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PREPARATION AND PROPERTIES OF COTTON STALK BARK FIBER

LONG LI, GUANZHONG SHENG, SHENGJING ZHANG and JIANLEI SUN

School of Textile and Materials
Xi'an Polytechnic University
P. R. China
e-mail: lilong2188@yahoo.com.cn

Abstract

Cotton stalk bark consists of cellulose and non-cellulose substances. The non-cellulose includes hemicelluloses, pectin, lignin, and wax. In this paper, alkali-H_2O_2 treatment was used to remove non-cellulose substance, and get and bleach cotton-straw bark cellulose fiber. Through orthogonal experimental, it showed that the condition of degumming was NaOH 8g/L, H_2O_2 9g/L at 100°C for 30 min. The morphology of the cellulose fiber was observed by using JSM5800 scanning electronic microscopy and crystallinity was calculated.

1. Introduction

Bast fibers are important fibers because they have biodegradable and eco-friendly characteristics. Efforts to exploit the wide use of bast fibers like hemp, kenaf, sisal, and jute have been an area of interest [2-4]. As agricultural waste, cotton stalk usually is used for paper-making, board-making, and building material. Beside, some cotton-stalk is burned. Cotton stalk bark comes from surface layer of cotton stalk. There is about 4 million tons of cotton stalk bark annual in China. Cotton stalk

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bark has cellulose and non-cellulose substances, such as hemi-cellulose, pectin, lignin, and wax. Therefore, the removal of most non-cellulose components is a prerequisite problem in order to make good use for textile resource.

Usually, bast fibers are processed with bacterial, chemical, and enzymatic methods. Chemical processing is effective for removing non-cellulose substances, and considerable research has focused on this method [6]. It is known that bleaching improves the appearance and colour of fibers and hydrogen peroxide is often used to bleach the fibers.

Earlier reports on the application of alkali-H_2O_2 one-bath process were used in dyeing and finishing [5]. Molecular weight of hemi-cellulose is lower than that of cellulose. Acid, alkali, and oxidizing agent have a strong action on hemi-cellulose. During degumming, the hemi-cellulose is removed through alkali solution treatment. Lignin is a complex amorphous polymer consisting of propane benzene cross-linked by ether bonds and C-C bonds; it can be resolved in alkali solution. During alkali-H_2O_2 one-bath treatment, alkali not only removes most of the hemi-cellulose, pectin, and lignin, but also provides H_2O_2 an alkaline environment to optimize the action of H_2O_2 and to remove lignin more efficiently. In this work, alkali-H_2O_2 was used to get natural cellulose fiber from cotton stalk bark (i.e., cotton stalk bark fiber). And the morphology and the crystallinity of the fiber were analyzed.

2. Experimental

2.1. Preparation of cotton-straw bast cellulose fiber

The cotton stalk bark was from country yard of Shaanxi, China. The components of cotton stalk bark are showed in Table 1.

Table 1. Components of cotton-straw bast

Component	Content, %
Wax	2.36
Hydrotropic matter	19.54
Pectin	3.34
Hemi-cellulose	25.88
Lignin	25.23
Cellulose	23.65

Cotton stalk bark was treated firstly by acid solution, and then treated by alkali-H_2O_2 solution under the experiment conditions. The preparation processing was following as:

Cotton stalk bark—water washing—alkali-H_2O_2 treating—water washing—beating—drying—natural cellulose fiber.

Alkali-H_2O_2 treating was done under the orthogonal experiment conditions. The experiment conditions were listed in Table 2. The treatment temperature of alkali-H_2O_2 treating was 100°C. The residual gum content of cotton stalk bark fiber was regarded as the evaluation index.

Table 2. Orthogonal condition

Factor	Level 1	Level 2	Level 3
NaOH content(A), g/L	6	8	10
H_2O_2 content(B), g/L	9	12	15
Bath ratio(C)	1:80	1:90	1:100
Time(D), min	25	30	35

2.2. Morphology structure

The morphology of the natural cellulose fiber was observed with a JSM5800 scanning electronic microscope (Electronic Co., Ltd., Japan). The fibers were coated with gold, and then the testing was done.

2.3. Flexibility test

The flexibility of fibers was tested with handle twister (as seen in Figure 1). The fibers are clamped, and then twirl the handle clockwise or counterclockwise up to the all fibers broken. The fibers with 40mm in length were cut by a cuter and about 3mg weight for each test. The flexibility of fibers was calculated according to Equation (1) [9].

$$D = \frac{n \times 40 \times 0.1}{L \times G}, \tag{1}$$

where D is flexibility, twist/m·tex; n is twist number of fibers broken; L is length of fibers, mm; and G is weight of fibers clamped, mg.

