

Effects of heat treatment on the properties of bamboo scrimber

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Abstract This study aims to investigate physical characteristic, mechanical properties, and chemical composition of heat-treated bamboo scrimber. Specimens were heated at 50–230 °C in laboratory conditions for 2 h. Test results of heat treatment samples were compared with the controls. Moisture absorption decreased slightly and then increased as temperature increased. It was probably due to changes of crystallinity and chemical structure. Mechanical properties varied greatly according to different temperature levels. Failure types reflected treatment temperature to some extent. Compressive strength reached a maximum when fiber bundles fractured neatly at 170 °C, which is a turning point for physical, mechanical, and chemical properties under this heat treatment condition. Increasing mechanical properties of bamboo scrimber after heat treatment was due to solidification of phenolic resin.

Keywords Bamboo scrimber · Heat treatment · Failure types · Mechanical properties · FTIR

Introduction

As a sustainable and environmentally natural material, bamboo resource is rich in China with fast growth and renewable characteristics. Due to its hollow culm, application of bamboo in construction is limited, although it has

high strength-to-weight ratio [1]. To improve it in building, some bamboo-based products, such as bamboo plywood, laminated bamboo lumber, bamboo particle board, and bamboo fiber reinforcing polymer composite, are produced [2]. Bamboo scrimber, a kind of bamboo products, is bundles of bamboo fiber with adhesive arranged in parallel under a press. The thought of bamboo scrimber is based on reconsolidated wood proposed by Coleman, bonding numerous wood splinters together [3, 4]. Compared with wood, mechanical strengths of bamboo scrimber can reach 6.36 times of *Pinus massoniana* and 11.03 times of *Cunninghamia Lanceolata* [5] that meet strength requirements of engineering material.

China developed bamboo scrimber successfully in 1990s. Raw material used for manufacturing bamboo scrimber at first was residues of bamboo and bamboo products. With increasing demand for bamboo scrimber, the products produced by residues had been unable to meet the demand of consumers, and then manufacturers used bamboo strips as raw material [6]. The fluffing method of bamboo fiber bundles was mainly manual operation in the beginning, and was replaced by mechanical fluffing later, improving production efficiency greatly [7]. In early stage of bamboo scrimber production, inner part of bamboo was removed, because interface bonding is weak between the outer part and inner part [8]. In 1990s, mechanical fluffing technology of including whole part of bamboo was developed successfully, which increased bamboo utilization up to more than 90 % [9]. Mature manufacturing technology and high strength of bamboo scrimber [5] make it a new engineering material. Now, research on mechanical properties of bamboo scrimber has been studied [2, 10], and however, its thermostability has not been reported.

Heat treatment is used to improve performance of wood and bamboo products [11, 12]. After heat treatment, wood

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becomes rather hydrophobic. The probable explanation of wettability change of wood after heat treatment may be plasticisation of lignin which leads to a reorganization of lignocellulosic polymeric components [13]. For softwoods, heat treatment protected wood surface to become rougher. Compressive strength and modulus of rupture (MOR) of samples decreased, while modulus of elastic (MOE) increased during heat treatment [14]. Bonding strength of PF-bonded laminated bamboo board glued skin-to-skin decreased after oil heat treatment, while the bonding strength of pith-to-pith bonded boards showed no significant difference before and after heat treatment [12]. Study of heat treatment on bamboo has been reported [15], and this area of bamboo scrimber needs to be explored.

Both physical and mechanical properties of bamboo scrimber have been studied [16]. Thermal treatment would have significant influence toward the adhesive of bamboo scrimber, which is the main factor related to mechanical property. However, there is little information about it. The main object of this work was to study the influence of heat treatment on bamboo scrimber.

Materials and methods

Materials

Neosino calamus affinis with 3–4 years age was used as raw material of bamboo scrimber. Untreated bamboo tubes were fluffed along longitudinal fiber direction, and then carbonized at a steam pressure of 0.37 MPa and a temperature of 115 °C. Carbonized bamboo fiber bundles (39.78 % of α -cellulose, 15.00 % of hemicelluloses, and 37.19 % of lignin) were immersed in a phenol formaldehyde resin (Dynea Co., Beijing City, China). The adhesive was controlled to 15 % of dry weight of bamboo scrimber, and the bamboo fiber bundles were pressed at 140 °C and 5.2 MPa for a holding time of 1 min/mm. Average density of bamboo scrimber board was 1.22 g/cm³.

