

## Effect of Steam Explosion on Degumming Efficiency and Physicochemical Characteristics of Banana Fiber

Zhanwu Sheng,<sup>1,2</sup> Jinhe Gao,<sup>1</sup> Zhiqiang Jin,<sup>1,3</sup> Haofu Dai,<sup>4</sup> Lili Zheng,<sup>1</sup> Bizun Wang<sup>1</sup>

<sup>1</sup>Haikou Experimental Station, Chinese Academy of Tropical Agricultural Sciences, Haikou 570101, China

<sup>2</sup>College of Food Science and Technology, Huazhong Agricultural University, Wuhan 430070, China

<sup>3</sup>Hainan Key Laboratory of Banana Genetic Improvement, Haikou 570101, China

<sup>4</sup>Institute of Tropical Bioscience and Biotechnology, Chinese Academy of Tropical Agriculture Sciences, Haikou 571101, China

Correspondence to: Z. Sheng (E-mail: shengzhanwu100@163.com)

**ABSTRACT:** A steam explosion degumming method coupled with the 16 (4<sup>4</sup>) orthogonal array design was used to investigate the efficient retting of fibers. Four parameters were examined to determine the residual gum and lignin content of retted fibers, and the optimum extraction conditions were determined to be the following: banana fiber moisture content of 10%, 1.2% NaOH, steam pressure of 1.75 MPa, and residence time of 90 s. Under optimized conditions, the experimental yield of residual gum ( $5.47 \pm 0.22\%$ ) and lignin ( $3.78 \pm 0.12\%$ ) agreed closely with the predicted yield. The chemical composition of the fibers was analyzed, and the retted fibers exhibited an increase in the cellulose content and a decrease in the lignin and hemicellulose contents. This result was further confirmed by XRD and Fourier transform infrared spectroscopy. The scanning electron microscopy analysis of the treated fibers showed a change in their surface morphology compared with that of the raw fibers. Their thermal characterization showed an enhanced thermal stability of the retted fibers compared to the raw fibers. © 2014 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2014**, *131*, 40598.

**KEYWORDS:** banana fiber; degumming; natural cellulose; orthogonal experiment; steam explosion

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### INTRODUCTION

The transition to a more sustainable bio-based economy includes a shift in the feedstock used by energy and chemical industries from petrochemical to renewable resources. As a major renewable resource, lignocellulosic agricultural byproducts will likely play an important role in this transition because these are a copious and inexpensive source of cellulose fibers. Natural cellulose fibers have properties and structures that make them suitable for various uses, such as the manufacturing of textiles, composites, pulp, and paper.<sup>1</sup> Cellulosic fibers for textile and paper pulp production are still important commercial non-food commodities.<sup>2</sup> The market niche for nature cellulose has been maintained, and numerous new markets are emerging due to the ecological “green” and low-carbon image of cellulosic fibers.<sup>3</sup>

Most cellulose fibers are primarily composed of cellulose, hemicellulose, and lignin. Cellulose, which is the most abundant natural renewable biodegradable polymer, is widely used in commercial materials because of its attractive properties, which include biocompatibility, biodegradability, renewability, and

thermal and chemical stabilities.<sup>4</sup> Hemicelluloses, lignin, and other non-cellulose component should partly separate, dissolve, and decompose during the retting process of cellulose fibers for textile use.<sup>5</sup> The retting of cellulose fibers involves the separation of the non-cellulose by mechanical methods or its dissolution and decomposition by physical–chemical and microbiological methods. The quality of the fiber and the ease with which it is spun into yarn fibers is largely determined by the efficiency of the retting process.<sup>6</sup>

Therefore, the determination of methods for effective degumming is necessary for the efficient utilization of lignocellulosic fibers. The degumming methods that are commonly used to obtain cellulose from fiber crops include mechanical, chemical,<sup>7</sup> physical–chemical,<sup>5,8</sup> and biological treatments.<sup>9</sup> Some of these processes are uneconomical, and their effluents also cause significant environmental problems. The industrial retting process needs to be enhanced by increasing the speed and controlling the process to improve the fiber quality and reduce the production costs.<sup>6</sup> The goal of this article was to develop a commercial method for the effective retting of raw banana fibers by

adopting the catapult explosion mode, which completes the explosion within 0.0875 s.<sup>10</sup>

To date, a large amount of work has been conducted to extract nanocellulose<sup>11–15</sup> and microfibrils<sup>16,17</sup> using steam explosion pretreatment. Steam explosion is an auto hydrolysis process that involves the use of saturated steam at high pressure followed by sudden decompression, which causes the substantial breakdown of the lignocellulosic structure, the hydrolysis of the hemicellulose fraction, the depolymerization of the lignin components, and defibrillation.<sup>18,19</sup> Kessler et al.<sup>5</sup> mentioned that steam explosion is a superior technique for the retting of flax fiber. Dupeyre and Vignon<sup>8</sup> indicated that steam explosion treatment is a fast and well-controlled process with a low-cost and very flexible treatment parameters and is well adapted for the processing of fiber degumming. The major factors that affect steam explosion degumming during the retting process are the residence time, pressure, moisture content, and the condition of preimpregnation.<sup>5,8</sup> The explosion speed will cause a qualitative change in the effect of the biomass pretreatment.<sup>10</sup> However, there is little information on the optimization of degumming technology by steam explosion for banana fibers destined for textiles. Furthermore, the retted efficiency and characterization of banana fiber has not yet been found in the relevant literature.

