CHARACTERIZATION OF PINEAPPLE LEAF FIBER FOLLOWING ENZYMATIC DEGUMMING

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Pineapple leaves are a rich resource of fiber with excellent properties. To overcome the processing bottleneck, the enzymatic degumming conditions with compound enzymes, comprising laccase and xylanase, on low-concentration alkaline-pretreated pineapple leaf fiber (PALF), were investigated. The cellulose content and fiber splitting degree were used as optimization indicators. Treated PALF characteristics were determined using microscopic morphology, XRD, and FTIR analysis techniques. The optimal conditions were found as: 0.5% laccase, 0.3% xylanase, bath ratio of 1:50, pH 4.6–5.0, and incubation at 50–55 °C for 4 h. Following enzymatic degumming, the cellulose content increased from 57.22 to 74.46%, the lignin and hemicelluloses contents decreased, a large number of colloidal impurities were hydrolyzed, and free fibers with smooth surfaces were released. The crystalline cellulose remained type I, and crystallinity increased from 36.46 to 46.73%. Low-concentration alkaline solutions, combined with compound enzymes that effectively hydrolyze colloids, resulted in mild enzymatic degumming that caused less damage to cellulose.

Keywords: PALF, enzyme, degumming, characteristics

INTRODUCTION

Pineapple is an herbaceous crop that is widely grown in tropical China, covering an area of 70,000 hm^2.[1] Pineapple leaves are an agricultural waste produced after pineapple harvest, with an annual output of up to 10 million tons in China. Pineapple leaves are generally discarded or burned, which not only has a negative impact on the environment, causing air pollution and smog, but also leads to wastage of resources and economic loss. Pineapple leaf fiber (PALF) is a natural fiber extracted from pineapple leaves by removing parts of the hemicelluloses, pectin, lignin, and other colloids. It is lightweight and biodegradable, has a high specific stiffness and strength, as well as antibacterial and deodorizing properties.[2,3] PALF has been included in the Chinese standard GB/T 11951-2018 "Natural Fiber Terminology". The exploitation and utilization of PALF are conducive to promoting positive circular and sustainable development of the pineapple industry, which, in turn, promotes the reuse of agricultural resources in the entire tropical region and compensates for the shortage of natural fibers.

PALF is similar to hemp and flax fibers; its single fiber is shorter, only 3–8 mm in length. Pineapple leaf processing fibers are composed of multiple fiber bundles tightly adhered to a colloid, and each fiber bundle consists of 10–20 single fibers. The cross-section of PALF is formed by the aggregation of similar round or oval cells, with some gaps; therefore, PALF has good moisture absorption and air permeability.[4] For further applications, PALF is most valuable as a bundle fiber obtained by the semi-degumming method, which is similar to hemp and flax fiber degumming.[5-7]

Natural fibers consist mainly of cellulose, hemicelluloses, and lignin, with cellulose being the main component. Cellulose is a long-chain polymer formed from repeated units of D-glucose that are joined by β-1,4-glycosidic linkages and held between chains by hydrogen bonds.[8-10] Hemicelluloses are a group of polysaccharides, consisting of pentose and hexose, which provide compatibility between the fiber and lignin. Because of their amorphous nature, they are

partially soluble in water and alkaline solutions.[11,12] Lignin is a highly cross-linked phenolic polymer, with an amorphous structure, which acts as a glue between individual cells. It maintains moisture in the fiber and determines the fiber bonding strength.[13,14]

The traditional extraction methods for PALF are mostly mechanical and natural degumming.[15-18] In recent years, chemical methods, such as acid and alkali treatments, have become more common.[19-23] During the acid treatment process, a condensation reaction occurs between lignin and sulfuric acid to form insoluble sulfated lignin, which is difficult to remove. In addition, the acidic solution is highly corrosive to the equipment. Chemical treatment damages fibers, reducing their strength, stiffness, and spinnability. At the same time, a large amount of wastewater is discharged, polluting the environment.[24-26]

In this study, a low-concentration alkali solution was used as a pretreatment agent, followed by an enzymatic treatment to extract PALF. The compound enzymatic degumming process for PALF was optimized. The microscopic morphology and physicochemical properties of PALF after degumming were characterized. The optimized conditions of enzymatic degumming are mild and specific, which is not only beneficial to the extraction rate and fiber splitting degree, but also causes less damage to cellulose. This research can contribute to the production of better-quality fibers and provide a reference for the application of PALF and the study of degumming systems.

