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Alkali Degumming and Refining Technology for Chinese Fevervine Fibre

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Abstract
In this paper, the Rubiaceae Chinese Fevervine stem was used as raw material, with pool dipping, manual decorticating, acid dipping and sodium hydroxide scouring twice as the degumming method, to produce Chinese Fevervine Fibre (CF fibre). Through an orthogonal experiment, optimised two-fold sodium hydroxide degumming using refining technology was achieved: in the first alkali degumming, the NaOH concentration was 1.4% at a temperature of 95 °C for 3.5 hours; in the second , the NaOH concentration was 0.8% at a temperature of 95 °C for 2.5 hours; and in the acid dipping, the sulfuric acid concentration was 6% at room temperature for 0.5 hours. The CF fibre produced has a spinnability of 39.90 mm length, a linear density of 2.82 dtex, a tenacity of 18.56 cN/dtex, and a break elongation of 2.81%.

Key words: *Chinese Fevervine fibre, alkali degumming; scouring, spinnability, fibre property.*

RESEARCH & DEVELOPMENT

Introduction

In recent years, there have been many reports on the development and application of new plant fibres, which generally belong to cellulose fibre. This research work focuses on how to degum and refine fibre so as to improve the fibre spinnability. Zhang Yi et al [1] modified pineapple leaf fibre with sodium hydroxide, and then treated it with laccase and acid xylanase. The biochemical combined degumming process was used to remove lignin and other colloids in the pineapple leaf fibre. Yin Yunlei et al [2] undertook the steam explosion-chemical combination method to refine hemp fibre, as a result of which fibre spinning performance was improved remarkably. Liu Fang et al [3] carried out the degumming of European hemp fibre by alkali degumming combined with the ultrasonic method, where the pectin removal rate and residual pectin rate of the final hemp fibre was 77.45% and 7.50%, respectively. In order to solve the problems of large water consumption and serious pollution in the traditional degumming process, Gao Shihui [4] combined supercritical fluid CO_2 and biological enzyme by the degumming method for apocynum bast fibre, and obtained good results. Zhang Qiuya et al [5] pretreated apocynum bast fibre with alkali and then degummed it with the Bio-degumming method, where the content of wax and lignin in the fibre decreased significantly. Ryszard Kozłowski et al [6-7] modified flax fibres and hemp fibres with the enzymatic cottonization process, the flax fibre linear density was decreased by 54% and the length by 66%. The hemp fibre linear density and length were decreased from 5.6 tex and 162 mm to 3.34 tex and 37.4 mm, respectively.

There are also many reports on the plant fibre degumming and refining methods, such as high-pressure alkali degumming, biochemical combined degumming, mechanical action and biological enzyme binding, laccase treatment, ultrasonic treatment, twice alkali boiling, etc. These methods were used in the treatment of cotton stalk fibre [8-10], carex meyeriana fibre [11], banana stalk bast fibre [12], mulberry bast fibre [13], and lotus stalk fibre [14], respectively.

Chinese Fevervine belongs to the Rubiaceae plant, the other name of which is Paederiae Herba. It is a perennial liana, growing on hillsides, in forests, on forest margins, and among valley side shrubs at an altitude of 200-2000 meters. The stem length is about 3~5 meters. The plant is widely distributed in the Asia tropical zone and can grow in harsh ground, such as on country roadsides, in rock apertures, brushwood, moats, field edges, etc. The distribution in China is in the Yangtze River basin and the southern region of the River, but it also occurs in North Korea, Japan, India, Myanmar, Thailand, Vietnam, Laos, Cambodia, Malaysia, and Indonesia [15].

Chinese Fevervine stem phloem under the epidermis is rich in cellulose fibre. In a previous work [16], Chinese Fevervine fibre (CF fibre) was obtained by natural pool dipping, manual decorticating, and sodium hydroxide scouring. The CF fibre produced possesses good spinnability with the following properties: cellulose content 82.82%, specific gravity 1.323 g/cm^3, length 41 mm, diameter 40.29 μm, linear density 2.93 tex, single fibre tenacity 55.4 cN, tenacity 18.9 cN/dtex, and break elongation 2.87%.

Based on the results of the previous research work [16], this paper uses the Chinese Fevervine stem as raw material to refine CF fibre by pool dipping, manual decorticating, acid dipping and twice-alkali degumming. The purpose was to minimise the fibre linear density as well as improve the softness and cohesiveness of the fibre under the condition of other properties making minor changes, so as to improve fibre spinnability.

Experiment

Chemicals and instruments

Main chemicals: sodium hydroxide, sulphuric acid, sodium silicate, Penetrant JFC. All are of chemical purity.

Main instruments: Y172 Hastelloy fibre slicing knife, YG004A Electronic fibre tenacity tester.

