

Effects of Acid Dye on the Performance of Bamboo-based Fiber Composites

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The objective of this study was to investigate the influence of acid dye on color change, modulus of rupture, modulus of elasticity, shear strength (parallel loading), thickness swelling rate, and water absorption rate of bamboo-based fiber composites from *Phyllostachys pubescens*. Bamboo fiber veneers were treated with acid black via three procedures: dyed in water at 20 °C, dyed in water at 90 °C, and dyed in ethanol at 75 °C. The samples dyed in ethanol at 75 °C and those dyed in water at 90 °C had a higher exhaustion rate than those dyed in water at 20 °C. The color change of bamboo fiber veneers exhibited similar variation trends by the three methods. The dyeing procedure slightly reduced the modulus of rupture and modulus of elasticity of bamboo-based fiber composites. Compared with the samples dyed in water, the samples dyed in ethanol had reduced shear strength (parallel loading) and modulus of elasticity. The thickness swelling rate and water absorption rate of bamboo-based fiber composite samples were reduced by the dye treatment.

Keywords: Dyeing; Bamboo scrimber; Colour difference; Bamboo-based fiber composites; Fibrosis bamboo veneers

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INTRODUCTION

Bamboo is abundantly available in many tropical and subtropical regions of the world and is an important product because of its fast growth rate, short rotation age, strength, easy machinability, and its great potential as a sustainable structural building material. Since the 1980s, many bamboo-based materials have been developed, such as bamboo plywood, particleboard, bamboo-laminated strand-board, and bamboo scrimber (Nugroho and Ando 2000; Matsumoto *et al.* 2001; Qin *et al.* 2012; Sahroni *et al.* 2012). These product types have been successfully developed in Asia. However, the sum value of all these bamboo products is not very high; therefore, improving the utilization ratio of bamboo is necessary (Zhang *et al.* 2013a). Developed at the Chinese Research Institute of Wood Industry, bamboo-based fiber composite manufacturing is a new technology for improving the utilization of bamboo resources. This technology has shown some important characteristics, such as using whole bamboo culms as a manufacturing unit, overcoming the bonding problems for outer and inner bamboo culms, and improving the bamboo utilization ratio to more than 90%. It has large potential markets in China and abroad (Yu *et al.* 2011).

There are many potential indoor and outdoor applications in which the composites can be used. For example, bamboo-based fiber composites can be used for both outdoor and indoor flooring, wood plank road, container flooring, garden landscaping, wind

turbine blades, concrete boards, horse stable boards, and furniture (Qin *et al.* 2009; Yu 2011; Zhang *et al.* 2011; Zhu *et al.* 2011; Zhu and Yu 2012; 2013b; Yu *et al.* 2014). However, the application of composites in exterior environments has been greatly limited by their surface color. Currently, only natural color and carbonized color are available in most bamboo markets, which can hardly satisfy the needs of consumers. To meet the demand for product diversification, it is imperative to improve the manufacturing process of bamboo-based fiber composites through appropriate dyeing techniques. This will not only broaden the scope of application of bamboo-based fiber composites, but also ease the severe shortage of valuable tree species.

A dyeing process is the most effective method for changing the appearance of these composites. Dyeing is a physicochemical process that results in the change of the surface color of a material. In recent years, many scholars have studied wood dyeing (Du *et al.* 2010; Guan and Guo 2010; Dogu and Grabner 2010); however, these studies are primarily aimed at wood with good dyeing permeability, and studies on dyeing technology and the dyeing process for bamboo have yet to be reported. Bamboo is a special type of polymer material, and its unique shape, form, and structure are different from those of wood. Bamboo has a dense outer covering and does not have crosswise organization, which makes it extremely difficult for dye solutions to penetrate deeply into the culm. During the production process for making bamboo-based fiber composites, bamboo culms are fluffed and flattened to improve the permeation of dye treatments for bamboo.

