Weathering Performance of Dyed Bamboo Fiber Composites

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Flattened and crushed fiber-bundles of moso bamboo (*Phyllostachys pubescens*) were dyed with a water or alcohol-based dye at 20 °C, 75 °C or 90 °C for 72 h, followed by drying and coating with PF resin. Hot-pressed bamboo fiber composites (BFC) were manufactured, and the weathering performance of the composites made from dyed and natural bamboo material were tested over 90 days of the Autumn/Winter season in Beijing. Dye was able to penetrate the bamboo tissue to create a rich red color that degraded in color faster than BFC made from un-dyed bamboo. BFC made from un-dyed tissue remained a lighter but more consistent color over the weathering period. Dying the bamboo in ethanol-based dye was effective at pigmenting the product, but made it more susceptible to color degradation as well as greater mass and thickness loss due to the chemical degradation of the lignin caused by ethanol. Higher temperature of water dying treatment (90 °C) gave the greatest resistance to mass and thickness loss during outdoor exposure, and the bamboo may have benefitted from the longer period of 'heat treatment' during dyeing. Further work is needed to quantify dye leaching from bamboo tissue and to develop more water-tight and color-fast, weather resistant dyes for bamboo products.

Keywords: Bamboo; Bamboo Fiber Composites; Dyeing; Weathering; Color change

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INTRODUCTION

Bamboo is a widespread, abundant natural resource covering the tropical and subtropical climate zones of the Asia Pacific (*ca.* 67%), the Americas (*ca.* 30%), and Africa (*ca.* 3%) (Huang *et al.* 2017). It is regarded as one of Asia's most important natural resources with significant potential applications in the building industry (Pei *et al.* 2016). The fast growing, high strength and versatility, and low cost of bamboo makes it a viable alternative/complementary building material to wood. Bamboo is a highly functionally graded composite with unique and different material characteristics to wood. Moso bamboo is a hollow culm with dense and waxy outer walls, with the tissue lacking cross-gain pores or large pits (Chen *et al.* 2020), which makes it much more difficult to treat with preservatives, stains and dyes, chemical modification agents or adhesive resins compared with wood. Therefore, significant culm crushing and flattening are needed to improve bonding and further treatment of bamboo fiber composites.

Over the past two decades, several bamboo-based composites have been developed in China and for domestic and export applications as exterior landscape and structural

building materials. Bamboo fiber composite (BFC) is being used to produce tough, durable planks and decking, container flooring, garden landscaping, horse stable fences, and furniture on account of its low cost, high strength, durability, and recyclability (Yu *et al.* 2014, 2019; Li *et al.* 2016). At present, the appearance of bamboo products are mainly dictated by the natural color of bamboo. Carbonization can help darken the color of the composite but to an extent limited by the maximum allowable temperature (220 °C) of heat treatment without severely degrading the fiber strength (Zhang *et al.* 2013b). The purpose of this research is to examine the feasibility of dyeing the bamboo fiber bundles to significantly broaden the color options of BFC products and to evaluate the changes in material color and weight/dimensional stability due to weathering.

Bamboo has a similar carbohydrate constituent content to wood and therefore behaves similarly to wood when exposed to outdoor weathering. During outdoor exposure ultraviolet (UV) radiation from sunlight, and action by wind, frost, rain and dew, dust, hot and dry climatic conditions all contribute to weathering (Baysal *et al.* 2016; Li *et al.* 2019; Topaloglu 2019). While limited studies have been reported on weathering of bamboo and bamboo composites (Qin and Yu 2009; Kim *et al.* 2008; Zhu *et al.* 2015; Yu *et al.* 2018; Azwa and Yousif 2019), the weathering of wood and wood composites has been comprehensively studied and reviewed, *e.g.* (Evans 2012). When exposed to solar radiation, wood experiences rapid color changes primarily due to photodegradation of lignin (Cui *et al.* 2004; Hon and Minemura 2000; Kishino and Nakano 2004; Tolvaj and Faix 1995). In the later stages of weathering, larger chemical modifications and breakdown of the wood surface layer occurs (Ayadi *et al.* 2003; Hayoz *et al.* 2003). UV radiation accounts for just 5% of total solar energy but is the most significant of the environmental factors that contribute to the surface deterioration and weathering of wood (Ayadi *et al.* 2003; Feist and Hon 1984; Evans 2012). Weathering eventually creates a whitish/grey, fissured, and friable surface of exposed cellulose as the photodegradation of lignin continues and fragments erode out of the surface from the actions of water, wind, and dust. Surface erosion can be accelerated in certain situations by the actions of insects, *e.g.* wasps, harvesting the soft exposed cellulose for use as nest-building material. Surface discoloration can also occur from the surface colonization by fungi and bacteria which contain pigments, *e.g.*, melanin in blue stain fungi (Zink and Fengel 1988). For bio-based composites bonded with adhesives, in addition to wood surface degradation, weathering also causes the eventual breakdown of the bonds due to delamination from shrinkage/swelling forces of the wood elements, and possible UV degradation of the adhesive in the surface layers (Evans 1989a,b).