According to FAO statistics, the planting area dedicated to bananas in China is nearly 403,300 hectares, and this area experienced an annual production of more than 1,070 million tons in 2011 (FAO, 2011), which results in a huge economic value. In commercial situations, after collecting a single bunch of bananas, many banana pseudo-stems are produced. To date, these have only been used as organic material and fertilizer in plantations in China. The aim of this article was to apply the catapult explosion mode, which completes an explosion within 0.0875 s,<sup>10</sup> to produce and characterize the potential of banana pseudo-stems as textile fibers. A steam explosion retting method coupled with an L16 (4<sup>4</sup>) orthogonal array design (OAD) was used to investigate the efficient degumming of the fibers. These retted fibers were characterized by Scanning Electron Microscopy (SEM), Fourier Transform Infrared (FTIR) Spectroscopy, XRD, and TG techniques. The developed process can be employed for the degumming of other natural fibers.

## EXPERIMENTAL

### Materials

Banana fiber extracted from *Musa spp.* Baxijiao was collected from the local farming community (Haikou, China). The chemicals used, namely sodium hydroxide, hydrogen peroxide, urea, sodium silicate, glacial acetic acid, and nitric acid, were all of commercial grade.

### Retting of Banana Fiber

The banana fibers were chopped to obtain a uniform size of approximately 10 cm. The fibers were impregnated with 1.0–1.8% sodium hydroxide solution (fiber to liquor ratio 1 : 20) at room temperature for 24 h. After bleaching, the fibers were thoroughly washed in water until they no longer contained alkali. Each pretreated sample (10 g) was exploded using a

QBS-80 steam explosion test bed (Hebi Steam Explosion Research Center, Hebi, China) with a specific initial moisture content (5–25%), steam pressure (1.45–1.85 MPa), and residence time (30–150 s). The pressure was immediately released. The steam-exploded fibers were bleached using a solution (fiber-to-liquor ratio of 1 : 15) of 2% NaOH, 1% sodium silicate, and 2% H<sub>2</sub>O<sub>2</sub> at 60°C for 2 h, and the samples were then bleached again using a solution of 1% NaOH, 1% sodium silicate, and 1% H<sub>2</sub>O<sub>2</sub> at 60°C for 2 h. After bleaching, the fibers were thoroughly washed in water until the pH of the solution returned to neutral, dried, and opened on standard opening and carding machines. All of the samples were tested in triplicate.

### Experimental Apparatus and Operation

The QBS-80 steam explosion test bed adopts the catapult explosion mode structure.<sup>10</sup> It equips a 400 mL cylinder and a complete set of piston drive system. The force of the piston drive system which is composed of a linear actuator and a solenoid valve comes from compressed air. The test bed equips a by-pass pipe and valve in addition to the steam supply pipe. Following the steam explosion pretreatment in the catapult explosion mode process, we first shut off the steam supply valve and triggered the switch of the piston drive device. The explosion was completed within 0.0875 s. The fibers were removed from the collecting tank and subsequently washed in water until the pH of the solution returned to neutral.

### Experimental Design

An orthogonal L16 (4<sup>4</sup>) test design was used to investigate the optimal retting conditions of banana fibers. As shown in Table I, the retting experiment was conducted with four factors and four levels: the concentration of NaOH (1.2%, 1.4%, 1.6%, and 1.8%), the initial moisture (relative water content) content of the banana fiber (5%, 10%, 15%, and 20%), the residence time (30 s, 60 s, 90 s, and 120 s), and the steam pressure (1.55 MPa, 1.65 MPa, 1.75 MPa, and 1.85 MPa). The range of each factor level was based on the results of preliminary experiments and references.<sup>5</sup> The raw banana fiber from *Musa spp.* Baxijiao (14.05%) contains higher lignin content than that from *Musa sapientum* (4.9%).<sup>14</sup> The most important factor in flax processing is the degree of retting.<sup>6</sup> The evaluation of the retting efficiency was based on the residual gum and lignin contents of the treated banana fiber, which were used as the dependent variables. The average residual gum and lignin contents of each trial and the average responses for individual factors at different levels were calculated and used to evaluate the efficiency and to optimize the experimental conditions of the retting processing. The retted fiber obtained from the above-described 16 tests was examined following the method described in “Retting of banana fiber”.

### Characterization of the Fiber

**SEM Analysis.** The SEM photographs of the raw and treated banana fibers were taken using a SEM model Hitachi-S4800 at an acceleration voltage of 1.0 kV. The samples were sputter-coated with gold particles and photographed.

**FTIR Spectroscopy.** FTIR analyses of the raw and treated banana fibers were performed to examine the structural changes

**Table I.** Performance of Individual Factors: Moisture Content, Concentration of NaOH, Residence Time and Steam Pressure at Different Levels

Level	Moisture content			Concentration of NaOH			Residence time			Steam pressure		
	RG	RL	Level	RG	RL	Level	RG	RL	Level	RG	RL	
5%	9.55 ± 0.14	7.89 ± 0.50	10	11.33 ± 1.09	10.32 ± 1.32	30	10.34 ± 0.21	10.14 ± 0.61	1.45	10.25 ± 0.58	10.12 ± 0.99	
10%	8.60 ± 0.30	8.39 ± 0.43	12	10.34 ± 1.32	9.02 ± 0.34	60	10.79 ± 0.23	9.59 ± 0.30	1.55	9.09 ± 0.63	9.14 ± 0.51	
15%	7.75 ± 0.07	7.73 ± 0.07	14	8.70 ± 0.63	8.02 ± 0.59	90	9.39 ± 0.54	8.49 ± 0.57	1.65	8.52 ± 0.82	7.89 ± 0.65	
20%	8.37 ± 0.44	7.83 ± 0.06	16	7.45 ± 0.99	7.52 ± 1.54	120	7.95 ± 0.39	9.37 ± 0.24	1.75	8.43 ± 0.42	8.14 ± 0.56	
25%	10.32 ± 1.21	8.54 ± 0.98	18	7.48 ± 1.01	7.25 ± 0.67	150	3.27 ± 0.38	7.89 ± 0.54	1.85	6.37 ± 0.91	7.61 ± 0.32	

RG: residual gum, RL: residual lignin.

in the fibers before and after treatment. The pellets were analyzed using a Nicolet 6700 spectrometer (Thermo Electron Corp., Madison, WI). The fibers were milled and mixed with KBr (sample/KBr ratio, 1/99) to prepare pastilles. The FTIR spectra were recorded at a spectral range 400–4000  $\text{cm}^{-1}$  with a resolution of 2  $\text{cm}^{-1}$ , and 20 scans were obtained for each sample. The spectral outputs were recorded in the transmittance mode as a function of the wavenumber.