Samples of 20 mm (Width) \times 20 mm (Thickness) \times 30 mm (Length) for compressive strength parallel to grain and 300 mm (Length) \times 20 mm (Width) \times 20 mm (Thickness) for bending properties were machined for the tests.

Heat treatment

Both bending and compression samples were separated into eight groups, each with 12 replicates, for different heat treatment temperatures: control (20 °C), 50, 80, 110, 140, 170, 200, and 230 °C. Heat treatment samples were put in a laboratory oven for 2 h. Then, the samples were placed at room temperature (20 °C, 60 % humidity) for 24 h. Color

measurements were performed in the middle part of samples using a colorimeter SC-80C, and $L^*a^*b^*$ values were measured. Four measurements were recorded on the sample surface of each treatment level. To observe density changes during heat treatment, measurements of 12 replicates of bending and compression samples were performed after different heat treatment levels, respectively.

Weight loss after heat treatment was calculated according to the formula:

$$WL = \frac{m_0 - m_1}{m_0} \times 100 \% \quad (1)$$

Moisture absorption at room temperature for 24 h was estimated by the following equation:

$$MA = \frac{m_2 - m_1}{m_1} \times 100 \% \quad (2)$$

where m_0 is the initial mass of sample before heat treatment, m_1 is the oven-dry mass of sample after heat treatment, and m_2 is the mass of treated samples at room temperature for 24 h.

Mechanical tests

Heat-treated samples were placed at room temperature for 24 h. Bending MOE and compressive strength parallel to grain were determined according to GB 1936.2–2009 [17] and GB 1935–2009 [18], respectively. Bending strength was tested according to GB 1936.1–2009 [19], using a three-point bending method. 180 replicates of the control sample for compressive strength parallel to grain were tested to compare failure types with heat-treated samples. Instron 5582 (Instron Co., Grove City, PA, USA) was used for the mechanical strength tests.

Fourier transform infrared spectroscopy (FTIR)

FTIR spectra were recorded as KBr disks on an FTIR spectrometer TENSOR 27 (Germany) at a wavenumber range of 4000–400 cm⁻¹. Three replicates of bamboo scrimber sample for each temperature (20, 110, 170, and 230 °C) were selected randomly and ground. The ground powder (2 mg) was mixed with KBr (200 mg) in an agate mortar to form pellets. Tests were repeated three times for each heat treatment temperature.

Results and discussion

Weight loss and density

Weight loss and density for heat-treated bamboo samples at different temperatures are shown in Fig. 1. Weight loss increased and density decreased while temperature

