DEGUMMING AND CHARACTERIZATION OF PALM FIBERS

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Palm fiber is an abundantly available, low-cost fibrous material that is not sufficiently valorized through the development of value-added products. Herein, palm fibers were degummed by acid treatment combined with alkali-peroxide bath. To explore the influence of various factors on the degumming results, the efficiency of degumming was assessed through single factor and orthogonal experiments. The optimal scheme for the preparation of palm fibers was obtained: the samples were pretreated with 0.06% sulfuric acid solution in an 80 °C water bath for 30 min, then treated with 20.0 g/L of NaOH, 40 mL/L of H_2O_2 and 2% Na_2SiO_3 for 2.5 h in a 95 °C water bath. The optimal degumming rate was 65.50%. The results showed that, after degumming, impurities, such as hemicelluloses and lignin, were basically removed, the main component of the fiber after degumming was cellulose. The crystallinity and thermal stability were improved, which was beneficial for the further development and utilization of the palm fiber.

Keywords: chemical composition, crystalline, fiber, infrared (IR) spectroscopy, microstructure

INTRODUCTION

In recent years, natural cellulose fibers isolated from lignocellulosic biomass have become increasingly popular in fiber-reinforced composites, in addition to their staple use in textiles, due to their abundant supply and excellent properties.[1-5] Therefore, the global demand for cellulosic fibers has drastically increased in recent decades. Traditionally utilized cellulose fiber resources, including cotton and other common bast fibers, are already thoroughly exploited, but they cannot satisfy the increasing needs.[6-8] Therefore, other less used fiber resources need to be investigated.

Palm fiber generally refers to the palm leaf sheath part, which is a natural fiber with excellent properties. It is generally characterized by low density, good elasticity, high water absorption, high availability and low price, but at present, only a small part of this biomass is used in production.[9] Valorizing the full potential of palm fibers would not only help protect the environment, but also would make up for the shortage of natural fiber resources. However, the content of cellulose in original palm fiber is relatively low, of about 30%, while the contents of lignin, hemicelluloses and other conjunctive components are relatively high, about 2/3 of the total, which leads to its low crystallinity. Also, because of this composition, palm fiber has poor spinnability and is not suitable for textiles. It is

necessary to enhance the cellulose content of palm fibers, by removing the non-cellulosic matrix, including hemicelluloses, lignin, pectin and waxes. In order to improve the performance of palm fiber products and the spinning quality of palm fiber, the gummy substances that make up the non-cellulose matrix should be partly removed by a process called "degumming". Fan *et al*. defined "degumming" as the removal of heavily coated, gummy material from the cellulosic part of plant fibers, emphasizing that it is necessary prior to industrial utilization of the fiber.[10] Degumming is a key process in textile production. A variety of degumming techniques, including mechanical, enzymatic, chemical or ultrasonic, have been investigated so far.[11-14] Although most degumming methods focus on hemp fibers, they will be considered here, due to the high similarity in composition between hemp and palm fibers.

In general, each degumming method has its own advantages and disadvantages.15 For example, the mechanical method has the advantage of being fast, but it involves high running costs and many factors can affect the final results. The biological enzymatic method has mild reaction conditions, is relatively environment-friendly and pollution-free, but the process stability is low, hindering the application of the process for large-scale production. The chemical method requires short reaction time and simple operation, but it may be discordant with environmental protection principles.[16] Therefore, to further use palm fiber, it is necessary to explore a better method of degumming.

Herein, we reported a novel method for the isolation of cellulosic fibers from the raw palm fibers by acid pretreatment, combined with alkali-peroxide bath degumming process. The effects of the dosages of sodium hydroxide and hydrogen peroxide, temperature and time on the degumming rate were discussed. The optimum technological parameters for the alkali-peroxide bath degumming were obtained. The structure and properties of the palm fiber were studied by scanning electron microscopy (SEM), X-ray diffraction (XRD), infrared spectroscopy and thermogravimetry.

