Effects of Different Pretreatments Combined with Steam Explosion on the Properties of Bamboo Fibers

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Bamboo pretreatment is a key technology for the preparation of bamboo fibers (BFs) for composites. This study examined the properties of BFs prepared by steam explosion (SE) BFs following pretreatment by enzyme, alkali, and salt. The microstructure, functional groups, crystallinity, and surface chemical elements of BFs were characterized by environmental scanning electron microscope (ESEM), Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), and X-ray photoelectron spectroscopy (XPS). The results indicated that bamboo could be separated into fiber bundles through SE after pretreatment. The separation of BFs pretreated by enzyme and alkali were better, but colloid remained and was able to stick the fibers together. Through performing different pretreatments before SE, the lignin and hemicellulose of BFs were partially removed, and alkali pretreatment had the best effect on lignin removal. The crystal structure of the BFs did not change significantly, and the crystallinity of BFs was highest at 2 MPa and 6 min when pretreated by alkali. The XPS results showed that the effect of alkali pretreatment at 2 MPa for 6 min was the best.

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Keywords: Bamboo fibers (BFs); Pretreatment; Steam explosion (SE); Microstructure; X-ray photoelectron spectroscopy (XPS)

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INTRODUCTION

Fiber-reinforced polymer composites often use synthetic fiber such as aramid, glass, or carbon, owing to their strength and availability. Composites are often used in many applications ranging from the aerospace industry to civil engineering and building applications (Manalo *et al*. 2015; Huang and Young 2018). As synthetic fibers are non-degradable, emit poisonous gases to the air when burned, and are expensive, natural fibers such as kenaf, flax, hemp, and sisal have gained attention as replacements for synthetic fibers in composites (Soutis 2005; Faruk *et al*. 2012; Sanjay *et al*. 2015). They possess fairly low density, renewability, biodegradability, low cost, low energy requirements, abundant availability, high strength, and high elastic modulus (Joshi *et al.* 2004; Li *et al.* 2007). Among many natural fibers, bamboo fiber (BF) has drawn particular attention due to its low density, high stiffness, high strength, and the rapid growth of bamboo, which makes them abundantly available. Particularly, BFs have been used for industrial applications as diverse as textiles, paper, and construction. Bamboo has long been regarded

as a composite material comprising reinforcing component fibers and a matrix comprised of parenchyma cells. In general, bamboo culm consists of about 50% parenchyma cells, 40% fibers, and 10% vascular bundles (vessels, sieve tubes with companion cells). BF is distributed in the internodes of vascular bundles as fiber caps or sheaths surrounding the conducting elements (including vessels, sieve tubes, and companion cells). Bamboo is a highly studied plant, especially in investigations on manufacturing fibers for textiles, fiber-reinforced composite materials, and other applications of the fiber. The major constituents of bamboo biomass are cellulose (45 to 55%), hemicellulose (15 to 25%), lignin (15 to 30%), pectin (0.5 to 1.5%), and other organic and inorganic compounds (Liu *et al.* 2011; Nayak and Mishra 2016; Tolessa *et al.* 2017; Rocky and Thompson 2018b). BFs exist in the bamboo structure in the form of bundles that are bonded (by multiple roots vertically and horizontally) to each other longitudinally and laterally and surrounded by basic tissue (Ray *et al*. 2005).

To make full use of the superior properties of BFs, a separation and extraction process is required to remove the basic tissues from bamboo and obtain bamboo fiber. The bamboo fiber cannot be obtained directly from the bamboo culm and several treatments are needed before the bamboo fiber can be obtained. The BFs can be prepared by physical methods (Okubo *et al*. 2004), chemical methods (Wang *et al*. 2007; Wang *et al*. 2017; Huang *et al*. 2019), biological methods (Zhang *et al*. 2009), and combined technology. The quality and strength of the fibers is directly affected by the separation method used. The physical method is simple, but the prepared fiber is coarse and hard. Chemical methods have high efficiency, but they are costly and polluting. Biological methods have very high requirements for process environment and are generally used for the refinement of coarse BFs (Zhao *et al*. 2021). Combined technology combines two or three preparation processes to produce BFs. It is useful to apply more than one treatment method in different stages of BF extraction to gain the utmost benefits. Since the complex structure of bamboo and its high lignin content hinder fiber extraction in a timely manner, it is difficult to extract fibers using only one technology. Thus, combined technology is more common and it accelerates the fiber production. For example, a combination of pretreatment or post-treatment of bamboo strips using chemicals or enzymes and steam explosion (SE) is one type of combined treatment that yields fibers (Fu *et al.* 2012). Pretreatment is a prerequisite processing step and it is also the most important and costly operation unit. Its options include physical pretreatment, chemical pretreatment, physicochemical pretreatment, and biological pretreatment (Chen and Liu 2015). Among them, the chemical method is usually used as a pretreatment for the preparation of BFs. Some types of enzymes, such as hemicellulase, cellulase, pectinase, and xylanase have been reported as effective to attack bamboo structure and dissolve binding materials (*e.g.* lignin, pectin) of the fibers (Rocky and Thompson 2018a). Enzyme treatment reduces the energy consumption and pollution of the fiber separation process (Wu *et al*. 2015). Salt pretreatment removes part of the lignin and hemicellulose from bamboo. Alkali pretreatment can effectively delignify cellulose, chemically expand cellulose, allow enzymatic saccharification of bamboo, and cut off chemical connection between hemicellulose and lignin, removing most of lignin and hemicellulose (Zhao *et al*. 2009; Yamashita *et al*. 2010; Wen *et al*. 2011; Sun *et al*. 2014; Kassaye *et al*. 2017; Zhang *et al*. 2018). It improves the interfacial bonding between bamboo fiber and matrix materials for composite preparations (Huang *et al*. 2012).