This study aims to explore new bamboo-based fiber composite products by treatment with acid black (C. I. Acid Black 2) dye. The color difference and the percentage of dye exhaustion of the bamboo fiber veneers is due to the accumulation of dye particles on the cell walls of raw bamboo culm. The distribution of dye particle aggregates in the bamboo cell structure was studied for the first time. The changes in board color and the physical and mechanical properties of the dyed bamboo-based composites from *Phyllostachys pubescens* were also investigated. This study will offer a reference for the study and production of bamboo-based fiber composites.

EXPERIMENTAL

Materials and Methods

Sampling and raw material

Bamboo (*Phyllostachys pubescens*) culms were obtained from the bamboo forest in Xuancheng, located in Southeast Anhui, China. The bamboo was 4 to 5 years old and had a diameter of about 80 to 100 mm and a wall thickness of about 7 to 12 mm.

Low-molecular weight phenol formaldehyde with the following parameters was obtained from Beijing Dynea Chemical Industry Co., Ltd.: a solid content of 45.59%, a viscosity of 36 CP·S, a pH of 10 to 11, and a water miscibility of 7 to 8.

Commercial-grade acid black dye (C. I. Acid Black 2; C.I. 50420), supplied by Qingdao Double-Peach Specialty Chemicals (Group) Co., Ltd. (China), is suitable for dyeing textile, wax varnishes, and wood. All other chemicals (acetic acid, sodium acetate, and ethanol) were of analytical grade and were used without further purification.

Treatment and composite preparation

Raw bamboo culms were cross-cut into two-meter-long segments. Using a bamboo culm splitter, each segment was split right down the middle into two or three parts. Each part was then pressed to form fiber veneer using a custom device in accordance with China patent no. 200920105914.9. The bamboo fiber veneer was dried to a moisture content of about 10%.

Bamboo fiber veneers with an equilibrium moisture content of 10 to 12% were dyed in a thermostatic water bath. In many preliminary experiments, we observed fiber veneers that had been dyed in water and in ethanol at different temperatures and determined that samples dyed in water at 90 °C and in ethanol at 75 °C (the temperature of the ethanol is lower than its boiling point, which is about 78 °C) have good color appearance and penetration depth. We therefore chose three parameters to dye the samples: water at 20 °C, water at 90 °C, and ethanol at 75 °C. In the dye-treated phase, the dye solutions were made by mixing the C. I. Acid Black with a surface active agent (3 g/L), acetic acid (3 g/L), and sodium acetate (1 g/L). The pH value was controlled at 5 to 6 (Tudor *et al.* 2013).

After the dye treatment, the bamboo fiber veneers were reconditioned to reach equilibrium by immersing them in phenol-formaldehyde adhesive for 5 min after dye treatment. The target resin content of the veneers was controlled at 15% (ratio of resin dry weight to the bamboo bundle's dry weight).

The veneers were then dried to achieve a moisture content of 12%. The dried veneers were reconstituted in a hot-pressing mold (300 mm × 170 mm × 20 mm) at 140 °C for 25 min. The thickness of the composites was set at 20 mm, and the target density was 1.1 g/cm³. Figure 1 shows the flow chart for making bamboo-based fiber composites.