EXPERIMENTAL

Materials

The raw palm fibers were collected from the trunk surface of palm trees in the gardens of Wuhan Textile University. The fibers were washed using tap water to remove dust and impurities, and then oven dried at 70^0 C for 24 hours. Sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2), sulphuric acid (H_2SO_4), sodium silicate (Na_2SiO_3) and other chemicals were of laboratory grade (Shanghai Aladdin Chemical Regent Inc., China) and used without further purification.

Degumming of palm fibers

Pretreatment of palm fibers

Palm fibers were pretreated with sulfuric acid solution with concentrations of 0.02, 0.04, 0.06, 0.08 and 0.10%, respectively, and soaked in an 80 °C water bath for 30 min. The pretreated palm fibers were then washed to neutral with purified water and oven dried for 24 hours at 70 °C. The optimum pretreatment processing conditions of the palm fibers were determined by the weight loss rate. Five replicates were taken for each experimental setting and the average value was calculated.

Alkali-peroxide treatment of palm fibers

After the pretreatment with sulfuric acid, the fibers were soaked in an alkali-peroxide bath. The main influencing factors of the alkali-peroxide treatment were: the concentrations of NaOH and H_2O_2, soaking time, temperature and bath ratio *etc*.[17] 2 g of pretreated palm fiber was placed in a beaker containing various concentrations of NaOH (10, 15, 20, 25, 30, 35 and 40 g/L) and H_2O_2 (15, 20, 25, 30, 35, 40 and 45 mL/L). The influence of reaction time (1.5, 2, 2.5, 3, 3.5, 4 and 4.5 h), and temperature (70, 75, 80, 85, 90, 95 and 100 °C) was also assessed. The concentration range was determined by single factor experiments, and then the orthogonal experiment was carried out to obtain the optimal process parameters. Thus, the degummed palm fibers were obtained for further tests.

Calculation of degumming rate

The degumming effect was assessed by evaluating the degumming rate. The degumming rate of palm fibers was calculated by Equation (1):[18,19]

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

where W_1 is the weight of palm fibers before degumming; W_2 is the weight of palm fibers after degumming.

Characterization

Scanning electron microscopy (SEM)

The surface morphology of extracted palm fibers was observed using a scanning electron microscope (JSM-6510 LV, JEOL, Japan). Prior to SEM evaluation, the samples were coated with a thin layer of gold by means of a plasma sputtering apparatus to avoid the charging effect. The SEM images was

captured at different magnifications, with an electron beam accelerating potential of 3 kV.

X-ray diffraction (XRD)

In order to investigate the crystallinity of untreated, acid pretreated and alkali-peroxide treated palm fibers, milled sample powders were analyzed at ambient temperature by step scanning on an X-ray diffractometer (PANalytical Empyrean, Panaco, Netherlands). The sample powders were examined in a maintained operating 2θ range between 5-45°, at a scanning speed of 5°/min. Then, the crystallinity of fibers was calculated with MDI Jade 6.

Fourier transform infrared (FTIR) spectroscopy

FTIR spectroscopic analysis was used to determine the chemical functional groups in the palm fibers before and after the treatments. This allows finding out the chemical changes occurring during the treatments, and thus, to evaluate the efficiency of each treatment. Powder samples of the control, acid pretreated, and alkali-peroxide treated fibers were dispersed into KBr pellets and the analysis was performed on a Nicolet iS50, Thermo Fisher, USA. All the samples were recorded in the range of 4000-400 cm^{-1}, with 16 scans in each case, at a resolution of 4 cm^{-1}.

Thermogravimetric analysis (TGA)

TG analysis was employed to investigate the thermal stability of fibers before and after the treatments. TG analysis of all the powdered samples was performed using a TG 209F1 Libra. Samples of approximately 5 mg were placed in a platinum crucible under nitrogen atmosphere, and heated at a heating rate of 20 °C /min, within the range from 30 °C to 800 °C. TG curves show the variation of sample weight and derivative weight with temperature.

RESULTS AND DISCUSSION

Degumming process of palm fibers

Acid treatment can effectively remove hemicelluloses and reduces the degree of fiber polymerization. At the water bath temperature of 80 °C, the bath ratio of 1:125, and the treatment time of 30 min, the optimum conditions of the pretreatment were determined by varying the concentration of the sulfuric acid solution.

