Effect of Alkaline Degumming on Structure and Properties of Lotus Fibers at Different Growth Period

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ABSTRACT

Lotus fibers are pulled from different period of lotus root. Alkaline was used as degumming agent to remove non-cellulose impurities of lotus fibers. Environmental scanning electron microscopy (ESEM), X-ray diffraction (XRD) and FTIR were used to characterize surface morphology and microscopic structure of lotus fibers before and after degumming. The effect of alkaline degumming on tensile properties of lotus fibers is investigated. The results show that impurities remove, hydrogen bond rupture, and crystallinity of partly armorphous chain occur during alkaline degumming. All of these changes play roles in influencing fiber tensile breaking force and elongation at break.

INTRODUCTION

In comparison with synthetic fibers, natural fibers are green and environmental friendly in environmental protection, comfort in dressing, and sometimes protective for people due to some natural beneficial ingredients. In recent years, various new natural fibers are developed and industrialized. Lotus (Nelumbo nucifera) is an aquatic vegetable which is extensively planted in China. It is reported that lotus root contains considerable amounts of effective ingredients including polyphenolic compounds, vitamins, and antioxidants [1-2]. More and more researchers, therefore, focus on application of lotus plants in food [3], medicine [4], and cosmetics [5]. Both the ingredients and abundant resources also inspire the development of the lotus fibers as a kind of textile fibers. Lotus fiber textiles become popular, especially in Japan and Myanmar [6-7]. All of these references, however, are lack of related quantitative data for production of lotus fiber textiles. The application of lotus plant in textile industry has not yet been deeply studied.

Since 2009, some systematically researches on lotus fibers have been done by Han et al [8-10]. Structure and physical properties of lotus fibers are clear to some extent, and non-cellulose impurities including pectin, hemicellulose and lignin are confirmed existence in lotus fibers. It is well-known that these impurities must be removed to ensure continuous spinning and yarn qualities. However, related quantitative data and mechanism are missing. The lack of data and theoretical support on impurities remove would limit further application of lotus fibers in textile industry.

In this paper, the degumming effect on lotus fibers at different growth period was assessed with crystalline and chemical change characterized by XRD and FTIR, morphological structure, and tensile properties. The quantitative data and mechanism analysis of degumming effect are carried out with NaOH as degumming agent [11]. The results are expected to provide valuable guidance for further textile producing process.

EXPERIMENTAL

Material

Fresh lotus root were collected from Jingzhou city of Hubei in China. The division of lotus root derived from different growth period of lotus plant is defined in *Table I*.

TABLE I. Division standard of lotus root fiber.

Period	Standard
1	Growing about 3 months. The lotus root is thin and crisp, and the cross section after broken is even.
2	Growing about 4 months. The lotus root is dark and stiff with obvious gum in surface. The cross section after broken is a little rough.
3	Growing about 5 months. The lotus root is very stiff and difficult to be broken. Once broken, the shape of cross section is irregular.

Fiber Extraction

The fibers are pulled from lotus root by hand, and *Figure 1* shows the pulling process. The length of the fibers from one lotus root is at least 10 cm. The fiber fineness shown in *Table II* was tested by YG252A optical microscopy (TECH, Beijing, China).

(a) Period 1 (b) Period 2

（c）Period 3

FIGURE 1. Pulling fibers from different growth period of lotus root.

TABLE II. Fiber fineness.

Period	1	2	3
Fineness/μm	11.69±0.17	12.78±0.22	15.38±0.22

As shown in *Figure 1(a)*, it is easy to pull fibers with high water content from fresh lotus of Period 1. However, the amount and fineness (See *Table II*) of the fibers are limited. At Period 2, the pulled fibers are plentiful and with strength, and the fineness increases from 11.69 μm to 12.78 μm. When lotus root continue growing to Period 3, there is no obvious difference in amount and appearance of the pulled fibers in comparison with that from Period 2. The fineness increases to 15.38 μm. Fibers pulled from different growth period are white and faint scent.