The objective of this study was to investigate the outdoor weathering performance of compressed bamboo fiber composite (BFC) made from bamboo subjected to three different dyeing regimes (water/alcohol solvent and ambient or >75 °C soaking temperature). BFC was manufactured from crushed flattened moso bamboo culms that was either not-dyed (natural) or dyed, and the changes in surface color, mass and dimensional stability monitored over a 90 days exposure period under mild weathering conditions.

EXPERIMENTAL

Materials

Dyes

Wood dyes may be based on either water or alcohol (*e.g.* ethanol) solutions, and these may be neutral, acidic or basic (Li *et al.* 2011; Jamili *et al.* 2019; Hu *et al.* 2016). Based on preliminary work (Hu *et al.* 2014), dying bamboo fiber bundles with acid-based dye produced acceptable color and luster but had reduced color penetration and uniformity. Furthermore, the acidic environment interfered with the pH conditions required for adequate curing of the phenolic resin adhesive during hot-pressing of BFC. Therefore, an alkaline stain (Basic Brown G) was selected and used in this study since it has positive charges in solution and therefore stronger affinity with the bamboo tissue with negative surface charges. Basic Brown G with a dye strength of 160%, was purchased from Baoding Baoman Chemical Plant (Hebei, China). The dye strength of a stain is related to its ability to impart stronger and/or different color to a material. The structure of Basic Brown G is shown in Fig. 1.

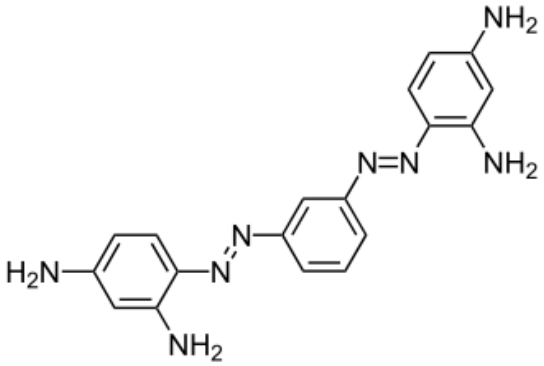


Fig. 1. Molecular structure formula of Basic Brown G

Bamboo

Moso bamboo (*Phyllostachys pubescens*) culms were harvested and converted to flattened fiber-bundles at a factory in Guangde, Anhui Province, located in southeast China. The bamboo was 4 y to 5 y old, and it had a diameter range of 70 mm to 90 mm, and a wall thickness range of 6 mm to 13 mm. The moisture content of the raw bamboo was 35% to 45% and density between 0.5 g/cm^3 and 0.6 g/cm^3. The culms were divided into three strips, which were then pressed through incised rollers and crushed into loose attached bundles and air-dried for a day or two to a moisture content of 10% before dyeing.

Adhesive

BFC was manufactured using a durable, thermosetting phenol-formaldehyde resin (PF) formulation. PFs are among the most used adhesives for exterior and semi-exterior products with excellent heat resistance, as well as water and weather resistant properties. The liquid PF resin was purchased from Beijing Dynea Chemical Co., Ltd. (Beijing, China), with a solids content of 46.56% and a viscosity of 42 cps.