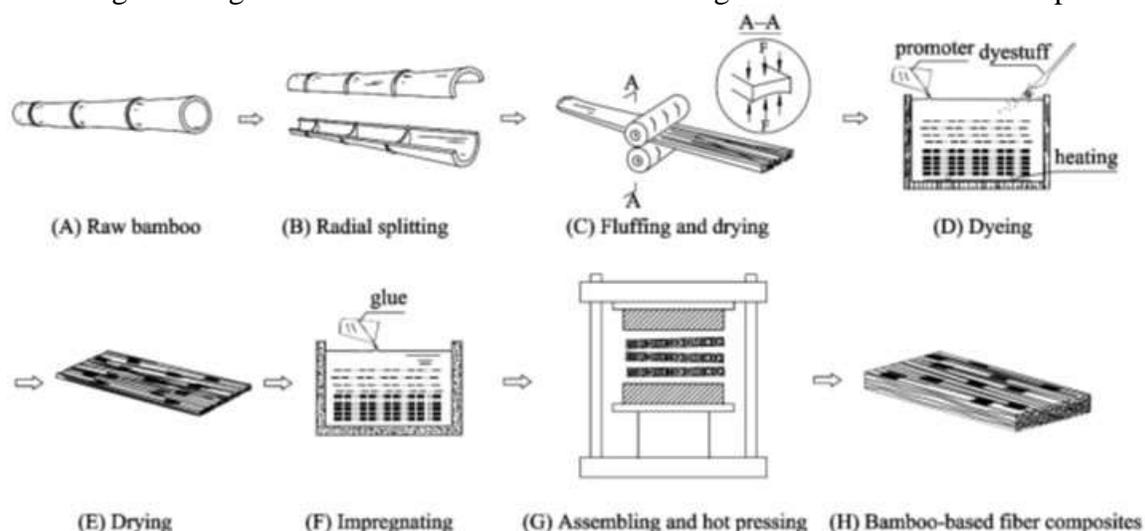


Fig. 1. Flow chart for making bamboo-based fiber composites

Evaluation of Properties

The surface of the veneers was rough because of fluffing by a pilot machine in accordance with China patent no. 200920105914.9 (Fig. 1C) (Yu *et al.* 2014). Therefore,

we chose raw bamboo culm as the object of study. Three raw bamboo sections of approximately 15 mm were collected for anatomical investigation. These sections were taken from the culms and macerated at 75 °C for 72 h. One was macerated with Acid Black 2 in ethanol, acetic acid, and surface active agent; the second was macerated with Acid Black G in water, acetic acid, and surface active agent; and the third was macerated with only water as the untreated control.

Transverse and radial sections (15- to 25- μm -thick) were cut from each bamboo section on a Reichert-Jung sliding microtome and mounted in a synthetic resin. Evaluation was performed using an BX-61(Olympus; Japan) light microscope, and images were taken using DP2-BSW(Olympus; Japan) Software. A DP71 digital camera was installed and adapted on the microscope. All microscopic investigation and evaluations were realized on transverse and tangential sections (Bond *et al.* 2008; Kim *et al.* 2008; Pouzoulet *et al.* 2013; Sint *et al.* 2013; Wahab *et al.* 2012).

The samples were mounted on an aluminum stub, and the surface sputter-coated with a gold alloy. The specimens then were analyzed with a scanning electron microscope (SEM; S-4800; Hitachi; Japan) at a working distance of 25 mm, an accelerating voltage of 10 kV, and a probe current of 6×10^{-10} A.

The dyeing of bamboo fiber veneers was accomplished using an exhaustive dyeing procedure. The absorbance of the dyes at their maximum wavelengths was evaluated using an ultraviolet spectrophotometer, model 721. Also, unfixed dye from the samples was extracted with hot water and measured in the same way. The extent of exhaustion of the dye on acid dyes was calculated as,

$$\text{Exhaustion \%} = (A_0 - A) / A_0 \times 100, (1)$$

where A_0 and A represent the absorbance of the dye solution before and after exhaustion, respectively (Kim and Sun 2002).

Color measurements of the bamboo veneer samples were made using a Chroma Meter CR400 with a D65 light source, 8-mm aperture size, and a 10° normal observer. Six measurements *per* specimen were carried out using a pattern that minimized the bamboo's structural influence on the results. Color evaluation was performed in the CIE Lab system according to the CIE International Commission on Illumination. In the CIE Lab systems, the L^* , a^* , and b^* components are based on a nonlinear transformation of the primary colorimetric values in the X, Y, and Z color space. The X, Y, and Z values themselves are calculated from the absorbance spectra. In the CIE Lab systems, the L^* axis represents the lightness of an object, varying from 0 (black) to 100 (white), and the a^* and b^* axes are the chromaticity coordinates ($-a^*$ for green, $+a^*$ for red, $-b^*$ for blue, and $+b^*$ for yellow). Color differences were calculated using the following equation (2),

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}, (2)$$

where ΔL^* , Δa^* , and Δb^* are the differences of the $L^*a^*b^*$ components between the dyed fibrosis bamboo veneers and the initial values. The resulting ΔE^* values indicate

the extent of the occurring change in color (Park *et al.* 2008; Liu *et al.* 2010; Ristić *et al.* 2010; Miklečić *et al.* 2012; Romagnoli *et al.* 2013).

