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Physicochemical and Thermal Properties of Lignocellulosic Fiber from *Gigantochloa Scortechinii* Bamboo: Effect of Steam Explosion Treatment

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Abstract: Bamboo is an abundant natural resource in Asia and one of the high potential fibers used to reinforce polymer composites. This article presents a comparative study on the physicochemical and thermal properties of bamboo fiber (BF) from *Gigantochloa scortechinii* for untreated BF, steam explosion (SE) treatment BF, and steam explosion followed by alkali (SE-alkali) treatment BF. The physicochemical and thermal properties of BF were determined using energy dispersive X-ray (EDX) spectroscopy and thermogravimetric analysis (TGA), while scanning electron microscopy (SEM) was used to examine the surfaces morphologies. A Fourier transform infrared (FTIR) spectroscopy was utilized to detect the presence of functional groups. TGA results showed that SE BF was significantly more thermally stable than the untreated BF and SE-alkali treatment BF. Major changes in chemical composition and surface morphology of the bamboo fibers indicated that hemicellulose and lignin were removed by SE-alkali treatment. In conclusion, the BF surface is physically and chemically modified by the SE-alkali treatment.

Keywords: Bamboo fiber, Steam explosion, Alkali treatment, Physicochemical, Thermal

Introduction

Environmental concerns, shortage of petroleum resources, needs for eco-products, and the demand for new materials to fulfill the requirement of the present technology have progressively encouraged the development of new eco-composites [1,2]. Industries have been developing new materials with lightweight and extreme tensile strength for aerospace and building industries, such as mining, tunnel, and automotive [3]. Natural fiber-reinforced polymer composites are advantageous over synthetic materials because of their enormous availability, sustainable reinforcement, lightweight nature, low cost, biodegradability, renewability, and excellent strength-to-weight characteristic [4,5].

However, many factors should be considered when natural fibers are used because of their incompatibility with the hydrophobic polymer matrix given by their hydrophilic nature [6-8]. Fiber and matrix adhesion also influenced the mechanical characteristics of natural fiber composites. The previous study [9] showed that untreated fibers have poor adhesion between the fiber and matrix. Despite this, high-level moisture absorption and poor wettability of non-polar plastics will also lead to poor adhesion [10-12]. Instead of breakage, the fibers have been pulled out during fracture because of weak adhesion between fiber and matrix. Dweiri and Azhari [13] observed that the failure morphology for polyamide 6/sugarcane bagasse composites was caused by voids and the broken fibers. The chemical structure and polarity of the fiber and matrix play an important role to enhance the adhesion [12]. Increasing the hydrophobicity of the natural fibers will result in enhanced mechanical properties and compatibility between the matrix and natural fibers [12]. Therefore, appropriate treatment should be employed to improve the adhesion between natural fibers and polymer matrices. Various types of fiber treatments or surface modifications have been performed to increase the adhesion between natural fibers and polymer matrices. These treatments include the application of silane coupling agent, alkali, permanganate, acetylation, benzoylation, and steam explosion (SE). Bamboo fiber (BF) composites prepared via SE treatment exhibited higher tensile strength compared with the same composites via mechanical extraction and alkali treatment [14].

The SE is a physical treatment method in producing fibers, because it involves hot steam with high temperature (180-240 °C) and high pressure (1-3.5 MPa), followed by explosive decompression [15]. The sudden release of pressure during the SE process with high temperature and high pressure has caused cellulose bundles to break and defibrillate [15]. The cellulose nanofibrils can be extracted using SE [16,17]. The smaller size of the treated fiber can increase thermal properties compared with untreated fibers [17]. The small-sized fibrils, such as pineapple leaves, from SE-alkali-treated fibril resulted in higher mechanical properties

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compared with alkali-treated pineapple leaves fibril [18]. The interfacial bonding between fiber and matrix is enhanced by changing the fiber surface with SE-alkali treatment. This process contributes to the absorption of energy during an impact test [19].

