EFFECT OF STEAM TREATMENT ON THE PROPERTIES OF *PHYLLOSTACHYS IRIDESCENS* BAMBOO COMPOSITE

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The objective of this paper was to propose a heat treatment method to change the surface color of a bamboo fiber reinforced composite (BFRC) and improve its dimensional stability. Oriented bamboo fiber mats (OBFMs) were heat-treated at 0.35, 0.40 and 0.45MPa steam pressures, respectively, for 140 min, and then used to make BFRC. The chemical composition, water extractives and pH value of OBFM, as well as the surface color, dimensional stability, mechanical properties of BFRC were studied. The results pointed out that the heat treatment can significantly change the surface color of the BFRC, as the board surface became darker. The dimensional stability of the BFRC was improved by reducing thickness swelling and wideness swelling. The mechanical properties were affected by a reduction in modulus of rupture (MOR), while an increase in the modulus of elasticity (MOE) was also observed. Chemical degradation occurred, mainly in relation to hemicelluloses contents, which is the main reason for the dimensional stability and reduction in MOR. The pH value decreased significantly, while the water extractive content increased, compared with the control sample. So, steam treatment can be a recommended method to improve the BFRC performance.

Keywords: oriented bamboo fiber mat (OBFM), bamboo fiber reinforced composite (BFRC), steam treatment, dimensional stability, mechanical property

INTRODUCTION

The manufacturing technology of bamboo fiber reinforced composite (BFRC) is based on the improvement of existing bamboo artificial board. In this technology, various bamboo resources are used as raw materials, whether it is large diameter bamboo or miscellaneous bamboo with path level, the bamboo can be effectively utilized, with a utilization rate above 90%. Compared with traditional bamboo scrimber, BFRC is a second-generation product from bamboo scrimber, which has a higher raw material utilization rate, greater production efficiency, and higher added value. As an advanced and frontier technology, the manufacture of BFRC has attracted extensive attention in the bamboo industry.[1]

In recent years, the heat treatment of bamboo has been widely used in Chinese bamboo enterprises for achieving deeper colored material, namely a carbonized color, which is very popular among customers of furniture and decorative materials. The heat treatment is a process that improves the properties of bamboo without using any toxic chemicals.[2] Qin L. studied the effect of the heat treatment on reconstituted bamboo lumber of *Neosinocalamus affinis* at temperatures of 160, 180 and 200 °C, and found that the thickness swelling and decay resistance of the material were improved after the heat treatment, but the strength properties were reduced.[3] A similar result was found when *Phyllostachys pubescens* bamboo was heat-treated under steam pressures of 0.40-0.50MPa for 1-2 h.[4] Many researches have been focused on BFRC,[5,6,7] but no effects of heat treatment on it were reported.

Phyllostachys iridescens bamboo is endemic to China. It is a common bamboo species, which can

be used as bamboo shoot, timber and as an ornamental plant. In this study, *Phyllostachys iridescens* bamboo was turned into OBFMs, using a new technology, which were heat treated at 0.35, 0.40 and 0.45 MPa steam pressure for 140 min. Then, the OBFMs were used to make BFRC. The effects of heat treatment on the surface color, dimensional stability, and mechanical properties of BFRC were investigated. In addition, the chemical composition, water extractives and pH value of OBFM were also studied.

EXPERIMENTAL

Materials

Phyllostachys iridescens bamboo aged 4 years was obtained from Anhui province. The matrix material used in this study was based on a commercially available low molecular weight phenol formaldehyde resin, supplied by Beijing Dynea Chemical industry Co., Ltd. The trade name is PF16L510 with the following parameters: 45.36% of solid content, 40.5 CPs of viscosity, 10-11 pH (at 25 °C), and ability of dissolve in water is 15.7 times.

Preparation of oriented bamboo fiber mat (OBFM)

The bamboo was sawn into a bamboo tube with the length of 2600 mm, and then was split longitudinally into two semicircular bamboo tubes. Thereafter, the inner nodes were removed and the semicircular bamboo tube was pushed into the fluffer along the grain direction. The bamboo tubes were fluffed along the longitudinal fiber direction to form a series of dotted and/or linear shaped cracks along the fiber direction. Consequently, a structural net of OBFM was formed by the interlaced bamboo fiber bundles, consisting of less than 5 vascular bundles and several ground tissues (see Fig. 1). Then, the OBFMs were dried in an oven to an approximate moisture content of 10%.

Heat treatment

The oriented bamboo fiber mats (OBFMs) with the moisture content of 9-10% were immersed in carbonation equipment, a horizontal carbonization furnace (THLφ1200X4300, made in China), controlled by steam pressure. In the experiment, steam pressures of 0.35 MPa (147 °C), 0.40 MPa (151 °C), and 0.45 MPa (155 °C) were set. After the heat treatment, the OBFMs were reconditioned to reach equilibrium.

