COMPARATIVE STUDY OF BAMBOO SHOOT SHELL FIBERS DEGUMMED BY IMPROVED SODIUM PERCARBONATE AND ALKALINE-OXYGEN METHODS

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Degumming is an essential and critical process for manufacturing bast fiber. It is generally believed to be an expensive process and causes some serious environmental problems. Therefore, it is imperative to develop novel or improved environmentally friendly degumming methods. Herein, we report a comparative study of degumming performance of bamboo shoot shell fibers treated by alkaline-oxygen and sodium percabonate, respectively, after dilute alkali pretreatment. Results indicate that fibers produced by the improved alkaline-oxygen method offered relatively higher cellulose content (71.67%) and crystallinity (65.12%) than those produced by the sodium percarbonate method, which yielded cellulose content of 64.42% and crystallinity of 60.07%. It was further observed that the improved alkaline-oxygen process did not only exhibit a better degumming rate, but also was relatively efficient.

Keywords: bamboo shoot shell fiber, degumming rate, sodium percarbonate, alkali-oxygen bath, chemical composition

INTRODUCTION

Alarmingly exhausting fossil fuel reserves have caused growing environment-related concerns, which firmly demand to prioritize the utilization renewable natural materials. Therefore, natural fibers have gained massive attraction in recent times due to their renewable source, biodegradable nature, sustainability and abundant availability.[1,2] Especially, biopolymers originating from cellulose have great potential for developing high-end functional products, presenting substantially high application value, particularly in the field of textiles and fiber-reinforced composites.[3,4] Among the variety of cellulose based natural fibers, bamboo fibers have received high recognition due to the fast growth rate, high output, and low cost. Bamboo shoot shell, the outer sheath of bamboo shoots, has been extensively used for slippers, animal feed, stickers and other applications. Therefore, it is essential to further explore the potential, compatibility and capacity of these bamboo shoot shells for high value-added applications.[5]

Degumming is a crucial process that improves the properties of cellulose bast fibers by removing a large number of non-cellulosic components and boosts their spinnability. For that purpose, various degumming technologies, including enzymatic, chemical or physical methods, can be employed.[6,7] Zhang *et al*. used steam explosion and Fenton reagent to degum kenaf fibers and achieved a linear density of 70 dtex.[8] Paramasivam *et al*. used pectinase, laccase and the combination of the two enzymes in different concentrations to treat banana fibers, which removed pectinolytic substances, leaving intact cells in the fiber cell walls, and smoothened the surface of resultant banana fibers.[9] Ahmed *et al*. reported the use of deep eutectic solvent (DES) *via* microwave energy (MWE) for degumming hemp bast fibers to generate pure hemp cellulose fibers for potential textile applications.[10] Therefore, it is essential to develop a novel

degumming method, which not only offers fast and economical degumming, but is also eco-friendly.[11]

Herein, improved alkali-oxygen and sodium carbonate methods were used for degumming of bamboo shoot shell fibers, preceded by a dilute alkali pretreatment. Factors affecting the process, such as the dosage of degumming chemicals, soaking temperature and time, were carefully analyzed. The results would provide reference for the future applications of bamboo shoot shell fiber.

EXPERIMENTAL
Materials
Mature natural bamboo shoot shells were collected from the bamboo planting garden of Wuhan Textile University, China. Sodium percarbonate ($2Na_2CO_3 \cdot 3H_2O_2$), 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 purchased from Aladdin Chemical Regent Inc., Shanghai, China. All the chemicals were used as received, without further purification.

Degumming process
The bamboo shoot shell was cut into strips, with the length of 2.5 cm and width of 0.5 cm, then soaked in a warm water bath (50 °C) to remove the impurities and fluff from the surface, and finally washed and oven dried for 24 h at 75 °C. Bamboo shoot shells were first pretreated with a dilute alkaline solution and then degummed with sodium percarbonate and alkali-oxygen method, respectively. The effects of the concentration of NaOH, H_2O_2 and sodium percarbonate, of soaking temperature and soaking time were carefully studied. Later, a thorough comparative analysis of the results of the two processes was carried out. Each determination was repeated thrice, and the average value was calculated.