Fiber Degumming

Degumming with caustic soda is a prevalent process due to rapid and thorough dissolution of impurities. Therefore, alkaline was selected as degumming agents for treatment of lotus fibers. The fibers were immersed into NaOH solution with 10 g/L, 20 g/L and 30 g/L, respectively, and boiled in YX autoclave with power of 2 kW at 110 °C for 1 h, 2 h and 3 h. The degummed fibers were neutralized with H_2SO_4 of 10 g/L and rinsed with tap water for several times till the washing solution was neutral. Then the fibers were loosened and dried.

Fiber Characterization

X-ray diffraction (XRD) patterns were collected by a diffractometer (D/max-2250PC,Japan) using Cu Kα radiation at 40 kV and 50 mA (λ=1.54 Å).The diffraction angle (2θ) scanned from 10° to 45°. MDI Jade 5.0 software was used to compute crystallite. Fourier transform infrared spectra (FTIR) were collected using a TENSOR37 FTIR spectrometer (Bruker Company). The samples were analyzed in 800 - 4000 cm^{-1} range with a 4 cm^{-1} resolution. Environmental Scanning electron microscope (Quanta 250 FEG, U.S.A) was used to observe longitudinal structure of fiber samples. The fiber samples were mounted with electrical conductive gum and then sputter-coated with gold prior to examination. The thickness of sputtered gold for SEM observation was 10 nm.

Fiber Tensile Property

A model YG001A fiber tensile tester (Taicang, Jiangsu, China) was used to test tensile property of lotus fibers. The pretension was 0.2 cN. The gauge length was 2 cm and the loading speed was 8 mm/min. The test was carried out at room temperature with relative humidity of (65±5) %. Each value was averaged from 50 tests.

RESULTS AND DISCUSSION

XRD Analysis on Lotus Fibers

In order to analyze internal microscopic structure of fibers, XRD patterns of fibers pulled from lotus root at different growth stage before and after degumming were recorded as shown in *Figure 2*, and also crystallite of respective samples were computed with MDI Jade 5.0 software (*Table III*).

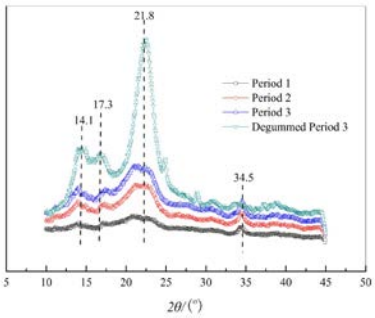


FIGURE 2. XRD pattern of lotus fibers.

TABLE III. Crystalline properties of lotus fibers.

2θ/(°)	Crystallite/nm			
	Period 1	Period 2	Period 3	Degummed Period 3
14.1	21.1	6.8	7.9	6.8
17.3	12.1	8.8	7.6	8.6
21.8	3.0	2.5	2.5	4.1
34.5	9.5	9.0	9.3	-

It can be seen from *Figure 2* that the lotus fibers at various growth period have 2θ diffraction peaks at 14.1˚, 17.3˚, 21.8˚, and 34.5˚, which are similar with typical cellulose I crystalline form [8]. With lotus root growing, 2θ diffraction peaks are more and more intense and sharp. In comparison with crystallite of period 1, the crystallite of period 2 and 3 gets smaller as illustrated in *Table III*. The results indicate that fiber crystallization is gradually improved during the whole growing stage. The most obvious increase in peak intensity occurs in spectrum of the degummed samples, and the respective peak except that of 34.5˚ is sharp and symmetrical. This could be due to formation of crystallized network by partly amorphous chains [11]. In addition, the disappeared peak at 34.5˚ and occurrence of some new peaks maybe indicate the transition possibility from cellulose I to cellulose II [12].

FTIR study

Figure 3 shows FTIR spectra of fibers at different growth period before and after NaOH treatment. The broad and strong peak at 3300-3400 cm^{-1} is attributed to hydroxyl group of cellulose I, which forms inter- and intra-molecular hydrogen bond. The peak at 1279-1368 cm^{-1} is due to stretching vibration of -CH_2 and -CH of cellulose. New band at 900 cm^{-1} occurs in spectrum of the degummed fiber. This band may be mostly assigned to bonding between cellulose and Na^+ due to partly substitution of hydrogen atom by sodium atom. It could be considered that hydrogen bond of cellulose I is disturbed by NaOH penetration. The band at 1733 cm^{-1} corresponds to carbonyl (C=O) stretching vibration, which is absent in spectrum of the degummed fibers. The carbonyl (C=O) appeared in spectra of lotus fibers at different growth stage derives from non-cellulose materials such as lignin, hemicelluloses, and pectin [13]. The disappeared band at 1733 cm^{-1} indicates that most non-cellulose materials are removed by degumming process.

