hydroxide content is 10, 20, 30, 40, 50, and 60 g/L, respectively. Treatment at 60°C for 40 min, bath ratio 1 : 50. The results are shown in Table I. It is shown that the fineness and length of fibers decrease as sodium hydroxide content rise. It is because that the noncellulose compositions were sufficiently decomposed by sodium hydroxide or effectively dissolved. When the treated fibers were drying, the fibers would not connect to each other again.

Breaking strength increases as sodium hydroxide content rise up to 50 g/L, but decreases when sodium hydroxide content is between 50 and 60 g/L. It may be explained that when the noncellulosic materials were partially removed, the interfibrillar region was likely less dense and less rigid, thereby making the fibrils more capable of rearranging themselves along the direction of tensile deformation. When the fiber is stretched, such rearrangement amongst the fibrils should result in better load sharing by them and hence result in an increase in fiber breaking strength. But the excessive removal of noncellulosic materials could also be negatively accompanied by a formidable decrease in the breaking strength of the fiber. Taking account of the spinnability of the fiber, which largely depends on its two important properties namely fineness and breaking strength,[18] our conclusion is that the sodium hydroxide content should be controlled at 30–50 g/L.

TABLE IV
Factors and Levels

	Factor		
Level	A: sodium hydroxide content (g L^{-1})	B: time (min)	C: temperature (°C)
1	30	20	40
2	40	40	60
3	50	60	80

Effect of treating time

The fiber from CS bark is put into the alkali solution. Sodium hydroxide content is 50 g/L. Treatment temperature is 60°C, bath ratio 1 : 50, and treatment time is 20, 40, 60, 80, and 100 min, respectively. The results are shown in Table II. It is shown that the fineness and length of fibers decrease as treating time increase, breaking strength increase as treating time increase up to 60 min, but decreases when time is between 60 and 100 min. So, the treating time should be controlled at 60 min. It may be explained that the extent of CS bark fiber swelling increased with time and was fairly complete with 60 min. As a result, the auxiliary agents react sufficiently with noncellulose materials, and make them decompose and dissolve in the treatment solution. At the same time, it may be also explained that decomposable and dissolvable materials have been completely removed in 60 min. So there is no need to continue treatment after 60 min.

Effect of treating temperature

The fiber from CS bark is put into the alkali solution. Sodium hydroxide content is 50 g/L. Treatment time 60 min, bath ratio 1 : 50, and treatment temperature is 40, 60, 80, and 100°C, respectively. The results are shown in Table III. It is shown that the fineness and length of fibers decrease as temperature increase, breaking strength increase as temperature increase up to 80°C, but decreases when temperature is between 80 and 100°C. So, the temperature should be controlled at 80°C. It may be explained that penetrability of auxiliary agents and swelling degree of fiber increased with the increase in temperature. As a result, the noncellulose compositions were decomposed or dissolved by alkali solution. Taking account of all the three indexes, the temperature should be controlled at 80°C.

TABLE V
$L_9(3^3)$ and Results

No.	Factor A	Factor B	Factor C	Fineness (tex)	Length (mm)	breaking strength (cN dtex^{-1})	Flexibility (T (m tex)$^{-1}$	Weight loss rate (%)
1	1	1	1	3.01	26.12	4.22	3.18	16.17
2	1	2	2	2.48	26.97	6.01	2.97	18.95
3	1	3	3	2.35	25.01	5.36	2.97	20.29
4	2	1	2	2.55	30.82	6.18	2.98	15.14
5	2	2	3	2.76	28.99	7.92	2.57	19.39
6	2	3	1	2.87	27.05	5.14	2.67	16.61
7	3	1	3	2.54	25.79	5.36	2.74	17.82
8	3	2	1	2.63	25.14	5.25	2.78	19.50
9	3	3	2	2.66	27.93	4.85	2.69	20.25

Orthogonal experiment

According to the above discussions, a $L_9(3^4)$orthogonal experiment was designed to optimize the condition for the modification. The factors, levels, and results are represented in Tables IV–VI.