SE uses high temperature and pressure steam to treat raw materials, resulting in component separation and structural changes through instantaneous pressure relief

(Chornet and Overend 1988). SE is an effective low cost and environmentally friendly method (Sheng *et al.* 2014). It is a highly efficient separation technology for wood fiber, and the process has undergone rapid development in recent years. SE, as a physical and chemical method, effectively separates the chemical components of wood fiber with no or few chemicals. During SE, saturated steam penetrates the fibers. When the steam pressure is released suddenly, hemicellulose is hydrolyzed, and hemicellulose-lignin bonds are also cleaved, which increases the solubility of lignin in alkaline or organic solvents (Li *et al*. 2007).

In this paper, BFs were prepared by SE after enzyme pretreatment, alkali pretreatment, and salt pretreatment. The effects of different pretreatments on the properties of BFs were investigated. Alkali pretreatment was selected to explore the influence of different pressure holding times of SE on BFs properties. The microstructure, functional groups, crystallinity, and surface chemical elements of the BFs were characterized by scanning electron microscope (SEM), Fourier-transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), and X-ray photoelectron spectroscopy (XPS).

EXPERIMENTAL

Materials

Five-year-old moso bamboo (*Phyllostachys pubescens*) was obtained from Zhejiang, China. The bamboo culm was cut and air-dried to moisture content of 8 to 12%. The bamboo slivers were obtained with a length of 15 cm and width of 2 to 3 cm. The sodium hydroxide (NaOH) and sodium sulfite (Na_2SO_3) were analytically pure and supplied by Chengdu Cologne Chemical Co., Ltd. The hemicellulase, pectinase, and xylanase were supplied by Shanghai Maclin Biochemical Technology Co., Ltd.

Preparation of Bamboo Fibers

The bamboo slivers were put into the enzyme solution, alkali solution, and salt solution for pretreatment. Enzyme pretreatment: The bamboo slivers were immersed in enzyme solution (hemicellulose: pectinase: xylanase=2:1:1) with a concentration of 0.6% at room temperature for 1 day. Alkali pretreatment: The bamboo slivers were immersed in NaOH and Na_2SO_3 solution (NaOH: Na_2SO_3=1:1) with a concentration of 5% at 100 ℃ for 4 h. Salt pretreatment: The bamboo slivers were immersed in Na_2SO_3 solution with a concentration of 5 % at 100 ℃ for 4 h. The pretreated bamboo slivers were rolled and divided into filaments.

One-hundred grams of bamboo slivers were weighed and put into the SE device after cleaning. The explosion pressure was set to 2 MPa for a duration of 4 min, and then the heating device was powered on. The saturated steam produced was fed continuously into the explosion device to reach the preset experimental pressure, the valve was closed, and the timer was started. After 4 min, the materials in the explosion chamber were sprayed into the receiving device; the BFs were collected, cleaned, and dried. On this basis, the alkali pretreatment was selected to explore the preparations of BFs with different pressure holding times. The explosion pressure was set as 2 MPa, and the pressure holding times were set as 4 min, 6 min, and 8 min. After SE, the BFs were collected, cleaned, and dried. The process is shown in Fig. 1.