The modulus of rupture (MOR), modulus of elasticity (MOE), and shear strength (parallel loading) (SS) were compared for dye-treated and untreated composites samples based on China Standard GB/T 17657-1999 and China Standard GB/T 20241-2006. Nine samples were obtained for each group to evaluate MOR, MOE, and SS.

The samples were cut from the composites according to the test standard. Thickness swelling (TS) and water absorption (WA) of the samples were determined to 0.001-mm accuracy at marked positions before and after immersion in boiling water for 4 h, drying in an oven at 63 °C for 20 h, then immersion in boiling water once again for 4 h. Dimensional stability was measured for nine samples in each group, and the results were averaged.

RESULTS AND DISCUSSION

The bamboo anatomical structure is illustrated in Figs. 2 and 3 for comparison.

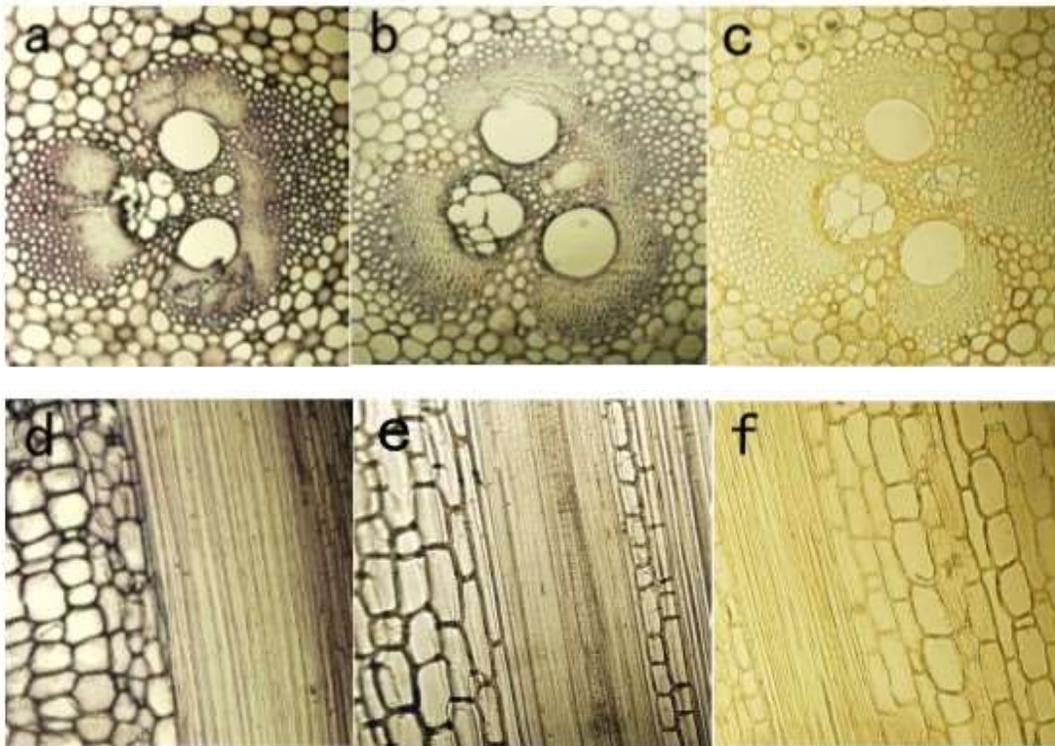


Fig. 2. Light micrographs of bamboo's anatomical structure. (a) Raw bamboo culm dye-treated in ethanol, transverse section; (b) Raw bamboo culm dye-treated in water, transverse section; (c) Untreated raw bamboo culm, transverse section; (d) Raw bamboo culm dye-treated in ethanol, radial section; (e) Raw bamboo culm dye-treated in water, radial section; (f) Untreated raw bamboo culm, radial section. Scale bars for (a), (b), (c), (d) (e), and (f) = 100 μ m

