(REFEREED RESEARCH)

PARAMETERS OPTIMIZATION FOR DEGUMMING OF COTTON-STRAW BAST FIBER

PAMUK BİTKİSİNDEN ELDE EDİLEN SAK LİFLERİNİN TERBİYESİNİN OPTİMİZASYONU

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ABSTRACT

Abstract: As a bast fiber, cotton-straw bast fiber consists of cellulosic and non-cellulosic substances, such as hemicellulose, pectin, lignin, and wax, and these noncellulosic substances affect the application of cotton-straw base fiber. Alkali-H2O2 treatment does not only remove hemicellulose, lignin and pectin effectively, but bleach fiber as well. Through orthogonal experimental, the degumming for first boiling and second boiling of alkali-H2O2 was investigated. Experimental results showed that the optimum combination of parameters for first boiling was 8g/L NaOH, 9g/L H2O2, 1:100 bath ratio and 30min treatment time by using mathematical statistic analysis. Optimum technological parameters of second boiling for degumming of cotton-straw base fiber were alkaline content 12g/L, hydrogen peroxide content 17g/L, temperature 80□, and time 60min by using the fuzzy comprehensive evaluation.

Keywords: Cotton-straw bast fiber, degumming, orthogonal experimental , alkali-H2O2 treatment, cellulose fiber, residual gum content, fiber fineness

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1. INTRODUCTION

Bast fibers are important fibers because they have biodegradable and ecofriendly characteristics. Efforts to exploit the wide use of bast fibers like hemp, coir, banana, sisal, and jute have been an area of interest(1-3). As agricultural waste, cotton-straw usually is used for paper-making, board-making, building material and feed(4). Beside, some cotton-straw is burned. Cotton-straw bast fiber comes from surface layer of cotton-straw. There is about 4 million tons of cotton-straw bast fiber annual in China. As a bast fiber, cotton-straw bast fiber has not been studied and used widely. Cotton-straw bast fiber consists of cellulosic and non-cellulosic substances, such as hemicellulose, pectin, lignin, and wax, and these noncellulosic substances affect the application of cotton-straw base fiber. The blends of the treated cotton-straw bast fibers/cotton were spun in rotor spinning, it was found that maximum blending ratio of treated cotton-straw bast fibers was 50 percent (5), the blending ratio is over 50 percent, the spinning could not be carried out. The poorer flexibility of treated cotton-straw bast fibers produced their poorer cohesion, which originated from residual noncellulosic substances in treated cotton-straw bast fibers. Therefore, the removal of most noncellulosic components is a prerequisite problem in order to make good use of cotton-straw base fiber.

Usually, bast fibers are processed with bacterial, chemical, and enzymatic methods. Bacterial processing depends on the weather, water quality, and other factors and often results in inconsistent fiber quality(6). Enzymatic processing method was developed for flax in the 1980s(7,8). Chemical processing is effective for removing noncelulosic substances, and considerable research has focused on this method (9,10). It is known that bleaching improves the appearance and color of fibers and hydrogen peroxide is often used to bleach the fibers(11). It is known that bleaching or similar treatment improve the appearance of fibers and hydrogen peroxide is often used to bleach the textile fibers.

Earlier reports on the application of alkali-H_2O_2 one-bath process were used in dyeing and finishing(12). In this work, alkali-H_2O_2 one-bath treatment for degumming and bleaching of cotton-straw bast fiber was investigated. The optimum parameters were obtained by using fuzzy orthogonal design.

2. EXPERIMENTAL

2.1 Material

Cotton-straw bast fibers were from Xi'an city of Shaanxi Province of China. The components of cotton-straw bast fiber are tested according to the Chinese Standard of GB5888-86**.** GB5888-86 is about method of quantitative analysis of ramie chemical components, of which wax content, water soluble content, pectin content, hemicellulose content, lignin content, and cellulose content can be tested according to GB5888-86. The results showed that the content of lignin and hemicellulose in cotton-straw bast fiber were 25.88 % and 25.23 %, respectively, which is higher than that of flax or ramie(13). So, the degumming parameters of cotton-straw bast fiber will be different from that of flax or ramie.

Molecular weight of hemicellulose is lower than that of cellulose, acid, alkali and oxidizing agent have a strong action on hemicellulose(14). During degumming, the hemicellulose is removed through alkali solution treatment. Lignin is a complex amorphous polymer consisting of propane benzene crosslikned by ether bonds and C-C bonds(15); it can be resolved in alkali solution. During alkali-H_2O_2 one-bath treatment, alkali not only removes most of the hemicellulose, 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(15).