**Thermal Characterization.** The thermogravimetric analysis (TGA: SDT Q600, TA instrument) of the untreated and treated banana fibers was conducted at a temperature interval of 30–800°C under a static air atmosphere with a flow rate of 100 mL/min using an alumina crucible with a pinhole. A constant heating rate of 10°C/min was maintained. The DSC measurements were conducted on a DSC Q100 V9.9 Build 275 differential scanning calorimeter. The dynamic DSC scans were conducted in a temperature range from –50 to 400°C at a constant heating rate of 10°C/min. The experiments were performed under an air atmosphere at a flow rate of 20 mL/min.

**X-ray Diffraction.** The X-ray diffraction (XRD) profiles of banana fibers before and after treatment were collected to examine the crystallinity of the samples in response to mechanical treatment. The samples were analyzed in powdered form using a Bruker D8 Advance X-ray Diffractometer with Cu K $\alpha$ 1 (1.5406 Å) and Ni-filtered radiation at a voltage of 40 kV and an intensity of 40 mA. The radiation was absorbed in the range of  $2\theta = 5\text{--}65^\circ$  at a speed of 2°/min. The crystallinity index of the fiber was calculated using the following equation:

$$\text{Crystallinity index} = \left( \frac{I_{\text{crystalline}} - I_{\text{amorphous}}}{I_{\text{crystalline}}} \right) \times 100$$

**Determination of the Chemical Composition and Physical Properties of the Fiber.** The chemical compositions of the fibers (holocellulose, lignin,  $\alpha$ -cellulose, and moisture contents) were estimated according to the following ASTM procedures: holocellulose, ASTM D 1104-56;  $\alpha$ -cellulose, ASTM D 1103-60; lignin, ASTM D 1106-56; and moisture contents, ASTM D4442-92. The difference between the values of holocellulose and  $\alpha$ -cellulose provides the hemicellulose content of the fibers. The fineness, brightness, breaking strength, and residual gum content of the raw and treated banana fibers were measured following the China National Standards (GB 5884-86, GB 5885-86, GB 5886-86, and GB 5889-86, respectively).

## RESULTS AND DISCUSSION

### Optimization of the Degummed Parameters of Banana Fiber

The OAD for the optimization of the retting conditions was based on the minimum residual gum and lignin contents of the sample. All of the parameters (A–D) were tested in a wider range prior to the OAD optimization to narrow down the ranges of the parameters tested (Table I). After implementing the 16 experimental trials based on the OAD16 (4<sup>4</sup>) matrix (Table II), the average responses (residual gum and lignin) to all four individual factors were obtained at the set levels (Table I). To develop a steam exploded retting method, various parameters play a significant role in the optimization of the experimental conditions. The moisture content of the banana fiber, the concentration of NaOH, the steam pressure, and the

**Table II.** Orthogonal Array Design and Results for the Four Variables Studied

Exp no.	A <sup>a</sup>	B	C	D	RG (%)	RL (%)
1	12	12	1.5	30	6.70 ± 0.32	8.58 ± 0.14
2	12	24	1.6	60	7.79 ± 0.23	7.86 ± 0.52
3	12	36	1.7	90	9.69 ± 0.73	6.33 ± 0.65
4	12	48	1.8	120	6.79 ± 0.54	11.75 ± 1.17
5	14	12	1.6	90	5.61 ± 0.65	5.99 ± 0.21
6	14	24	1.5	120	7.96 ± 0.75	8.78 ± 1.26
7	14	36	1.8	30	6.83 ± 0.21	5.16 ± 0.43
8	14	48	1.7	60	7.30 ± 0.22	6.30 ± 0.37
9	16	12	1.7	120	7.86 ± 0.45	9.73 ± 0.46
10	16	24	1.8	90	8.43 ± 0.64	7.10 ± 0.68
11	16	36	1.5	60	7.95 ± 0.12	6.89 ± 0.14
12	16	48	1.6	30	6.81 ± 0.06	6.08 ± 0.53
13	18	12	1.8	60	6.65 ± 0.74	9.04 ± 0.21
14	18	24	1.7	30	9.56 ± 1.09	12.13 ± 1.38
15	18	36	1.6	120	7.14 ± 0.16	4.60 ± 0.16
16	18	48	1.5	90	5.89 ± 0.39	5.58 ± 0.14
$K_1^b$ RG	7.74	6.71	6.63	6.91		
$K_2$ RG	6.93	8.44	6.84	7.42		
$K_3$ RG	7.76	7.90	8.60	7.48		
$K_4$ RG	6.81	6.20	7.18	7.44		
Range <sup>c</sup> RG	0.95	2.24	1.98	0.57		
$K_1$ RL	8.63	8.34	7.46	7.99		
$K_2$ RL	6.56	8.97	6.13	7.52		
$K_3$ RL	7.45	5.75	8.62	6.25		
$K_4$ RL	7.84	7.43	8.26	8.72		
Range RL	2.07	3.22	2.49	2.47		

RG: residual gum, RL: residual lignin.

<sup>a</sup>A: concentration of sodium hydroxide (%), B: moisture content of fiber (%), C: steam pressure (MPa), D: residence time (s).