Chemical analysis of OBFM

The bamboo flour used for chemical analysis was made according to GB/T 2677.1-93. Samples for each treatment were sawn into small pieces and milled. Powdered samples were sieved into three fractions; the middle fraction (0.25 mm to 0.42 mm) was used for chemical determinations. The measurements of the holocellulose content, α-cellulose content, hot water extractives content and pH value were done according to the methods mentioned in our previous paper.[8]

Preparation of bamboo fiber reinforced composite (BFRC)

The solid content of phenolic-formaldehyde resin was adjusted to 13%. To obtain a uniform glue spread, the OBFMs were immersed in the above resin for several minutes at room temperature. Thereafter, the OBFMs were taken out and placed vertically for several minutes until the adhesive on the surface stopped dropping. The amount of spread glue was controlled to about 8% of the oven dry weight of the OBFM during the glue dipping process. Then, the glued OBFMs were dried at 40 °C in an oven to a moisture content of 10-12%. The OBFMs were weighed out and were assembled along the grain direction with the outer layer outward and the inner surface inward, so as to form a slab. The heat-in and cold-out technology was used. The slab was put into the press machine, when the temperature of the hot platen was around 145 °C.

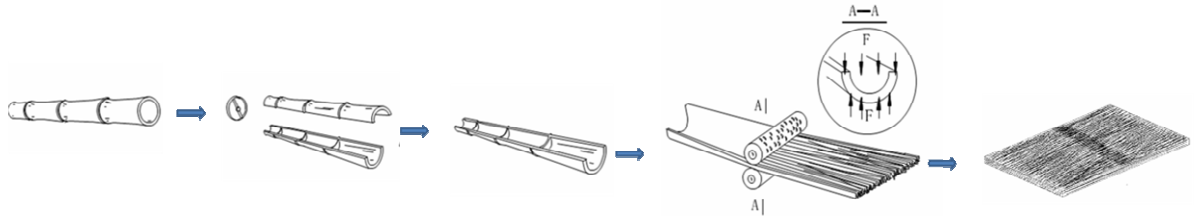

Figure 1: Process flow diagram for OBFM; (A) Raw bamboo; (B) Splitting; (C) Semicircular bamboo tube; (D) Fluffing; (E) Bamboo fiber mat

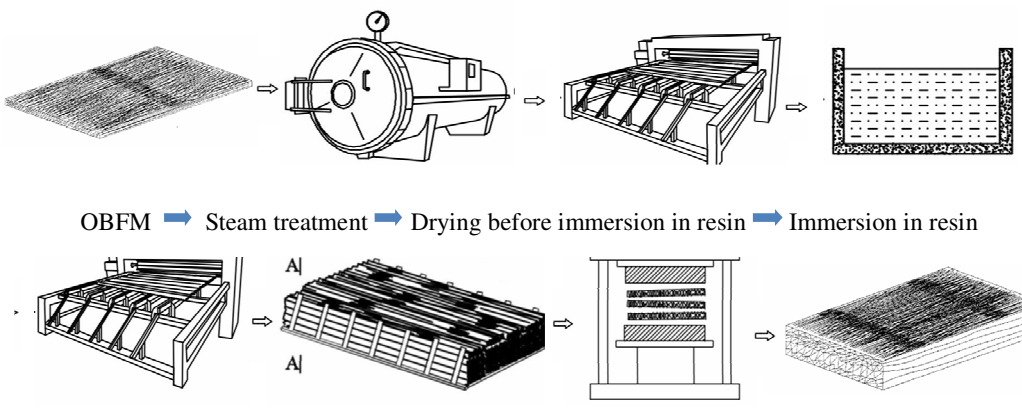

Figure 2: Process flow diagram for BFRC

Next, the pressure was increased due to the introduced superheated vapor to the press. The pressure was kept at 4.5 MPa, and the temperature was kept at 145 °C for a holding time of 1 min/mm. Then, the cold water was introduced into the press to decrease the temperature to 40 °C, and then the pressure was released. Finally, the slab was taken out from the press and the needed assembly product of several BFRCs was obtained. The nominal dimensions of the BFRC were 2600 mm × 1300 mm × 25 mm in length, width and thickness, respectively. All specimens were conditioned in a controlled environment room at 20 °C and 65% relative humidity (RH) for two weeks before testing. The density and moisture content of BFRC were of 1100 kg/m^3 and 8-10%, respectively.