Table VI shows that the sodium hydroxide content and treatment temperature are the two most important factors for fineness and length of the fiber, the optimum condition for fineness is $A_3B_2C_3$, and the optimum condition for length is $A_2B_1C_2$. The optimum condition for breaking strength is $A_2B_2C_3$, the optimum condition for flexibility is $A_1B_1C_2$, and the optimum condition for weight loss rate is $A_2B_1C_1$. Under the treating condition of $A_2B_1C_2$, the fineness is 2.55 tex, length 30.82 mm, breaking strength 6.18 cN/dtex, flexibility is 2.98/T (m tex)$^{-1}$ and weight loss rate is 15.41%. Under the treating condition of $A_2B_2C_3$, the fineness is 2.76 tex, length 28.99 mm, breaking strength 7.92 cN/dtex, flexibility is 2.57/T (m tex)$^{-1}$, and weight loss rate is 19.39%. With the view that the primary purpose for alkali solution treatment is to remove noncellulose materials and so as to decrease the stiffness of the fiber, coarseness and weight loss rate, the reasonable treating condition is selected as $A_2B_1C_2$, i.e., sodium hydroxide 40 g/L, time 20 min, and temperature 60°C.

TABLE VI
Results Analysis

Index	Factor	Level 1	Level 2	Level 3	Effect
Fineness (tex)	A	2.62	2.73	2.61	0.12
	B	2.70	2.62	2.63	0.08
	C	0.84	2.56	2.55	0.29
Length (mm)	A	26.03	28.95	26.29	2.92
	B	27.58	27.03	26.66	0.92
	C	26.10	28.57	26.60	2.47
Breaking strength (cN dtex^{-1})	A	5.20	6.41	5.15	1.26
	B	5.25	6.39	5.12	1.27
	C	4.87	5.68	6.21	1.34
Flexibility/T (m tex)$^{-1}$	A	3.04	2.74	2.73	0.31
	B	2.97	2.77	2.78	0.20
	C	2.87	2.88	2.76	0.12
Weight loss rate (%)	A	18.47	17.05	19.19	2.14
	B	16.34	19.28	19.05	2.94
	C	17.43	18.11	19.17	1.74