A natural fiber reinforced polymer composites have been chosen for current materials because of their superior characteristics and response to green technology. Several natural fibers with excellent mechanical properties are bamboo, jute, cotton, kenaf, and coir [20-24]. Bamboo is one of the fibers with excellent mechanical properties because of its weight and longitudinally aligned fibers in its body [25]. It is widely distributed worldwide and belongs to the *Gramineae* family and *Bambuseae* subfamily [26]. Besides that, bamboo is also one of the lignocellulose materials for thermoplastic composites that can reduce the use of fossil fuel-based materials and environmental impact [27].

Among jute, coir, and bamboo, Young's modulus of BF was the highest one [28]. Several studies [29-31] have shown that bamboo has a great potential for reinforcing thermoplastic composites and is a promising substitute for wood polymer composites. The usage of bamboo fibers in a composite is widely accepted because of their biodegradability, sustainability, recyclability, good mechanical properties, and low cost [32-35]. Other studies have reported that bamboo contains 60 % of cellulose and 32 % of lignin [36]. Lignin has low compatibility with most thermoplastics and tends to give poor mechanical properties for polymer composites. Various BF reinforced biocomposites have been developed to investigate surface morphologies, mechanical properties, thermal properties, and water absorptions. This study was performed to determine the effect of the SE followed by alkali treatment on the physicochemical, thermal, and morphological properties of the BF. In order to analyze the

Figure 1. Conditions of (a) bamboo, (b) raw-cut, (c) chipped, (d) fine chipped, (e) ground, (f) milled - untreated BF, (g) SE BF, and (h) SE-alkali treatment BF.

physicochemical and thermal properties of a modified surface, the energy dispersive X-ray (EDX) spectroscopy and thermogravimetric analysis (TGA) were used. The scanning electron microscopy (SEM) was utilized to examine the surface morphology of bamboo fibers before and after the treatment. In addition, the Fourier transform infrared (FTIR) spectroscopy was utilized to detect the presence of functional groups.

Experimental

Materials

Bamboo culms from a 3-year-old *Gigantochloa scortechinii* were obtained from the Forest Research Institute Malaysia (FRIM) in Kepong, Selangor, Malaysia. The raw bamboos were cut into pieces approximately 5 cm long. The samples were dried at room temperature for 3 days before being crushed by a string crusher machine (Guangdong Jiangmen Electric Machinery Co. Ltd.) and grinded by a ball mill machine (Hitachi) for 24 h.

Bamboo Fiber Treatment

Steam Explosion Treatment

BF was treated by SE at 200 g scale with a maximum pressure of 700 kPa, at a fixed high temperature of 160 °C, and a maximum time of 30 min using SE plant at FRIM laboratory. Then, the SE BF was dried at 60 °C for 24 h in the oven.

The Steam Explosion Followed by Alkali Treatment

The SE BF was soaked in 5 % sodium hydroxide at room temperature for 30 min [37]. The treated fibers were then thoroughly rinsed with distilled water until the pH of the fibers became neutral (pH 7). Then, the fibers were oven-dried at 103 °C for 24 h to eliminate moisture in the fibers completely [27]. Figure 1 shows all the conditions of all samples involved in this work. Thermal gravimetric analysis (TGA) and differential thermogravimetry (DTG) were used to evaluate the thermal degradation and the stability of the bamboo fibers. The TGA and DTG of untreated BF, SE BF, and SE-alkali treatment BF were conducted using a TA Instruments Q series thermal gravimetric analyzer (Model TGA Q500) to evaluate remaining mass fraction with temperature. The test was performed under a nitrogen atmosphere with a heating rate of 10 °C/min at room temperature (25 °C) up to 600 °C.

Morphological and Chemical Characterization

The surface morphologies of the untreated BF, SE BF, and SE-alkali treatment BF were examined using a SEM with a model VPSEM LEO 1450 with an INCA 2000 OXFORD EDX. The samples for SEM/EDX was then coated with a thin layer of gold to avoid electrostatic charge [38]. The EDX spectroscopy is an important technique for elemental analysis and dependent on the significant interaction of the sample and the source of X-ray excitation [39]. In this study, the EDX spectroscopy and SEM were combined to observe and investigate the surface morphology and perform element analysis of the untreated BF, SE BF and SE-alkali treatment BF.