Dye treatment

Flattened, dried bamboo bundles were steeped in a steam-heated dye bath shown schematically in Fig. 2. From preliminary work (Hu *et al.* 2014) it was found that dyeing in water at 90 °C and in ethanol at 75 °C (slightly below its boiling point of 78 °C) produced good color appearance and penetration depth. Thus, three treatments were used here: dyeing in water-based solution at 20 °C or at 90 °C and ethanol-based solution at 75 °C. The dye solutions were produced by mixing C.I. Basic Brown G with surface active agent (3g/L), acetic acid (3g/L), and sodium acetate (1g/L). The pH was controlled to between 5 and 6. The bamboo-to-dye solution was 1:10 by weight, and the total dyeing time was 72 h. This is three times as long as needed for wood veneer due to the lower permeability of bamboo to treatment solutions.

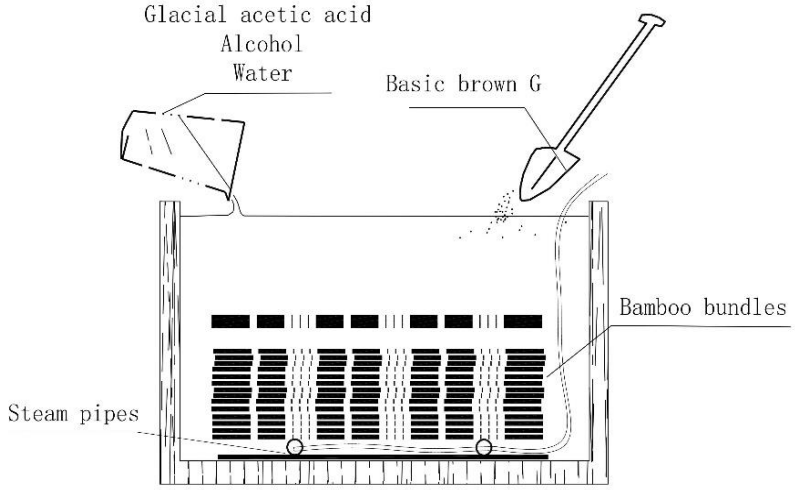


Fig. 2. Schematic diagram of dye bath for bamboo fiber bundles.

After dyeing, the bamboo bundles were oven dried at 75 °C for 24 h to 10 to 12% MC, weighed, and then immersed in a diluted PF resin bath for 5 minutes to achieve a target resin addition of about 15% (ratio of dry resin weight to dry bamboo weight). Resin coated bundles were air dried for 1 to 2 days then re-weighed to determine resin dosage. Bundles were assembled symmetrically in a mold along the grain direction, with the outer denser side of the culm wall being placed facing outwards to maximize bending strength properties.