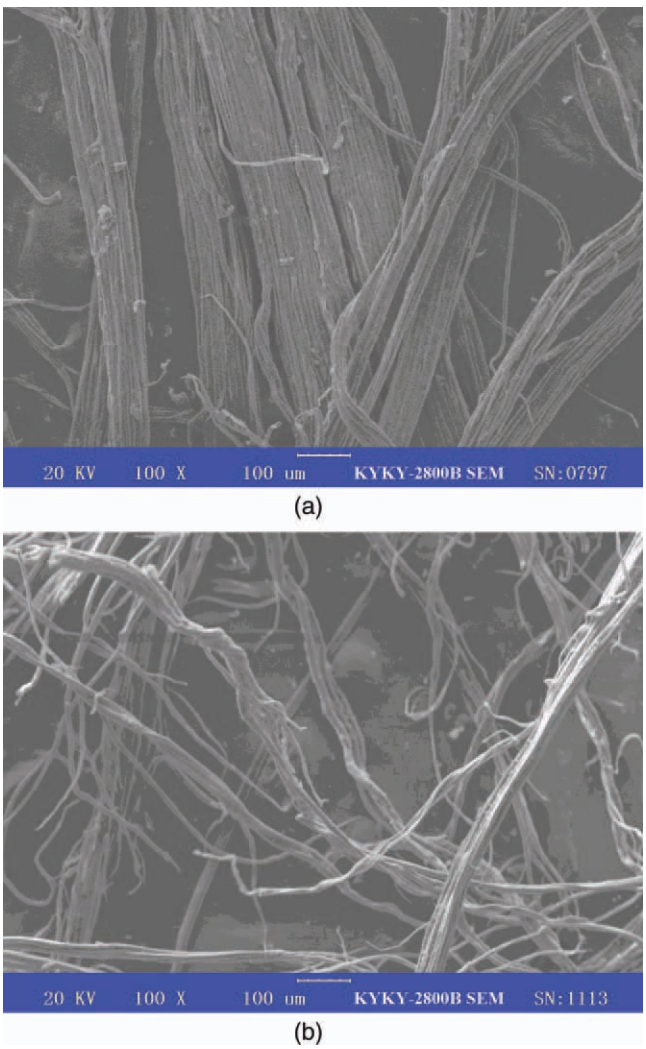

Figure 1 The morphology of the fiber: (a) morphology before treating (100×), (b) morphology after treating (100×). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

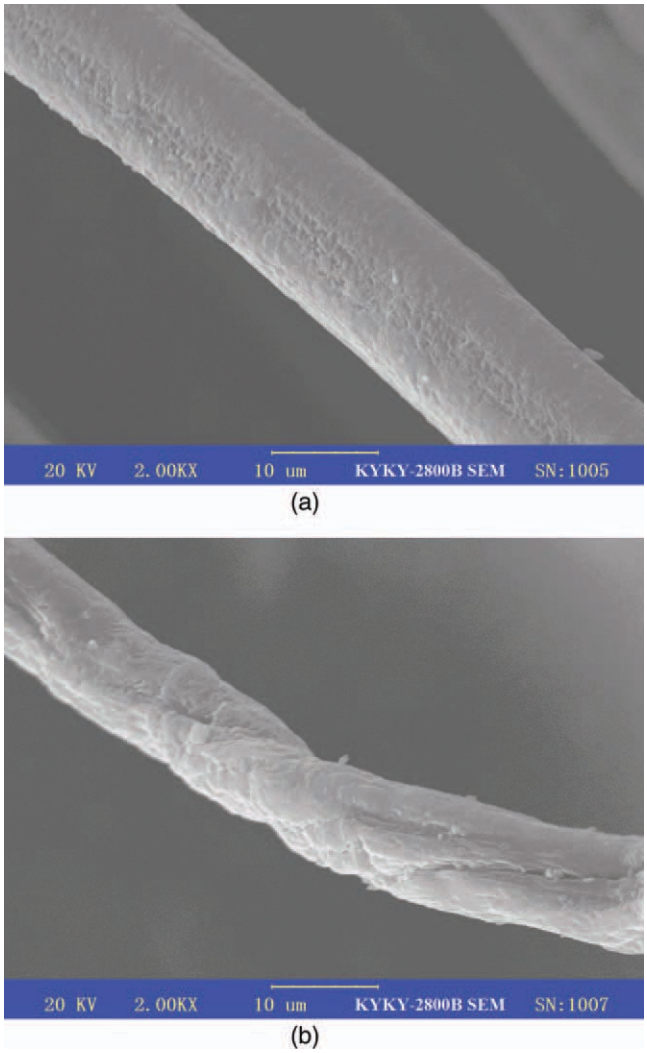

Figure 2 Morphology of single cell, (a) morphology before treating (2000×), (b) morphology after treating (2000×). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