FTIR Analysis

FTIR spectroscopy was utilized to study the effects of treatment on the chemical constitution of the BF. The FTIR spectrum of the untreated BF, SE BF, and SE-alkali treatment BF were recorded using a Nicolet 6700 Thermoscientific FTIR spectrometer.

Results and Discussion

Thermal Analysis

The TGA and DTG curves of the untreated BF, SE BF, and SE-alkali treatment BF are presented in Figures 2-4, respectively. Table 1 shows the values of the decomposition temperature and weight loss of the samples. The thermal analysis of bamboo fibers was marked within the temperature range of 25-600 °C. Figures 2-4 show the earlier weight loss occurred between 50 °C and 125 °C with temperature peaks

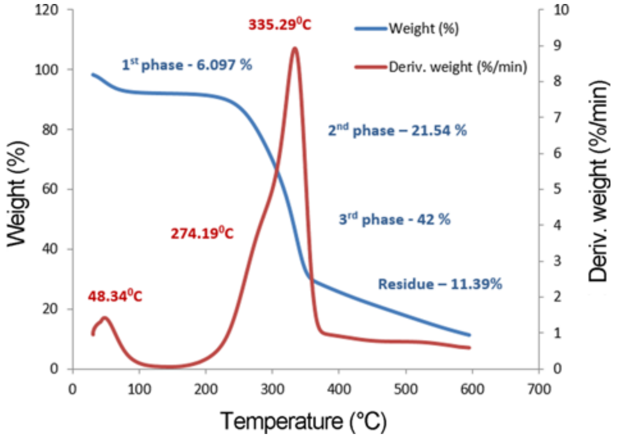

Figure 2. TG and DTG curves for untreated BF.

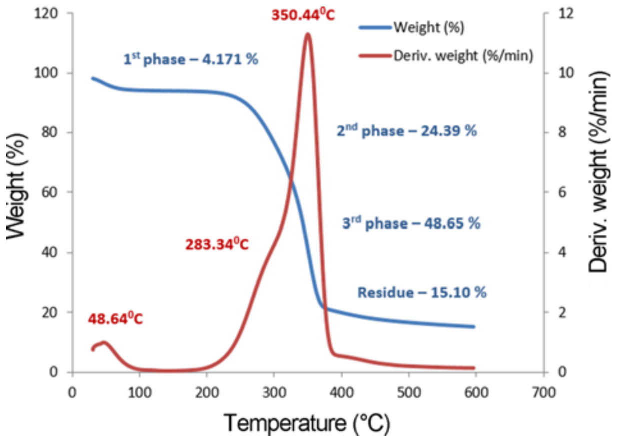

Figure 3. TG and DTG curves for SE BF.

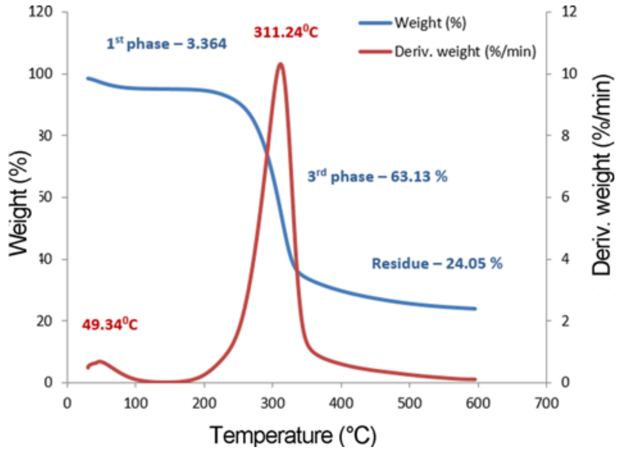

Figure 4. TG and DTG curves for SE-alkali treatment BF.