Dilute alkali pretreatment
An amount of 10 g of bamboo shoot shell was placed in 10 g/L NaOH (mass ratio of fibers to solution) solution mixed with 3 wt% Na_2SiO_3 at a bath ratio of 1:40. Then, samples were soaked in a hot water bath (90 °C) for 3 h. The pretreated bamboo shoot shells were washed with deionized water until the pH of drain water was neutral, and then oven dried for 24 h at 60 °C.

Alkali-oxygen treatment
An amount of 2 g of alkali-pretreated bamboo shoot shell was placed in a beaker containing various concentrations of NaOH (10, 12.5, 15, 17.5, 20 and 22.5 g/L) and H_2O_2 (12.5, 15, 17.5, 20, 22.5 and 22.5 mL/L). The influence of reaction time (1.5, 2, 2.5, 3 and 3.5 h), and temperature (70, 75, 80, 85, 90 and 95 °C) was also assessed to examine their effect on the subsequent bamboo shoot shell fibers. The concentration range was determined by the single factor experiment, and then an orthogonal experiment was carried out to obtain the optimal process.

Sodium percarbonate treatment
An amount of 2 g of alkali-pretreated bamboo shoot shells was placed in a beaker with sodium percarbonate (12, 16, 18, 20, 22 and 24 g/L) and H_2O_2 (12, 16, 20, 24, 28, 32 mL/L). Reaction time (0.5, 1, 1.5, 2, 2.5 and 3 h), and temperature (70, 75, 80, 85, 90 and 95 °C) were varied to investigate the effects of time and temperature on the resultant bamboo shoot shell fibers. The concentration ranges were also determined by the single factor experiment, and then an orthogonal experiment was carried out to obtain the optimal process.

Calculation of degumming rate
The degumming effect was assessed by determining the degumming rate. The degumming rate of bamboo shoot shell fibers was calculated by Equation 1:[12]

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

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

Characterization
The content of cellulose, hemicelluloses, lignin and pectin of the samples was determined by chemical analysis with five replicated samples and the average values with standard deviation were reported according to the literature reported.[13] The surface morphology of all samples coated with a thin layer of gold by means of a plasma sputtering apparatus was observed using a Scanning Electron Microscope (JSM-6510 LV, JEOL, Japan), with an electron beam. Accelerating potential of 5 kV was used for SEM analysis. The crystallinity and surface chemistry of the samples, before and after the treatment, was determined by X-ray diffraction (D/max-2550pc, RIGAKU) and Fourier transform spectroscopy (Nicolet iS50, Thermo Fisher, USA). The crystallinity index (CrI) of the material was calculated using Equation 2:[14]

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

where CrI represents the relative degree of crystallinity, I_{200} is the maximum intensity of the (200) lattice diffraction at $2\theta = 22.3°$, and I_{am} is the intensity of diffraction around $2\theta = 15.9°$.

The thermal stability of the samples, before and after the treatment, was examined by thermogravimetric analysis (TGA TG 209 F3), at a heating rate of 10 °C/min, and in the temperature range of 20-600 °C.

RESULTS AND DISCUSSION
Degumming process of bamboo shoot shell fiber
According to a large number of single factor experiments, orthogonal experiments were designed

for the various factors involved, such as the mass concentration of sodium hydroxide, sodium percarbonate, and hydrogen peroxide, reaction time and temperature, and three levels were set for each factor. The range of the experiment was finally determined in Tables 1 and 3, and the test results were shown in Tables 2 and 4, respectively.