2.2 First boiling experimental

Cotton-straw bast fibers are treated firstly by acid solution before first boiling. Acid treatment condition is following as:

H_2SO_4(98%)content 5 g/L, $NaSO_3$ content15 g/L, bath ratio1:30, treatment temperature 60℃, treatment time 2.5h

Removing noncellulosic substances from cotton-straw bast fiber is main objective of this experiment. Through the experiment, it was found that two stage processes to cotton-straw fiber could remove more noncellulosic substances. Therefore, the two stage processes to cotton-straw fiber were used in this work.

Orthogonal design parameters for first boiling of cotton-straw bast fiber are showed in Table 1. The all treatment temperature is 100°C.

Table 1. Orthogonal design parameters for first boiling

Level	NaOH content (g/L)	H_2O_2 content (g/L)	bath ratio	treatment time(min)
1	6	9	1 : 80	25
2	8	12	1 : 90	30
3	10	15	1 : 100	35

2.3 Second boiling experimental

After first boiling, the cotton-straw bast fiber is treated again by alkali-H_2O_2, which is called second boiling. Orthogonal design parameters for second boiling are showed in Table 2. Bath ratio is 1:100. After second boiling, the fibers are washed using H_2SO_4 solution. Washing conditions: H_2SO_4(98%)content 1 g/L, bath ratio 1:30, time 30min. Washing temperature is room temperature.

Table 2. Orthogonal design parameters for second boiling

level	NaOH content (g/L)	H_2O_2 content (g/L)	temperature(°C)	treatment time(min)
1	8	15	60	40
2	10	17	80	60
3	12	19	100	80

2.4 Testing

The residue gum content of fibers(RG) in % was calculated using the Equation 1.

$$RG \text{ in } \% = [(M_1 - M_2)/M_1] \times 100 \quad (1)$$

Where, M_1 and M_2 are the gum content of the raw cotton stalk bark and the gum content of degummed samples, respectively.

The fibers were conditioned in a standard testing atmosphere of 21℃ 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 Chinese Standard for Test method for linear density of jute and kenaf fibres-Cut middles method (GB/T12411.3-90).

The residual lignin content of the fibers was tested according to Chinese Standard for Method of quantitative analysis of ramie chemical component (GB5889-86).

The infrared spectroscopy curve of the fibers was analyzed using FT-IR spectrometer at spectrum range 4000-500cm^{-1}, resolution 4 cm^{-1}, and scanning frequency 32.

3. RESULTS AND DISCUSSION

3.1 First boiling experimental result

The residual gum content of cotton-straw bast fiber is regarded as an evaluation index for first boiling. The experimental result is showed in Table 3. Using mathematical statistic (16), absolute difference analysis result of residual gum content is showed in Table 4.

Based on the absolute difference in Table 4, experimental factors influence the ability of residual gum content is listed as H_2O_2 content > bath ratio> NaOH content> treatment time. To reduce the residual gum content of cotton-straw bast fiber in first boiling, based on the calculated minimum value of different parameters at each level in Table 4, selecting the corresponding level as optimum treatment condition of each factor. So the optimum combination of parameters is 8g/L NaOH, 9g/L H_2O_2, 1:100 bath ratio and 30min treatment time.

Table 3. Orthogonal experimental result for first boiling

treatment No.	NaOH content (g/L)	H_2O_2 content (g/L)	bath ratio	treatment time(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	7.98

Table 4. Analysis of absolute difference of the residual gum content for first boiling

level	NaOH content	H_2O_2 content	bath ratio	treatment time
1	6.643	5.947	7.170	6.643
2	6.247	6.687	6.887	6.627
3	7.083	7.340	5.917	6.703
Absolute difference	0.836	1.393	1.253	0.076

3.2 Second boiling experimental result

After second boiling, the residual gum content and fibers linear density are regarded as evaluation indices for cotton-straw bast fiber. The experimental result is showed in Table 5. Using mathematical statistic, absolute difference analysis results of residual gum content and linear density are showed in Table 6 and Table 7, respectively.

Based on the absolute difference of residual gum content in Table 6, experimental factors influence the ability of residual gum content is listed as temperature >treatment time>H_2O_2 content > NaOH content. To reduce the residual gum content of cotton-straw bast fiber in second boiling, the combination of parameters is 8g/L NaOH, 19g/L H_2O_2, 80°C temperature, and 80min treatment time.