As shown in Fig. 2, a remarkable increase in color was observed in the bamboo's transverse and radial sections. Because parenchymatous cells are crushed in the process

of hot-pressing, we paid more attention to the color change on the cell wall of the vascular bundle of bamboo. In Fig. 2a, it is apparent that the vascular bundle of bamboo in the transverse section was nearly completely black. From the radial section in Fig. 2d, one can also observe that the color of fibers was nearly completely black. In addition, the color of raw bamboo culm treated in ethanol was slightly blacker than the color of bamboo treated in water. This result shows that the method of dyeing in ethanol was helpful for improving color strength.

SEM images of dyed raw bamboo culm and untreated raw bamboo culm with acid black by SEM are depicted in Fig. 3. As shown in Fig. 3b, the untreated raw bamboo culm sample had a comparably smooth surface, and the image of the dyed raw bamboo culm sample in Fig. 3a indicates that acid dye accumulates on the surface of the fiber cell as a fine spherical particle. Figures 3c and 3d indicate that dye particles accumulated on the surface of parenchymatous cells. In Figs. 3a and 3c, one can observe pit diameters in cells of approximately 3 to 10 μm . These results indicate that there were favorable factors for the water-soluble acid dye molecules moved into the bamboo from the pit.

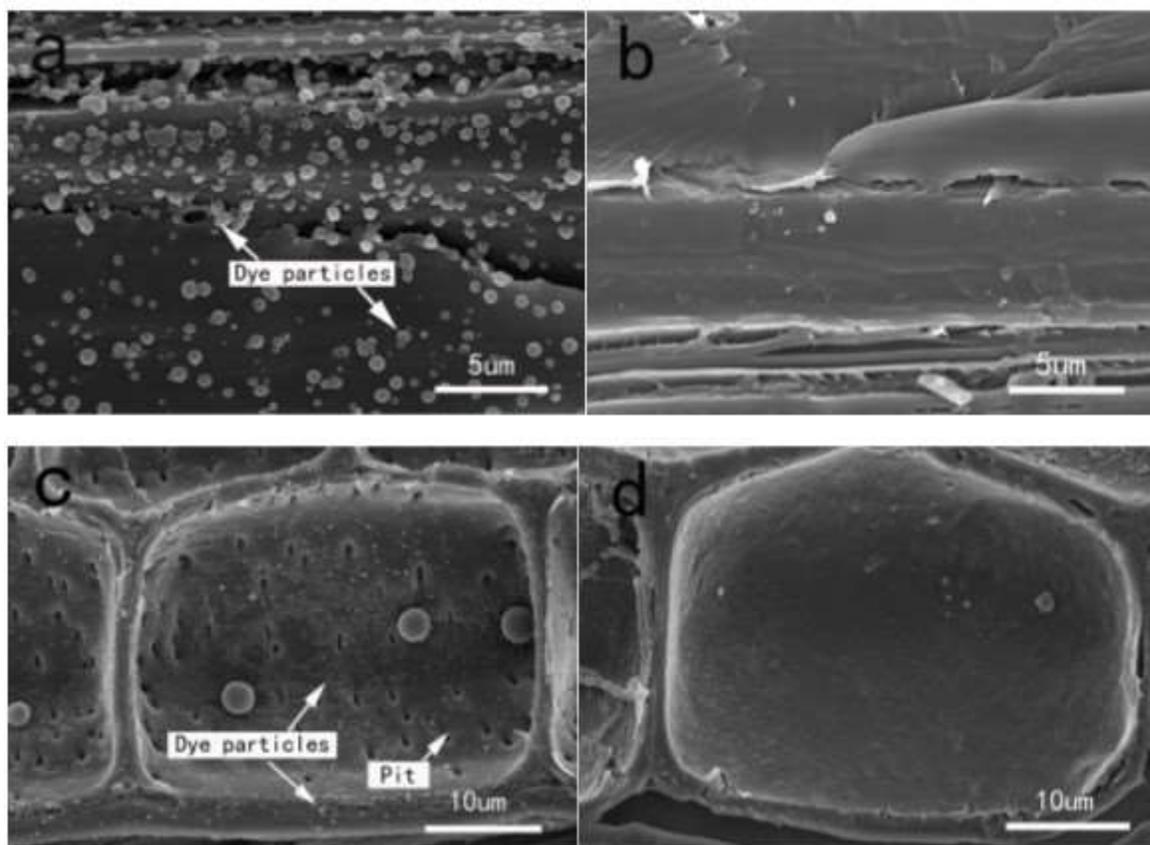