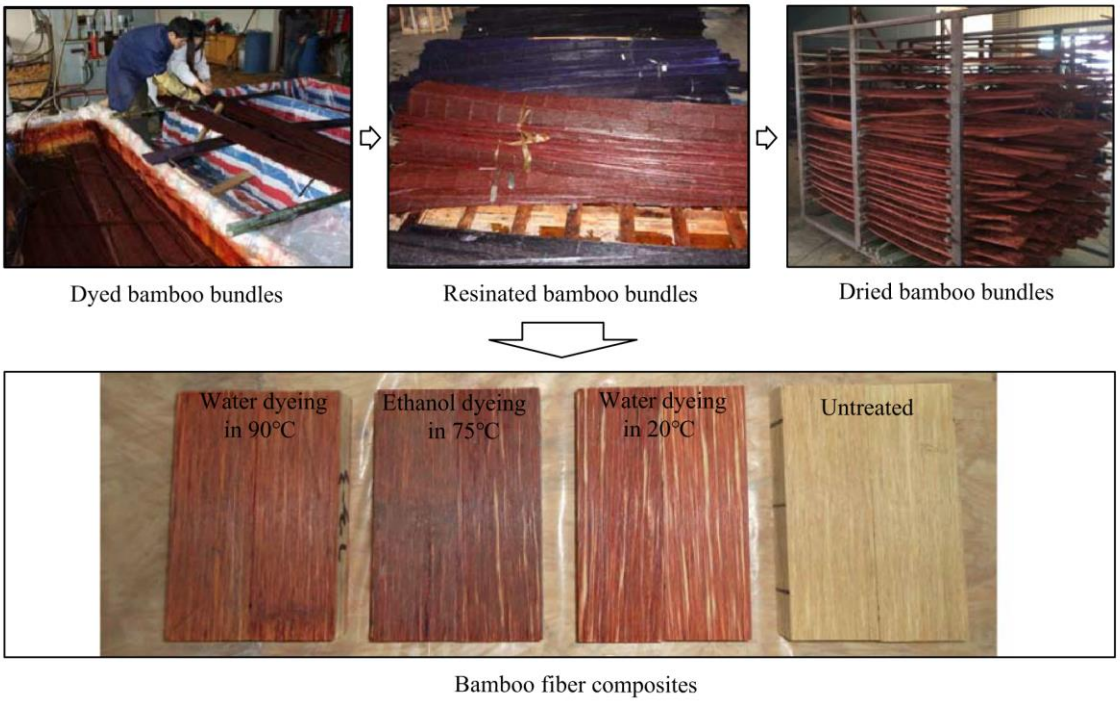


Fig. 3. Preparation of dyed BFC

The mold containing the mats was transferred to a single-opening hydraulic hot-press for pre-compaction and consolidation (Rao *et al.* 2019) followed by hot pressing at 140 °C for 30 min to a thickness of 20 mm. Hot press time was 1.5 min/mm x slab thickness for adequate resin curing. BFC slabs were cooled to room temperature for 30 min before demolding. BFC made using the same procedure from un-dyed bamboo bundles were used for the control. For each of the four treatment groups (un-dyed and three dyeing regimes), two BFC slabs were pressed. Images of the dyeing process and the appearance of the cut slabs are shown in Fig. 3.

Evaluation

Weathering experiments

The pressed BFC slabs were cut into six adjacent samples, each measuring 40 mm × 40 mm × 20 mm and conditioned to constant weight for 2 days in ambient room conditions. A total of 12 specimens per treatment were cut. The initial weight and thickness (h_0) at the center of each specimen were recorded. To visualize the dye particle distribution in the bamboo tissue, prepared surfaces of dyed bamboo and BFC specimens were examined using scanning electron microscopy (SEM; JSM-5500LV, JEOL Ltd., Tokyo, Japan).

A total of 48 weathering specimens were arranged on a south-facing weathering rack inclined 15° on the roof of the Chinese Academy of Forestry in Beijing (latitude 40.00N, longitude 116.14E), China. Specimens were exposed from November 2018 to February 2019 for a period of 3 months (90 days), with the climatic conditions from the China Meteorological Administration during this period shown in Table 1. Unexposed BFC made from un-dyed and dyed stock served as controls for the mass loss, thickness and color change experiments.

Table 1. Climate Conditions during the Experiment

Month	High Temperature (°C)	Low Temperature (°C)	Average High Temperature (°C)	Average Low Temperature (°C)
November	17	-5	12	1
December	13	-9	6	-5
January	10	-9	5	-5
February	9	-10	4	-4

During the weathering period the highest and lowest recorded temperatures were 17 °C and -10 °C, respectively. The total number of sunny days was 69, along with 8 cloudy days and 4 rainy/snowy days. The total estimated radiation for the period was 2325 MJ/m^2.

Every 15 days the specimens were recorded for color change, and change in mass and specimen thickness. The color changes in the specimens were determined using the standard chromaticity system $L^*a^*b^*$ color space (ISO/CIE 1976). Values for brightness (L^*), red to green spectrum (a^*), and yellow to blue spectrum (b^*) were recorded every 15 days using a spectrophotometer and colorimeter.

Total color difference ΔE in the CIE Lab space was calculated by determining the Euclidean distance between different colors according to Eq. 1,

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

The mass (m_n) and thickness (h_n), and the change relative to initial mass and thickness recorded and plotted. The mass loss was calculated according to Eq. 2,

$$M\,(\%) = (m_n - m_0) / m_0 \times 100\% \quad (2)$$

where M is the mass loss (%), m_0 is the original specimen mass (g), and m_n is the sample mass after treatment (g).

The % thickness change was calculated according to Eq. 3,

$$H\,(\%) = (h_n - h_0) / h_0 \times 100\% \quad (3)$$

where H (%) is the thickness loss (%), h_0 is the original specimen thickness (mm), and h_n is the thickness of specimen at time of measurement (mm).