at 48.34 °C, 48.64 °C, and 49.34 °C in DTG curves because of the 6.097 %, 4.171 %, and 3.364 % weight loss of the untreated BF, SE BF, and SE-alkali treatment BF, respectively. The hemicellulose degradation was marked by the weight loss between 175 °C and 300 °C in Figures 2 and 3, where the peak degradations of the untreated BF and SE BF at 274.19 °C and 283.34 °C were 21.54 % and 24.39 %, respectively. In contrast, the SE-alkali-treatment BF did not show any peak in this phase because of the depletion of lignin and dissolution of hemicellulose during the treatment (Figure 4). Previous works [39] also showed similar results. The cellulose degradation starts after the complete degradation of hemicellulose [17]. Figures 2-4 show that the degradation temperature of cellulose ranged from 310 °C to 600 °C. A slight shift of the peaks at 335.29 °C, 350.44 °C, and 311.24 °C corresponding to 42.00 % (untreated BF), 48.65 % (SE BF), and 63.13 % (SE-alkali treatment BF), respectively, was clearly noted (Table 1). The untreated BF showed the lowest percentage (11.39 %) of ash content compared with the SE BF (15.10 %) and SE-alkali treatment BF (24.05 %). The SE-alkali treatment BF decreases the hemicellulose and lignin content and shows the lowest silica (Si) content. This study is in agreement with the findings of Kaushik *et al.* [16], who found that hemicellulose and lignin components in wheat straw decreased after the SE process because the steam had the lignocellulosic structure disintegration, partial hydrolysis of the hemicellulosic fraction, and depolymerization of the lignin. However, the ash residue of SE-alkali treatment BF shows the highest value compared to untreated BF and SE BF, which may occur due to the chemical components of the fiber caused by the process of SE-alkali treatment increasing the percentage of cellulose in the fiber.

In general, the thermal decomposition of natural fibers has three phases [39,40]. The first one is known as moisture evaporation, followed by hemicellulose and cellulose decomposition and decomposition of lignin, with ash as the residue. The result of bamboo fibers degradation occurred between 50 °C and 125 °C is almost in agreement with a study by Wang *et al.* [41], who showed that the first degradation occurs at approximately 100 °C. At this stage, the bound water and volatile extractives evaporated and inclined to move to the surface of the fiber [42]. The chemical treatment is one of the important factors that reduce the hydrophilic of bamboo fibers due to hemicellulose and lignin content [41]. SE-alkali treatment BF had the lowest percentage of weight loss, followed by SE BF and untreated BF (Table 1). This phenomenon could be explained by the highest moisture content observed in the untreated BF compared to the SE BF and SE-alkali treatment BF. The result of SE-alkali treatment BF is similar to works done by Rashid *et al.* [39], who observed sugar palm fibers treated with alkali treatment have no peak at the hemicellulose decomposition treatment (Figure 4). After the SE treatment, the fiber bundles decreased with increasing individual filament fiber [43].

The SE treatment had opened up the cellulose fiber, weakened the lignocellulosic structure, and increased the extractability of lignin during the subsequent kraft pulping process [44]. Hence, the SE treatment breaks up the fiber and liberates embedded impurities. At the same time, the size of the fibers is reduced up to 90 % after the treatment [16,17,45,46], resulting in improved mechanical properties of the composite with better dispersion in the matrix. The alkali treatment can eliminate a large amount of hemicellulose, lignin, and other impurities in the cellulosic fibers, resulting in a rougher surface of the fiber [47]. Apparently, the SE-alkali treatment could dissolve the hemicellulose completely, which was consistent with findings by Rashid *et al.* [39].