Table 1
Levels of orthogonal factors in the degumming experiment on palm fibers

Levels	Factors			
	A	B	C	D
	NaOH (g/L)	H_2O_2 (mL/L)	Time (h)	Temperature (°C)
1	15	30	2.5	85
2	20	35	3	90
3	25	40	3.5	95

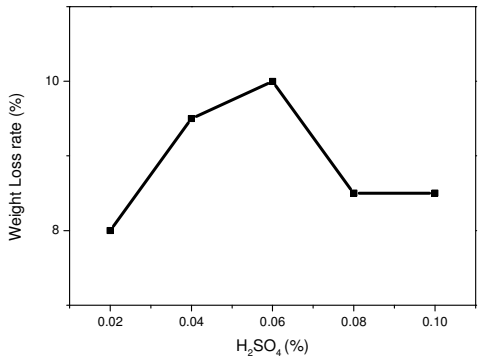

Figure 1: Effect of H_2SO_4 concentration on degumming of palm fiber

As shown in Figure 1, the highest weight loss rate was 10.00%, when the sulfuric acid concentration was 0.06%. With the continuous increase of sulfuric acid concentration, the curve tends to be stable. Thus, 0.06% was chosen as the optimum pretreatment concentration for sulfuric acid.

On the basis of single factor experiments, the effects of various factors on the alkali-peroxide degumming process were investigated, and the optimal degumming process of palm fibers was explored by the orthogonal experiment. R is the "Range" value, the larger the R value is, the greater the influence of this factor. The data

shown in Tables 1 and 2 reveal the following sequence: $R_A > R_B > R_D > R_C$, which means that the most important effect on the alkali-peroxide degumming of palm fibers was exerted by the sodium hydroxide concentration, followed by the hydrogen peroxide concentration, temperature and time, respectively. The optimum alkaline-peroxide treatment conditions for palm fibers were found as: NaOH 20 g/L, H_2O_2 40 mL/L, temperature 95 °C, and time 2.5 h.

Therefore, the optimum treatment conditions for preparing palm fibers were as follows: the samples were firstly pretreated with 0.06% sulfuric acid solution in an 80 °C water bath for 30 min, then treated with 20.0 g/L of NaOH, 40 mL/L of H_2O_2 and 2% Na_2SiO_3 for 2.5 h in a 95 °C water bath. The final degumming rate of palm fibers was 65.50%.

Table 2
Orthogonal design for the degumming process

No.	A	B	C	D	Degumming rate (%)
①	1	1	1	1	62.10
②	1	2	2	2	60.55
③	1	3	3	3	66.50
④	2	1	2	3	65.45
⑤	2	2	3	1	64.45
⑥	2	3	1	2	68.50
⑦	3	1	3	2	64.15
⑧	3	2	1	3	65.45
⑨	3	3	2	1	63.30
k_1	189.15	191.70	196.05	189.85	
k_2	198.40	190.45	189.30	193.20	
k_3	192.90	198.30	195.10	197.40	
R	9.25	7.85	6.75	7.55	

Palm fiber micro-structure

As shown in Figure 2 (a) and (b), the raw untreated fibers were cylindrical, with a few spherical cavities. The fiber surface was quite rough, obviously coated with a large amount of waxes, fatty deposits and possibly silica, which normally accumulate in the epidermis of plants and offer a certain mechanical support for plant growth, protecting plants from pathogens and insects.[20] Figure 2 (c) and (d) reveal that, after the sulfuric acid pretreatment, the surface of the fiber was smoother, as the impurities from the surface of the fiber have been partially removed. As can be seen from Figure 2 (e) and (f), after the alkali-peroxide treatment, the surface of the fiber became even smoother. The longitudinal vertical lines became much clearer, indicating that the binding substances on the surface of the fiber were basically removed, the alkali broke the pectins in the ribbons, without attacking the cellulose in the fibers.[21] Also, comparing the micrographs in Figure 2 (b), (d) and (f), it can be clearly remarked that the diameter of the fiber slightly decreased after the treatments. Compared with untreated fiber, NaOH treatment provided an increase in the surface area of palm fiber, due to the elimination of the binding substances from the surface of the fibers and thus, the release of fibrils.[22,23]