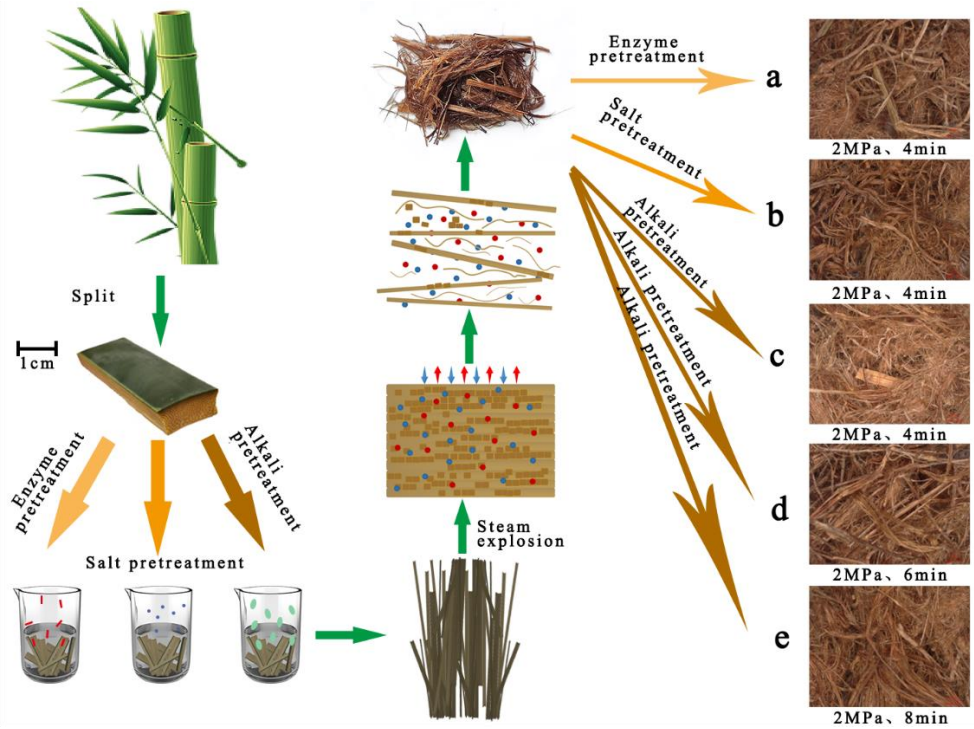

Fig. 1. Flow chart for BFs prepared by SE after different pretreatments

Surface Morphology and Microscopy

Length and diameter of BFs were measured with vernier calipers and a digital microscope (VHX-7000, China), respectively. Thirty replicates were measured for each sample. The morphology and surface of the BFs were observed with a digital microscope (VHX-7000, China) and a field emission scanning electron microscope (SEM, Quanta 200, FEI, Hillsboro, OR, USA).

Chemical Composition Test

The surface chemistry of the fibers was revealed by KBr-Fourier transform infrared (FTIR) and X-ray photoelectron spectroscopy (XPS) analyses. KBR-FTIR analysis was run on a spectrometer (VERTEX 80 V, Bruker, Karlsruhe, Germany). About 1 mg of BFs was milled to small particles and then mixed with KBr and pressed into a small disc about 0.5 mm thick. The FT-IR spectra were recorded over a spectral range of 4000 to 500 cm^{-1} at a resolution of 4 cm^{-1}, with a total of 64 scans for each sample. Three replicated were measured for each sample. XPS experiments were performed using an XPS device (AXIS UltraDLD, Shimadzu, UK) with Al Kα radiation source. The C1s spectra and O1s spectra were analyzed using the XPS Peak software.

The crystallinity of different pretreatments of BFs was measured with an X-ray diffractometer with a CuK radiation source (XRD, Ultima IV, Rigaku, Tokyo, Japan). The crystallinity index (Crl) was the relative crystallinity, I_{200} was the maximum intensity of the (200) diffraction peak, and I_{am} was the amorphous diffraction intensity. The crystallinity index was calculated by Formula 1:

$$Crl=[(I_{200} - I_{am})/I_{200}] \times 100\% \quad (1)$$

RESULTS AND DISCUSSION

Surface and Microscopic Morphology

Table 1 and Fig. 2 show the size and surface morphology of BFs prepared by SE after different pretreatments. BFs pretreated by alkali had more uniform distribution compared with BFs pretreated by salt and enzyme. With the increase of SE holding time, part of fiber bundles was separated into single fibers. The average lengths of a and b were small, while those of c, d, and e were large. The average diameters and diameter variabilities of c, d, and e was small, the average diameters of a and b were large, which indicated that alkali pretreatment could increase the length of BFs and decrease the diameter of BFs compared with enzyme pretreatment and salt pretreatment. The general trend was that the fineness decreases with the increase of length. This indicates that the higher the average length of the fibers, less delignification occurred (Rocky and Thompson 2018a). The average length of c was largest, and the average diameter of c was smallest, which resulted in higher dispersion in the composite. Thus, the mechanical properties of composites could be improved, indicating that the treatment effect of c is best.

