EXTRACTION AND CHARACTERIZATION OF CELLULOSE FIBERS FROM CATTAIL LEAVES: MORPHOLOGICAL, MICROSTRUCTURAL AND THERMAL PROPERTIES

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Due to the excessive exploitation of petrochemical resources, the development of new materials, especially natural fiber materials, has attracted increasing attention and extensive concern in academic and industrial fields. As a typical natural plant fiber, cattail has a chemical composition similar to that of hemp. However, its traditional degumming process is not only costly, but also prone to causing severe environmental pollution. In the present study, improved degumming methods were adopted: after dilute alkali pretreatment, cattail fibers were subjected to alkali-peroxide and pectinase treatments, respectively. The results indicated that both processes (dilute alkali pretreatment followed by pectinase treatment, and dilute alkali pretreatment followed by alkali-peroxide treatment) exhibited excellent degumming effects, and most of the non-cellulosic components (*e.g*., hemicelluloses, lignin and pectin) were effectively removed. Specifically, after pectinase treatment, the cellulose content and crystallinity were 60.37% and 59.43%, respectively. In contrast, after alkali-peroxide treatment, the content of cellulose and the crystallinity were 52.42% and 51.13%, respectively. In terms of degumming rate, the alkali-peroxide treatment achieved a degumming rate of approximately 40%, while the pectinase treatment yielded a degumming rate of more than 55%. Furthermore, the pectinase degumming was mild and caused little damage to fiber structure. Collectively, the cattail fibers prepared by the improved degumming methods are expected to become a promising alternative material in the textile industry.

Keywords: cattail fiber, pectinase, cellulose

INTRODUCTION

At present, natural fibers have attracted growing attention owing to their low cost, biodegradability, excellent mechanical properties as well as sustainable and abundant supply.[1,2] Among the wide variety of natural fiber sources, cattail (*Typha orientalis*) is a perennial aquatic plant, widely distributed in marshes, wetlands, lakes and ponds,[3] and its leaves possess thermal insulation and heat-clearing properties.[4] It can be exploited to produce novel plant cellulose fibers, which can diversify the feedstock for textile products and meet the current demand for green development.[5,6]

To improve the performance of cattail fiber, it is essential to enhance its spinnability through degumming treatment. As known, cattail fiber has a chemical composition similar to that of hemp, and it is usually degummed by conventional high temperature alkali boiling and bleaching processes.[7] Lin *et al*. extracted ramie fibers using a deep eutrophilic solvent (DES), and the results showed that the reusable degumming solution could still effectively remove most non-cellulose components from raw ramie even after four repeated degumming cycles.[8] Koschevic *et al*. bleached cattail with hydrogen peroxide, and non-cellulosic components such as the lignin, pectin and waxes were effectively removed.[9] Cui *et al*. conducted chemical dewaxing and bleaching treatment on cattail fibers, and the

results indicated that hemicelluloses and lignin in cattail fibers were successfully removed, with the cellulose content increasing from 41.66±1.11% to 89.72±1.07%.[10]

Each degumming method has its own advantages and disadvantages. The biological degumming process features simple reaction conditions, but exhibits high selectivity, which hinders its large-scale industrial production. Chemical degumming has the advantage of short reaction time, but it tends to generate waste liquid, which is unfavorable from an environmental perspective. Physical degumming is associated with high costs, and the degumming effect is affected by multiple factors during the reaction process. Therefore, to efficiently extract high-quality cattail fiber, it is necessary to explore a simple and efficient degumming method.[11,12]

Hence, the traditional degumming process of cattail fiber was improved in this study: pretreatment with a small amount of alkali was performed first, followed by alkali-peroxide treatment and pectinase treatment, respectively. The factors affecting the degumming process during the experiment, including pectinase concentration, sodium hydroxide concentration, hydrogen peroxide concentration, the reaction temperature and the time, were systematically investigated. Scanning electron microscopy (SEM), Fourier transform infrared (FT-IR) spectroscopy, and thermogravimetric analysis (TGA) were employed to compare and analyze the degumming effects of the two methods from the aspects of fiber morphology, chemical composition, and thermal stability. The extracted cattail fibers were fully characterized, aiming to provide a theoretical and experimental reference for the development and application of cattail-based products.