The cellulose degradation needs a high amount of energy compared to the hemicellulose degradation due to the higher crystalline chains in cellulose. Alkali treatment removes non-cellulosic material and retains the high degree of

Table 1. Decomposition temperatures and weight losses of untreated BF, SE BF, and SE-alkali treatment BF

BF treatment	1st phase		2nd phase		3rd phase		% Residue at 600 °C
	T_{peak} (°C)	Max weight loss (%)	T_{peak} (°C)	Max weight loss (%)	T_{peak} (°C)	Max weight loss (%)	
Untreated BF	48.34	6.097	274.19	21.54	335.29	42.00	11.39
SE BF	48.64	4.171	283.34	24.39	350.44	48.65	15.10
SE-alkali treatment BF	49.34	3.364	-	-	311.24	63.13	24.05

structural order, thus indirectly increased the degradation temperature [16]. For the bamboo fibers, the degradation temperatures of hemicellulose, cellulose, and lignin ranged from 175 °C to 600 °C, which was quite a huge range most likely due to the Si content in the bamboo fibers. In general, Si has high thermal stability due to its strong chemical bonds [48].

Thus, the SE treatment had released lignin from the cell wall towards the fiber surface [49]. Lignin has low compatibility with most thermoplastics and tends to give poor mechanical properties for polymer composites. The lignin from extract free-bamboo and SE pulp of bamboo started to thermally lose their weight at 130 °C and 110 °C, respectively [49].

The maximum value of thermal degradation of untreated BF, SE BF, and SE-alkali treatment BF are 335.29, 350.44, and 311.24 °C, respectively. It is shown that SE BF has the greatest thermal stability followed by untreated BF and SE-alkali treatment. This result can be explained by high thermal stability caused by the presence of Si in the outer surface. Through the SEM observation, the existence of Si on the external surface of SE BF and untreated BF is clearly visible and supported by EDX analysis that was conducted in this study.

Surface Morphology and Elemental Analysis

Figure 5 shows the SEM observations of the untreated BF, SE BF, and SE-alkali treatment BF. It was noted that the untreated BF in Figure 5a contains wax and impurities. In Figure 5b, however, shows that the SE treatment has broken down the fiber structure, but the wax and impurities could still be observed on the surface of the fiber [17]. The surface treatment of the lignocellulosic fiber could improve surface wettability by removing the wax and non-cellulose constituents and increase the tensile strength of the fiber [50].

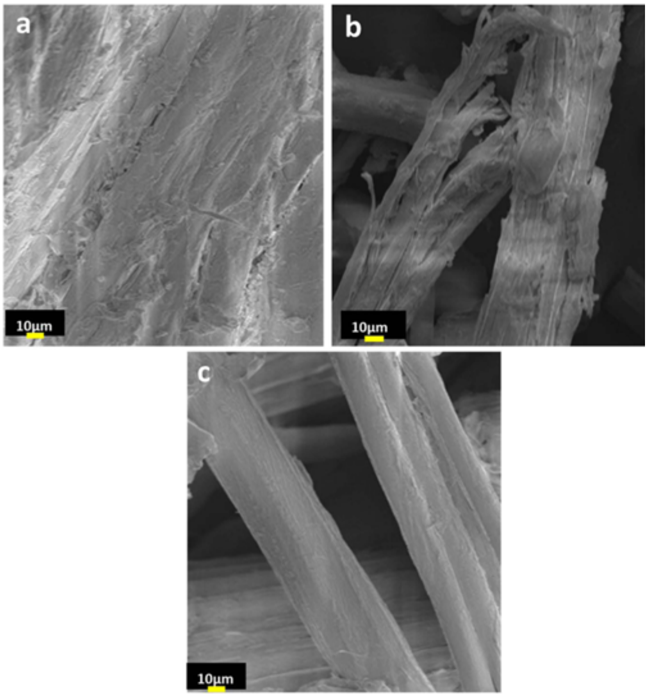

Figure 5. Scanning electron micrographs of (a) untreated BF, (b) SE BF, and (c) SE alkali treatment BF.