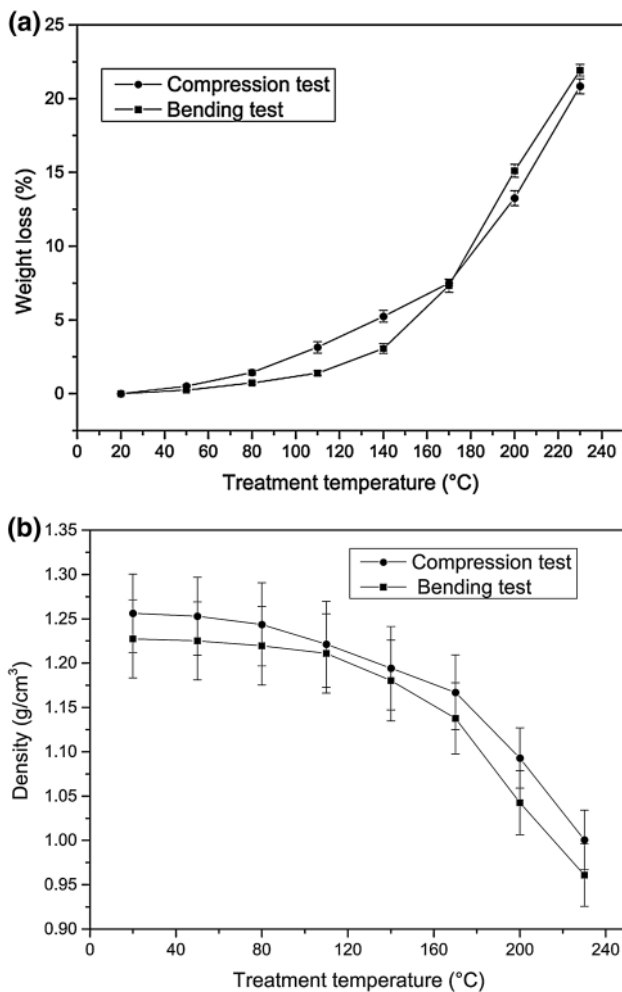


Fig. 1 Influence of temperature on weight and density. **a** Weight. **b** Density

increased. Noticeable mass evolution was observed for temperatures between 170 and 230 °C. This phenomenon can also be observed in graphs of density change with temperature, whose slopes grew larger at high temperature. Density change graphs of compression sample and bending sample were almost parallel to each other at low (20–80 °C) and high (170–230 °C) temperatures. Changes of mass loss and density of bamboo scrimber during heat treatment can be explained by chemical modifications after heat treatment. Polysaccharide (cellulose and hemicelluloses), whose branched structure and amorphous tissues are more susceptible to thermal degradation than other components, decreases steadily during heat treatment [20]. α -Cellulose in bamboo decreases from 41.54 to 26.61 % as temperature increases from ambient temperature to 200 °C [21]. Hemicelluloses decompose into xylose, galactose, rhamnose, and arabinose with treatment time [22]. Contents of hemicelluloses and lignin increased with heat treatment because of the reduction of relative amount of

cellulose [23]. Weight loss of phenolic below 150 °C is mainly evaporation of water, free phenol and free formaldehyde. Dibenzyl ether bond in phenolic structure begins to react when temperature reaches 150 °C, causing oxygen loss in the form of water and carbon dioxide, and generate methine bond [24].

Color changes and moisture absorption

Color changes of bamboo scrimber after heat treatment are shown in Fig. 2 and Table 1. Original light color of samples turned to be dark brown after heat treatment as wood [25], especially at temperatures of 200 and 230 °C. L^* , represents lightness stability [14], decreased significantly above 170 °C, indicating a tendency of bamboo scrimber surface to become darker. Increased and decreased values show a tendency of surface to become reddish and greenish for a^* , and yellowish and bluish for b^* [14]. Surfaces of bamboo scrimber turned to be greenish and bluish after heat treatment at 230 °C. Rate of color change was lower below 170 °C than that above 170 °C, and this tendency is consistent with mass loss. Change of color at different temperatures may be due to degradation of lignin, a typical Gramineae lignin composed of mixed dehydrogenation polymer of coniferyl, sinapyl and *p*-coumaryl alcohols [23], on which chromophoric groups (carbonyl and unsaturated bond) exist. Lignin is relatively stable at room temperature but is easily oxidized with forming chromophoric groups at a high temperature [26, 27]. The β -O-4 linkages, major linkage types in lignin, are cleaved significantly during steam explosion treatment (210 °C) [23], forming free hydroxyl group. In the presence of auxochrome group (hydroxy, carboxyl, and ether bond), color of bamboo scrimber grew dark [28]. Blue shift of methoxy (lignin) is another reason for color change of bamboo. Peak of methoxy shifts to higher wave numbers as temperature increases, and wavelength comes down [29]. Of visible light, the smaller the wavelength, the darker the color is. Cracks appeared on the surface of both compression and bending samples when temperature reached 170 °C. This may be due to curing of adhesive that caused stress concentration on sample surfaces.