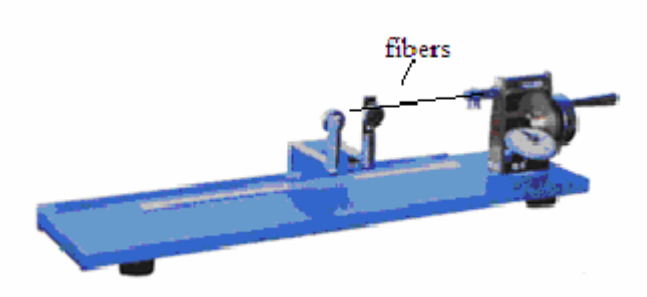

Figure 1. Handle twister.

2.4. Fiber length

The single fibers length was tested by using a ruler, and then the length of fiber was the average of 30 fibers.

2.5. Desorbing moisture

Dry the fibers in an oven of 105°C up to a constant weight W_1(g), then watering the humidify the dried fiber to $2W_1$(g). Put the humidified the fiber in atmosphere room (RH25%, 33°C) for different times, then weighted its weight, respectively, W_2(g). The desorbing moisture of the fibers was calculated as following:

$$\text{Desorbing moisture of fibers } (\%) = [(W_2 - W_1)/W_1] \times 100\%.$$

2.6. Crystallinity

The X-ray diffraction of the fiber was tested with a D/MAX-2400 X-ray diffraction analyzer (Rigaku Co., Ltd., Japan). The test conditions were as follows: voltage = 46kV, current = 100mA, Cu Kα radiation, scanning scope $2\theta = 6 \sim 36°$, and scanning speed = 8°/min. Then, the crystallinity and crystalline index were calculated based on the X-ray diffraction result.

2.7. TG analysis

The weight loss rate was tested with Du-Pont951 thermogravimetric analyzer. Test was done under nitrogen gas, and temperature 30-500°C, temperature rise rate 30°C/min.

3. Results and Discussion

3.1. Orthogonal analysis

The residual gum content after the alkali-H_2O_2 treating is shown in Table 3.

Table 3. Orthogonal result

No.	A, g/L	B, g/L	C	D, min	Residual gum content, %
1	6	9	1:80	25	6.43
2	6	12	1:90	30	6.87
3	6	15	1:100	35	6.63
4	8	9	1:90	35	5.81
5	8	12	1:100	25	5.52
6	8	15	1:80	30	7.41
7	10	9	1:100	30	5.60
8	10	12	1:80	35	7.67
9	10	15	1:90	25	7.89

Using mathematical statistic theory [8], absolute difference was calculated, the result is shown in Table 4.

Table 4. Analysis of absolute differences

Level	NaOH conc.	H_2O_2 conc.	Bath ratio	Time
1	6.64	5.95	7.17	6.64
2	6.25	6.69	6.89	6.63
3	7.08	7.34	5.92	6.70
Absolute difference (R)	0.83	1.39	1.25	0.07

Table 4 shows that the factors influencing residual gum content was in order: H_2O_2 content > bath ratio > NaOH content > treatment time, and the combination of parameters was NaOH 8g/L, H_2O_2 9g/L, treatment time 30 min, and bath ratio 1:100.

3.2. Morphology of the fiber

Under the optimum treating conditions, the cotton stalk bark fiber was prepared. The morphology of the fibers was observed by using JSM5800 scanning electronic microscopy. It is showed that the surface of the fiber is not smooth (shown in Figure 2), the degummed fiber is not single fiber state, which is called a *technology fiber*. The morphology of the fibers is not regular. The morphology of the fiber is influenced by treating condition.