Table 1. Sizes of BFs Prepared by SE after Pretreatment

Sample	Length (mm)		Diameter (mm)		Aspect Ratio
	Average	Range	Average	Range	
a	48.81 (13.43)	23.42-69.33	0.33 (0.10)	0.14-0.55	148
b	49.65 (11.92)	25.99-63.02	0.45 (0.19)	0.14-0.87	110
c	60.26 (13.23)	29.01-81.19	0.25 (0.12)	0.09-0.52	241
d	64.93 (11.16)	45.87-81.18	0.24 (0.09)	0.12-0.44	232
e	58.85 (12.66)	22.36-80.06	0.28 (0.12)	0.13-0.55	210

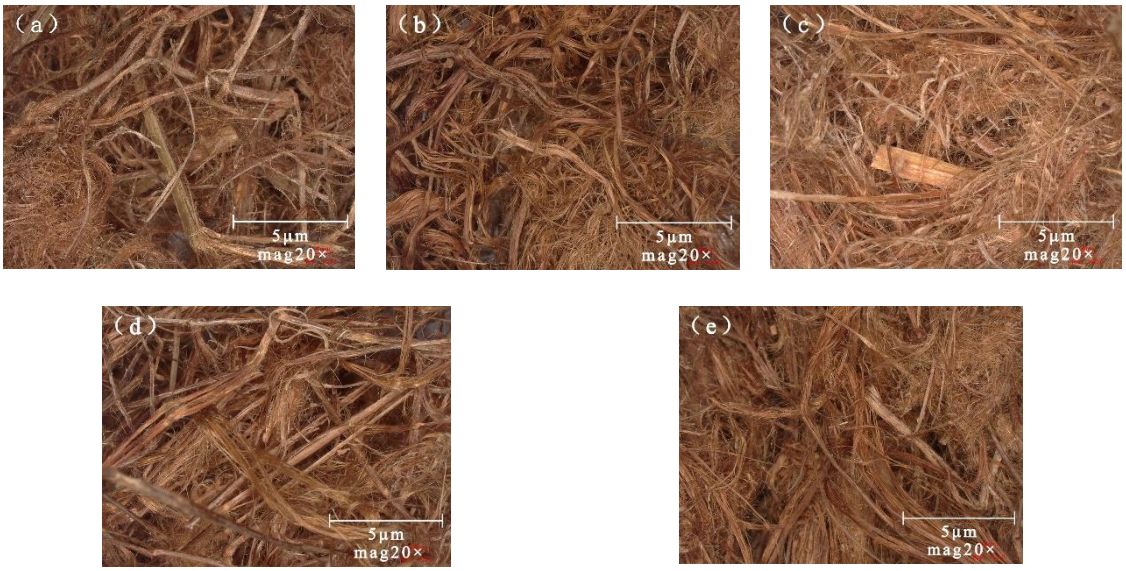

Fig. 2. Morphology of BFs prepared by SE after different pretreatments

Observations of SEM pictures in Fig. 3 showed that the fibers in the separation methods were longitudinally aligned, and they were bonded together by lignin and hemicellulose (Phong *et al*. 2012). Through the SE process, the bamboo slivers were

exposed to a highly wet state with high temperature and high steam pressure. The steam could infiltrate the cell walls and pores between fibers. Under the combined effect of the steam and heat, the fiber began to soften and change the chemical structure. Low molecular weight substances started to dissolve out from the fibers, and the connection between fibers began to weaken (Luo *et al*. 2014). The cell wall of BFs produced by pretreatment combined with SE was damaged to varying degrees, and the fiber bundles were separated and became loose from each other. The fiber surface was torn with many fragments and cracks. The surface of BFs pretreated by enzyme was relatively uneven (Fig. 2a), the dispersion was high and with a large amount of granular material adhering to the surface. The dispersion of BFs pretreated by salt was low, and granular material appeared on the surface of BFs (Fig. 2b). These granular materials may have been fragments of the fibrous cell wall. Under the action of high temperature and high pressure, the cellulose, hemicellulose, and lignin structure were destroyed. The instantaneous pressure relief gave rise to the accumulation of dissolved hemicellulose degradant products and lignin on the fiber surface (Luo *et al*. 2014). The surface of BFs pretreated by alkali had been stripped (Fig. 2c-e). When the holding time was 4 min, BFs were still bonded together in bundles, indicating that SE treatment had removed part of the colloid between BFs, but there was still a certain amount of colloid to stick the fibers together, keeping the material in the shape of fiber bundles. With increasing pressure holding time, most lignin content was extracted outside the fiber bundle, and it became significantly condensed on fiber surfaces under the high temperature and pressure (Phong *et al*. 2012), the dispersion degree became higher. The alkali pretreatment was more effective for BFs extraction than enzyme pretreatment and salt pretreatment, and the dispersion uniformity of BFs was significantly improved when the holding time was increased.