<sup>b</sup> $K_i^A = \sum \text{RG yield at } A_i$ . Values were mean of three determinations.

<sup>c</sup>Range RG =  $\max\{K_i^A\} - \min\{K_i^A\}$ .

residence time are generally considered to be the most important factors that affect the retting efficiency of banana fiber.<sup>5,8</sup>

The moisture content also plays an influential role in the severity of pretreatment because it greatly influences the ability of heat and chemicals to penetrate fibers.<sup>20</sup> As shown in Table I, when the other three factors, i.e., concentration of NaOH, steam exploded time, and pressure, were fixed to 1.4%, 90 s, and 1.55 MPa, respectively. The residual gum of banana fibers decreases with an increase in the initial moisture content, reached its lowest value ( $6.43 \pm 0.13\%$ ) at a moisture content of 15%, and then started to increase. The variation in the residual lignin of banana fiber is consistent with the change in the residual gum. The ability of heat to penetrate the fibers is reduced when the moisture content of the samples increase, which results in an uneven treatment of the substrate.<sup>20</sup> The residual gum and lignin (%) were affected by different concentrations of NaOH (1.2–1.8%), as shown in Table I, when the other three factors, i.e., moisture content, residence time, and steam pressure, were fixed to 15%, 90 s, and 1.55 MPa, respectively. The residual gum and lignin of the banana fiber decreases with an increase

in the concentration of NaOH because the alkali treatment of the natural fiber leads to the swelling of the fiber and a subsequent increase in the absorption of moisture.<sup>11</sup> In addition, the sodium hydroxide-catalyzed steam explosion was effective in the removal of lignin and hemicelluloses.<sup>21</sup> The residual gum and lignin (%) were differentially affected by different residence times (30–150 s), as shown in Table I, when the other three factors, i.e., moisture content, concentration of NaOH, and steam pressure, were fixed to 15%, 1.4%, and 1.55 MPa. The residual gum of banana fiber decreases with an increase in the residence time. The results reveal that the steam cooking time did result in some alterations in the non-cellulose components. The residual gum and lignin (%) were differentially affected by different steam pressure (1.45–1.85 MPa), as shown in Table I, when the other three factors, i.e., moisture content, concentration of NaOH, and steam exploded time, were fixed to 15%, 90 s, and 1.55 MPa, respectively. The residual gum of banana fiber decreases with an increase in the steam pressure.

The investigated levels of each factor were selected depending on the above experimental results of the single-factor

**Table III.** Chemical Composition and Physical Properties of Raw and Treated Banana Fiber

pre-impregnated degumming	Raw banana fiber	Treated banana fiber
$\alpha$ -cellulose (%)	43.13 $\pm$ 1.66 <sup>a</sup>	69.22 $\pm$ 2.15
Lignin (%)	14.05 $\pm$ 0.26	3.90 $\pm$ 0.16
Hemi-cellulose (%)	18.85 $\pm$ 2.32	6.89 $\pm$ 0.67
Holocellulose (%)	61.98 $\pm$ 1.98	76.11 $\pm$ 3.21
Residual gum (%)	36.31 $\pm$ 2.12	7.94 $\pm$ 0.65
Moisture content (%)	10.31 $\pm$ 0.98	10.55 $\pm$ 0.34
Breaking strength (cN)	345.37 $\pm$ 4.32	156.37 $\pm$ 3.22
Fiber brightness (ISO)	15.24 $\pm$ 0.23	51.02 $\pm$ 0.23
Fiber fineness (dtex)	89.09 $\pm$ 2.31	225.50 $\pm$ 3.57

<sup>a</sup> Mean  $\pm$  SD (N = 3).

experiment. The independent variables with the four variation levels are listed in Table II. All of the selected factors were examined using an orthogonal L16 (4<sup>4</sup>) test design. The analyses of the results of the orthogonal test, which were performed using the statistical software SPSS 14.0, are presented in Table II. Although the minimum yields of residual gum and lignin (%) were 5.89  $\pm$  0.39% and 4.60  $\pm$  0.16, respectively, the corresponding retted conditions cannot be chosen as the best technology.

For the orthogonal analysis, the values of K, k, and R were calculated using the statistical software. The factors that influence the residual gum and lignin were listed in a decreasing order according to the R value as follows: B > C > A > D and B > C > D > A. Thus, the minimum yield of the residual gum (5.61  $\pm$  0.65%) was obtained when the moisture content of the banana fiber, the concentration of NaOH, the steam exploded pressure, and the time were B<sub>2</sub> C<sub>3</sub> A<sub>3</sub> D<sub>3</sub> (10%, 1.75 MPa, 1.6%, and 90 s, respectively). The minimum yield of lignin (4.60  $\pm$  0.16%) was obtained when the moisture content of the banana fiber, the concentration of NaOH, the steam exploded pressure, and the time were B<sub>2</sub> C<sub>3</sub> A<sub>1</sub> D<sub>4</sub> (10%, 1.75 MPa, 1.2%, and 120 s, respectively). To decrease the cost of production and time for industrialization, the optimal technological conditions is the following: B<sub>2</sub> C<sub>3</sub> A<sub>1</sub> D<sub>3</sub> (10%, 1.75 MPa, 1.2% and 90 s). Using confirmatory tests, we obtained the following lowest yields of residual gum and lignin contents in the retted banana fibers: 5.47  $\pm$  0.22% and 3.78  $\pm$  0.12%, respectively.