EXPERIMENTAL

Materials and chemicals

The raw materials used in this study were mature cattail plants from the gardens of Wuhan Textile University (Fig. 1). Pectinase, 98% sulfuric acid (H_2SO_4), sodium hydroxide (NaOH), 30% hydrogen peroxide (H_2O_2), sodium silicate (Na_2SiO_3), sodium tripolyphosphate ($Na_5O_{10}P_3$), and other chemicals of laboratory grade were procured from Aladdin Chemical Regent Inc., Shanghai, China. All the chemicals were used as received, without further purification.

Figure 1: Cattail plants (a) and dry mature cattail leaves (b)

Degumming processes

Figure 2 shows the degumming process of cattail fibers. The leaves were crushed, and then soaked in a hot water bath (50 °C) to remove the impurities and fluff from the surface, finally washed and oven dried for 24 h at 75 °C. Cattail fibers were pretreated with dilute alkali solution and then degummed with alkali-peroxide and pectinase, respectively. The degumming effects (the dosages of NaOH, H_2O_2 and pectinase, soaking temperature and soaking time) of different processes were compared and discussed. Three replicates were taken for each experimental setting and the average value was calculated.

Alkali pretreatment

Cattail fibers (5 g) were immersed in a 10 g/L sodium hydroxide (NaOH) solution with a fiber-to-solution mass ratio of 1:40. The solution was supplemented with sodium silicate (Na_2SiO_3) at a mass fraction of 3 wt%. Subsequently, the mixture was placed in a hot water bath maintained at 90 °C for 3 h of soaking treatment. After

the pretreatment, the cattail fibers were repeatedly rinsed with deionized water until the pH value of the effluent reached neutrality. Finally, the fibers were thoroughly rinsed again and oven-dried at 60 °C for 24 h to obtain pretreated cattail fibers, which were used for subsequent degumming experiments.

Alkali-peroxide treatment

The main influencing factors of alkali-peroxide degumming treatment included the concentrations of sodium hydroxide (NaOH) and hydrogen peroxide (H_2O_2), soaking time, reaction temperature, and bath ratio. For the experiment, 2 g of alkali-pretreated cattail fibers were placed in a beaker containing NaOH solutions with various concentrations (12, 14, 16, 18, 20, and 22 g/L) and H_2O_2 solutions with different concentrations (12.5, 15, 17.5, 20, 22.5, and 25 mL/L). Additionally, the effects of reaction time (1.5, 2, 2.5, 3, and 3.5 h) and reaction temperature (70, 75, 80, 85, 90, and 95 °C) on the degumming effect were systematically assessed. The optimal concentration ranges of NaOH and H_2O_2 were first determined through single-factor experiments, and then orthogonal experiments were conducted based on these ranges to obtain the optimal process parameters for alkali-peroxide degumming.

Pectinase treatment

Similar to the alkali-peroxide degumming process, the pectinase degumming treatment also had a range of influencing factors, including pectinase concentration, hydrogen peroxide (H_2O_2) concentration, soaking time, and reaction temperature. For the single-factor experiments, 2 g of alkali-pretreated cattail fibers were placed in a beaker, and the experimental variables were set as follows: pectinase concentration (12, 16, 18, 20, 22, and 24%), reaction pH (3, 3.5, 4, 4.5, 5, and 5.5), reaction time (11, 12, 13, 14, 15, and 16 h), and reaction temperature (40, 45, 50, 55, 60, and 65 °C). The optimal ranges of each influencing factor were first determined through single-factor experiments, and then orthogonal experiments were further conducted to optimize the process parameters and obtain the optimal conditions for pectinase degumming.

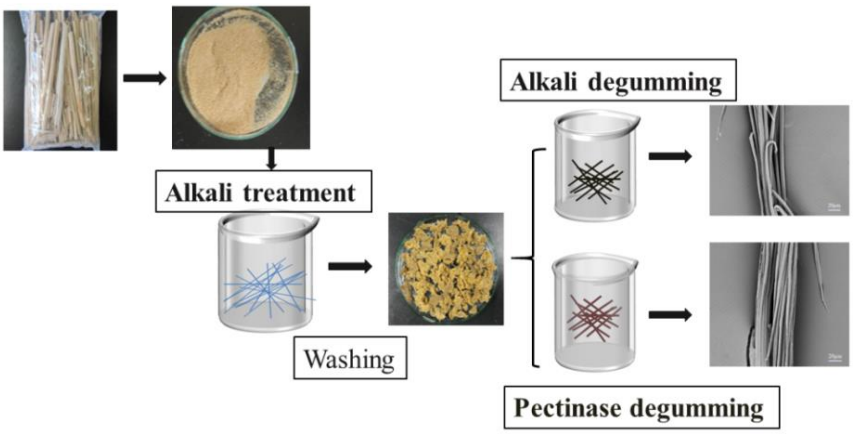

Figure 2: Degumming process of cattail fibers

Calculation of degumming rate

The degumming effect was assessed by determining the degumming rate. The degumming rate of fibers was calculated by the following formula:[13]