Palm fiber crystalline structure

The X-ray diffractograms of the untreated, sulfuric acid pretreated and alkali-peroxide treated fibers are shown in Figure 3. The diffractograms reveal three main reflections that correspond to 2θ values of approximately 16°, 22°, and 34°. These peaks are attributed to the cellulose crystalline structure. The peak near 16° is a (1-10)/(110) overlapped reflection and the peaks around 22° and 34° were assigned to the (200), and (004) reflections, respectively.[24] These findings imply that the degumming treatment did not change the crystal structure and cellulose types of palm fibers. The crystallinity of untreated palm fiber was about 42.28%, which was slightly improved after the acid pretreatment to about 46.74%. However, the crystallinity of the final fiber reached 56.77% after the alkali-peroxide treatment, due to the efficient removal of non-cellulosic components forming the amorphous region.[25] The alkali treatment for a prolonged period of time may lead to molecular

degradation of cellulose, which affects fiber crystallinity.[26] In our study, the absorption peaks of the fibers subjected to acid pretreatment and alkali-peroxide treatment revealed stronger intensity than those of the raw untreated ones, which means the crystallinity increased due to the treatments applied.

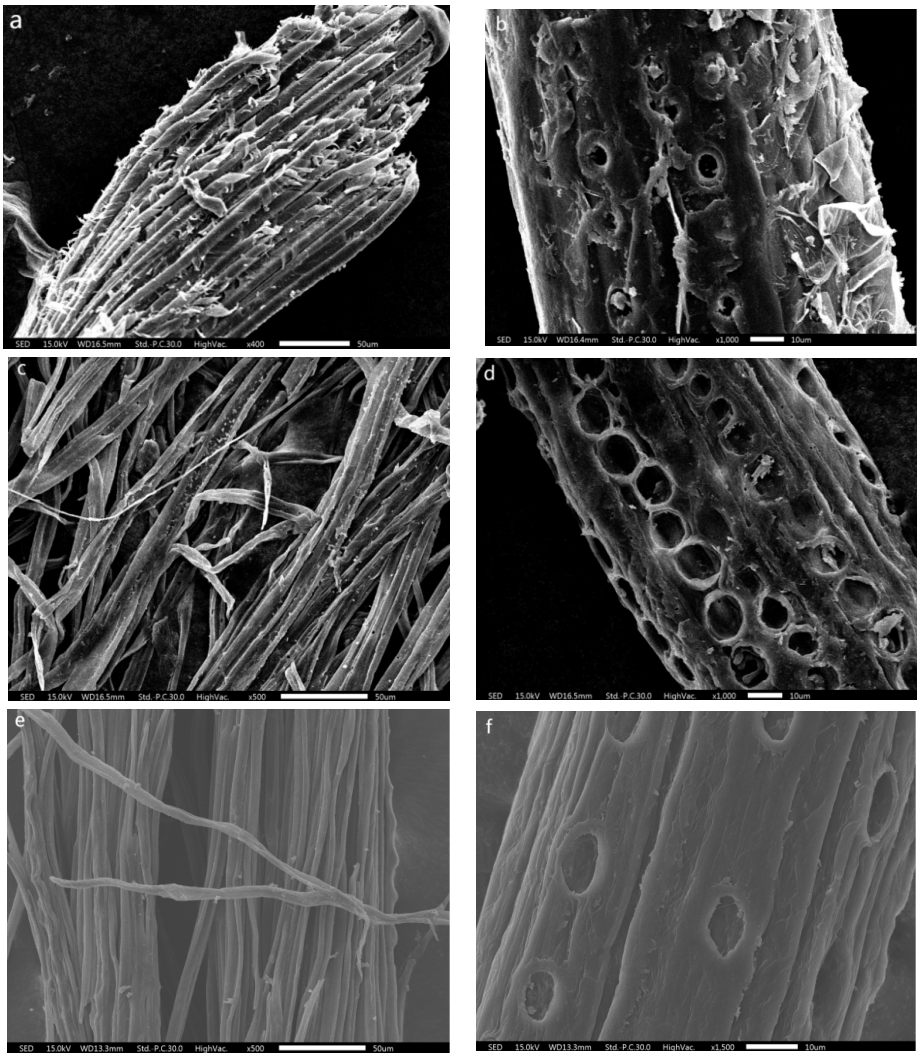

Figure 2: SEM images of palm fibers before and after different treatments; (a) and (b) untreated; (c) and (d) sulfuric acid pretreated; (e) and (f) alkali-peroxide treated palm fiber