The compatibility between fiber and polymer matrix depends on the surface morphology, including their level of smoothness and roughness, and this can be observed from SEM images [19,51]. The removal of unwanted surface impurities in order to produce a cleaner and rougher surface would enhance the fiber and matrix adhesion, and this is attributed to the mechanical locking between the fiber surface and the matrix [19,52]. The possible explanation for the decrease in flexural strength of bamboo/epoxy composites is due to the smooth surface fiber, hence leads to a less efficient stress transfer at high loads where fiber failure starts to occur [53]. The number of anchorage points can be increased through rough surfaces, thus offering a good fiber-matrix mechanical interlocking [51]. The treated BF with benzoic acid was hardly pulled out of the PVC matrix with a little rough surfaces because of better interfacial adhesion [54]. Figure 5c shows that the surfaces became rougher and clean after the alkali treatment, suggesting a complete removal of wax and impurities. This unwanted wax protects the outer surface of the fiber from the environmental conditions and does not stick firmly onto the fiber [39]. BF reinforced semirigid polyvinyl chloride (PVC) composites study by our group (unpublished data) showed that the tensile strength for SE-alkali treatment BF reinforced semirigid PVC was higher than untreated BF reinforced semirigid PVC and semirigid PVC.

Hemicellulose is known as the most hydrophilic and thermally unstable substance [55], and lignin may migrate to the fiber's surface if heated above the glass transition temperature [56]. The presence of the impurities can be seen by SEM (Figure 5b and 5c) and approved by EDX (Table 2). The impurities like hemicellulose and lignin should be removed as they would affect the adhesion between the fiber and matrix in polymer composites. In this study, SE-alkali treatment had removed hemicellulose and lignin from the bamboo fibers successfully, as confirmed by the TGA results (Table 1). In short, the SEM results in Figure 5 show that SE and SE-alkali treatments modified the surface properties of the fiber compared with the untreated BF.

Figure 6 shows the corresponding EDX analysis of all types of bamboo fibers. It was noted that the main elements of BF structure were C, O, and Si. Figure 6a shows a high intensity of Si for untreated BF compared with the SE BF (Figure 6b) and SE-alkali-treated BF (Figure 6c). Table 2 indicates elements weight percentages of untreated BF, SE BF, and SE-alkali treatment BF as determined by EDX analysis. The untreated BF showed the highest Si content