Table 2 presents the order of factors influencing the degumming using the sodium percarbonate method. R stands for the "Range" value. The larger the R value was, the greater the influence of the factor. In the sodium percarbonate degumming experiment, R for A (NaOH), B (Sodium percarbonate), C (Temperature) and D (Time) was 2.21, 3.26, 3.94 and 4.42, respectively. It was noticed that the effect of soaking time (D) displayed relatively higher influence than soaking temperature (C). Additionally, the concentration of H_2O_2 (B) helped comparatively more in regulating degumming performance than sodium percarbonate (A). The optimal scheme of the orthogonal experiment is $A_3B_3C_3D_2$, *i.e.*, sodium percarbonate of 24 g/L, hydrogen peroxide of 32 mL/L, soaking temperature of 95 °C, and treatment time of 2.5 h. The degumming rate of 55.48% was obtained under the optimized process conditions.

Table 1
Levels of orthogonal factors in sodium percarbonate degumming experiment

Levels	Factors			
	A NaOH, $g \cdot L^{-1}$	B Sodium percarbonate, $g \cdot L^{-1}$	C Temperature, °C	D Time, h
1	20	24	85	2
2	22	28	90	2.5
3	24	32	95	3

Table 2
Orthogonal design and test data of sodium percarbonate degumming

Test No.	A	B	C	D	Test index Degumming rate (%)
1	1	1	1	1	48.81
2	1	2	2	2	47.13
3	1	3	3	3	50.29
4	2	1	2	3	46.14
5	2	2	3	1	53.12
6	2	3	1	2	52.66
7	3	1	3	2	55.18
8	3	2	1	3	45.30
9	3	3	2	1	52.38
k_1	48.74	50.04	48.92	51.66	
k_2	50.64	48.52	48.55	47.24	
k_3	50.95	51.78	52.86	47.24	
R	2.21	3.26	3.94	4.42	
Main→Secondary				DCBA	
Optimal scheme				$A_3B_3C_3D_2$	

Table 3
Levels of orthogonal factors in alkaline oxygen degumming experiment

Levels	Factors			
	A NaOH, $g \cdot L^{-1}$	B H_2O_2, $mL \cdot L^{-1}$	C Temperature, °C	D Time, h
1	17.5	20	85	2.5
2	20	22.5	90	3
3	22.5	25	95	3.5

Table 4
Orthogonal design and test data of alkaline oxygen degumming

Test No.	A	B	C	D	Test index Degumming rate (%)
1	1	1	1	1	48.15
2	1	2	2	2	57.97
3	1	3	3	3	62.85
4	2	1	2	3	61.35
5	2	2	3	1	55.59
6	2	3	1	2	53.32
7	3	1	3	2	57.88
8	3	2	1	3	60.59
9	3	3	2	1	56.64
k_1	56.32	55.79	54.02	53.46	
k_2	56.75	58.05	58.65	56.39	
k_3	58.37	57.60	58.77	61.60	
R	2.05	2.26	4.75	8.14	
Main→Secondary				DCBA	
Optimal scheme				$A_3B_2C_3D_3$	

Table 5
Mean square errors from SPPS software for two degumming processes

Sodium percarbonate degumming	A NaOH, $g \cdot L^{-1}$	B $2Na_2CO_3 \cdot 3H_2O_2$, $g \cdot L^{-1}$	C Temperature, °C	D Time, h
Mean square error	4.290	7.981	17.134	18.555
Alkaline oxygen degumming	A NaOH, $g \cdot L^{-1}$	B H_2O_2, $mL \cdot L^{-1}$	C Temperature, °C	D Time, h
Mean square error	3.494	4.284	22.038	50.950