In Fig. 3, moisture absorption declined slightly when temperature was below 170 °C, and increased when temperature exceeded 170 °C. Contact angle of oil heat-treated bamboo (180 °C) increased after oil extraction due to changes to the chemical components of bamboo [12]. At low temperature, increased crystallinity [30], loss of hemicelluloses or fragile pentoses and hexoses, reduction of OH bonds, structural modifications, and chemical changes of hemicelluloses [25] can lead dimensional stabilization and decrease hygroscopicity and swelling of

Fig. 2 Discoloration of the control and heat-treated bamboo scrimber samples. From *left to right* are the control samples and samples exposed to 50, 80, 110, 140, 170, 200, and 230 °C for 2 h. **a** Bending samples. **b** Compression samples



Table 1 Color changes of bamboo scrimber samples after heat treatment

Temperature (°C)	L^*	a^*	b^*
20	39.04 (0.45)	9.96 (0.21)	23.71 (1.04)
50	37.14 (0.65)	9.59 (0.49)	24.87 (0.74)
80	38.24 (0.49)	10.15 (0.32)	23.84 (0.50)
110	37.25 (0.083)	8.55 (0.091)	24.43 (0.14)
140	37.05 (0.41)	9.37 (0.31)	25.14 (0.34)
170	34.18 (0.19)	10.32 (0.49)	27.83 (0.52)
200	26.76 (0.64)	10.15 (1.54)	24.75 (1.42)
230	14.21 (0.27)	-0.54 (0.81)	20.57 (1.14)

Values in parentheses denote standard deviation

bamboo. At 230 °C, moisture absorption increased may be because crystal structure of cellulose, where water is not easy to enter [31], was damaged. Crystallinity decreases at the later stages of heat treatment due to depolymerization and degradation in both crystalline and non-crystalline regions [30]. And water entered amorphous area, increasing moisture absorption. Decreased density and destruction of chemical structure may also be the reasons of increasing moisture absorption at high temperature. Moisture absorption of compression sample was greater than that of bending sample probably, because specific surface area of compression sample was larger than bending sample.

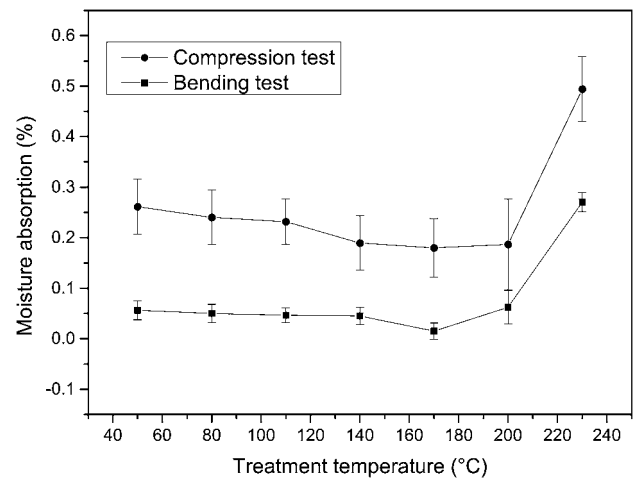


Fig. 3 Influence of temperature on moisture absorption

Failure types and mechanical properties

For failure types, no significant difference was found between the control and heat-treated bending samples; therefore, compression specimens parallel to grain were observed. Failure types of the control samples for compressive strength are diagonal failure (Fig. 4a), end cracking (Fig. 4b), and Y-type failure (Fig. 4c). For diagonal failure type, dislocation of fiber occurred along shear plane (diagonal plane) during compression process. Dislocation fiber was out of the sample surface, as shown in

Fig. 4 Failure types of compression samples parallel to grain at room temperature. **a1** Diagonal failure. **a2** Diagonal failure. **b** End cracking. **c** Y-type failure