Fig. 3. SEM images of raw bamboo culm in radial section untreated and after dye treatment. (a) Raw bamboo fiber after dyed in ethanol ; (b) Raw bamboo fiber untreated; (c) Dye particle attached to bamboo parenchymatous cell after dyed in ethanol; (d) Undyed parenchymatous bamboo cell

The exhaustion rate and lightness of bamboo fiber veneers are illustrated in Fig. 4 for comparison.

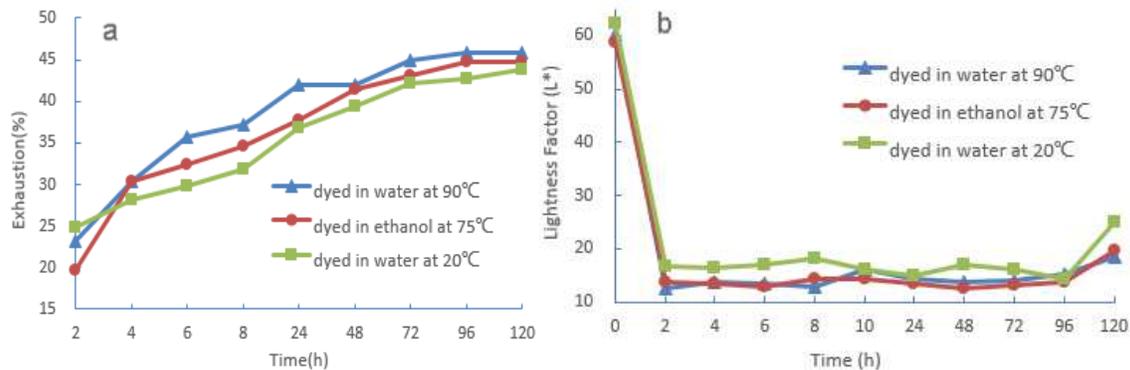


Fig. 4. Exhaustion rate and lightness of fibrosis bamboo veneers

As can be seen from Fig. 4a, with increasing dyeing time, the exhaustion rate increased significantly for samples dyed in ethanol at 75 °C and those dyed in water at 90 °C. This is because when the surface tension of ethanol decreases, dye enters the bamboo's interior easily. The increase in temperature, which enhanced the collision frequency among molecules, is also helpful for improving the exhaustion rate.

The effects of dyeing on color changes in bamboo fiber veneer samples are shown in Fig. 4b. Remarkable color changes in the samples were observed after dye treatment. The lightness change of bamboo fiber veneers were found to have basically similar variation trend by the three methods. When the bamboo was dyed in the ethanol source, its surface color changed rapidly from light to dark, as indicated by the decreasing value of lightness (L^*) during the first 2 h, after which the change rate slowed.

Figure 5 shows the changes in color difference and surface color of bamboo-based fiber composites.