### Chemical Composition and Properties of Banana Fibers

The chemical compositions of the raw and treated fibers are shown in Table III. The treated fibers showed higher percentages of cellulose and lower percentages of hemicelluloses, lignin, and residual gum compared with untreated fibers. The alkali pretreatment removes a certain amount of lignin, hemicelluloses, and wax because some alkali labile linkages (ether and ester linkages) between lignin monomers or between lignins and polysaccharides may be broken during the alkali treatment process.<sup>22</sup>

During the steam explosion process, banana fibers are both mechanically defibrated and chemically modified.<sup>23,24</sup> The fiber

is separated by mechanical action during the high-pressure discharge, the lignin and hemicelluloses are softened and dissolved by the high temperatures in the steam exposition process, and this process paves the way for the formation of water-soluble phenolic contents and sugar.<sup>18,22</sup> The combination of steam explosion with an alkaline medium result in the hydrolysis of the hemicelluloses within the fiber, and the resulting sugars can be washed out in water to yield a residue of cellulose and lignin. This process also leads to the cleavage of lignin–hemicelluloses bonds. The reaction results in the increased solubility of the lignin alkaline solvent and in an enhanced water solubilization of hemicellulose.

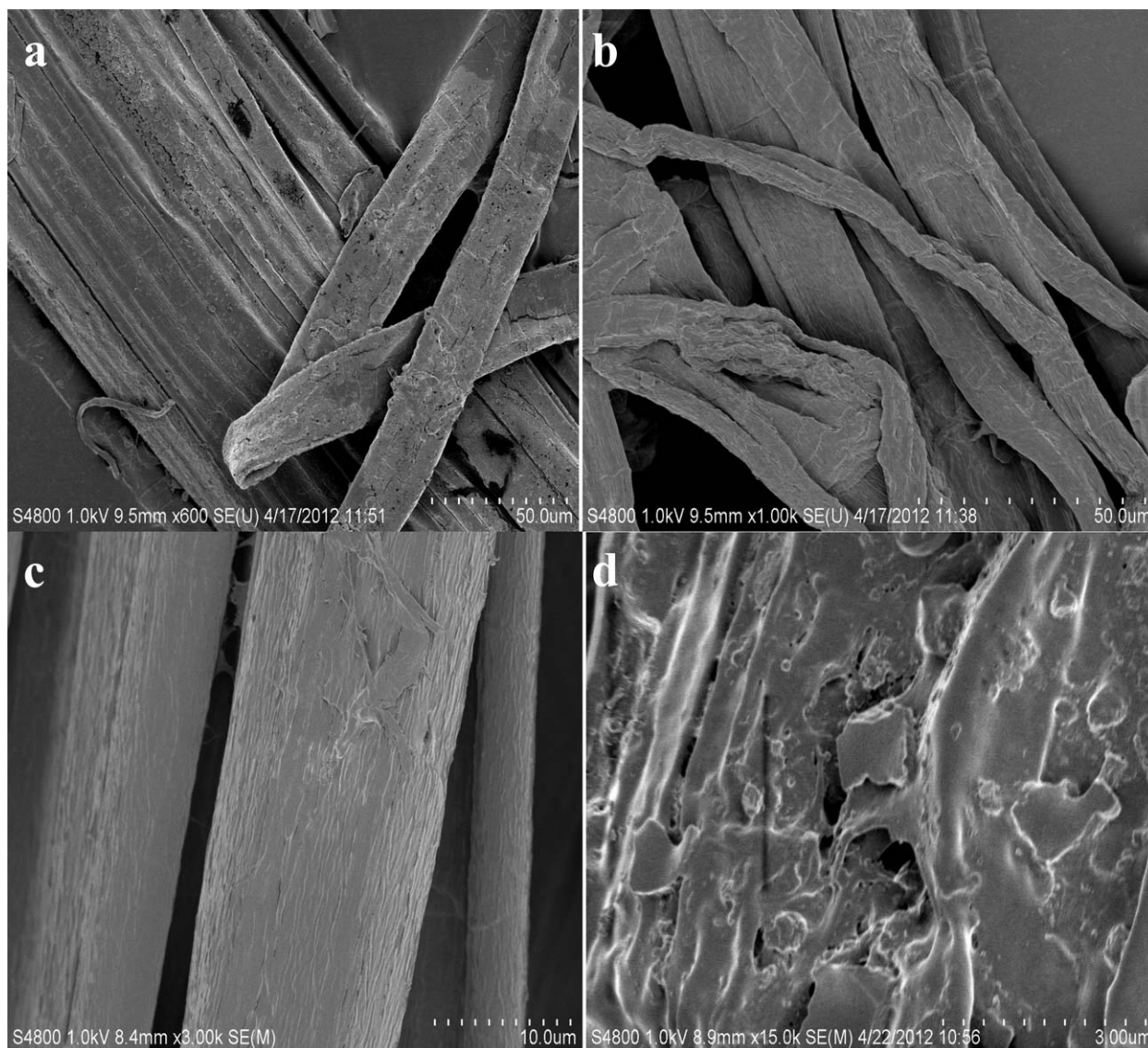
However, the lignin and hemicelluloses cannot be removed completely by the steam exploded treatment because this process only results in changes in the arrangement of the macromolecular chain. Bleaching processes to remove the majority of the residual gum and lignin component were conducted using a solution of NaOH, sodium silicate, and hydrogen peroxide. This process results in the further solubilization of hemicelluloses and lignin. The bleaching treatment breaks down phenolic compounds or molecules with chromophoric groups present in lignin and removes the byproducts of this breakdown, which results in a whitening of the pulp.<sup>22</sup> Lignin was oxidized and became soluble in the solution during the bleaching process.

The moisture content exhibited a slight increase in the treated fiber compared with the raw fiber. This increase is due to the increase in the cellulose content during the process. The moisture absorption rate increases with an increase in the cellulose content because the chemical structure of cellulose contains three free –OH groups. When the fibers were exposed to alkali media, the swelling of the fibers and the development of hydrophilic ionic groups on the surface was induced, which also promotes the absorption of moisture by capillary action.<sup>25</sup> The presence of residual alkali remaining in the microfibers after treatment can also cause moisture absorption. Furthermore, the hemicellulose removal after treatment increases the moisture sorption.