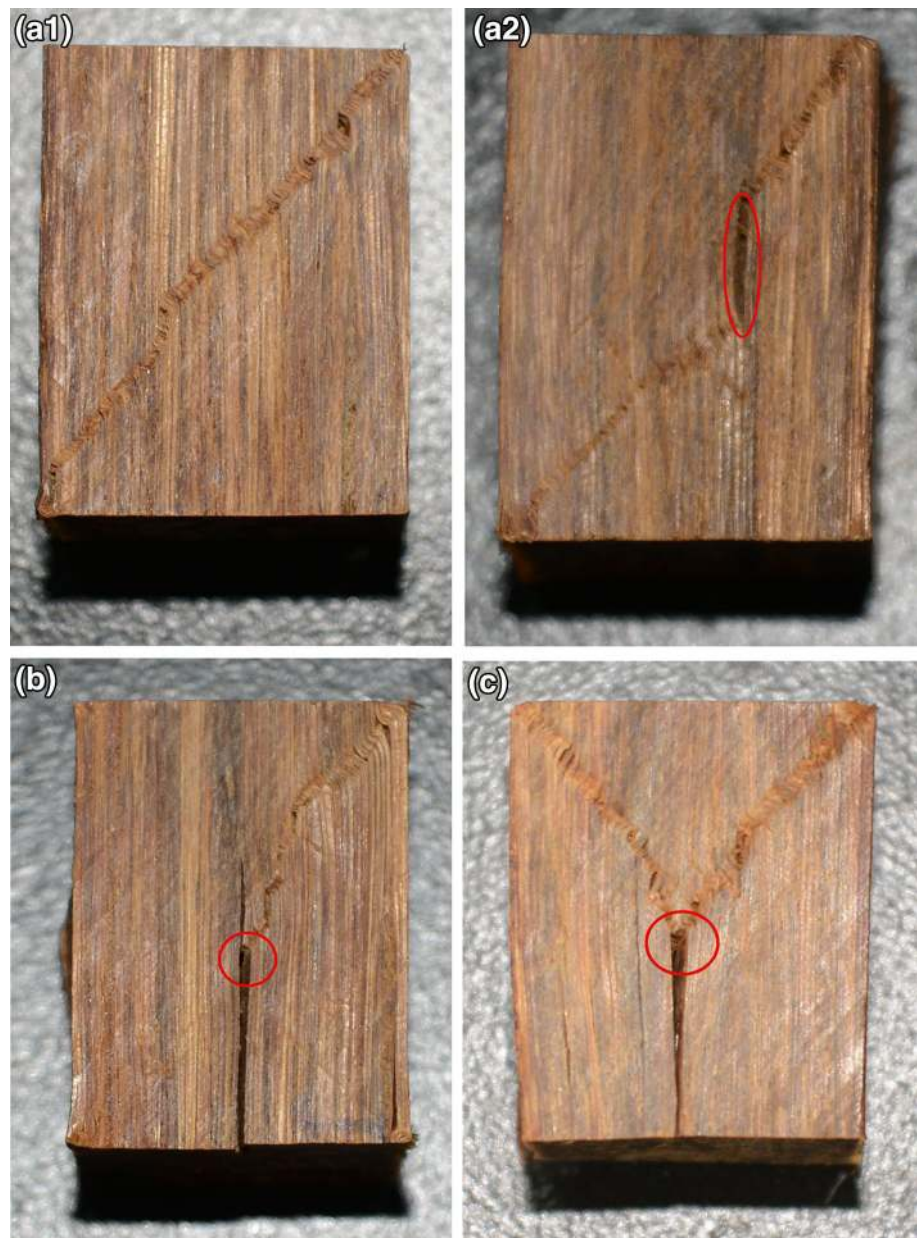


Fig. 4a1. In the process of stress transfer, adhesive layer delaminated and stress released, as shown in Fig. 4a2. If adhesive layer cannot withstand stress transfer and cracked to the end, end cracking failure type formed (Fig. 4b). For Y-type failure samples, dislocation fibers appeared on the two shear planes and intersected in the middle of the sample. Adhesive layer cannot afford intersecting stress and cracking to the end of the sample. Gap at the crack of Y-type failure sample is larger than that of end cracking sample.

Proportion of the three failure types were 35.56, 24.44, and 40.00 %, respectively. Compressive strengths were 104.51 MPa (± 11.45 MPa), 105.38 MPa (± 12.71 MPa),

and 104.87 MPa (± 10.14 MPa) for diagonal failure, end cracking, and Y-type failure, respectively. It can be seen that failure types have no significant effect on mechanical strength of the control bamboo scrimber.

Failure types of heat-treated samples were significantly different from the control samples (Fig. 5). Compared with the control samples, delamination of adhesive layer of heat-treated sample was much more serious. It was probably caused by solidification of adhesive during process of water evaporation. Fiber bundles fractured neatly at temperature of 170 °C; however, this phenomenon is not found in the control samples. At this temperature, water in bamboo scrimber evaporated, causing fiber brittleness



Fig. 5 Failure types of compressive strength parallel to grain at different temperatures. From left to right are the control sample and samples exposed to 50, 80, 110, 140, 170, 200, and 230 °C for 2 h