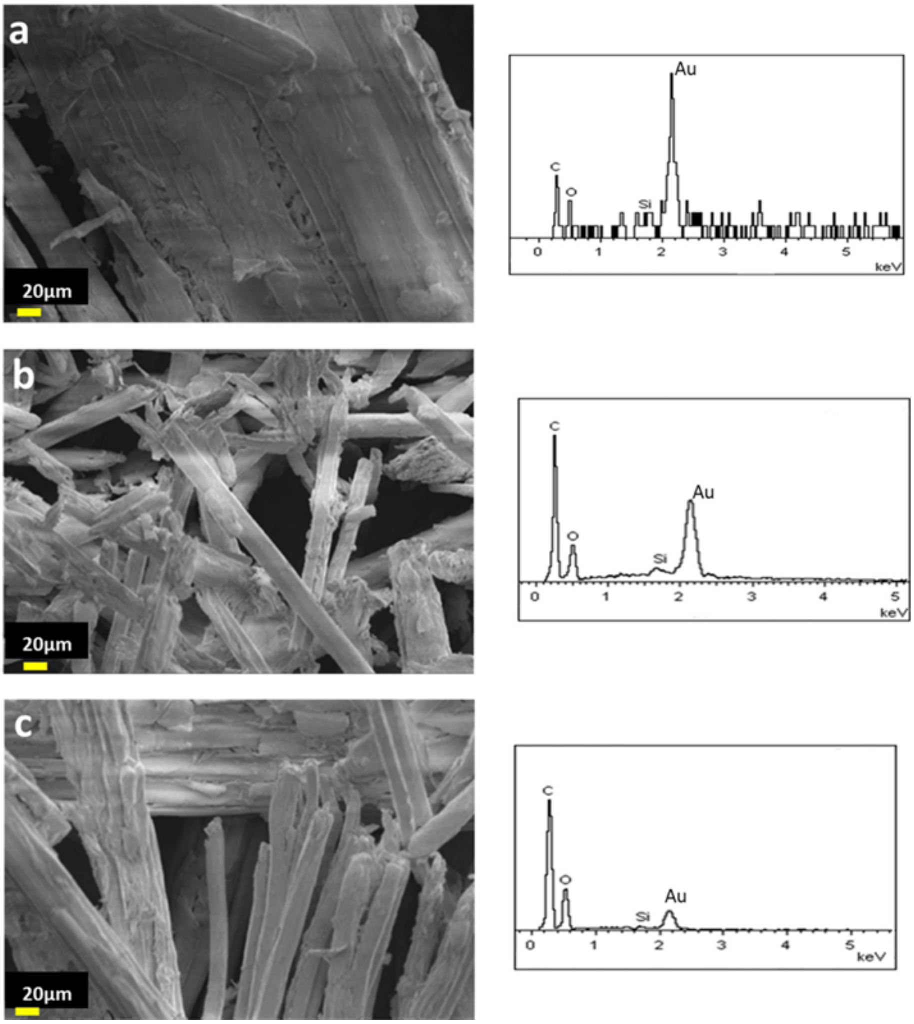

Figure 6. Scanning electron micrographs and energy dispersive X-ray (EDX) images of (a) untreated BF, (b) SE BF, and (c) SE-alkali treatment BF.

Table 2. EDX elemental analysis of untreated BF, SE BF, and SE-alkali treatment BF

Type of BF treatment	Weight percentage (%)			
	C	O	Si	Total
Untreated BF	89.91	3.96	6.14	100
SE BF	72.01	27.48	0.51	100
SE-alkali treatment BF	67.99	31.93	0.08	100

(6.14 %), whereas the silica content of the SE BF and SE-alkali treatment BF was observed to be reduced down to 0.51 % and 0.08 %, respectively.

The reduction of Si content also played an important role in addition to the removal of wax and impurities in order to enhance the fiber and matrix adhesion in polymer composites. The removal of hemicellulose and Si also increased the surface roughness [55]. Polymer composites with low Si content have high resistance to impact force, because of high silica and carbon content increase brittleness, which reduces absorbed impact energy [57]. The depletion of Si content would also affect the ultimate degradation of the bamboo fibers. This phenomenon caused the lower degradation temperature of the fibers in the SE-alkali treatment BF, as discussed in the TGA results.

Fourier Transforms Infrared (FTIR) Profile

Figure 7 illustrates the FTIR spectra of untreated BF, SE BF, and SE-alkali treatment BF. Table 3 displays the characteristic bands of the FTIR spectra for the untreated BF, SE BF, and SE-alkali treatment BF.

The spectrum showed broadband within the range of 2990-3700 cm^{-1}, which was caused by the -OH group in the fibers [41,58]. For the untreated BF, the peak at 3347 cm^{-1} corresponded to the stretching of the hydroxyl group because of the vibration of the hydrogen bond between

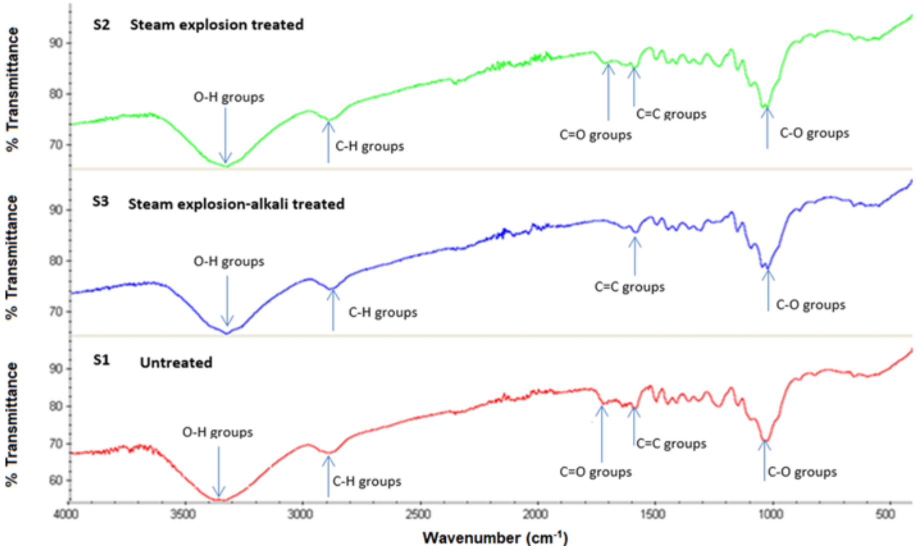

Figure 7. FTIR spectra of (S1) untreated BF, (S2) SE BF, and (S3) SE-alkali treatment BF.