Color measurement

The color of BFRC was measured with a spectrophotometer (Mercury 2000) under a D65 light source and an observer angle of 10°. The sensor head of the spectrophotometer was 8 mm in diameter. Color was expressed according to the CIE ($L^*a^*b^*$) system. The differences in ΔL^*, Δa^*, and Δb^* and the total color change (ΔE^*) were calculated with the following formulas:

$$\Delta L^* = L^* - L_0^*$$
$$\Delta a^* = a^* - a_0^*$$
$$\Delta b^* = b^* - b_0^*$$
$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (1)$$

where L^* represents lightness with values varying from 0 (black) to 100 (white). The parameters a^* and b^* describe the chromatic coordinates on green-red (a^*) and blue-yellow (b^*) axes. Color was measured for each group at 20 points on the surface of the BFRCs, which were sanded, and the results were then averaged. L_0^*, a_0^*, and b_0^* are the reference values.

Dimensional stability and mechanical strength of BFRC

The control and heat-treated BFRCs were evaluated according to ASTM D1037 for dimensional stability and mechanical properties of modulus of rupture (MOR) and modulus of elasticity (MOE).

Statistical analysis

One-way statistical analysis (ANOVA) was performed on the data to evaluate the effect of steam treatment on the color, by using SAS software (Version 8.0, SAS Institute, NC, USA).

RESULTS AND DISCUSSION

Changes in chemical composition of OBFM

During the heat treatment process, the chemical components in the bamboo produced chemical reactions, which affected the contents of holocellulose and α-cellulose, as shown in Table 1.

When samples were treated at different steam pressures for 140 min, a decrease in the holocellulose content with increasing steam pressure was found. The relative decrease in holocellulose content was 6.65% between 0.35 MPa and 0.45 MPa. The decrease in holocellulose content indicated the degradation of holocellulose after steam treatment.

The α-cellulose content increased slightly by less than 3.0% compared with the control sample. However, no apparent change in the α-cellulose content was found when the samples were treated at 0.35 MPa to 0.45 MPa steam pressures. The same result was also reported for wood. According to a study on wood, other components accompanying wood cellulose are involved in the

increase of crystallinity after heat treatment.[9]

The most obvious change was in hemicelluloses content, which decreased by approximately 39.06% to 58.95% after the heat treatment. Hemicelluloses degrade first (between 160 °C and 260 °C), because their low molecular weight and branching structures facilitate faster degradation compared with other components present in wood.[10] Windeisen *et al*. observed that the amount of polysaccharide decreased as the treatment temperature increased.[11]

Table 1
Changes in chemical composition of OBFM

Steam pressure, MPa	Holocellulose (A), %	α-cellulose (B), %	Hemicelluloses (A-B), %
Control	67.41	44.29	23.12
0.35	59.13	45.04	14.09
0.40	56.85	45.60	11.25
0.45	54.65	45.16	9.49

Note: Hemicelluloses (%) = Holocellulose (%) – α-cellulose (%)

Table 2
Values of pH and water extractives forOBFM

Steam pressure, MPa	pH value	Hot water extractives, %
Control	5.32	5.74
0.35	4.06	10.17
0.40	4.13	11.21
0.45	3.83	15.00

Changes in pH value and water extractive content of OBFM

As shown in Table 2, the pH value was reduced clearly compared with that of the control sample and reached a maximum reduction of 28.00% after the steam treatment. The steam pressure (0.35 MPa to 0.45 MPa) had a slight effect on the pH value, the relative change was lower than 0.30.

The contents of water extractives increased apparently, which was 1.77 times as much as that of the control samples when the samples were treated at 0.35 MPa, and followed by a relative increase at a higher steam pressure, it was 2.61 times as much as that of control samples when the samples were treated at 0.45 MPa. The increase in water extractives showed that the steam treatment resulted in many more small soluble molecules due to the degradation of hemicelluloses and cellulose.

Color measurement of BFRC

The specimens became visibly darker and the color changed to a relatively more pleasant reddish hue after the heat treatment. The visual observations described above can be confirmed by the objective color measurement.

The color evolution of the samples is presented in Table 3. A decrease in the L* values was associated with a darker color. The lightness decreased continuously with increasing steam pressure. According to the research of Tjeerdsma *et al.*, a deeper wood color is mainly due to the formation of benzoquinone by oxidation.[12] Windeisen *et al.* studied the thermal treatment of beech, and after elemental analysis concluded that the carbon content increased with increasing temperature.[11] The increase in the content of carbon indicated the loss of functional groups, which contain oxygen (such as carboxyl, hydroxyl acetyl).

The yellow hue (b* coordinate) had a similar trend as L*, a significant change was found when the materials were treated at a steam pressure of 0.35 MPa. Lower values were detected with increasing treatment pressure. This indicates that the surface color closes to the center axis direction of the yellow-blue axis. The change in the red color represented by a* coordinate followed

another pattern. A significant increase was observed when the samples were heat-treated at a steam pressure of 0.35-0.45 MPa. The coordinate a* shifts positively, indicating that the color of the bamboo became more red.