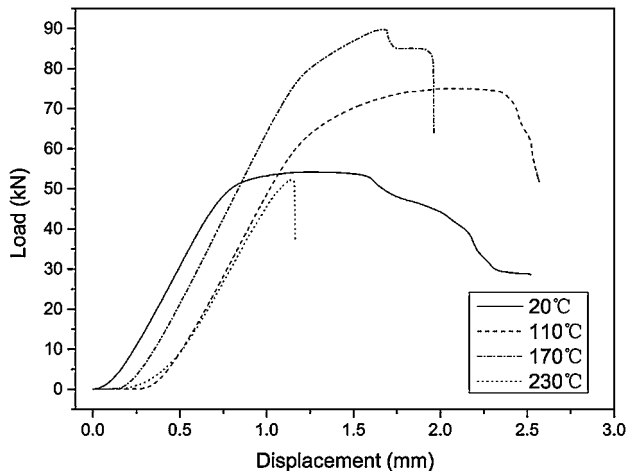


Fig. 6 Load–displacement curves of the control sample and heat-treated samples

increased [32], and fiber bundles fractured neatly. Fiber bundles pulled out of the sample plane at 200 and 230 °C. This may be due to thermal degradation of adhesive which could not play a bonding role in the sample.

Comparison of load–displacement curves (compressive strength) between the control sample and heat-treated samples is shown in Fig. 6. With the increase in load, plastic deformation occurred in the control sample, as shown in Fig. 6. Dislocation fiber on sample surface, caused by plastic deformation, can be seen in all control samples (Fig. 4). Brittle fracture became obvious as temperature increased. The phenomenon of dislocation fiber reduced with increasing temperature in Fig. 5. This is due to increasing of brittleness after exposure to higher temperature [32]. Load–displacement curve of heat-treated sample at 230 °C showed brittle rupture without yield phenomenon, and specimen fractured suddenly.

Mechanical properties of bamboo scrimber were affected by temperature significantly in Fig. 7. Heat treatment influences mechanical properties of wood adversely [33, 34]. However, as temperature rose, mechanical properties of bamboo scrimber increased first and then decreased. 170 °C was the turning point of mechanical properties of bamboo scrimber under this heat treatment condition. Bending strength and bending MOE