EXPERIMENTAL

Materials

Pineapple leaves used in this study were provided by China South Subtropical Crops Research Institute.

Xylanase and laccase were purchased from SuKehan Biological Engineering Co., Ltd. (China). Other chemical reagents were all commercially available and of analytical grade.

PALF degumming method

For efficient degumming of PALF, a low-concentration alkaline solution, containing 1% (w·v^{-1}) NaOH, 2% (w·v^{-1}) Na_2CO_3, 1% sodium polyphosphate, and 2% (w·v^{-1}) osmotic agent JFC, was used as a pretreatment agent. A compound enzyme composed of laccase and xylanase, with concentration levels of 0.1, 0.3 and 0.5% (w·v^{-1}), was then used to degum the pretreated PALF. Degumming conditions were as follows: bath ratio levels: 1:30, 1:40, 1:50, 1:60 and 1:70 (w·v^{-1}), temperature levels: 35, 40, 45, 50 and 55 °C, pH levels 4.2, 4.6, 5.0 and 5.4, and 4 h processing time. After degumming, the fibers were rinsed to neutral pH and dried under ambient conditions. A process flow diagram is presented in Figure 1.

PALF fiber characterization

Chemical composition

Quantitative analysis, in accordance with the Ramie Chemical Components Method GB5889-86, was used to determine the chemical composition of PALF.

Fiber splitting degree

The degummed PALF was randomly extracted and 20 mm was cut from the middle section of a 5 mg sample, and the number of fibers was recorded. The formula for calculating the splitting degree was as follows:[27]

$$N_m = (20 \times n)/G \quad (1)$$

where *Nm* is the degree of fiber splitting, *n* is the number of fibers, and *G* is the weight of the fibers.

Morphological analysis

Scanning electron microscopy (SEM) was used to examine PALFs. Stem segments were mounted on carbon SEM stubs and gold-coated for observation under a Quanta 200 (Frequency Electronics, Inc., USA) scanning electron microscope at 15 kV.

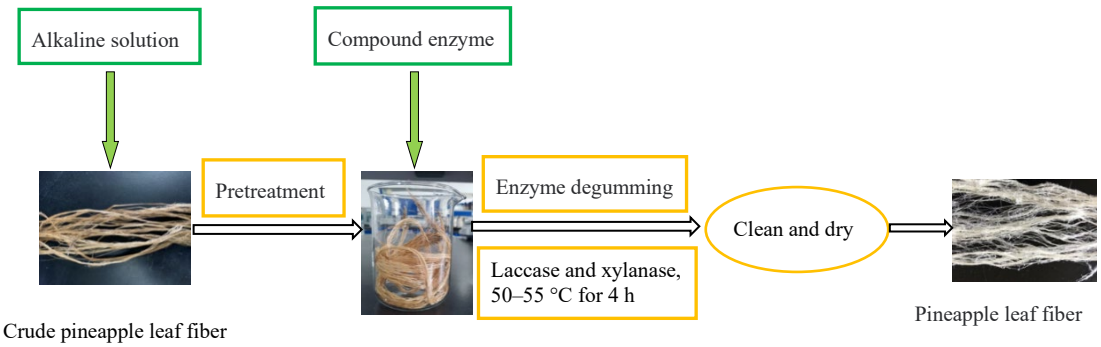

Figure 1: Process flow diagram of PALF degumming

Fourier transform infrared spectroscopy (FTIR) analysis

An appropriate amount of the fiber sample was mixed with KBr-milled powder, and FTIR (PE Specdrum100, USA) was carried out in the range of 400–4000 cm^{-1} to determine the chemical functional groups of the PALF.