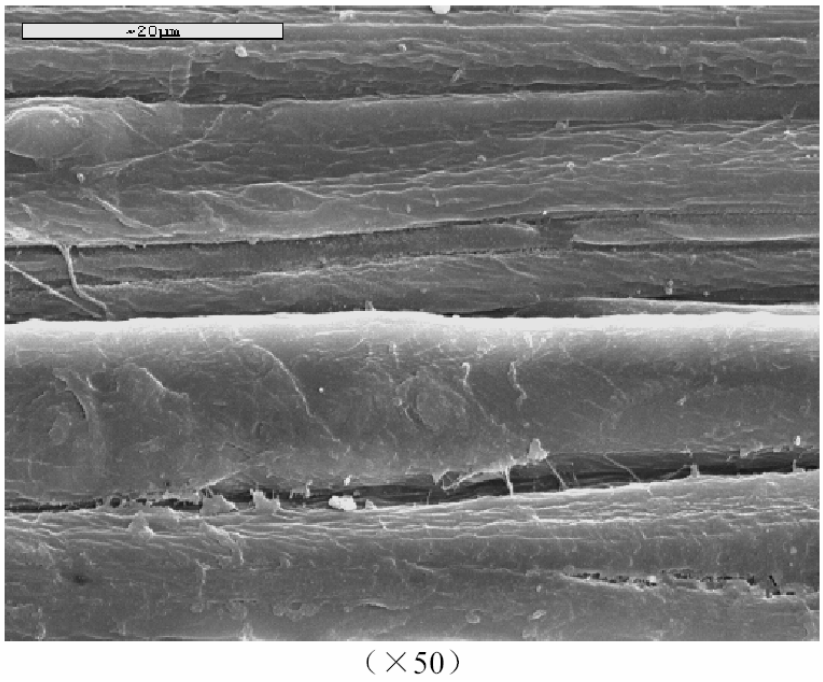

Figure 2. Fiber morphology.

3.3. Flexibility

The flexibility of the fiber is 2.93 twist/m·tex. So, the flexibility of cotton-straw bark fibers is poor.

3.4. Fiber length

The fiber length was tested through random taking fiber from the sample. The length of 100 fibers is shown in Figure 3. From the result, 18 fibers length is between 160mm~140mm, 20 fibers length is between 140mm~120mm, 18 fibers length is between 120mm~100mm, and 17 fibers length is between 100mm~80mm. So, the fibers length has a serious difference. It is because of fibers morphology structure.

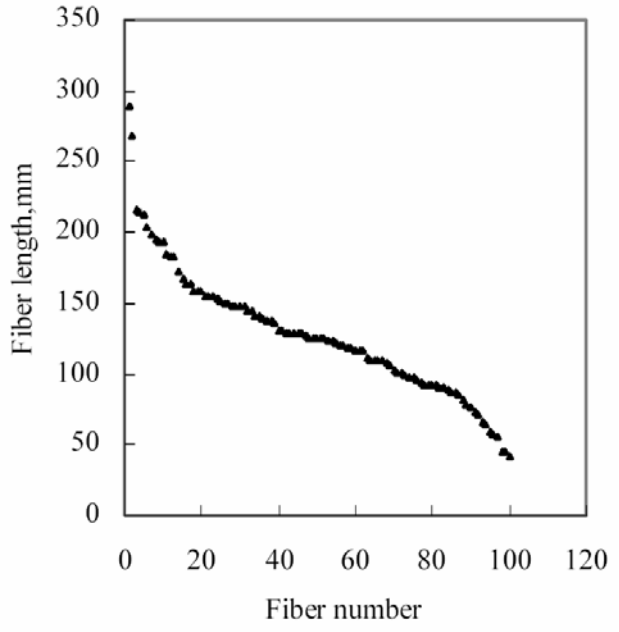

Figure 3. Fiber length distribution.

3.5. Desorbing moisture

The desorbing moisture of the fibers is shown in Figure 4. From the result, the fiber has fast desorbing moisture within 5 hours, then desorbing moisture decreases. When the time is 6 hours, the fibers reach its regain. The fast desorbing moisture may be related with the atmosphere condition.

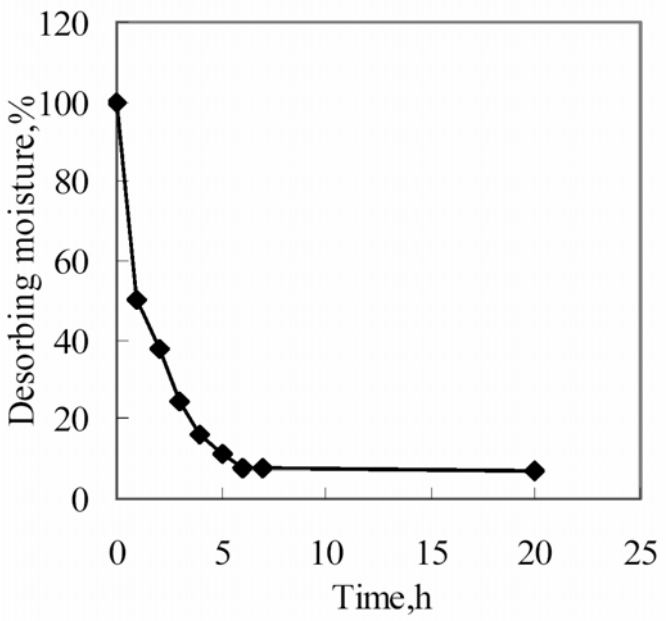

Figure 4. Desorbing moisture of the fibers.

3.6. Crystallinity of the fiber

The X-ray diffraction of the fiber is showed in Figure 5.