Table 3. Characteristic bands of the FTIR spectrum for the untreated BF, SE BF, and SE-alkali treatment BF

Probable group assignment	Wavenumber (cm^{-1})		
	Untreated BF	SE BF	SE-alkali treatment BF
O-H stretching vibration in cellulose, hemicellulose and lignin	3347.0	3332.3	3337.5
C-H stretching in methyl and methylene groups	2895.2	2901.4	2897.9
C=O stretching vibration of hemicellulose and pectin	1733.5	1724.9	-
C=C stretching of aromatic rings of lignin	1602.7	1602.8	1591.9
C-O stretching of C-O in removing hemicellulose and lignin	1035.3	1033.9	1033.2

molecules [58]. After the BF surface treatment, the peaks shifted to 3332.3 cm^{-1} and 3337.5 cm^{-1} for the SE BF and SE-alkali treatment BF, respectively. This finding proved that there are the water molecules in the bamboo fibers, which is supported by other studies that were related to the -OH groups present in natural fibers [39,59]. The peaks within the range of 2920-2915 cm^{-1} were attributed to C-H, which is mainly present in cellulose and hemicellulose (lignin polysaccharides) [39,58]. The peaks at 2895.2 cm^{-1} (untreated BF), 2901.4 cm^{-1} (SE BF), and 2897.9 cm^{-1} (SE-alkali treatment BF) indicated the C-H stretching vibration from CH and CH_2 [60]. The peaks slightly shifted because of the SE and SE-alkali treatments. The peak of the SE-alkali-treatment BF showed lower intensity compared with those of the other fibers indicating the removal of non-cellulosic content. The peaks at 1602.7 cm^{-1}, 1602.8 cm^{-1}, and 1591.9 cm^{-1} in the untreated BF, SE BF, and SE-alkali treatment BF represented the aromatic C=C stretch of the aromatic rings of lignin [39,61]. The intensity of the SE-alkali treatment BF was depleted, which indirectly indicates the partial removal of lignin. The strong peak at 1733.5 cm^{-1} was attributed to the stretching of C=O in the carboxylic group of the untreated BF. For the SE BF, the peak shifted to 1724.9 cm^{-1}, indicating the removal of hemicellulose, lignin, and impurities. Disappearnce of the C=O peak for the SE-alkali treatment BF indicated that large amounts of impurities (hemicelluloses and lignin) were eliminated during SE-alkali treatment. The peaks at 1035.3 cm^{-1} (untreated BF), 1033.9 cm^{-1} (SE BF), and 1033.2 cm^{-1} (SE-alkali treated BF) showed the characteristics of the C-O group from the cellulose in the fiber [61].

Conclusion

This study was carried out to investigate the physico-chemical and thermal properties of untreated BF, SE BF, and SE-alkali treatment BF. Thermal analysis showed that the alkali treatment after the SE process decreased the thermal characteristics of bamboo fibers. The SE BF demonstrated the highest thermal decomposition compared with untreated BF and SE-alkali treatment BF. The EDX results showed that the SE-alkali treatment BF had lower percentages of Si than untreated BF and SE BF. The hydrolysis of the hemicelluloses and lignin was confirmed by FTIR, where the peak of the SE-alkali treatment BF showed lower intensity compared with untreated BF and SE BF indicating the removal of non-cellulosic content. The SEM analysis indicates that SE-alkali treatment BF had clean and rougher surfaces compared to untreated BF and SE BF in which wax and impurities were clearly observed on the surface of the fiber. In conclusion, stronger fiber can be produced with a high percentage of cellulose by reducing lignin, hemicellulose, and Si after SE-alkali treatment.

Acknowledgments

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