Raw material preparation

Chinese Fevervine is a perennial plant. In the autumn, its mature stalk (preferably two years or more) is used as raw material. After 30 days of natural pool dipping, the phloem and stem xylem were separated by the action of microorganisms, and the skin manually peeled off. The phloem is rich in cellulose fibres, which were washed and dried for use.

Experimental design and steps

The overall experimental process is as follows: acid dipping – washing – first alkali scouring – washing – acid washing – washing – second alkali scouring – washing – acid washing – washing – drying – oiling – opening – property testing.

In the process, the technology factors of acid dipping and two-fold alkali scouring are important for the fibre properties, thus we take the acid dipping concentration, twice alkali scouring concentration, and the two-fold alkali scouring time as five factors, with four levels for each factor, An $L_{16}(4^5)$ orthogonal experiment [17] was designed, shown ***Table 1***.

In the experiment, the acid dipping temperature was room temperature, the acid dipping time 0.5 h; the mass percentage of sulfuric acid and alkali is shown in ***Table 1***; the alkali scouring temperature is 95 °C, the bath ratio 1:5, and the mass concentrations of stabiliser sodium silicate and penetrant JFC in the scouring liquid weas 0.1% and 0.05%, respectively.

Cleaning: rinse with room temperature water, the purpose was to remove the colloid, impurities, dust, etc. that had fallen off from the fibre. Acid washing: 0.1% dilute sulfuric acid at room temperature was used to neutralise residual alkali, and also to remove some of the gum and impurities. Drying: drying at 110 °C for 90 minutes in an oven. Oiling and opening: hemp fibre softening oil was sprayed onto the CF fibre. which was then sealed in a plastic bag for at least 24 hours to fully soften the fibre and then opened with a small opening machine to obtain refined CF fibre. Property testing: the CF fibre length, linear density, tenacity, elongation at break, etc. were tested and analysed with the aim of obtaining optimal conditions for acid dipping and the two-fold alkali scouring process.

Table 1. *Orthogonal experimental program.*

Experiment number	First alkali scouring concentration, %	First alkali scouring time, h	Second alkali scouring concentration, %	Second alkali scouring time, h	Acid dipping concentration, %
1	1.0	2.5	0.6	1.5	4
2	1.0	3.0	0.8	2.0	6
3	1.0	3.5	1.0	2.5	8
4	1.0	4.0	1.2	3.0	10
5	1.2	2.5	0.8	2.5	10
6	1.2	3.0	0.6	3.0	8
7	1.2	3.5	1.2	1.5	6
8	1.2	4.0	1.0	2.0	4
9	1.4	2.5	1.0	3.0	6
10	1.4	3.0	1.2	2.5	4
11	1.4	3.5	0.6	2.0	10
12	1.4	4.0	0.8	1.5	8
13	1.6	2.5	1.2	2.0	8
14	1.6	3.0	1.0	1.5	10
15	1.6	3.5	0.8	3.0	4
16	1.6	4.0	0.6	2.5	6

Table 2. *Fibre property test results.*

Experiment number	Length, mm	Linear density, dtex	Tenacity, cN/dtex	Elongation at break, %
1	42.1	3.13	19.56	3.02
2	42.0	3.08	19.44	2.99
3	41.8	2.99	19.35	2.89
4	39.9	2.96	19.23	2.81
5	41.0	2.95	19.22	2.80
6	39.9	2.94	19.21	2.80
7	39.9	2.89	19.19	2.79
8	40.0	2.88	19.18	2.83
9	39.8	2.79	18.99	2.81
10	39.7	2.80	18.98	2.81
11	39.6	2.81	18.78	2.75
12	39.6	2.78	18.72	2.79
13	39.3	2.94	18.73	2.88
14	39.2	2.93	18.78	2.84
15	39.3	2.80	18.42	2.69
16	39.2	2.77	18.33	2.70

CF fibre property testing method

Fibre length test: approximately 1 gram of CF fibre was randomly selected and the length of a single fibre measured with a velvet board, tweezers, ruler and other tools to obtain the average length fibre. Linear density test: the linear density was tested by the mid-section cutting and weighing method with a Y172 Hastelloy fibre slicing knife. The strength and elongation at break were tested with a YG004A Electronic fibre strength tester, with each sample being tested 30 times and the average value taken as the final result.

Experiment results and analysis

Experiment results

Test results of the length, linear density, tenacity and elongation at break of the refined CF fibre obtained from 16 experimental schemes are shown in ***Table 2***.

Analysis of results

The average fibre property values and extreme difference (Extr-diff) were calculated with respect to the five factors and four levels according to ***Table 1*** of the orthogonal experimental scheme and ***Table 2*** of the fibre property test results. The results calculated are listed in ***Table 3***.