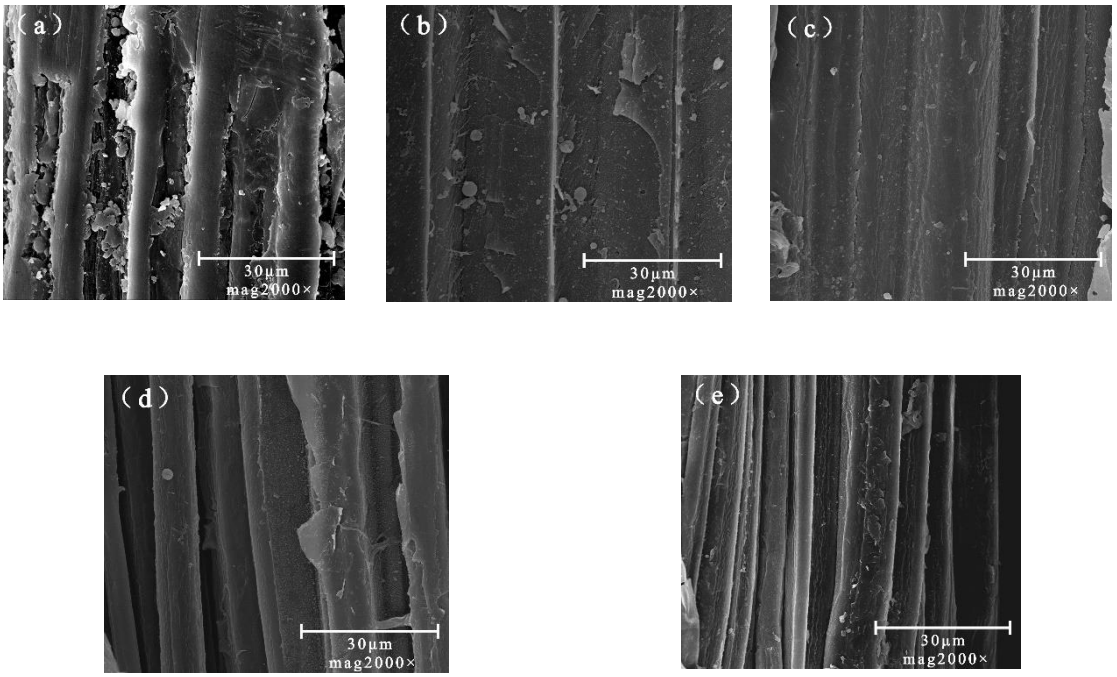

Fig. 3. The microstructure of BFs prepared by SE after different pretreatments

Chemical Compositions

Figure 4(a) shows the FT-IR spectra of BFs resulting from different pretreatments combined with SE. There was an evident band at 1730 cm^{-1} in the BFs pretreated by enzyme and salt, which was attributed to the carboxyl groups and ester groups in hemicellulose (Zhang *et al*. 2016). The band at 1730 cm^{-1} disappeared or its intensity decreased in the BFs pretreated by alkali, indicating that more hemicellulose and lignin were removed compared to enzyme pretreatment and salt pretreatment. The decrease in the intensity of the bands at 1509 cm^{-1} and 1460 cm^{-1}, which were the characteristic band of lignin, confirmed the partial removal of lignin in the BFs pretreated by alkali with the pressure holding times of 4 min and 6 min, indicating that alkali pretreatment had a good removal effect on lignin. The decrease in the intensity of the bands at 1080 cm^{-1}, which were the characteristic band of cellulose, indicating that part of cellulose had been degraded during SE when the SE pressure holding time was 4 min and 6 min. With the increase of pressure holding time, the peak showed a trend of weakening first and then strengthening. The alkali pretreatment was relatively effective for the removal of lignin and hemicellulose, and the best removal was achieved at 2 MPa and 6 min.

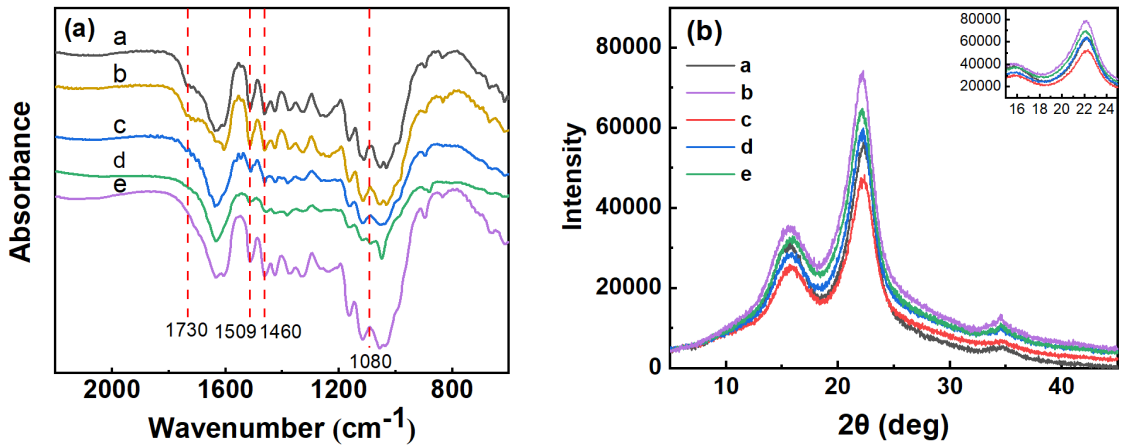

Fig. 4. FT-IR spectra (a) and XRD patterns (b) of BFs prepared by SE after different pretreatments

The XRD patterns of BFs prepared by different pretreatments are shown in Fig. 4(b). The distinct peaks at 15.9°, 21.8°, and 34.7° corresponded to (1-10) and (110), (200) and (040) lattice planes, respectively (Yang *et al*. 2021), which is a characteristic for the typical cellulose I (French 2014). It is obvious that the crystal form of BFs cellulose prepared after different pretreatments had not changed, and it was still typical cellulose I. The peaks at 2θ of 18° and 22° in the XRD patterns of the BFs prepared by different pretreatment were ascribed to (110) and (200) reflection of the crystalline structure of typical cellulose I (22). The pretreatment of bamboo removed some components such as hemicellulose and lignin so that the fiber structure was arranged orderly; hence, the crystallinity was increased.