FTIR analysis of palm fiber

FTIR spectra of the untreated, sulfuric acid pretreated and alkali-peroxide treated palm fibers are presented in Figure 4. They all exhibit typical vibration bands that mainly correspond to cellulose, hemicelluloses and lignin. The peaks ranging from 3000 cm^{-1} to 3600 cm^{-1} correspond to -OH stretching vibrations, mainly attributed to the large amount of hydroxyl groups in cellulose fibers and lignin.[19] After the fibers were subjected to sulfuric acid pretreatment and alkali-peroxide treatment, the relative intensities around 3000-3600 cm^{-1} decreased, suggesting that the content of hydroxyl groups was significantly reduced and after the treatment. The hydrogen bond structures in cellulose were partly broken and this may lead to a decline in the mechanical properties of degummed fibers. The absorbance at 1620 and 1458 cm^{-1} corresponds to the aromatic skeletal vibrations, ring breathing with C-O stretching, and the absorbance at 1098 cm^{-1} corresponds to the -C-O-C stretching, which are the characteristic peaks of pectins. After the acid pretreatment and the alkali-peroxide treatment,

the relative intensities of this characteristic peak decreased, which suggested that the content of pectins was significantly reduced. The absorption bands at 1731 and 1255 cm^{-1}, corresponding to the carbonyl groups and the C-O stretching of the acetyl group in the hemicelluloses of the untreated fibers, decrease in intensity after the acid pretreatment, completely disappearing after the alkali-peroxide treatment. The changes in these characteristic peaks indicate that the sulfuric acid pretreatment and the alkali-peroxide treatment significantly removed the hemicelluloses, lignin and other impurities. Thus, the FTIR studies confirmed the reduction in the content of non-cellulosic components upon the treatment of palm fibers.[27-29]

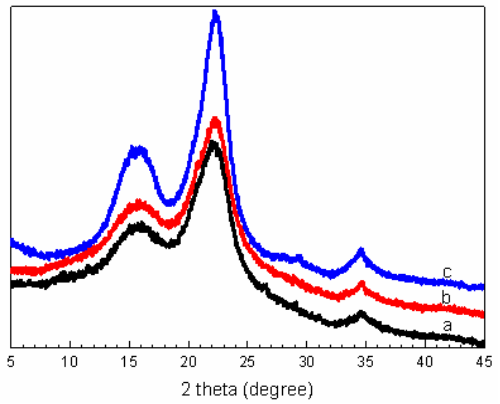

Figure 3: X-ray diffractograms of untreated (a), sulfuric acid pretreated (b) and alkali-peroxide (c) treated palm fibers

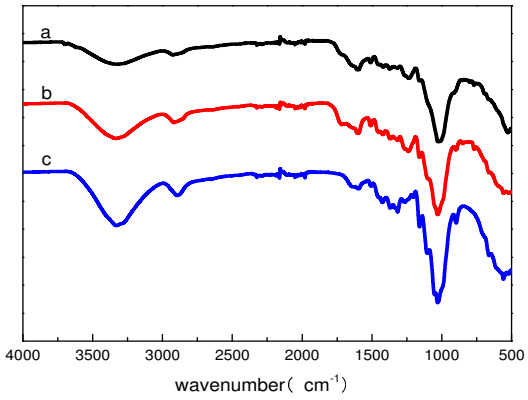

Figure 4: FTIR spectra of untreated (a), sulfuric acid pretreated (b) and alkali-peroxide (c) treated palm fibers