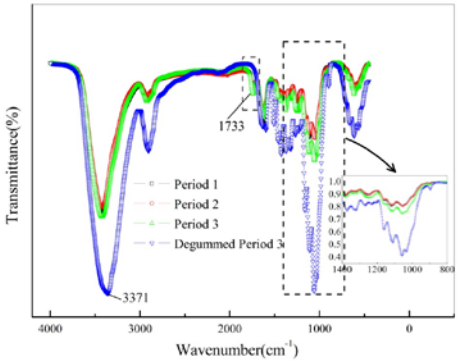
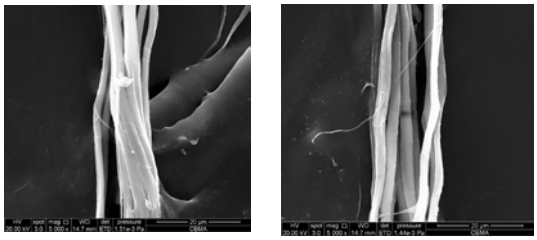


FIGURE 3. FTIR spectra of lotus fibers.

Meanwhile, the intensity of peak at around 1232 cm^{-1} attributed to acetyl ester weakens in spectrum of the degummed fibers. This could be due to rupture of linkage between cellulose and non-cellulose resulted from saponification reaction. The weakened intensity at 1232 cm^{-1} further confirms the removal of non-cellulose materials.

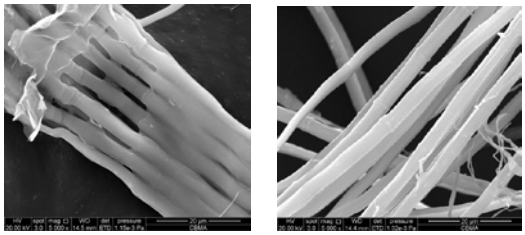
Morphology Comparison

Figure 4a and *Figure 5a* show the ESEM morphology of lotus fibers at different periods. It can be seen that fiber surface is coarse and fibers are connected by binding agents like pectin to fiber bundles. With the mature of lotus root, the binding agents gradually increase in quantity. After degummed by NaOH, the binding agents are apparently removed as shown in *Figure 4b* and *5b*. Fibers are individual in longitudinal view. In addition, no obvious corrosive dents are observed in surface of the degummed lotus fibers. This indicates that most of binding agents could be removed while no apparent damage to fibers at the present treatment conditions.



(a) (b)

FIGURE 4. Morphology of lotus fiber at Period 2 before (a) and after(b) alkaline boiling (5000×).



(a) (b)

FIGURE 5. Morphology of lotus fiber at Period 3 before (a) and after (b) alkaline boiling (5000×).

Effect of Alkaline Concentration on Fiber Mechanical Properties

Table IV shows the effect of NaOH concentration on fiber tensile properties. Fibers pulled from Period 2 and Period 3 are selected as fiber samples, and duration time is 1 h. It is seen from *Table IV* that fibers pulled from Period 2 is stronger than that from Period 3. Alkaline treatment significantly reduces fiber tensile breaking force, and about 50% of fiber breaking force is kept after degumming. This decrease, however, is not obvious with further increase of NaOH concentration and even the breaking force of fibers increases from 2.185 cN to 2.37 cN for Period 2 and from 1.72 to 2.37 cN for Period 3. Like most of bast fibers, the actual lotus fibers are fiber bundles bonded with non-cellulose matters as illustrated in *Figure 4* and *Figure 5*. It could be conducted, therefore, that both fiber internal structure and non-cellulose binder contribute to fiber strength. As far as fiber internal structure is concerned, hydrogen bond and strong crystalline regions contribute to fiber breaking force. During degumming process, the penetration of NaOH into fiber amorphous regions interrupts the inter- and intra-molecular hydrogen bond, which has been confirmed by FITR analysis. As a result, the fiber breaking force is weakened rapidly when treatment with 10 g/L NaOH for 1 h. obviously, the non-cellulose matters that bind fiber cells to fiber bundles are also removed with increase of NaOH concentration. When tensile force is loaded on fibers, the possibility of partially align of fiber cells increases due to the removal of binding agents, and thus increase load bearing capacity [11].