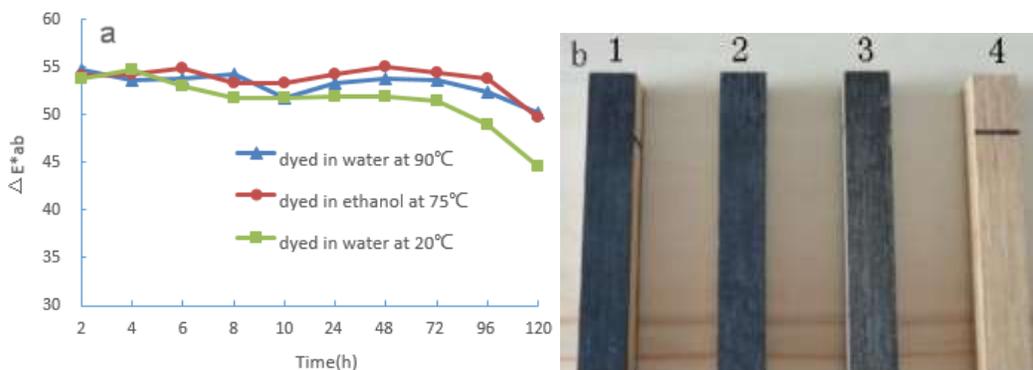


Fig. 5. The color difference and surface color of bamboo-based fiber composites: (1) bamboo-based fiber composites dyed in ethanol at 75 °C; (2) bamboo-based fiber composites dyed in water at 90 °C; (3) bamboo-based fiber composites dyed in water at 20 °C; (4) untreated bamboo-based fiber composites

Figure 5a shows that ΔE^* increased with the temperature and duration of the treatment. For a given treatment time, the values of ΔE^* for samples dyed in ethanol solution at 75 °C and treated with water at 90 °C were generally greater than those treated with water at 20 °C. These samples tend to show slightly higher color differences

when samples had been dye-treated at 75 °C and treated with water at 90 °C. Samples treated with ethanol solution and treated in water at 90 °C had more violent collisions among molecules than those treated in water at 20 °C. The decomposition of hemicelluloses and the chemical changes of extractives are also accountable for such changes in color (Martina *et al.* 2012; Nguyen *et al.* 2012). In Fig. 5b, we can see that the appearance was consistent with the results. In addition, the ΔE^* for samples dye-treated in ethanol at 75 °C and treated in water at 90 °C were very similar.

Figure 6 shows changes in the mechanical properties of bamboo-based fiber composites.

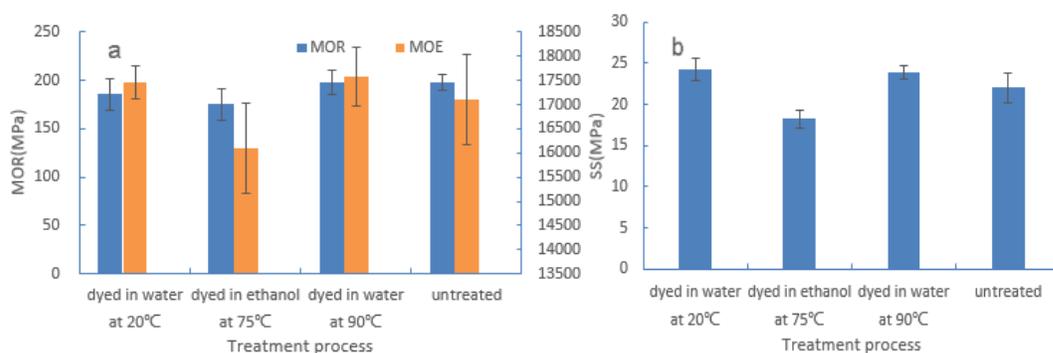


Fig. 6. Mechanical properties of bamboo-based fiber composites

The composite samples of bamboo dyed in water at 20 °C and treated with water at 90 °C showed better performance than the MOR of those dyed at 75 °C in an ethanol solution. The MOE value also decreased for samples dyed in ethanol at 75 °C. The results of this experiment can be explained by the fact that MOR loss corresponds to changes in cell wall components, such as decomposition of hemicelluloses or demethoxylation of lignin, during dye treatment (Qin and Yu 2009; Yildiz *et al.* 2011; Zhang *et al.* 2013c). This also explains why the stable MOE and MOR showed a remarkable loss after heat treatment.