After steam explosion degumming, the fibers were isolated from the raw banana fiber, and the tensile properties of the treated fibers were determined (Table III). The results show that treatment with steam explosion resulted in a numerical increase in the brightness and fineness of the retted fibers compared with the raw material. However, only the breaking strength of the treated fiber was markedly lower than that of the raw fiber. This result is due to the retting process, which results in the degradation of pectin and hemicellulose around and between fiber bundles.<sup>9</sup> In spinning tests on modern rotor spinning frames, treated fiber is blended with viscose, and blended yarns with 50/50 treated fiber/viscose counts of Nm 30 can be achieved. The reason for this is the higher degree of freeness and the more homogenous and cleaner nature of the material obtained by steam explosion, which significantly improves the spinnability.<sup>5</sup>

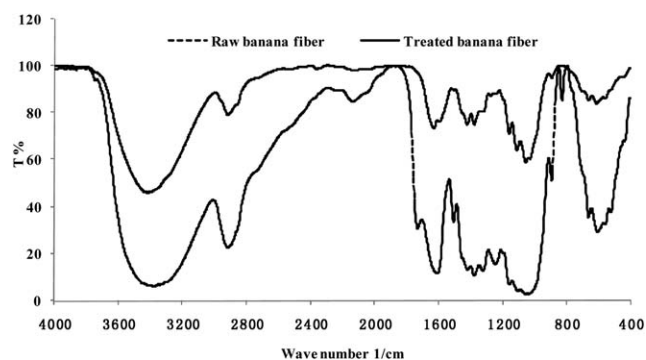
### SEM Analysis

The alkaline impregnation, steam explosion, and bleaching process result in structural and chemical changes on the fiber



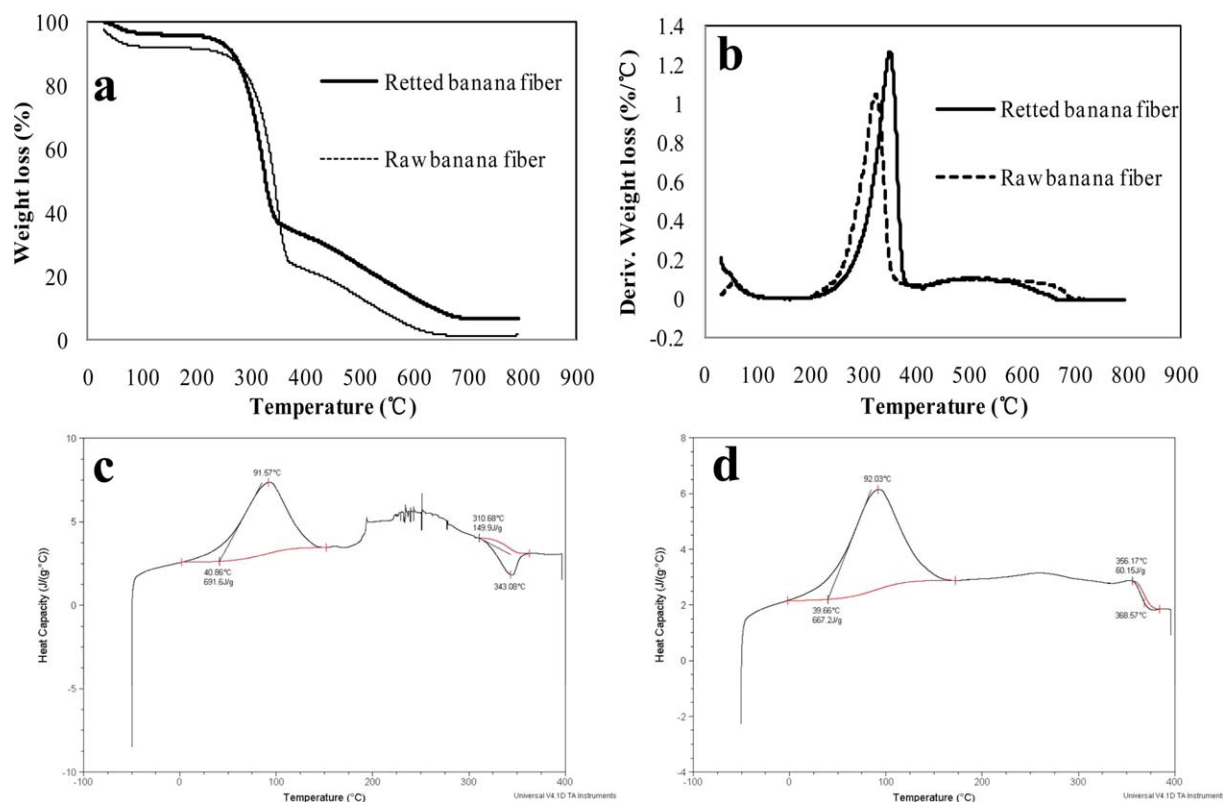
**Figure 1.** SEM of raw (a) and treated banana fibers (b–d).

surfaces. SEM micrographs of the raw and treated banana fiber were taken to investigate the structure of these fibers. The SEM micrographs are shown in Figure 1(a–d). Figure 1(a) reveals that the fibrils are associated in bundles, and the surface of the