X-ray diffraction (XRD) analysis

The crystallinity of PALF was measured using an X-ray diffractometer (Bruker-AXS-D8, Germany). The test voltage was 40 kV, current was 100 mA, and diffraction angle ranged from 5º to 60º. The X-ray diffraction patterns of PALF were fitted and analyzed, and the crystallinity was calculated with the following formula:[28]

$$CrI\ (\%) = (I_{002} - I_{amp})/I_{002} \quad (2)$$

where CrI is the crystallinity (%), I_{002} is the maximum intensity of the I_{002} lattice diffraction angle, and I_{amp} is the scattering intensity of amorphous background diffraction.

RESULTS AND DISCUSSION

Compound enzyme PALF degumming

Effect of pH on PALF degumming

The degree of dissociation of the fibers extracted by the compound enzyme can be influenced by the pH of the degumming system. When the pH of the degumming system reaches an appropriate value, the enzymatic reaction efficiency of the compound enzyme and the hydrolytic efficiency of pectin, hemicelluloses, and other colloids increases, resulting in effective decomposition of colloids between bundles and single fibers, followed by the gradual splitting of the fibers. As shown in Figure 2, the cellulose content of PALF reached approximately 65% in the pH range of 4.2–5.4, and no significant differences were observed in the cellulose content within this pH range. This suggested that the pH range suitable for compound enzyme degumming is wide, which is conducive to the application of compound enzymes. As shown in Table 1, the fiber splitting degree of PALF first increased and then decreased with increasing pH. The fiber splitting degree reached the highest level of 200 yarns at pH 5.0, which was 60 yarns higher than the minimum splitting degree (pH 5.2). Therefore, the optimal degumming pH was selected as pH 4.6–5.0.

Effect of temperature on PALF degumming

Temperature affects the activity of the enzyme and the transfer rate of substances in the reaction system, which in turn affects PALF degumming. In the temperature range of 35–50 °C, the activity of the compound enzyme increased gradually, and the cellulose content of PALF increased rapidly (Fig. 3). When the temperature reached 50 °C, the cellulose content stabilized above 66%. Temperature also had a similar effect on the fiber splitting degree (Table 1). Therefore, the optimum temperature was selected to be 50–55 °C.

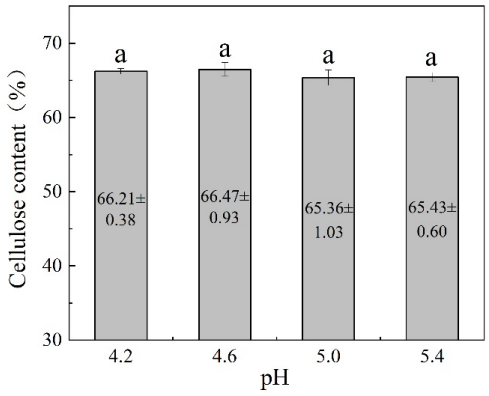

Figure 2: Effect of pH during PALF degumming on cellulose content (different lower case letters indicate significant differences ($p<0.05$))

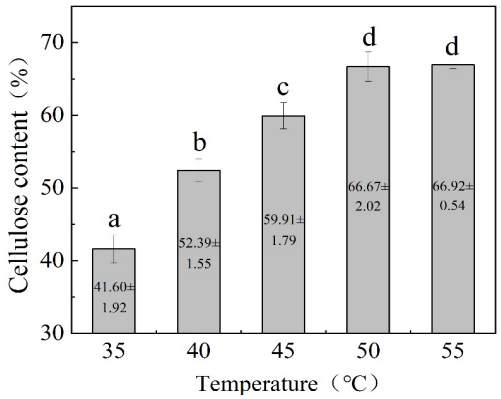

Figure 3: Effect of temperature during PALF degumming on the cellulose content (different lower case letters indicate significant differences ($p<0.05$))