$$\text{Degumming rate} = \frac{W_1 - W_2}{W_1} \times 100\% \qquad (1)$$

where W_1 and W_2 are the weights of samples before and after degumming, respectively.

Chemical analysis

The cellulose, hemicellulose, lignin and pectin contents of the fiber samples were determined by chemical analysis according to the literature reported (GB5889-86:1986).[14] Five replicate samples were used for each test and the average values with standard deviation were reported.

Scanning electron microscopy (SEM)

The surface morphologies of all samples were observed using a scanning electron microscope (JSM-6510 LV, JEOL, Japan), with an electron beam accelerating potential of 5 kV. Prior to SEM evaluation, the samples were coated with a thin layer of gold by means of a plasma sputtering apparatus.

X-ray diffraction (XRD) measurement

The crystallinity of cattail fiber before and after treatment was determined by an X-ray diffractometer (D/max-2550pc, Rigaku). The experimental conditions were as follows: Cu Target K_α radiation (X-ray wavelength 0.154 nm), tube voltage 40 kV, tube current 30 Ma for scanning, scanning angle 5-40° and scanning speed 5 °/min. The crystallinity index (CrI) of the material was calculated using Equation 2:[15]

$$CrI(100\%)=(I_{200}-I_{am})/I_{200}\times 100 \quad (2)$$

where CrI represents the relative degree of crystallinity, I_{200} is the maximum intensity of the (200) lattice diffraction at $2\theta = 22.4°$, and I_{am} is the intensity of diffraction at around $2\theta = 18.2°$.

Fourier transform infrared spectroscopy (FTIR)

The treated and untreated sample powder was pressed into KBr pellets. The spectra were recorded on a Fourier transform spectrometer (Nicolet iS50, Thermo Fisher, USA) in the range of 4000-400 cm^{-1} with 16 scans, at a resolution of 4 cm^{-1}.

Thermogravimetric analysis (TGA)

TG analysis was employed to investigate the thermal stability of fibers before and after treatment. 3-5.0 mg powder samples were put into the differential thermal gravimetric analyzer (TG 209 F3) sample pool for thermal performance measurement, at a heating rate of 10 °C/min in the temperature range of 20-600 °C.

RESULTS AND DISCUSSION

Degumming process of cattail fibers

Orthogonal experiments were designed for the various factors involved, *i.e.* the mass concentration of sodium hydroxide, the concentration of pectinase, the concentration of hydrogen peroxide, reaction time and temperature; three levels being set for each factor. The range of the experiment was finally determined, as shown in Tables 1 and 3. The test results and related analysis are shown in Tables 2 and 4.

The orthogonal experiment was designed to optimize the pectinase degumming method and the alkali-peroxide degumming process of the cattail fibers. As shown in Table 2, the order of factors affecting the degumming effect of the alkali-peroxide method is the following: (C) temperature > (D) time > (A) NaOH mass concentration > (B) H_2O_2 concentration. The optimal scheme of the orthogonal experiment for alkali-peroxide degumming is: $A_3B_3C_2D_3$, which corresponds to a NaOH concentration of 22 g/L, a hydrogen peroxide concentration of 25 mL/L, a reaction temperature of 90 °C, and a treatment time of 3.5 h. Under these optimal conditions, the degumming rate of cattail fibers reached 40.12%.

For the pectinase degumming method, the order of factors influencing the degumming effect was the following: (H) time > (E) pectinase concentration > (F) pH > (G) reaction temperature. The optimal orthogonal scheme of the orthogonal test is: $E_2F_2G_3H_2$, that is, a pectinase concentration 18%, pH of 2.5, temperature of 55 °C, and treatment time of 13 h. The corresponding degumming rate was 55.23%.