**Figure 2.** FTIR spectra of raw and treated banana fibers.

fiber is smooth due to the presence of waxes and oil. Figure 1(b) clearly shows individual fibers after the removal of hemicelluloses, lignin, and pectin, which are the cementing materials around the fiber bundles, after the treatments. During the alkaline impregnation, steam explosion, and bleaching processes, the lignin becomes depolymerized and oxidized by hydrogen peroxide. Lignin oxidation leads to lignin degradation and to the formation of hydroxyl, carbonyl, and carboxylic groups, which facilitate the solubilization of lignin in an alkaline medium.<sup>13</sup> As a result, the defibrillation of the fiber occurs due to the removal of the cementing materials, as shown in Figure 1(b). It is clear from Figure 1(c) that the average diameters of the fibers are approximately 5–10  $\mu\text{m}$ , which is lower than the average size of the fiber bundles before treatment. This reduction in the particle size is due to the dissolution of hemicelluloses and lignin, as confirmed by the chemical analysis and FTIR graphs, which are shown in Table III and Figure 2. Figure 1(d) shows that the fibers have a rough surface and that most of the



**Figure 3.** TG (a), DTG (b), and DSC (c, d) heating curves of raw and treated banana fibers. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

fiber bundles are entirely individualized; this result is attributed to the steam explosion. The steam explosion obviously destructurized the fibers and increased the reactive area, which is beneficial for enhancing the cellulose accessibility.

### FTIR Analysis

FTIR spectroscopy is a non-destructive method used to investigate the physicochemical properties of lignocellulosic materials. Figure 2 shows the FTIR spectrum of the raw and treated banana fibers. The hydrophilic tendency of untreated and treated fibers is reflected in a broad band in the 3700–3100  $\text{cm}^{-1}$  region, which is attributed to the hydroxyl (OH) group present in the main component. In the spectrograms (Figure 2), a band of strong intensity at  $\sim 3400 \text{ cm}^{-1}$  in the spectra of the raw and treated banana fibers arises due to the stretching vibrations of the hydrogen-bonded –OH group. A band of medium intensity at  $\sim 2900 \text{ cm}^{-1}$  is due to the aliphatic saturated C–H stretching vibration in cellulose and hemicelluloses.<sup>12</sup> In the spectrum obtained for treated fiber, a sharpening of the peak at 2916  $\text{cm}^{-1}$  was observed. This sharpening can be attributed to the increase in the crystallinity of the fibers, which reflects an increase in the percentage of the cellulose components.<sup>14</sup> In the spectrum of raw banana fiber, the prominent band at 1734  $\text{cm}^{-1}$  may be due to the uronic esters and acetyl groups from the hemicelluloses or to the ester bonds of the carboxylic groups of the p-coumaric and ferulic acids from lignin.<sup>26</sup> This peak disappeared in the treated banana fiber, which indicates that the treatment process effectively broke the ester bond of

the hemicelluloses and lignin and thereby removed them. The peak at 1630  $\text{cm}^{-1}$  is primarily due to the bending mode of the absorbed water with some contribution from carboxylate groups.<sup>27</sup> The peak at 1509  $\text{cm}^{-1}$  was attributed to vibrations of the aromatic ring of lignin,<sup>26</sup> and this band also disappeared in the treated banana fiber. In the spectrum obtained for treated fiber, we can observe that the treated fibers also contain a characteristic peak due to lignin components in the region of 1425  $\text{cm}^{-1}$ , which indicates that the complete removal of lignin was not achieved. The weak peak at 1380  $\text{cm}^{-1}$  was assigned to O–H bending.<sup>27</sup> At 1050  $\text{cm}^{-1}$ , a strong band due to the skeletal vibration of the C–O–C pyranose ring was observed.<sup>28</sup> The peak at 897  $\text{cm}^{-1}$  is characteristic of the  $\beta$ -glucosidic bonds between sugar units. A small band appeared at 713  $\text{cm}^{-1}$ , which indicates that phase  $I_{\beta}$  cellulose is the predominant form in the treated banana fiber.<sup>29</sup>

### Thermal Analysis

The thermal study involved the analysis of the weight loss of the fiber as a function of temperature. Figure 3(a) and (b) show the thermogravimetric (TG) and derivative thermogravimetric (DTG) curves of the raw and treated banana fibers, respectively. A small weight loss was found from 50 to 100°C, and this loss is primarily attributed to moisture and solvent evaporation or to low-molecular-weight compounds remaining after the isolation procedures.<sup>30</sup> The temperature range between 200 and 300°C is primarily due to the depolymerization of non-cellulose, such as hemicelluloses, and the cleavage of glycosidic

linkages of cellulose to form levoglucosan (1,6-anhydro- $\beta$ -D-glucopyranose) and carbon residues.<sup>31</sup> The decomposition of lignin occurs in the region from 200 to 500°C.<sup>32</sup> The thermogram of the raw banana fiber [Figure 3(a)] indicated a greater resistance to degradation in the range 340–500°C due to the presence of larger amounts of hemicelluloses and lignin. As shown in Figure 3(b), the maximum degradation rate was found during the  $\alpha$ -cellulose decomposition of raw and treated banana fibers at 322.81°C (mass loss = 54.72%) and 349.04°C (mass loss = 42.78%), respectively.

Table IV shows that the degradation temperature of raw fiber is lower than that of treated fiber because the cellulose of raw banana fiber is organized into fibrils, which are surrounded by a matrix of lignin, hemicellulose, and pectins. Hemicelluloses are located within and between the cellulose fibrils and decomposed before lignin and cellulose due to the presence of acetyl groups.<sup>33</sup> This strong association between the cellulose fibrils and hemicellulose is hypothesized to decrease the average crystallinity of the cellulose fibrils.<sup>15</sup> These impurities may initiate more active sites and accelerate the initiation of thermal degradation.