Meanwhile, according to the data listed in Table 4, the order of factors in the alkaline-oxygen method can be established as a function of the R value. Thus, time (D) had the highest effect on degumming process performance, followed by temperature (C), as the R values for A (NaOH), B (H_2O_2), C (Temperature) and D (Time) were 2.05, 2.26, 4.75 and 8.14, respectively. Furthermore, it was noticed that the concentration of H_2O_2 had a relatively higher impact on the degumming, when compared with that of NaOH concentration. The optimal scheme of the orthogonal experiment is $A_3B_2C_3D_3$, which is alkali (22.5 g/L), hydrogen peroxide (22.5 mL/L), temperature (95 °C), and treatment time (3.5 h). The degumming rate of 60.34% was achieved under these optimized process conditions. Meanwhile, the degumming rate (55.48%) reached by the sodium supercarbonate method is lower than that obtained by the alkaline-oxygen method (60.34%), indicating that the degumming effect of the alkaline-oxygen method is relatively higher than that of the sodium supercarbonate method in this respect.

The mean square errors (as shown in Table 5) were also analyzed by the F-test used in ANOVA by the SPPS software in order to determine whether the factors had a significant influence on the experimental indicators. The results were consistent with that of range value.

Chemical composition analysis

Table 6 displays the chemical composition of all the samples. It is apparent that the bamboo shoot shell mainly contained cellulose, hemicelluloses and lignin. The cellulose content in bamboo shoot shell was relatively lower with the mass fraction (41.12%) than the content of pectin, lignin, and hemicelluloses, whose collective mass fraction was about 56.74%.[15] After the alkaline oxygen treatment, the cellulose content significantly improved to 71.67%, indicating successful degumming *via* the improved alkaline oxygen method. After the percarbonate treatment, the mass percentage of cellulose also significantly improved to 64.42%, indicating efficient degumming *via* this method. Still, as can be observed, after alkaline oxygen degumming, the cellulose mass content in the resultant bamboo shoot shell fibers (71.67%) was

significantly higher than that in the sodium percarbonate degummed fibers (64.42%). It was further observed that the content of hemicelluloses decreased substantially after the alkali pretreatment. However, there was no apparent change in the content of lignin and pectin. This decrement of hemicelluloses was attributed to their low degree of polymerization, making them easily soluble in alkaline solutions. Moreover, it is well-known that cellulose is a long-chain molecule and is resistant to alkaline solutions.[16] On the other hand, the lignin content decreased only after the alkaline oxygen and sodium percarbonate treatments, this decline in the lignin content was credited to the presence of H_2O_2, which oxidized the lignin molecules. After oxidization, lignin was easily dissolved in the alkaline solution.[17] Finally, it was witnessed that both sodium percarbonate and alkaline oxygen methods were capable of successfully resolving the tangled structures of cellulose, hemicelluloses, lignin and pectin. As a result, both processes were successfully used to extract cellulose.

Table 6
Major chemical components of the samples before and after different treatments

Component	Cellulose, %	Hemicelluloses, %	Lignin, %	Pectin, %
Untreated	41.12±1.82	34.10±0.75	15.50±0.79	7.14±0.37
Pretreated	50.04±1.83	26.59±0.68	12.12±0.73	4.69±0.31
Sodium percarbonate treated	63.42±1.88	17.47±0.56	10.45±0.67	3.67±0.23
Alkaline oxygen treated	70.67±1.92	13.51±0.51	8.34±0.62	2.75±0.19