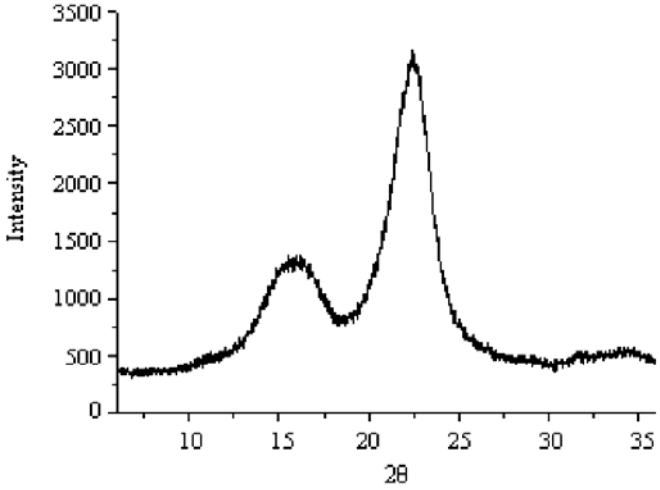

Figure 5. X-ray diffraction of cotton-straw bast cellulose fiber.

Crystalline peak and non-crystalline peak of X-ray diffraction diagram is decomposed by using the Gause function in Origin7.5 software. According to calculating method [7], the crystallinity (C_r) and crystalline index (C_rI) are calculated by the following equations:

$$C_r(\%) = S_e /(S_e + S_n), \tag{2}$$

where S_e is crystalline peak area and S_n is non-crystalline peak area.

$$C_rI(\%) = (I_{002} - I_{amorph})/ I_{002}, \tag{3}$$

where I_{002} is maximum diffraction peak intensity of main crystalline peak (002) and I_{amorph} is diffraction peak intensity of amorphous region at $2\theta = 18^{\circ}$.

On the basis of the Equations (2) and (3), the results of C_r and C_rI are listed in Table 5. As shown by the result in Table 5, the C_r and C_rI values of cotton-straw bast cellulose fiber are less than those of cotton [1]. So, as textile material, cotton-straw bast cellulose fiber dyes easily.

Table 5. C_r and C_rI values

S_e	S_n	I_{002}	I_{amorph}	C_r(%)	C_rI(%)
7690.5	7340.4	3163	766	51.16	75.78

3.7. TG analysis

The TG and DTG curves were shown in Figure 6. It is shown that weight loss happens, when temperature is 0-100°C because of water loss. The fibers begin thermal decomposition at 250°C, the weight loss is serious.

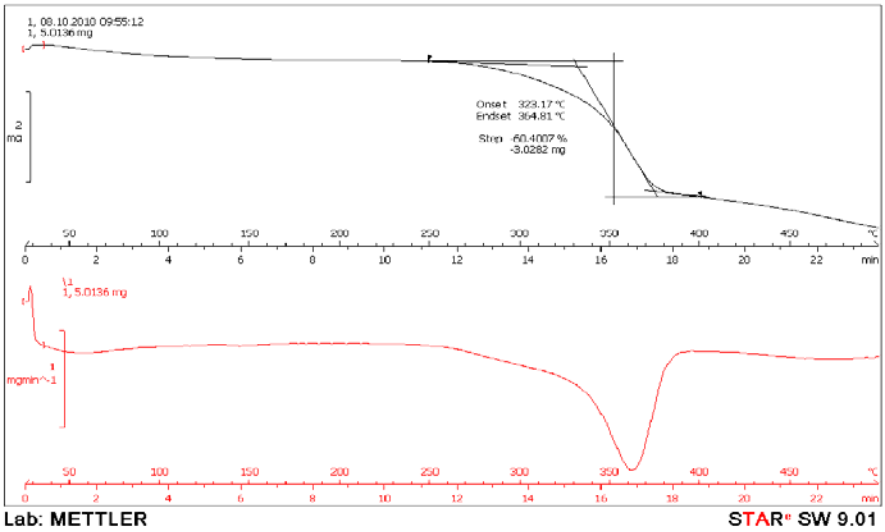

Figure 6. The TG and DTG curves.

The temperature of maximum thermal weight loss rate is 365°C. The thermal weight loss is 5.67% when temperature is 0-100°C; the thermal weight loss is 60.4% when temperature is 250-400°C.

4. Conclusion

Cotton stalk bark fiber is made by using alkali-H_2O_2 treatment. The surface of the fiber is not smooth, and the fiber is a technology fiber. The crystallinity and crystalline index of the fiber is lower compared with cotton fiber. The fiber is stiff, so it must be treated by softness agent before carding. The fiber has fast desorbing moisture, and the fibers begin thermal decomposition at 250°C.

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