As shown in Fig. 5, in the same SE process, the crystallinity of BFs pretreated by three methods did not differ, and the crystallinity of salt pretreatment was slightly higher than the alkali and enzyme pretreatments (Fig. 5a-c). In the same pretreatment process, the pressure holding time would affect the steam penetration strength, the degradation of hemicellulose in raw materials, and the removal of lignin. With increasing pressure holding

time, the crystallinity of BFs increased first and then decreased, indicating that cellulose was degraded after 6 min, so the crystallinity of BFs decreased. The crystallinity of BFs was the highest at 2 MPa and 6 min.

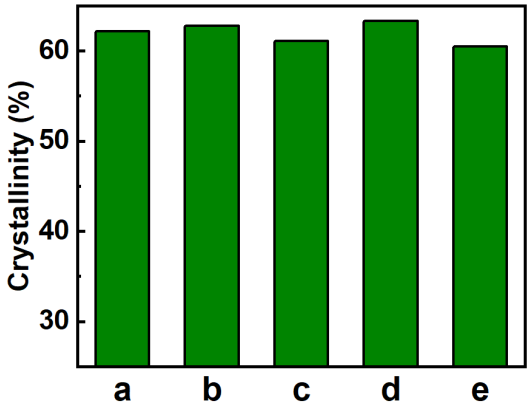

Fig. 5. Crystallinity of BFs prepared by SE after different pretreatments

Surface Chemical Properties

The chemical elements of the surface of BFs prepared by different pretreatments were studied by XPS. The XPS spectra revealed that carbon and oxygen, the peaks of which occur at about 284 and 532 eV, respectively, were the major elements in the sample.

To evaluate the surface chemical elements of BFs prepared by different pretreatments, the C1s XPS spectra of the samples were processed, and the results are shown in Fig. 6. The C1s XPS spectra correspond to four types of carbon atoms are expressed as C1 to C4. The C1 peak corresponds to groups with C-C or C-H, mainly originating from lignin and extractives. The C2 peak corresponds to groups with C-O, has been shown to be mainly derived from cellulose and hemicellulose. The C3 peak corresponds to groups with C=O or O-C-O, and mainly originates from cellulose and hemicellulose. The C4 peak corresponds to groups with O-C=O, is primarily from hemicellulose and extractives (Johansson *et al*. 1999, 2004; Laine *et al*. 1994).

As shown in Fig. 6 and Table 2, C1 and C2 components were the major constituents of carbons on the surfaces of BFs. The constituents of BFs prepared by different pretreatments also varied. The C1 components of BFs pretreated by enzyme were the lowest, indicating that enzyme pretreatment had removed more lignin. The C2 components of BFs pretreated by alkali and salt were low, indicating that alkali and salt pretreatments had partially removed cellulose and hemicellulose. The C3 components of BFs pretreated by alkali and salt were low, which indicated that the content of cellulose and hemicellulose on the surface of BFs was low. The content of C4 components did not show much difference between different pretreatments and SE holding time. The distributed material on the surface of BFs was mainly comprised of lignin and various extractives, and the distribution of cellulose and hemicellulose was significantly lower than that of lignin and extractives. This may be because in the process of preparation of BFs, bamboo internal cellular elements destruction and separation was mainly in the intercellular layer as well as between the primary wall and secondary wall, and the main chemical components of the intercellular layer and the primary wall of the cell were lignin and hemicellulose. Accordingly, the surface of BFs was characterized by a high content of lignin and

extractives. The C1s of BFs pretreated by alkali of different pressure holding times were not significantly changed, indicating that the increase of pressure holding time had little effect on the surface chemical composition of BFs.