Thermal behavior of palm fiber

Figures 5 and 6 exhibit the TG and DTG diagrams of palm fibers. The thermal decomposition of the palm fibers reveals three main stages. The first stage is due to the hydrophilic characteristic of natural fibers. All the TG curves showed an initial small drop between 50 and 150 °C, which corresponded to a weight loss of approximately 5% absorbed moisture from the surfaces of all the samples, including chemisorbed water and/or intermolecularly H-bonded water. The second stage was the fiber degradation stage, the glycoside bond in the fiber chemical structure began to break, producing volatile compounds. The initial decomposition temperature of untreated palm fibers was 298 °C, it was 289.5 °C after the acid pretreatment, and rose to 322.6 °C after the alkali-peroxide treatment. The decrease in the onset degradation temperature for the acid pretreated sample indicated that its thermal stability was low, after the alkali-peroxide treatment, the degradation temperature increased significantly, implying an improvement in the thermal stability of palm fibers. Analyzing the DTG curves in Figure 6, some difference can be noted among the curves, namely, the untreated and acid pretreated samples showed three peaks, while the alkali-peroxide treated one exhibited only two peaks. The higher water content in the untreated and the acid pretreated palm fibers explained a slower weight loss, compared to the alkali-peroxide treated palm fibers. The high amount of moisture in the fibers acts as a thermal barrier, therefore, the peaks shifted along to high-temperature regions.[30] In the third stage, from the maximum decomposition to 800 °C, the weight loss was slow, and the residue in the degraded fibers is carbonized. The residual char content increased from 1.88% to 20.68% upon treatment. Overall, the thermal stability of palm fibers was improved by the chemical treatment.

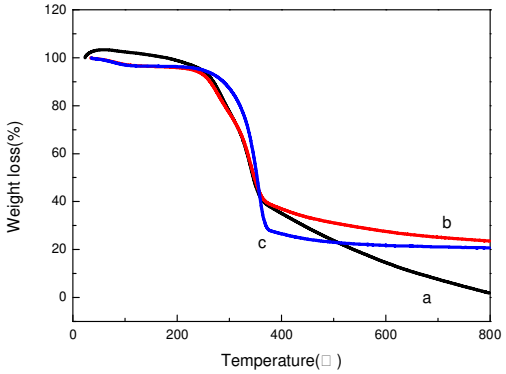

Figure 5: TG diagram of untreated (a), sulfuric acid pretreated (b) and alkali-peroxide (c) treated palm fibers

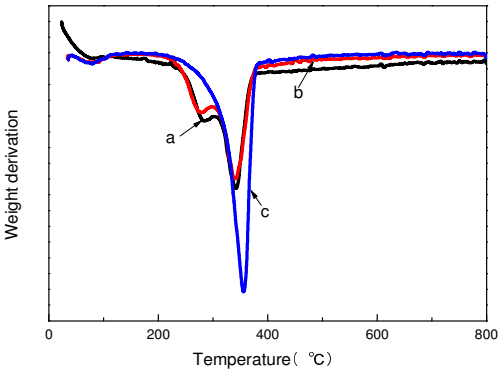

Figure 6: DTG diagram of untreated (a), sulfuric acid pretreated (b) and alkali-peroxide (c) treated palm fibers

CONCLUSION

In this work, the optimum parameters for the degumming process of palm fibers have been determined by single factor experiment and the orthogonal experiment. It was found that the optimum treatment conditions for removing non-cellulosic components from palm fibers were as follows: pretreatment with 0.06% sulfuric acid solution in an 80 °C water bath for 30 min, then treatment with 20.0 g/L of NaOH, 40 mL/L of H_2O_2 and 2% Na_2SiO_3 for 2.5 h in a 95 °C water bath. FTIR analysis revealed that the characteristic peaks of hemicelluloses and pectins became lower in intensity or completely disappeared after the treatments, and the main component of the treated palm fibers was cellulose. The crystallinity and thermal stability of the treated palm fibers were enhanced. The degumming treatment of palm fibers increases the content of cellulose, by removing most of other components, and thus improves the properties of the obtained cellulose fibers, making them suitable for further development and utilization. Based on their renewability and biodegradability, the prepared palm fibers can be considered for applications in functional fabrics, such as filters, thermal insulation materials, sound absorbers, *etc*.

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