Pairwise comparison showed that there was no significant difference between water-dyed and untreated samples, which indicates that MOR and MOE values were not influenced by the water treatment.

The composite samples of bamboo dyed at 20 °C and those treated with water at 90 °C had better performance than those dye-treated at 75 °C in ethanol solution in terms of shear strength (parallel loading). The results of this experiment can be explained by the fact that the bamboo fiber veneer was effectively softened by hydrothermal treatment. This process improved the permeation effect, which effectively promoted gluing but caused a serious loss in hemicellulose, resulting in a lower shear strength for samples dyed in ethanol.

The water resistance of bamboo-based fiber composites is illustrated in Fig. 7 for comparison.

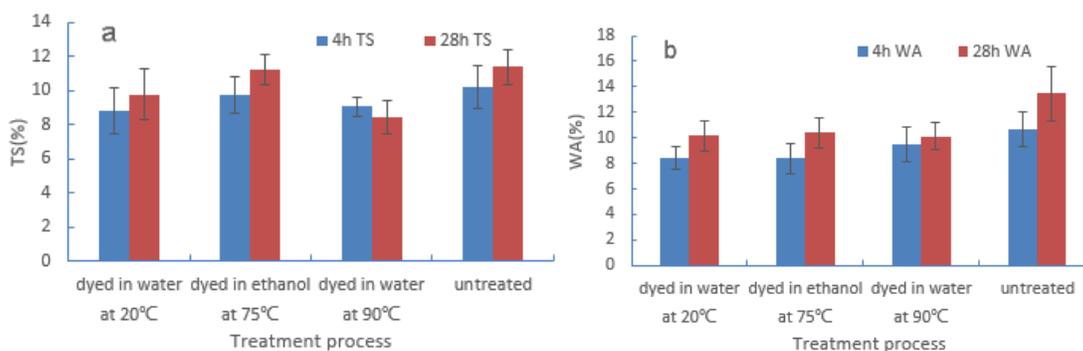


Fig. 7. Water resistance of bamboo-based fiber composites

According to Fig. 7a, the thickness swelling (TS) of dye-treated samples after 28 h was lower than that of untreated samples. The same trend also occurred for TS measured after 4 h. This change can be explained by the increase in the number of hydroxyl groups after the veneers were dyed in the solution. The changes in hygroscopicity and dimensional stability are caused by chemical changes in the cell wall fiber, such as a reduction in the hydroxyl group content of wood or the formation of cross-linking during heated treatment. Increasing crosslinking makes the molecules less elastic and reduces the possibility for enlarging cellulose microfibrils, thus reducing wood's ability to absorb water. This is in accordance with the water absorption results. Figure 7b shows dye-treated bamboo-based fiber composites samples demonstrated higher water absorption than untreated samples after 4 and 28 h of treatment, respectively.

CONCLUSIONS

1. The results showed that the color of bamboo fiber veneers reddens as a result of different oxidative and hydrolytic reactions taking place during dyed heat treatment. The dye treatment changed the color of the veneers from cream to light black with a decreased lightness factor (L^*). Color changes in the veneer are related to chemical changes, hemicellulose degradation, and slight carboxyl formation. The total color change of samples dyed in ethanol at 75 °C was higher than that of both samples dyed in water at 20 °C and those dyed in water at 90 °C. The greatest color change was observed in samples dyed in ethanol at 75 °C for 120 h. This might be related to the effect of ethanol, which increased the dye uptake through the internal pores of bamboo fiber veneer.
2. The MOR, MOE, and shear strength (parallel loading) of untreated bamboo-based fiber composite samples were remarkably better than those of dyed samples. Dyed samples also demonstrated higher dimensional stability than the untreated samples. Heating with ethanol negatively affected the mechanical strength of bamboo-based fiber composites more than heating with water did, which shows that bamboo-based fiber composites are more sensitive to these types of ethanol processes. Strength properties also decreased with dye treatment.

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