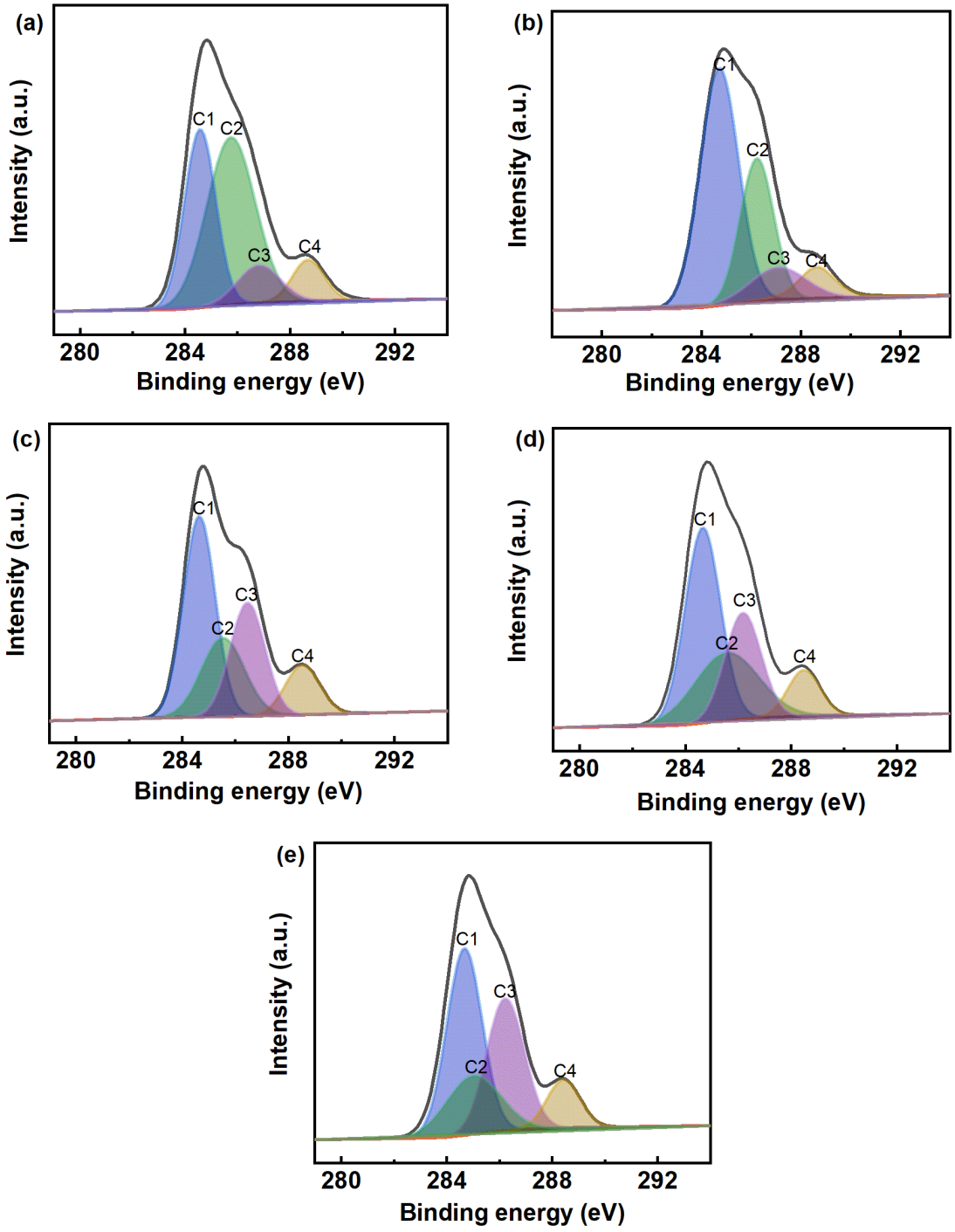

Fig. 6. C1s map of BFs prepared by SE after different pretreatments

To evaluate the surface chemical elements of BFs prepared by different pretreatments, the O1s XPS spectra of the samples were processed, and the results are shown in Fig. 7.