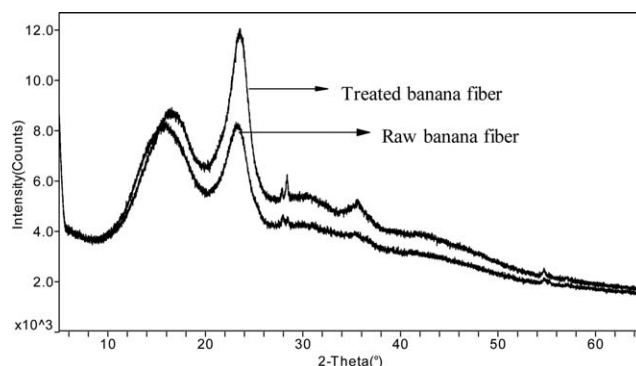
The raw banana fiber shows a higher ash content at 800°C (Table IV) because it contains a higher lignin content. The treated fiber shows a lesser ash content, which indicates the removal of lignin. As shown in Table IV, as the fiber is processed from raw fiber through steam treatment, an increase in the value of the temperature is observed in different weight loss regions. This finding is due to the increase in the cellulose content because a greater crystalline structure requires a higher degradation temperature, which enables the fiber to resist drastic heat and processing conditions.<sup>15</sup> These results corroborate the results obtained from the chemical composition analysis and FTIR measurements.

**Differential Scanning Calorimetry (DSC).** The heating DSC curves for raw and treated banana fibers are shown in Figure 3(c) and (d). The DSC curves of all of the samples reveal the

**Table IV.** Peak Temperature at Various Mass Loss Percentages and Thermal Behavior of Raw and Treated Banana Fiber

Fiber characteristics	Raw banana fiber	Treated banana fiber
Peak temperature (°C)	322.81 ± 0.32 <sup>a</sup>	349.04 ± 0.72
% of degradation	54.72 ± 0.51	42.78 ± 0.27
Ash content at 800°C (%)	7.02 ± 0.32	1.72 ± 0.12
5% Mass loss (°C)	224.75 ± 0.77	47.20 ± 0.78
10% Mass loss (°C)	267.80 ± 0.21	249.50 ± 0.47
15% Mass loss (°C)	282.65 ± 0.65	286.35 ± 0.98
20% Mass loss (°C)	293.59 ± 0.35	302.75 ± 0.12
25% Mass loss (°C)	300.85 ± 0.86	313.65 ± 0.34
Total mean value (°C)	273.93 ± 19.51	238.89 ± 18.65

<sup>a</sup>Mean ± SD (N = 3).



**Figure 4.** XRD patterns of raw and treated banana fibers.

evaporation of water at approximately 90–100°C and the degradation of  $\alpha$ -cellulose at approximately 310–370°C. This finding was also confirmed with the TG results. The enthalpy values associated with moisture desorption and  $\alpha$ -cellulose decomposition are marked in the curves. The raw fibers exhibit lower enthalpy values than the treated fiber due to increases in the amount of crystalline cellulose during different chemical processes, such as alkaline impregnation, steam explosion, and bleaching. Additionally, the increased crystallinity percentage of the fibers and H-bonding located within cellulose chains leads to increases in the enthalpy values.<sup>15</sup> Therefore, it can be concluded from these results that the retted banana fibers exhibit enhanced thermal properties compared with the untreated fibers. The DSC and TGA results prove that the retted banana fibers have enhanced thermal properties and can be processed at elevated temperatures compared with their non-treated counterparts.

#### XRD Analysis

Cellulosic fibers consist of three major components: lignin, hemicelluloses, and  $\alpha$ -celluloses. Cellulose shows a crystalline nature, whereas lignin is amorphous in nature. As a result, the crystallinity of the fibers should increase after treatment. As shown in the XRD graph (Figure 4), it is clear that the treated banana fibers show a crystalline nature, which indicates changes in the crystalline proportions of the fiber after treatment. The peaks observed near 23.87 represent diffractions of the (002) crystal plane, which indicates that the nature of the cellulose crystal in treated banana fiber is cellulose type  $I_{\beta}$ .<sup>34</sup> This result further confirms the FTIR findings. The sharp peak in the XRD pattern of the treated fibers exhibits a higher crystallinity, which is attributed to the more efficient removal of non-cellulosic polysaccharides and the dissolution of amorphous zones. The percent crystallinity calculated from the areas under the peaks and the amorphous regions in the graphs were 11.8% for raw banana fiber and 53.9% for treated banana fibers. It is clear that the raw fiber is almost amorphous with very little crystallinity and that the treated banana fibers show a crystalline nature. The XRD pattern of retted fiber, which exhibit an increased crystallinity, gives a relatively intense peak at  $2\theta = 23.87^\circ$ . Similar results have been reported by Abraham et al.<sup>11</sup>. Therefore, the above results demonstrate that hydrolysis took place preferentially in the amorphous region.



## CONCLUSIONS

The raw banana fibers were successfully retted by steam explosion. The optimum degumming conditions were determined to be the following: moisture content of 10%, 1.2% NaOH, steam pressure of 1.75 MPa, and residence time of 90 s. Under these optimized conditions, the experimental yield of residual gum ( $5.47 \pm 0.22\%$ ) and lignin ( $3.78 \pm 0.12\%$ ) agreed closely with the predicted yield. The hydrolysis of the hemicelluloses and lignin was confirmed by chemical composition, FTIR, and XRD. The SEM analysis of the treated fibers showed a change in their surface morphology compared with that of the raw fibers. The thermal analysis showed that the thermal stability of the treated fibers was improved.

## APPENDIX

1. Count testing of ramle fiber, Chinese National Standard, GB 5884-86.
2. Testing method of whiteness of ramle fiber, Chinese National Standard, GB 5885-86.
3. Testing method of single fiber breaking tenacity of ramle, Chinese National Standard, GB 5886-86.
4. Testing method of length of ramle fiber, Chinese National Standard, GB 5886-86.

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