As L*, a*, b* were affected by the heat treatment, the final color change (ΔE*) was significant. The ΔE* changed little when OBFMs were steam treated at 0.35 MPa and 0.40 MPa, but it increased apparently when OBFMs were steam treated at 0.45 MPa. According to the results obtained by Varga *et al*., the main factors of the color change were the dissolution and oxidation of wood components and the decomposition of extractives.[13] To explain the reasons of bamboo color change, more research needs to be done.

Dimensional stability of BFRC

The dimensional stability results, *i.e.* thickness swelling (TS) and wideness swelling (WS), of the water immersion test (63 °C, 24 h) are shown in Table 4. The values of TS and WS decreased by maximum 25.08% and 19.55%, respectively, after the heat treatment, compared with the control specimens. The TS decreased with increasing steam pressure, but WS changed slightly when specimens were heat-treated at 0.35-0.45 MPa.

Table 3
Results of color measurement of BFRC

Steam pressure, MPa	Color in CIE system			Color difference			
	L^*	a^*	b^*	ΔL^*	Δa^*	Δb^*	ΔE^*
Control	70.18 ^{A} (3.65)	6.35 ^{A} (13.53)	29.17 ^{A} (6.37)				
0.35	45.19 ^{B} (3.81)	8.75 ^{B} (10.51)	18.88 ^{B} (7.90)	-24.99	2.4	-10.29	27.13
0.40	45.77 ^{B} (7.58)	9.63 ^{C} (8.60)	19.92 ^{C} (13.37)	-24.41	3.28	-9.25	26.31
0.45	42.82 ^{C} (13.42)	9.16 ^{C} (15.67)	17.43 ^{D} (15.71)	-27.36	2.81	-11.74	29.90

Coefficient of variation (CV) is given in parentheses. All data in Variance and one-way ANOVA tests were done at a confidence level $p<0.05$. Values followed by a different letter within a column are statistically different at $P = 0.05$ (ANOVA Single Factor and t-test)

Table 4
Results of TS and WS of BFRC

Steam pressure, MPa	TS, %	WS, %
Control	6.30 (0.19)	1.79 (0.06)
0.35	5.26 (0.54)	1.56 (0.30)
0.40	4.88 (0.74)	1.44 (0.18)
0.45	4.72 (0.25)	1.62 (0.11)

Standard Deviation (SD) is given in parentheses

Table 5
Effect of steam treatment on the mechanical properties of BFRC

Steam pressure, MPa	MOR, MPa	MOE, MPa
Control	223 (6.98)	19008 (4.88)
0.40	214 (2.24)	23790 (6.90)
0.45	165 (1.29)	21983 (11.00)

Coefficient of variation (CV) is given in parentheses

This implies that the BFRC becomes more stable after being heat-treated at higher steam pressure. The organic components of bamboo are similar to those of wood. According to the research on wood, decreased swelling of heat-treated wood is mainly due to the reduction of the hydroxyl group content in wood[14,15] or to the formation of cross-linking during the heat treatment.[12] A cross-linking increment makes the molecules less elastic and reduces the possibility to enlarge the cellulose microfibrils, which reduces the ability to absorb water.

Mechanical properties of BFRC

As shown in Table 5, the heat treatment resulted in a reduction of MOR, the value of MOR decreased by 26.01% when the material was steam-treated at 0.45 MPa. However, the MOE increased by 14-15% after the heat treatment, compared with the control specimens. The decrease in the MOR can be explained by thermal degradation. The loss of bending properties are highly associated with the degradation of carbohydrates; the loss of MOR corresponds to a decrease in the hemicellulose content.[16] Sivonen *et al*. found that the deterioration of the hemicellulose starts at or below 180 °C, which produces acetic acid, helping to degrade the structure of the hemicellulose.[17] Cellulose and lignin deteriorate at higher temperatures and at a slower rate than hemicellulose. However, other components accompanying wood cellulose are involved in the increase of crystallinity after the heat treatment; this may increase the rigidity of the material,[11] which may explain the change in MOE.

CONCLUSION

The surface color of BFRC became darker after the steam treatment, and the total color change ΔE^* increased with increasing steam pressure. The results obtained can be used in practice, for example to meet certain costumer requirements regarding the color of bamboo products.

The dimensional stability of BFRC was improved as the thickness swelling and wideness swelling were reduced with increasing steam pressure. In addition, the mechanical properties of BFRC were affected with a reduction in MOR and an increase in MOE. The hemicelluloses content and pH value decreased, while the water extractives increased after the heat treatment. The degradation of hemicelluloses may explain the reduction in MOR and the improved dimensional stability, while the decrease in the pH value and the increase in water extractives indicate the production of more small soluble molecules after the heat treatment.

As a conclusion, the steam treatment can be used as a method to improve the properties of BFRCs to meet certain product specifications.

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