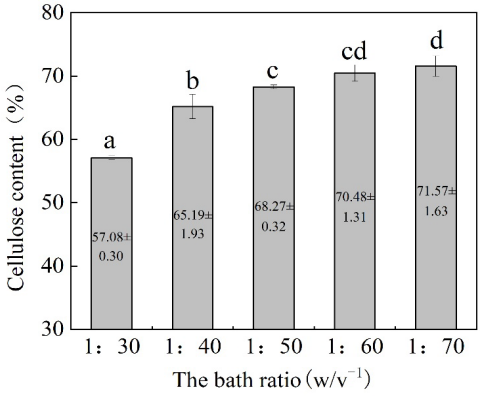

Figure 4: Effect of bath ratio during PALF degumming on the cellulose content (different lower case letters indicate significant differences ($p<0.05$))

Table 1
Effects of various factors on PALF splitting degree

pH	Splitting degree (N_m)	Bath ratio ($w \cdot v^{-1}$)	Splitting degree (N_m)	Temperature (°C)	Splitting degree (N_m)
4.2	150	1:30	130	35	140
4.6	180	1:40	200	40	160
5.0	200	1:50	180	45	180
5.2	140	1:60	180	50	190
5.4	160	1:70	180	55	190

Effect of bath ratio on PALF degumming

The compound enzymes were added at 3% and the bath ratio was adjusted to 1:30–1:70 ($w \cdot v^{-1}$). As shown in Figure 4, the fiber cellulose content increased dramatically when the bath ratio was increased from 1:30 to 1:50 ($w \cdot v^{-1}$); however, this increase stabilized when the bath ratio exceeded 1:50. This may be because, at small bath ratios, there is less degumming solution, tight adhesion of the fiber, insufficient water absorption of the fiber, and lack of effective swelling, resulting in the compound enzyme not fully contacting the substrate. At a bath ratio of 1:50, the fiber was completely immersed in the degumming solution with sufficient swelling, high enzymatic reaction efficiency, and an excellent degumming effect. As shown in Table 1, with an increase in the bath ratio, the degree of fiber splitting gradually decreased and stabilized, which is consistent with the change in the cellulose content. Considering the cost of the procedure, the optimal degumming bath ratio was established as 1:50.

Orthogonal test

Orthogonal table L_9 (3^4) was used for the degumming test of the PALF with the compound enzyme. The k represents the mean value of the results for the corresponding factors at a certain level, and the range (R) analysis directly reflects the influence of the reaction factors on the indices; the greater the range, the greater is the influence of this factor on the cellulose content. As shown in Table 2, the primary factor affecting the cellulose content in PALF was laccase concentration and the secondary factors were xylanase concentration and time. Considering the cellulose content and enzyme cost factors, the optimal PALF degumming process of the compound enzyme was 0.5% ($w \cdot v^{-1}$) laccase, 0.3% ($w \cdot v^{-1}$) xylanase, and a 4 h processing time.

Chemical composition analysis

The PALF is composed of cellulose, hemicelluloses, lignin, pectin, cerolipoid, and hydrotrope, which overlap with each other and bond closely to the fiber. In the degumming system, the action of xylanase and laccase hydrolyzes the ester and glycosidic bonds between hemicelluloses-cellulose and lignin-cellulose, and breaks the bond between the colloid and fiber. The colloid falls off the fiber surface and the fiber is released. In this study, chemical composition of PALF was determined before and after degumming (Table 3). The cellulose content of PALF increased from 57.22% to 79.46% after compound enzyme

degumming, and the colloids were largely removed. The hemicellulose content decreased from 21.32% to 8.52%, and the lignin content decreased from 11.99% to 5.03%, which also confirmed that xylanase and laccase could effectively remove colloids from PALF.