Morphological structure

Figure 1 illustrates the morphology of bamboo shoot shell fibers before and after the degumming process. It is obvious from Figure 1a that the surface of the untreated bamboo shoot shell was rough and covered with a lot of colloids. It can be seen that there was an apparent decline in the joints of fibers and the fiber surface became relatively smooth after the alkaline pretreatment (Fig. 1b).
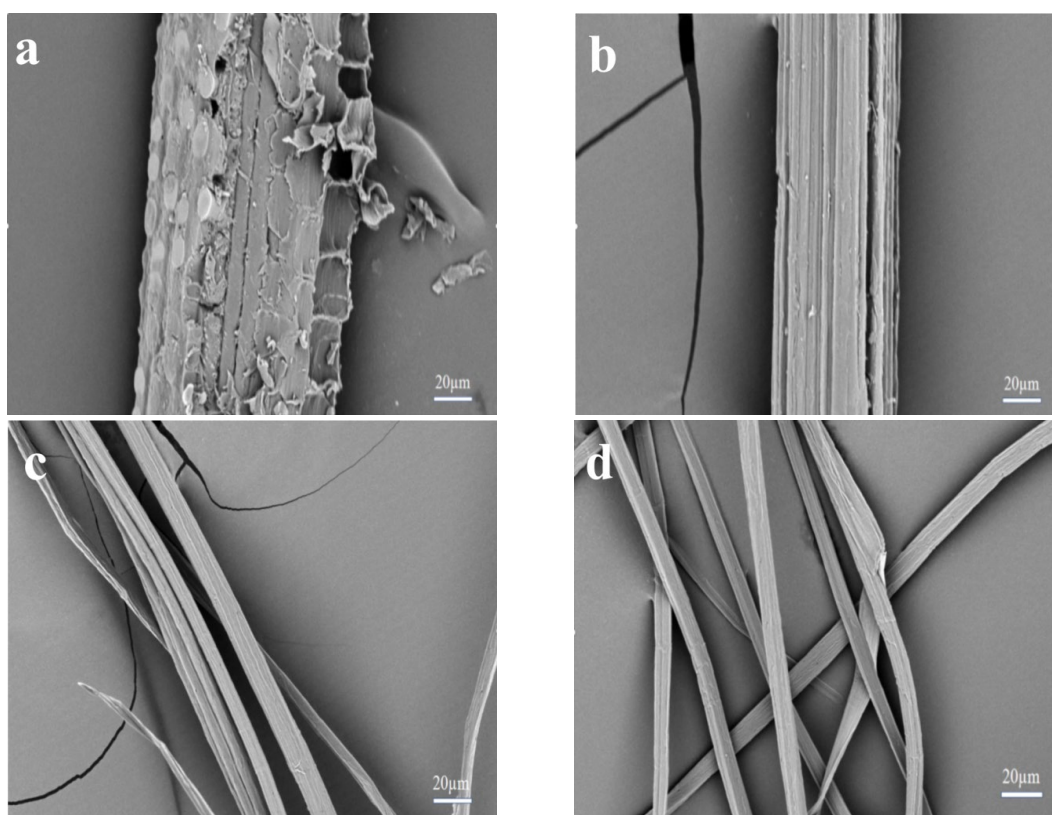

Figure 1: SEM images of (a) untreated, (b) pretreated, (c) sodium percarbonate treated, and (d) alkaline oxygen treated samples
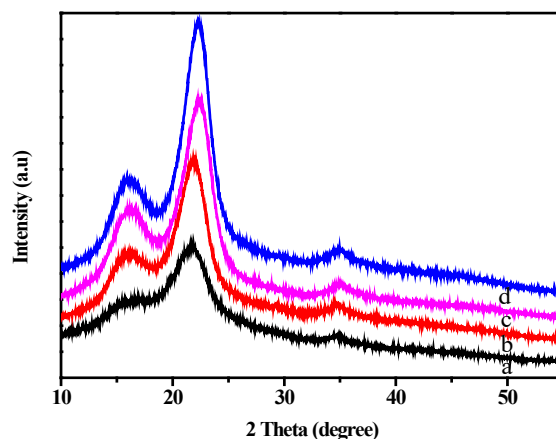
Figure 2: X-ray diffraction patterns of (a) untreated, (b) pretreated, (c) sodium percarbonate treated and
(d) alkaline oxygen treated samples

Meanwhile, it can be seen that the bamboo shoot shell is composed of a large number of fibers glued together. Further, it is visible that both the sodium percarbonate (Fig. 1c) and the alkaline-oxygen treatments (Fig. 1d) transformed the glued fiber bundles into individual bamboo shoot shell fibers, with a diameter of tens of microns. The resultant fibers also had comparatively much smoother fiber surface than the untreated sample. It was also noticed that the fibers produced by sodium carbonate degumming still presented partial attachment of fibers, while those degummed by the alkaline-oxygen process were completely separated, with no attachment of adjacent fibers.[18] It was established that the degumming performance of the alkaline-oxygen treatment was certainly higher than that of the sodium supercarbonate degumming.