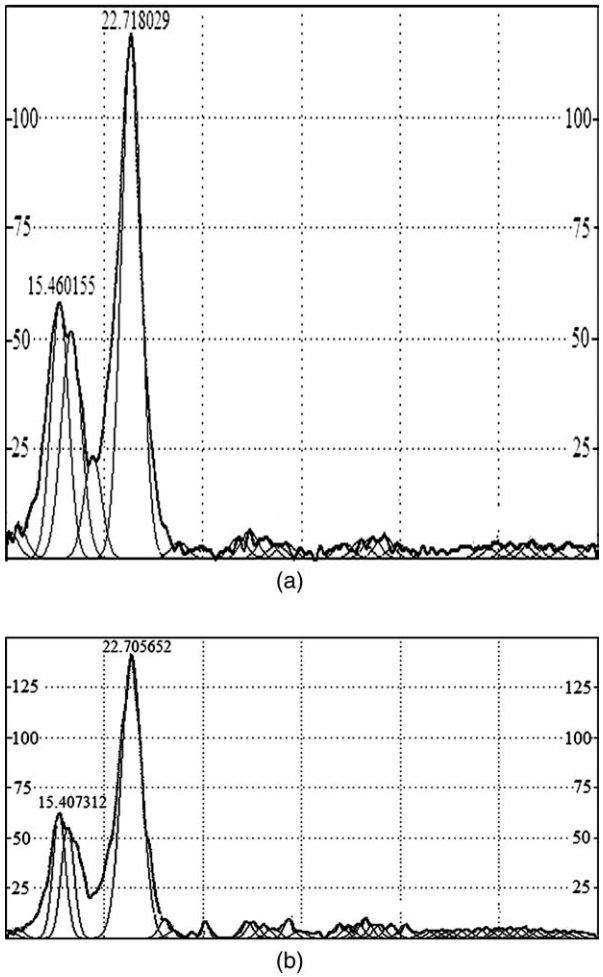

Figure 3 X-ray diffraction result: (a) X-ray diffraction curve before treating, (b) X-ray diffraction curve after treating.

The morphology

Figure 1 shows the morphology of the fiber before and after treating. The treating conditions is following as: sodium hydroxide 40 g/L, time 20 min, and temperature 60°C, and bath ratio 1 : 50. Figure 2 shows the morphology of single cell of CS bark. It is obtained that there is a good separation of fiber after treating as seen from Figure 1. And the surfaces of the fiber and single cell after treating are bright compared with before treating. The surface of single cell is not smooth before and after treating, the single cell has convolutions after treating as seen from Figure 2. From the morphology, it is obtained that natural CS bark fibers is "technology fiber," that is, single cells bonded together form a fiber by noncellulose substance. With different treating conditions, the fineness and the length of the fiber may be changed owing to single cell separating from the fiber. The different amount of noncellulose substance removed by the chemically treating results in different separating degree, which leads to various weight loss rates.

X-ray diffraction result

The results of X-ray diffraction for the fibers before and after treating are shown in Figure 3. Crystalline peak and noncrystalline peak of X-ray diffraction diagram is decomposed using the Gause function in Origin7.5 software. The crystallinity (C_r) is calculated by the following eq. (3)[19]:

$$C_r = \frac{S_e}{S_e + S_n} \times 100\% \quad (3)$$

In which, C_r is crystallinity; S_e is crystalline peak area; S_n is noncrystalline peak area.

TABLE VII
The Crystallinity of the Fibers Before and After Treating

Sample	Before treating	After treating
C_r (%)	68.55	62.53

The crystallinity of the fibers before and after treating is listed in Table VII. The crystallinity of the fibers after treating is less than that of the fiber before treating. It may be explained that cellulose swell in aqueous alkali solution where ionized H^+ in hydroxyl groups are replaced by Na^+ to combine into stable compounds, and the lattice of cellulose I changes after removal of reagent, resulting in the change in crystal structure of treated fibers. Wang et al.[20] also reported that the crystallinity of flex fiber decreased through alkali-urea treating. The crystallinity of the natural CS bark fibers after treating may contribute to flexibility rising.

CONCLUSIONS

Sodium hydroxide treating of natural CS bark fiber influences the fineness, length, breaking strength, and flexibility of the fiber. The fineness and the length of the fiber decrease when sodium hydroxide content increases. Compared with before treating, the crystallinity of the fibers after treating decreases, and the separation of fibers is good after treating. The surfaces of the fiber and single cell after treating are bright compared with before treating. The surface of single cell is not smooth before and after treating, the single cell has convolutions after treating.

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