Table 5. Orthogonal experimental result for second boiling

No.	NaOH content (g/L)	H_2O_2 content (g/L)	temperature (°C)	treatment time(min)	residual gum content(%)	fiber fineness (tex)
1	8	15	60	40	5.54	4.46
2	8	17	80	60	5.11	3.85
3	8	19	100	80	5.60	3.02
4	10	15	80	80	5.48	3.66
5	10	17	100	40	5.58	2.96
6	10	19	60	60	5.42	3.86
7	12	15	100	60	5.61	2.77
8	12	17	60	80	5.53	3.78
9	12	19	80	40	5.14	4.41

Table 6. Analysis of absolute difference of the residual gum content for second boiling

level	NaOH content	H_2O_2 content	temperature	treatment time
1	5.417	5.543	5.497	5.420
2	5.493	5.407	5.243	5.380
3	5.427	5.387	5.597	5.537
Absolute difference	0.076	0.156	0.354	0.157

Table 7. Analysis of absolute difference of the linear density for second boiling

level	NaOH content	H_2O_2 content	temperature	treatment time
1	3.777	3.630	4.033	3.943
2	3.493	3.530	3.973	3.493
3	3.653	3.763	2.917	3.484
Absolute difference	0.284	0.233	1.116	0.456

Based on the absolute difference of linear density of fibers in Table 7, experimental factors influence the ability of residual gum content is listed temperature>treatment time>NaOH content >H_2O_2 content. To reduce the linear density of cotton-straw bast fiber in second boiling, the combination of parameters is 10g/L NaOH, 17g/L H_2O_2, 100°C temperature, and 80min treatment time.

From the analysis results of absolute difference of the residual gum content and linear density for second boiling, the combination of parameters for the residual gum content and linear density are different. To evaluate the residual gum content and linear density and obtain a reasonable combination of parameters, fuzzy mathematical theory is used for analysis of orthogonal experimental results in second boiling.

3.3 Fuzzy method evaluation for Second boiling

Based on fuzzy mathematical theory(17), the subordination degree and comprehensive evaluation value for residual gum content and linear density are showed in Table 8.

Based on the result in Table 8, four fuzzy sub-sets for experimental factors are following as:

$\underset{\sim}{C}_1$ =(0.324,0.330,0.346), $\underset{\sim}{C}_2$ =(0.256,0.392,0.352),

$\underset{\sim}{C}_3$ =(0.196,0.419,0.385), $\underset{\sim}{C}_4$ =(0.281,0.428,0.296)

According to the principle of maximum subordination degree, individual factor influencing degree is following as:

$\underset{\sim}{C}_1$ =0.346, $\underset{\sim}{C}_2$ =0.392, $\underset{\sim}{C}_3$ =0.41, $\underset{\sim}{C}_4$ =0.428

So, the ability of individual factor influencing fuzzy comprehensive evaluation value is listed as: $\underset{\sim}{C}_4 > \underset{\sim}{C}_3 > \underset{\sim}{C}_2 > \underset{\sim}{C}_1$, that is, treatment time>temperature> H_2O_2 content > NaOH content

According to individual factor influencing degree, the corresponding level is selected as optimum treatment condition of each factor. So, the optimum parameters for degumming of cotton-straw bast fiber are 12g/L NaOH, 17g/L H_2O_2, 80℃ temperature and 60min treatment time.

For level combination of all possible 81 factors, optimum combination is C_{13} C_{22} C_{32} C_{42}. The subordination degree is 0.346∧0.392∧0.419∧0.428=0.346, that is, the condition of serious influencing comprehensive evaluation value is 12g/L NaOH, 17g/L H_2O_2, 80℃ temperature and 60min treatment time.

Under the conditions of 8g/L NaOH, 9g/L H_2O_2, 1:100 bath ratio and 30min for first boiling and 2g/L NaOH, 17g/L H_2O_2, 80℃ temperature and 60min for second boiling, the residual gum content and the residual lignin content of the fibers is 5.52% and 9.34%, respectively. The infrared spectroscopy curves of cotton-straw bast fibers and flax fibers are shown in Figure1 and Figure 2, respectively. Table 9 shows infrared transmittance peaks (cm^{-1}) of cotton-straw bast fibers and flax fibers. Compared with the infrared spectroscopy curve of flax fibers, it is showed that the cotton-straw bast fibers has a serious infrared transmittance peak at 3746.7cm^{-1}, other peaks are similar to the peaks of flax fibers.