X-ray diffraction analysis

Figure 2 displays the XRD patterns of bamboo shoot shell fibers before and after the degumming process. Characteristic diffractive peaks at 2θ of 15.9 (1$\bar{1}$0/110), 22.3 (200) and 34.5° (004), respectively, correspond to typical crystalline cellulose type I structure.[14,19] The presence of these peaks in all the samples determined that the crystal form of the bamboo shoot shell fibers did not change after the degumming. The crystallinity of untreated fibers was 43.53%, which slightly increased after the pretreatment to 50.12%. Furthermore, it was observed that the fibers degummed with alkali-oxygen offered comparatively higher crystallinity (65.12%) than those degummed with sodium percarbonate, which had a crystallinity of 60.07%. Both of them are higher than our earlier reported crystallinity value

(58.7%), when bamboo shoot shell fibers were extracted by the ultrasound-assisted alkali-oxygen bath method.[20] This improvement in the crystalline region of bamboo shoot shell fibers after the degumming process was attributed at an increase in the proportion of crystalline region, owing to the removal of lignin, hemicelluloses, pectin and water solutes from the fiber matrix.[17] Additionally, higher crystallinity for alkaline-oxygen degummed fibers indicated better degumming performance, compared to the sodium percarbonate degumming method.

Infrared analysis

Figure 3 depicts the FTIR spectra of the bamboo shoot shell samples before and after different treatments. The strong absorption band at 3339 cm^{-1} corresponds to the stretching vibration of -OH group. The existence of the methyl group was confirmed by the presence of a typical peak at 2903 cm^{-1}. Similarly, another peak at 1050 cm^{-1} attributed to the stretching vibration of the C-O group in C-O-C confirmed the presence of cellulose. Other absorption peaks at 1643 cm^{-1}, 898 cm^{-1}, 1740 cm^{-1} and 1256 cm^{-1} correspond to C=O in the cellulose acetyl group, the expansion vibration of glucose-glycoside, C=O in acetyl group, and the symmetric tensile vibration of the C-C group in the aromatic ring representing the presence of lignin, respectively.[21] Further, it can be seen from Figure 3 that the intensity of the characteristic peaks of cellulose in the degummed bamboo shell fibers increased compared to that for the untreated samples, indicating the increment in the cellulose content after degumming.[22] Lastly, the decrease or disappearance of the characteristic peak of lignin at

1740 and 1256 cm^{-1} for the treated samples indicates that the degumming treatment effectively removed non-cellulosic components, such as hemicelluloses and lignin. The results are well in line with the findings of XRD and chemical composition.
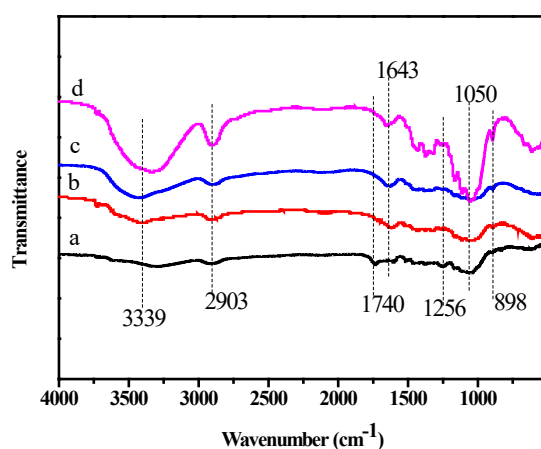