TABLE IV. Effect of alkaline concentration on tensile properties of lotus fibers.

Conc. of	Period 2		Period 3	
NaOH	Breaking	Elongation	Breaking	Elongation
(g/L)	force (cN)	(%)	force (cN)	(%)
0	5.31±0.38	1.98±0.23	3.94±0.34	1.95±0.28
10	2.65±0.23	1.60±0.24	2.65±0.23	1.59±0.24
20	2.185±0.27	1.90±0.35	1.72±0.47	1.50±0.30
30	2.37±0.37	1.40±0.29	2.37±0.37	1.40±0.29

Effect of Alkaline Treatment Time on Fiber Mechanical Properties

The effect of alkali treatment time on fiber tensile properties are also studied, and the results are shown in *Table V*. Fibers pulled from Period 3 are selected as fiber samples, and concentration of NaOH is 20 g/L. The influence and the reasons of treatment time on fiber tensile properties are similar with that of alkaline concentration. This further identifies that NaOH degumming significantly reduces fiber tensile breaking force whatever concentration and treatment time. In addition, it is seen from *Table V* that the elongation at break increases with treatment time. After a long period of boiling, it is expected that NaOH gradually penetrates amorphous areas and porous interstice of fibers, leading to fiber swelling. Thus, fibers become soft, and consequently the elongation at break of lotus fiber by different alkaline duration time increases.

TABLE V. Effect of duration time on tensile properties of lotus fibers.

Time(h)	Breaking force (cN)	Elongation(%)
0	3.94±0.34	1.95±0.28
1	1.72±0.47	1.50±0.30
2	1.70±0.51	2.0±0.23
3	2.13±0.32	2.1±0.24

According to the above analysis, the degumming process of lotus fibers with NaOH is simulated as illustrated in *Figure 6*. The simulation describes that the chemical and physical change of lotus fibers with NaOH degumming treatment. Chemically, partial rupture of hydrogen bond occurs due to alkaline disturbance, which reduces fiber breaking force. Physically, it is seen from *Figure 6* that the binding agents of non-cellulose matters are removed by dissolving NaOH solution. Single fibers are possible to be parallel for each other, and part amorphous chains are oriented. As a result, the tensile breaking force of lotus fibers maybe experienced increase. The contribution of chemical and physical to tensile properties are competed during degumming process. The present study shows that rupture of hydrogen bond is dominant when treatment with NaOH of 10 g/L for 1 h. Further increase concentration of NaOH or duration time, parallel of single fibers in lotus fiber bundles resulted from removal of binding agents and the oriented amorphous chains play dominant role in increasing tensile breaking force and elongation at break.

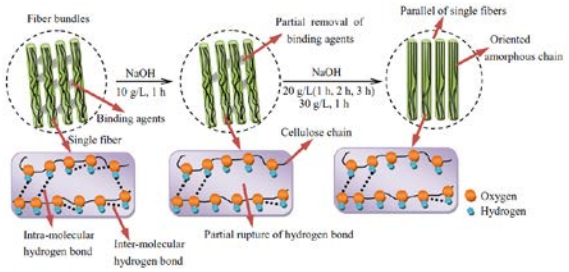


FIGURE 6. Simulation schematic for the present degumming process.

CONCLUSION

Lotus fibers are pulled from lotus root at different growth stage by hand. This paper addresses the effect of alkaline degumming on lotus fibers. XRD analysis indicates that the crystalline structure improves after alkaline treatment, and there occurs the transition possibility from cellulose I to cellulose II. FTIR results confirm the removal of non-cellulose impurities and rupture of hydrogen bond. In combination with microscopic and mechanical analysis, the perfected crystalline structure, impurities removal and hydrogen bond rupture are responsible for fiber tensile properties.

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