Table 8. The subordination degree and comprehensive evaluation value

No.	NaOH content (g/L)	H_2O_2 content (g/L)	temperature (°C)	treating time (min)	residual gum content	fiber fineness	comprehensive evaluation value, b_n
1	8	15	60	40	0.14	0	0.07
2	8	17	80	60	1	0.361	0.681
3	8	19	100	80	0.02	0.852	0.436
4	10	15	80	80	0.26	0.473	0.367
5	10	17	100	40	0.06	0.888	0.474
6	10	19	60	60	0.38	0.355	0.368
7	12	15	100	60	0	1	0.5
8	12	17	60	80	0.16	0.402	0.281
9	12	19	80	40	0.94	0.03	0.485
$\sum b_{i1}$	1.187	0.937	0.719	1.029			
$(\sum b_{i1})'$	0.324	0.256	0.196	0.281			
$\sum b_{i2}$	1.209	1.436	1.533	1.567			
$(\sum b_{i2})'$	0.330	0.392	0.419	0.428			
$\sum b_{i3}$	1.266	1.289	1.41	1.084			
$(\sum b_{i3})'$	0.346	0.352	0.385	0.296			

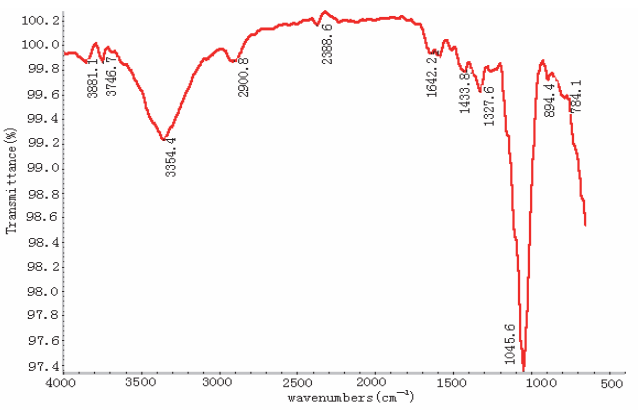

Figure1. The infrared spectroscopy curve of cotton-straw bast fibers

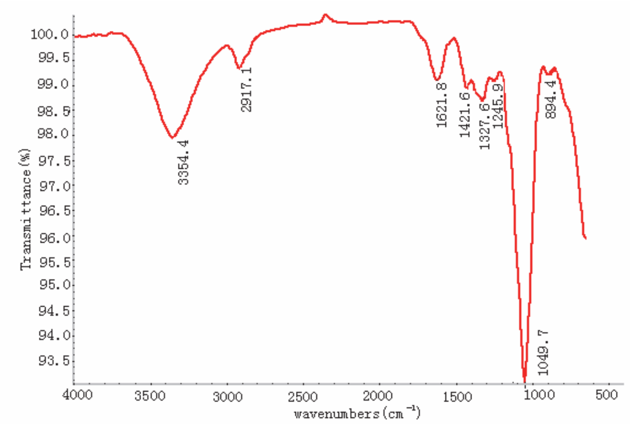

Figure 2. The infrared spectroscopy curve of flax fibers

Table 9. Infrared transmittance peaks (cm^{-1})

possible assignment	infrared transmittance peaks (cm^{-1})
hydroxyl OH- stretching vibration in cellulose, hemicellulose, and lignin	3746.7
hydrogen bond OH stretching vibration	3354.4
C-H_2 asymmetrical stretching vibration	2900.8
C-H_2 symmetrical stretching vibration	2388.6
C-C stretching vibration	1642.2
C-H bending vibration	1433.8
O-H In-plane bending	1327.6
C-O stretching vibration	1045.6
alkene C-H out-of - plane bending vibration	894.4
O-H out-of -plane bending	784.1

4. CONCLUSIONS

Cotton-straw bast fiber contains high content of content of lignin and hemicellulose before degumming. After second boiling, the residual gum content reduces seriously. Using the fuzzy comprehensive evaluation for residual gum content and linear density of fibers, optimum technological parameters of secone boiling for degumming of cotton-straw base fiber are alkaline content 12g/L,hydrogen peroxide content 17g/L,temperature 80°C,time 60min.

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