Table 1
Orthogonal single factor levels for alkali-peroxide treatment

Levels	Factors			
	A NaOH Concentration ($g\cdot L^{-1}$)	B H_2O_2 Concentration ($mL\cdot L^{-1}$)	C Temperature (°C)	D Time (h)
1	18	20	85	2.5
2	20	22.5	90	3
3	22	25	95	3.5

Table 2
Orthogonal table of alkali-peroxide treatment

Test No.	A	B	C	D	Degumming rate (%)
1	1	1	1	1	30.51
2	1	2	2	2	39.50
3	1	3	3	3	33.20
4	2	1	2	3	39.50
5	2	2	3	1	31.53
6	2	3	1	2	32.52
7	3	1	3	2	33.52
8	3	2	1	3	33.00
9	3	3	2	1	39.10
k_1	34.40	34.51	32.01	33.71	
k_2	34.52	34.68	39.37	35.18	
k_3	35.21	34.94	32.75	35.23	
R	0.81	0.43	7.36	1.52	
Main→Secondary					CDAB
Optimal scheme					$A_3B_3C_2D_3$

Table 3
Orthogonal single factor levels for pectinase treatment

Levels	Factors			
	E Pectinase concentration (%)	F pH	G Temperature (℃)	H Time (h)
1	16	2	45	13
2	18	2.5	50	14
3	20	3	55	15

Table 4
Orthogonal table of pectinase treatment

Test No.	E	F	G	H	Degumming rate (%)
1	1	1	1	1	57.21
2	1	2	2	2	55.51
3	1	3	3	3	55.50
4	2	1	2	3	54.50
5	2	2	3	1	59.11
6	2	3	1	2	55.00
7	3	1	3	2	53.01
8	3	2	1	3	53.50
9	3	3	2	1	56.50
k_1	56.07	54.91	55.24	57.61	
k_2	56.20	56.04	55.50	54.51	
k_3	54.34	55.67	55.87	54.50	
R	1.86	1.13	0.63	3.11	
Main→Secondary					HEFG
Optimal scheme					$E_2F_2G_3H_1$

Meanwhile, a comparison of the orthogonal experiment results between the two methods showed that the degumming rate of the alkali-peroxide method (approximately 40%) was lower than that of the pectinase method (approximately 55%), indicating that the pectinase degumming method exhibited a better degumming effect than the alkali-peroxide method.

Chemical composition analysis

As can be seen from Table 5, cattail leaves mainly contain cellulose, hemicelluloses, lignin, and other components. The raw cattail leaves have a low cellulose content, with a mass fraction of 37.32%,[16] and a high content of pectin, lignin and hemicelluloses, with a total mass fraction of about 44.56%. The cellulose content after pectinase treatment (60.37%) was significantly higher than that after alkali-peroxidase treatment (52.42%). After the initial dilute alkali pretreatment, the hemicellulose content decreased significantly, but the lignin and pectin contents did not change significantly. This is because hemicelluloses have a low degree of polymerization and are easily soluble in alkali solutions. In addition, cellulose is alkali resistant.[17] However, after the alkali-peroxidase and pectinase treatments, the lignin content decreased, which is due to lignin being oxidized in the presence of hydrogen peroxide and thus easily soluble in alkaline solutions.[18] The gummy material that bonded the fibers was removed due to the action of enzymes.[16] The degradation of pectin by pectinase caused a significant change in the structure of the gummy material, and the stability of the gummy complex was greatly damaged. Due to the action of the enzyme, the gummy material that binds the fiber was removed, and the non-cellulose components, such as hemicelluloses, lignin and pectin, could be successfully removed by both pectinase and alkali-peroxidase methods, ultimately realizing the extraction of cellulose.