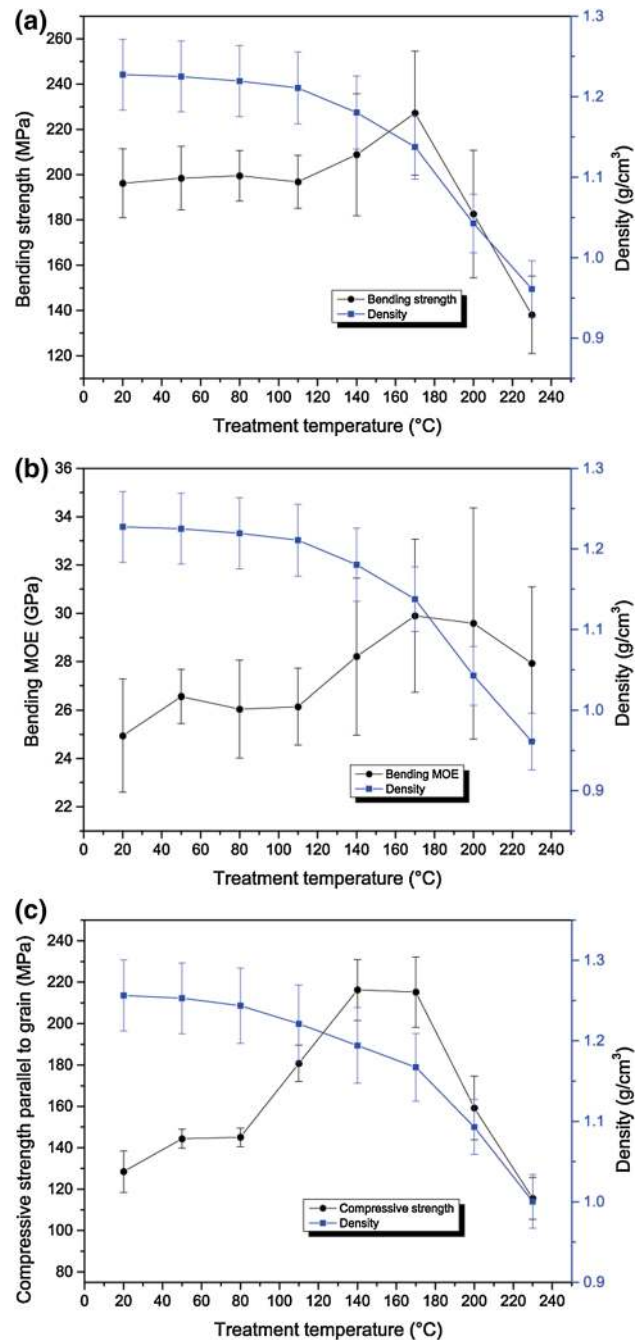


Fig. 7 Mechanical properties and density of bamboo scrimber at different temperatures. **a** Bending strength. **b** Bending modulus of elastic (Bending MOE). **c** Compressive strength

Table 2 Characteristic bands of infrared spectra of bamboo scrimber

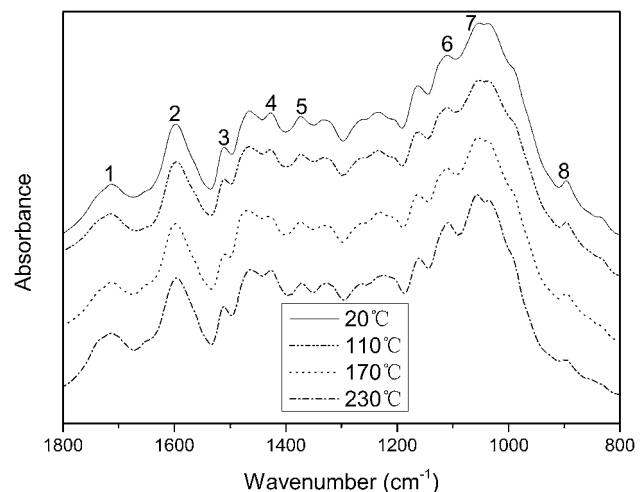
Number	Wavenumber (cm ⁻¹)	Absorption peak location and assignment
1	1712	C=O stretching of xylan (hemicellulose)
2	1596	C=C stretching of benzene ring (phenolic resin)
3	1510	Carbon skeleton stretching of benzene ring (phenolic resin and lignin)
4	1427	Aromatic skeletal vibrations and C–H stretching (lignin)
5	1373	C–H deforming and stretching (cellulose and hemicelluloses of bamboo)
6	1108	C–H stretching of aromatic ring (phenolic resin and lignin)
7	1053	C–O stretching
8	897	Anomeric carbon (C ₁) stretching (cellulose of bamboo)

reached maximums at 170 °C, and then declined. However, decreased bending MOE values at 230 °C were still higher than the control sample. Compressive strength increased fast at 110 and 130 °C, and decreased greatly above 170 °C. Mechanical property of heat-treated bamboo decreases slightly at 150 °C; however, it declines significantly when temperature reaches to 200 °C [15]. It is because high temperature causes certain damage on cell walls of the sample [35]. Compared with bamboo, changes of mechanical properties of bamboo scrimber are probably because of the adhesive in it. As can be seen from Fig. 7, there is no obvious correlation between density and mechanical properties at relatively low-temperature environment. However, mechanical properties declined when density decreased significantly above 170 °C.

Color and failure types to some extent reflected strength performance of heat-treated bamboo scrimber under this experimental condition. Specimens had high mechanical properties when fiber bundles fractured neatly (170 °C). Color of the samples at 200 and 230 °C were much darker than other samples, and mechanical strength became low when color changed brown obviously.

FTIR analysis and chemical components

Table 2 and Fig. 8 are characteristic bands and FTIR spectrogram of bamboo scrimber. There was no significant difference of FTIR curves between 20 and 110 °C. Relative intensities of the peak at 1373 cm⁻¹, representing C–H deforming and stretching of cellulose and hemicelluloses, decreased slightly at 170 °C. However, for phenolic resin, cross-linking reaction proceeds rapidly when temperature reaches 150 °C [36]. Then, a structure of three-dimensional networks, having high mechanical strength, can be formed by solidification. Mechanical strength of phenolic resin-derived spherical activated carbon reaches maximum when temperature reached 180 °C [36]. Therefore, mechanical properties of bamboo scrimber reached maximum at 170 °C probably due to curing of phenolic resin inside the

**Fig. 8** Fourier transform infrared spectroscopy (FTIR) spectrogram of bamboo scrimber in different temperatures

material. Tendency of peaks for 230 °C was similar to 170 °C, but degradation was more intense.