Table 2
Orthogonal test results on degummed PALF

Test number	Laccase (%)	Xylanase (%)	Time (h)	Cellulose content (%)
1	0.1	0.1	3	59.67
2	0.1	0.3	4	59.71
3	0.1	0.5	5	62.85
4	0.3	0.1	5	60.57
5	0.3	0.3	3	63.30
6	0.3	0.5	4	68.55
7	0.5	0.1	4	65.25
8	0.5	0.3	5	71.27
9	0.5	0.5	3	66.94
k_1	60.74	61.83	63.30	
k_2	64.14	64.76	64.50	
k_3	67.82	66.11	64.90	
R	7.08	4.28	1.59	

R: range; k represents the mean value of the results for the corresponding factors at a certain level

Table 3
Chemical composition of PALF

Chemical composition (%)	Cerolipoid	Hydrotrope	Pectin	Hemicellulose	Lignin	Cellulose
Raw PALF	5.40	1.94	2.13	21.32	11.99	57.22
Degummed PALF	2.46	2.69	1.84	8.52	5.03	79.46

SEM observation

A scanning electron microscope was used to observe the microscopic morphology of the PALF (Fig. 5). Non-degummed (raw) PALFs (Fig. 5 (a)) were wrapped in a large number of colloids and bonded together into sheets. Fragments and debris were attached to the epidermal tissue. After enzymatic degumming (Fig. 5 (b)), the PALFs were separated and the fiber surface was smooth, which might be due to the removal of a large number of colloid impurities during the process of compound enzyme degumming, thus releasing free fibers. This was consistent with the increase in the degree of fiber splitting.

FTIR analysis

The infrared spectrum analysis of the functional group changes in the degummed (Fig. 6 (b)), compared to the raw (Fig. 6 (a)) PALF, was performed. Characteristic absorption peaks were observed at wavelengths of 3450 and 2918 cm^{-1}, which are attributable to the O-H of cellulose molecules and stretching vibration caused by the phenylpropane units of cellulose and lignin -C-H ($-CH_3$, $-CH_2-$), respectively. The absorption peak at 1732 cm^{-1}, attributed to the hemicellulose acetyl group and carbonyl group,[29-31] was obviously weakened in the degummed PALF. The absorption peak at 1595 cm^{-1}, caused by the C=C stretching vibration of lignin,[32] was also significantly weakened in the degummed PALF. This shows that the lignin in the raw PALF was effectively degraded by the compound enzyme system. Stretching vibrations caused by the C-O-C and β-1,4-glycosidic bonds of cellulose and hemicelluloses at 1032, 1429, 1163, and 896 cm^{-1} were observed, which correspond to the characteristic absorption peaks of cellulose type I.[9,33] This shows that the degummed PALF still has the basic chemical structure of cellulose, indicating that the enzyme treatment destroys the amorphous region of the fiber, without affecting the structure of cellulose.

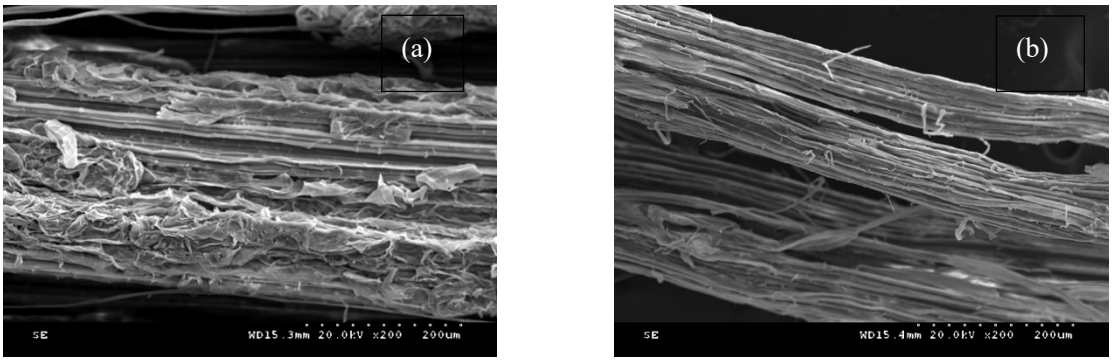

Figure 5: SEM images of (a) raw and (b) degummed PALF (×200)