Table 5
Compositions of cattail fibers before and after degumming treatments

Component (%)	Cellulose	Hemicellulose	Lignin	Pectin
Untreated	37.32± 1.82	21.5± 0.75	14.92± 0.79	8.14± 0.56
Pretreatment	43.04± 1.67	18.59± 0.68	12.56± 0.46	6.69± 0.31
Alkaline oxygen treatment	52.42± 1.98	13.47± 0.56	10.38± 0.53	5.39± 0.22
Pectinase treatment	60.37± 1.43	10.51± 0.51	7.27± 0.72	3.83± 0.14

Morphological structure

Figure 3 shows the SEM images of cattail fibers before and after different degumming procedures. As shown in Figure 3a, the surface of untreated cattail fiber is rough and uneven, covered with a large amount of colloidal substances that bind the fibers together. Figure 3b presents the cattail fibers after dilute alkali pretreatment, and the colloidal substances on the fiber surface were significantly reduced. Meanwhile, it can be observed that the cattail fiber is aggregated into bundles by a large number of single fibers through colloidal adhesion,[19] which is because non-cellulosic components, such as hemicelluloses and lignin, were not successfully dissolved or removed by the dilute alkali pretreatment. Figure 3c and d show the fibers after alkali-peroxide and enzyme treatment, respectively. It can be seen from the figures under the chemical treatment conditions (alkali-peroxide treatment), almost all the components that bind the cellulose fibril structure of the cellulose were removed; cattail fibers were separated into individual single fibers with a bright and clean surface. In comparison, the colloidal substances on the fiber surface were further removed after pectinase treatment, whereas some fibers still remained adhered in the alkali-peroxide treatment group.[20] In this respect, the effect of the pectinase treatment was better than that of the alkali-peroxide method.

X-ray diffraction analysis

Figure 4 shows the X-ray diffraction (XRD) patterns of cattail fibers before and after different degumming treatments. It can be seen that the patterns of fibers before and after treatment are similar, and the main diffraction peaks appear at 2θ 15.6 and 21.7, respectively, which correspond to the (110) and (200) crystal planes of cellulose. The crystallinity of untreated cattail fiber was 35.28%, while that of the fibers after the initial dilute alkali pretreatment was 46.31%. The crystallinity of pectinase treated and alkali-peroxide treated samples was 51.13% and 59.43%, respectively. After a series of treatments, the crystallinity of cattail fiber was significantly improved,[21] which is attributed to the

removal of non-cellulose components from the amorphous region, resulting in the treated fibers containing more crystalline cellulose regions. Meanwhile, it could be seen that the crystallinity of cattail fiber after pectinase treatment was slightly higher than that after alkali-peroxide treatment, indicating that the enzyme degumming treatment had a better effect on improving fiber crystallinity.

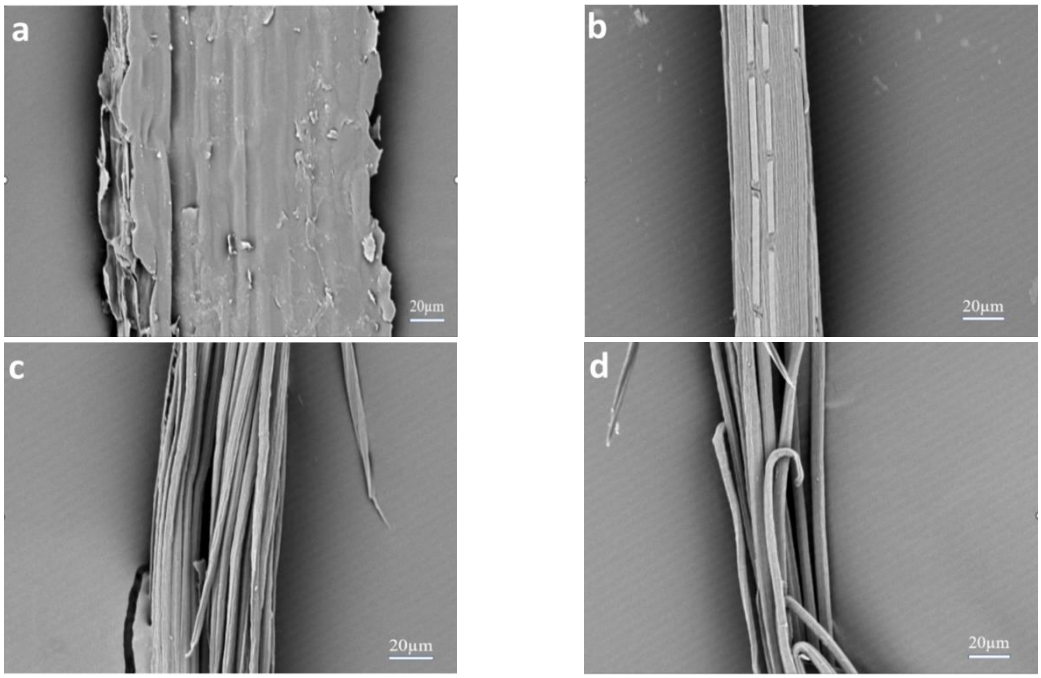

Figure 3: SEM images of cattail fibers; (a) untreated, and subjected to (b) pretreatment, (c) pectinase treatment, and (d) alkali-peroxide treatment