Table 3. *Average CF fibre property values and extreme difference.*

Properties	Four levels and Extr-diff	Five factors				
		First alkali scouring concentration, %	First alkali scouring time, h	Second alkali scouring concentration, %	Second alkali scouring time, h	Acid dipping concentration, %
Length, mm	1	41.45	40.55	40.20	40.20	40.28
	2	40.20	40.20	40.33	40.23	40.23
	3	39.68	40.15	40.20	40.43	40.15
	4	39.25	39.68	39.70	39.73	39.93
	Extr-diff	2.20	0.87	0.63	0.70	0.35
Linear density, dtex	1	3.04	2.95	2.91	2.93	2.90
	2	2.92	2.94	2.90	2.93	2.88
	3	2.79	2.87	2.90	2.88	2.91
	4	2.86	2.85	2.89	2.87	2.91
	Extr-diff	0.18	0.10	0.02	0.06	0.03
Tenacity, cN/dtex	1	19.39	19.13	18.97	19.06	19.04
	2	19.20	19.10	18.95	19.03	18.99
	3	18.87	18.94	19.08	18.97	19.00
	4	18.57	18.87	19.22	18.96	19.00
	Extr-diff	0.82	0.26	0.27	0.10	0.05
Elongation at break, %	1	2.93	2.88	2.82	2.86	2.83
	2	2.81	2.86	2.82	2.86	2.82
	3	2.79	2.78	2.84	2.80	2.80
	4	2.78	2.78	2.82	2.78	2.80
	Extr-diff	0.15	0.10	0.02	0.08	0.03

From the Max-diff of the four levels in ***Table 3***, among the five influencing factors, the alkali concentration and alkali scouring time of the first alkali scouring have a significant effect on the fibre properties, followed by the second alkali scouring effect. The acid dipping concentration has little effect, hence the first alkaline scouring process is mainly considered when selecting optimised technology, followed by the second alkaline scouring process.

All factors have little effect on the fibre length and elongation at break. It can be considered that CF fibre has good acid and alkali resistance as well as good flexibility and tensile strength.

The fibre linear density gradually decreased with an increase in the alkali concentration and alkali scouring time of the first alkaline scouring, which was beneficial to refining the fibre. However, the fibre length, strength and elongation at break gradually decreased. After comprehensive consideration, the optimal conditions for the first alkali scouring is an alkali concentration of 1.4%, and alkali scouring time of 3.5 h.

Along with an increase in the alkali concentration and scouring time of the second alkali scouring, the changes in fibre length, fibre linear density, strength and elongation at break are similar to those of the first alkali scouring; but the fibre property Extr-diff is much reduced. Therefore, the conditions of the second alkali scouring have a small effect on the fibre properties. After comprehensive considertaion, the optimal conditions of the second alkali scouring are an alkali concentration of 0.8%, and alkali scouring time of 2.5 h.

It is seen from the Extr-diff that the acid dipping concentration has little effect on all fibre properties, but in the experiment, it was found that acid dipping facilitates the peeling off of the surface crust from the fibre-containing phloem, as well as the removing of hemicellulose and lignin. After comprehensive consideration, the optimal conditions for acid dipping are an acid concentration of 6%, and acid dipping time of 0.5 h.

In summary, the optimal conditions were obtained: in the first alkali degumming: alkali concentration 1.4%, at temperature 95 °C, for 3.5 hours; in the second alkali degumming: alkali concentration 0.8%, at temperature 95 °C, for 2.5 hours; and in acid dipping: sulfuric acid concentration 6%, at room temperature, for 0.5 hours.

Verification experiment of optimal conditions

Using the optimal conditions above, a verification experiment was repeated 3 times, and the average property values of the CF fibre were as follows: linear density 2.82 dtex, length 39.90 mm, tenacity 18.56 cN/dtex, and elongation at break 2.81%. The experimental results show that the length, strength and elongation at break are slightly reduced comparing with a previous work [13]: length 41 mm, tenacity 18.90 cN/dtex, and elongation at break 2.87%; however. the reduction has little negative effect on the overall fibre spinnability. The fibre linear density is 0.11 dtex lower than that of the previous work – 2.93 dtex [13]; the fibre is refined to a certain extent, and the spinnability is improved.

Conclusions

Through small-scale planting experiments, it was found that this plant is easy to cultivate, and has a high stem and medium fibre yield [16].

The length of CF fibre is about 40 mm, which is similar to cotton-type chemical fibre; the linear density is slightly thicker than that of cotton and thinner than that of wool, the elongation at break close to that of natural plant fibres, the strength much higher than that of ordinary natural plant fibre, and it has better spinnability.
After planting, the ground part of the vine can be harvested from September to October every year and can be dried in the sunshine. The whole herb is a kind of Chinese Medicine, which has the functions of dispelling wind and dampness, eliminating food accumulation, promoting blood circulation, and relieving pain [15]. Therefore, it is necessary to conduct further study of whether CF fibre has

medical and health care functions. If true, planting, developing and using this fibre will achieve better economic benefits and have a good development prospect.

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