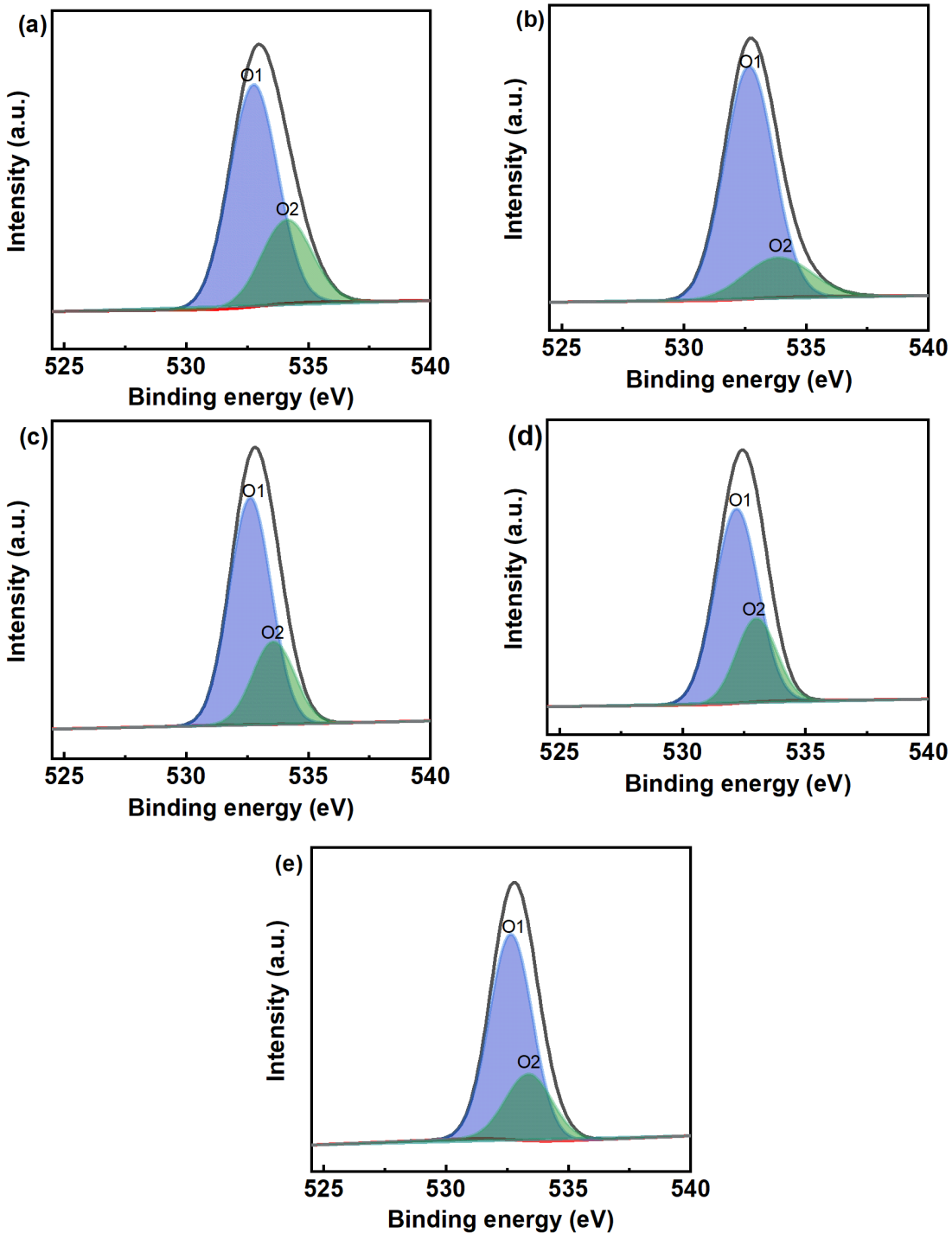

Fig. 7. O1s map of BFs prepared by SE after different pretreatments

The O1s spectrum presents two oxygen components. The O1 peak originates from O-C=O, and this component has been proposed to be associated with lignin and extractives.

The O2 peak originates from C-O and is associated with hemicelluloses and cellulose. The O1 component of BFs pretreated by alkali and salt increased compared with BFs pretreated by enzyme, whereas the O2 component decreased. This indicated that alkali and salt pretreatments decreased the carbohydrate content and increased the lignin and extractives content of BFs surfaces. With the pressure holding time increasing, the O1 component of BFs pretreated by alkali decreased first and then increased. The O2 component behaved conversely, which indicated that the surface of c become poorer in lignin and richer in cellulose and hemicellulose.

Table. 2. Analysis of XPS Peak Areas of BFs Prepared by SE after Pretreatment

Sample	C1(%)	C2(%)	C3(%)	C4(%)	O1(%)	O2(%)
a	33.55	48.71	9.76	7.98	71.74	28.26
b	41.42	21.84	25.53	11.21	73.22	26.78
c	41.82	26	23.23	8.95	71.66	28.34
d	40.3	19.2	30.59	9.92	74.03	25.97
e	53.54	27.96	10.55	7.96	81.06	18.94

CONCLUSIONS

1. The bamboo fibers (BFs) surface prepared by different pretreatments combined with steam explosion (SE) had been damaged to varying degrees. The surfaces of BFs pretreated by enzyme were relatively uneven. The alkali pretreatment was more effective for BFs extraction than enzyme pretreatment and salt pretreatment, and the dispersion uniformity of BFs was significantly improved when the holding time was increased.
2. The lignin and hemicellulose components of BFs were partially removed, and alkali pretreatment was the most effective for lignin removal. With increasing pressure holding time, the crystallinity of BFs pretreated by alkali presented a downward trend after rising first.
3. At 2 MPa and 6 min, the crystallinity of BFs was the highest. C1 and C2 components were the major constituents of carbons on the surfaces of BFs. The C2 components of BFs pretreated by alkali were lowest, indicating that alkali pretreatment had removed more cellulose and hemicellulose. The increase of pressure holding time had little effect on the surface chemical composition of BFs. The surface of BFs pretreated by alkali and then subjected to SE at 2 MPa and 6 min become poorer in lignin and richer in cellulose and hemicellulose. It was preliminarily found that the effect of alkali pretreatment, 2 MPa, 6 min was the best.

ACKNOWLEDGMENTS

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