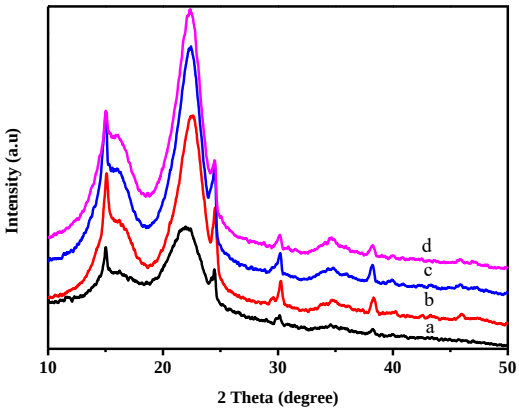

Figure 4: XRD patterns of (a) untreated cattail fibers, and after (b) pretreatment, (c) pectinase treatment, and (d) alkali-peroxide treatment

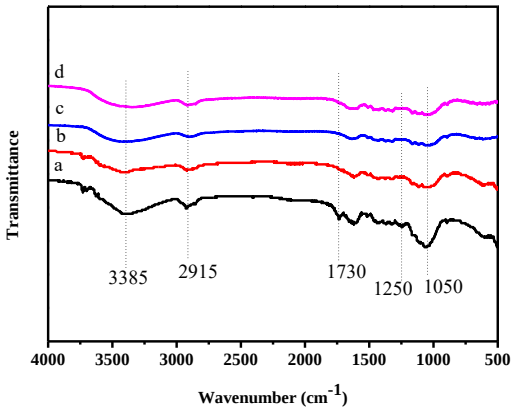

Figure 5: FTIR spectra of (a) untreated cattail fibers, and after (b) pretreatment, (c) pectinase treatment, and (d) alkali-peroxide treatment

FTIR analysis

Figure 5 shows the IR spectra of cattail fibers before and after different degumming treatments. The strong absorption peak in the range of 3000-3600 cm^{-1} is attributed to the stretching vibration of -OH groups in cellulose fiber and lignin. After the degumming treatment, the relative intensity at 3000-3600 cm^{-1} decreased, which indicates that the content of hydroxyl group decreases. The decrease in peak intensity after degumming indicates that some non-cellulosic components substances, such as lignin and hemicelluloses, were partially removed during the degumming process.[22] The absorption peak at 2915 cm^{-1} corresponds to the asymmetric stretching vibration of methyl group, the absorption peak at 1050 cm^{-1} is caused by the C-O stretching vibration of the six-membered ring skeleton of

cellulose. The absorption peak at 1730 cm^{-1} is assigned to the stretching vibration of C=O in hemicelluloses, while the peak at 1250 cm^{-1} is the vibration peak of aromatic ring skeleton in lignin. The significant reduction in the intensity of the peaks at 1730 cm^{-1} and 1250 cm^{-1} after treatment indicates that the non-cellulosic components in cattail fibers were effectively removed by the degumming processes.[23]

Thermal performance

Figure 6 shows the thermal behavior of cattail fiber before and after the treatments. The first mass loss of cattail fibers before and after the degumming treatment occurred in the temperature range of 30–150 °C, due to the evaporation of moisture adsorbed by the fibers.[24] The second mass loss occurred in the range of 200 to 400 °C, indicating the degradation of the major components. Specifically, the decomposition temperature of hemicelluloses is 210-320 °C, that of lignin is 200-600 °C, while that of cellulose ranges from 320 °C to 400 °C. It can be seen that the decomposition temperature of untreated cattail fiber is relatively low, starting at 250 °C. Due to the high decomposition temperature of lignin, the control and alkali treated cattail fibers can continue to decompose at 400 °C,[24] indicating that degumming treatment can remove substances with lower decomposition temperature. Additionally, the temperature range of the significant mass loss stage of degummed cattail fibers was higher; this is because part of the pectin in the fibers was removed after degumming, leading to an increase in the proportion of crystalline regions and thus an improvement in the thermal stability of the fibers.