Figure 3: FTIR spectra of (a) untreated, (b) pretreated, (c) sodium percarbonate treated, and (d) alkaline oxygen treated samples
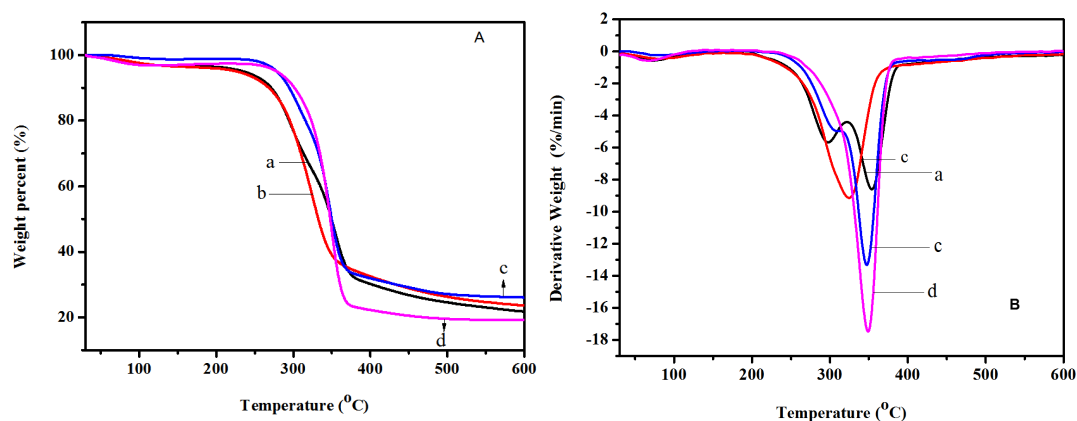

Figure 4: TG (A) and DTG (B) curves of (a) untreated, (b) pretreated, (c) sodium percarbonate treated and (d) alkaline oxygen treated samples

Thermal performance

Figure 4 shows the TG and DTG curves of bamboo shoot shell fibers before and after the degumming process. The first weight loss in bamboo shoot shell fibers both before and after the degumming treatments took place between 30 °C and 150 °C, which was attributed to the evaporation of water absorbed by the fibers.[23] The second weight loss stage was witnessed between 200 and 400 °C, which was credited to the lysis of cellulose. During this weight loss, decomposition of hemicelluloses and lignin took place at 210-320 °C and 320-400 °C, respectively. Moreover, it was noted that the decomposition of the untreated and alkali-pretreated bamboo shoot shell samples begins at 250 °C, indicating their relatively lower thermal stability, compared to that of the degummed samples, which started to decompose at relatively higher temperature. Since lignin decomposes in a higher temperature range (200-600 °C), even after the complete decomposition of cellulose and hemicelluloses, lignin continued to decompose after 400 °C. The results indicate that the degumming successfully removed even the components having higher decomposition temperature.[24] After degumming, the bamboo shoot shell fibers displayed weight loss in a comparatively higher temperature range. This improved thermal resistance was ascribed to the removal of colloids located among the fibers and enhanced the proportion of the crystalline area. All in all, both degumming treatments improved the thermal stability of the resultant bamboo shoot shell fibers.

CONCLUSION

In summary, bamboo shoot shell fibers were successfully degummed by improved

alkaline-oxygen and sodium percarbonate methods, respectively. The outcomes of the two degumming processes were compared. The fibers degummed by the alkaline-oxygen method displayed relatively higher cellulose content (71.67%) than those degummed by the sodium percarbonate method, which offered the cellulose mass content of 64.42%. It was further witnessed that the alkaline-oxygen method did not only produce a higher degumming rate, but also was a relatively fast and efficient degumming method, compared to the sodium percarbonate treatment. The obtained bamboo shoot shell fibers have can be potential used in composite reinforcement and other perspective applications.

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