In Table 3, ratios are relative intensities of peaks at 1596, 897, 1712, and 1427 cm⁻¹ against peak at 1108 cm⁻¹. Ratios of peak height between 1596 cm⁻¹ (phenolic resin) and 1108 cm⁻¹ decreased as temperature went up. There was no significant difference of peak height ratio between 20 and 110 °C, at which water, free phenol, and free formaldehyde volatilize [24]. However, significant differences existed between 170 and 230 °C samples. Phenolic resin decomposed more serious at 230 °C, because difference of peak height ratio between 170 and 230 °C was higher than that between 110 and 170 °C. Regularity of molecular chain of phenolic resin decreased when temperature is beyond 180 °C. This is due to severe degradation of hexamethylenetetramine into ammonia and methylene [36]. Tendency of peak height ratios between 897 cm⁻¹ (cellulose of bamboo) and 1108 cm⁻¹ is similar to the ratios between 1596 and 1108 cm⁻¹, showing degradation of bamboo cellulose at high temperature. This is consistent with diminished relative intensity at

Table 3 Relative intensities of C–O and C₁ stretching against typical bands

Temperature (°C)	I ₁₅₉₆ /I ₁₁₀₈	I ₈₉₇ /I ₁₁₀₈	I ₁₇₁₂ /I ₁₁₀₈	I ₁₄₂₇ /I ₁₁₀₈
20	4.98 (a)	0.93 (a)	1.85 (b)	0.78 (a)
110	4.95 (a)	0.84 (a)	2.11 (a)	0.73 (a)
170	4.41 (b)	0.61 (b)	1.59 (c)	0.71(a)
230	2.92 (c)	0.25 (c)	1.28 (d)	0.71(a)

895 cm⁻¹, representing cellulose in bamboo, after heat treatment (200 °C) [21]. The ratios between 1712 cm⁻¹ (hemicellulose) and 1108 cm⁻¹ increased at 110 °C and showed significant difference between 20 and 110 °C. This may be due to carbonized process of bamboo fiber bundles at 115 °C, at which hemicelluloses are more susceptible than cellulose and lignin [31]. When bamboo scrimber was heated to 110 °C, content of hemicelluloses remained unchanged and phenolic resin was affected, so the ratio between them rose. As temperature continued to rise, hemicelluloses decomposed and ratios decreased. Non-conjugated carbonyl group stretching in xylan of bamboo hemicelluloses decreased after heat treatment, indicating that polyoses are considerably more susceptible to thermal degradation than other bamboo components [21]. Ratios of 1427 cm⁻¹ (lignin) against 108 cm⁻¹ decreased as temperature increased; however, no significant differences existed among 20, 110, 170, and 230 °C. It is probable that lignin is more difficult to decompose than polysaccharide [37]. Relative lignin contents in heat-treated bamboo samples increases as temperature reaches 200 °C due to reduction of relative amount of cellulose [21]. It can be explained that decline of mechanical strength was due to beginning degradation of bamboo and phenolic resin at high temperature (200 °C).

Conclusion

1. Rising temperature could cause weight and density of bamboo scrimber decreasing. The effect was great from 170 to 230 °C. Moisture absorption for heat-treated samples increased progressively when temperature exceeded 200 °C.
2. There is no significant effect of failure types on mechanical strength of the control samples. However, mechanical properties of heat-treated bamboo scrimber reached maximums when fiber bundles fractured neatly in this experiment environment. Plastic deformation occurred during compression process for the control sample. Brittle failure became more obvious with increasing temperature for heat-treated samples.

3. In this research, solidification of adhesive inside the material was the main reason for increased mechanical properties. Degradation of phenolic resin and bamboo at high temperature resulted in decline of mechanical properties. Factors affecting heat treatment of bamboo scrimber should be further studied.

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