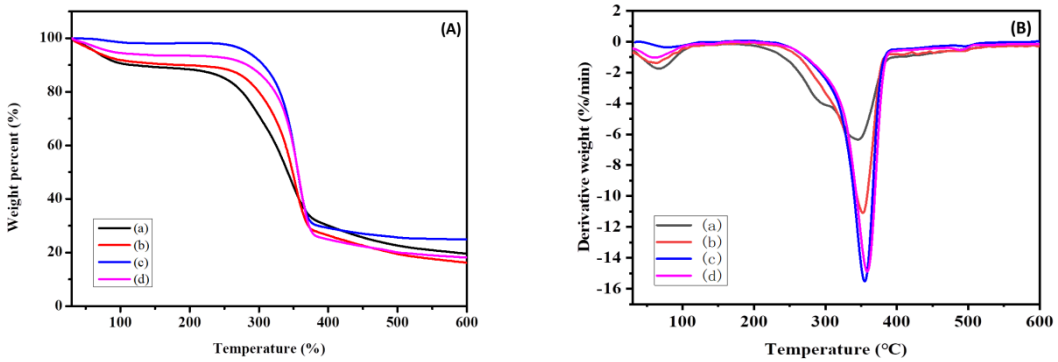

Figure 6: TG (A) and DTG (B) curves of (a) untreated cattail fibers, and after (b) pretreatment, (c) pectinase treatment, and (d) alkali-peroxide treatment

CONCLUSION

In this study, the optimal degumming processes of *Typha orientalis* (cattail) fibers were systematically explored using the alkali-peroxide method and pectinase method, with dilute alkali pretreatment as the preliminary step. Comprehensive characterization via chemical analysis, scanning electron microscopy (SEM), X-ray diffraction (XRD), Fourier transform infrared (FT-IR) spectroscopy, and thermogravimetric analysis (TGA) was conducted to evaluate the degumming effects of the two methods, and the key findings and conclusions are summarized as follows. Both the alkali-peroxide and pectinase degumming methods effectively removed most non-cellulosic components (including pectin, lignin, and hemicelluloses) from cattail fibers after dilute alkali pretreatment. Chemical analysis results showed that the cellulose content of cattail fibers increased from 37.32% (raw sample) to 52.42% after alkali-peroxide treatment and 60.37% after pectinase treatment, confirming the effectiveness of both degumming processes in purifying cellulose. The pectinase degumming method exhibited superior performance compared to the alkali-peroxide method. Orthogonal experiments revealed that the optimal degumming rate of the pectinase method was 55.23% (under conditions: 18% pectinase, pH 2.5, 55 °C, 13 h), which was significantly higher than the 40.12% degumming rate of the alkali-peroxide method (under conditions: 22 g/L NaOH, 25 mL/L H_2O_2, 90 °C, 3.5 h). SEM observations further demonstrated that pectinase treatment resulted in cleaner fiber surfaces and more

complete separation of single fibers, with fewer residual colloidal substances compared to the alkali-peroxide treatment. The degumming treatment significantly improved the structural and thermal properties of cattail fibers. XRD analysis indicated that the crystallinity of cattail fibers increased from 35.28% (untreated) to 59.43% (alkali-peroxide treated) and 51.13% (pectinase treated), attributed to the removal of amorphous non-cellulosic components. FT-IR spectra confirmed the effective removal of lignin and hemicelluloses, as evidenced by the reduced intensity of characteristic peaks at 1730 cm^{-1} (C=O in hemicelluloses) and 1250 cm^{-1} (aromatic ring in lignin). TGA results showed that degummed fibers had higher thermal stability, with a higher temperature range for significant mass loss, due to the increased proportion of crystalline cellulose regions. The pectinase degumming method had notable advantages of simple operation, mild reaction conditions, and minimal damage to fiber structure, which is more conducive to the subsequent application of cattail fibers compared to the alkali-peroxide method.

In conclusion, both the alkali-peroxide and pectinase methods can effectively degum *Typha orientalis* fibers, but the pectinase method is more recommended for industrial application due to its better degumming efficiency and milder reaction conditions. This study provides a theoretical and experimental basis for the efficient utilization of *Typha orientalis* resources and the development of high-value cattail fiber products, with broad application prospects in the textile and green material fields.

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