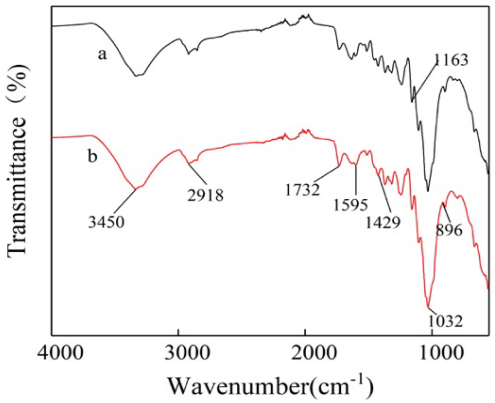

Figure 6: FTIR spectra of (a) raw and (b) degummed PALF (characteristic peak values indicated on the graph)

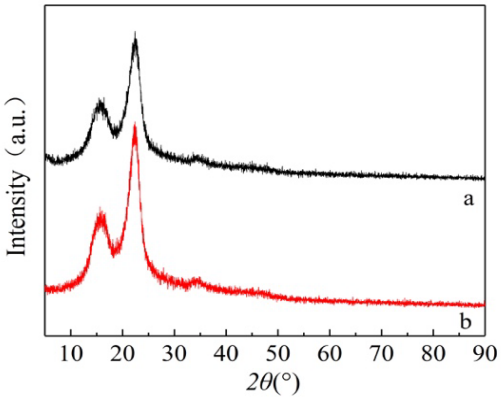

Figure 7: X-ray diffraction patterns of (a) non-degummed and (b) degummed PALF

XRD analysis

XRD analysis was performed on the degummed (Fig. 7 (b)) and raw (Fig. 7 (a)) PALF. Two diffraction peaks were observed in both degummed and non-degummed PALF at 2θ = 22.5° and 16.5°. These diffraction peaks belong to the (200) and (100) crystal planes, respectively, which are attributed to type I cellulose.[34,35] This indicates that the compound enzymatic degumming did not change the crystal structure of the pineapple leaf cellulose. The crystallinity of the degummed PALF increased from 36.46% to 46.73%. This change indicates that compound enzyme degumming can effectively destroy the amorphous region composed of adhesive impurities, such as hemicelluloses, pectin, and lignin, and improve the crystallinity of cellulose. Thus, the approach investigated in the present work, involving a low-concentration alkali pretreatment of PALF, followed by a compound enzymatic degumming process, proved successful. From the perspective of the degumming time, enzymatic degumming takes 1-2 hours longer than chemical degumming, but it is significantly shorter than microbial degumming. Also, the enzymatic and chemical degumming processes are simpler than microbial degumming, as microbial degumming involves pre-culture and fermentation of the strains.[36-38] From the perspective of degumming costs, chemical degumming has the lowest operating costs, as the price of enzymes leads to higher costs for enzymatic degumming, while microbial degumming requires increased equipment investment. For further research prospects, the authors suggest the following two aspects: firstly, in the follow-up processing stage, it is necessary to further improve the removal rate of colloidal impurities, therefore, more efficient enzymes must be screened. This can be achieved by adding other enzyme systems. Secondly, exploring the reuse of the degumming enzyme solution to reduce the cost of degumming and the discharge of wastewater may be a meaningful point in the future.

CONCLUSION

In this study, PALF was treated with a compound enzyme comprising laccase and xylanase. The effects of degumming conditions on

the cellulose content and splitting degree of pineapple fiber were discussed, and the microstructure, chemical composition, FTIR and XRD analysis results for pineapple fiber, before and after degumming, were compared. After degumming, the cellulose content and the degree of splitting of the PALF reached 74.46% and 190 Nm, respectively. Many adhesive impurities among the fibers were removed and the fibers were in a free state, with a smooth surface. The cellulose crystalline structure of the degummed fibers did not change, remaining as type I, while the crystallinity increased from 36.46% to 46.73% after hydrolysis in the amorphous region.

Pineapple leaves are one of the main sources of agricultural waste in the tropical and subtropical regions. Owing to the lack of research on the processing technology of PALF, exploration of the utilization of PALF is limited. This study will help expand the application range of PALF and promote the comprehensive utilization of tropical crop waste, which will provide additional income for farmers.

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