RESULTS AND DISCUSSION

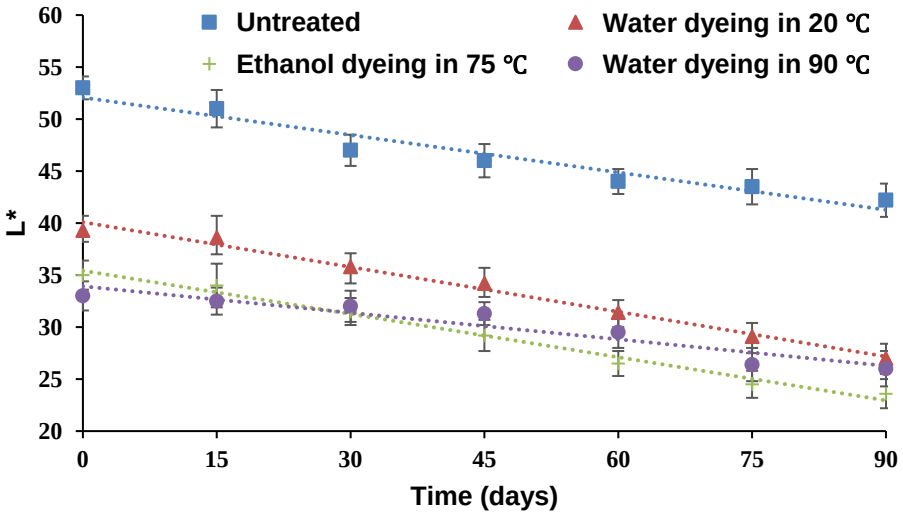
Color Change

Figure 4 shows the changes in color indices of dyed and natural BFC samples over the 90 days weathering period. While starting at different levels, the values of L^*, a^*, and b^* of the test groups all decreased linearly with exposure time. The ethanol-dyed specimens degraded in brightness, L^*, at a faster rate than the others (Fig. 4a). The possible reasons for this are discussed in the section on mass and thickness loss.

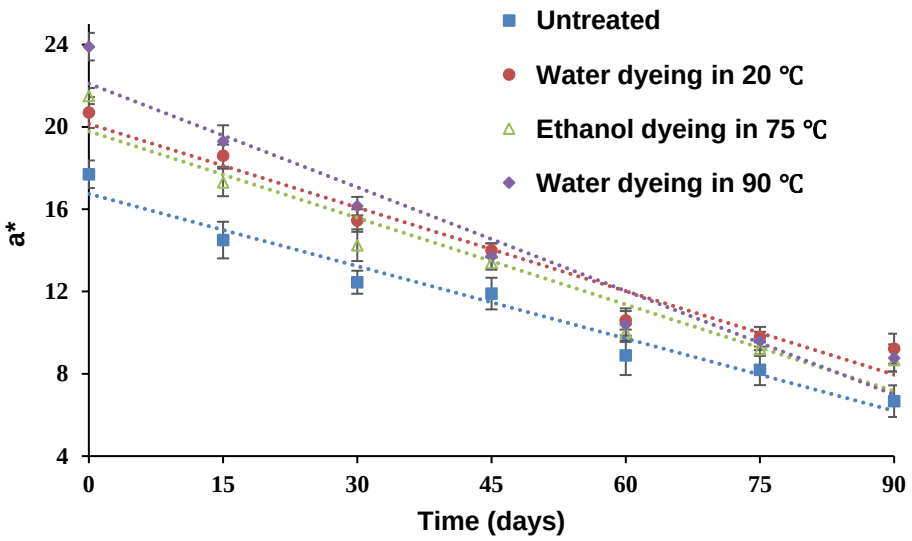
From Fig. 4a-c, all dyed samples started off with lower brightness indices (L^*) as well as red-green (a^*) and yellow-blue (b^*) than the un-dyed controls, with decline in L^* and a^* indices occurring at a similar rate for all groups over the 90 days weathering period. Change in b^* was minimal for the dyed groups compared with the natural BFC. Adding

dye to the bamboo reduces the rate of color change in the red-green spectrum (Chee *et al.* 2019). The total change in color ($\triangle E^*$) of the surface was greater if the bamboo was dyed before manufacturing BFC, as indicated in Fig. 4d. The color change in the un-dyed BFC tapered off after about 60 days of weathering, but the fast rate of change continued in the dyed specimens until about day 75. Despite starting out a darker color, the dyed material was less color-fast than the natural material, fading from its rich reddish color to a lighter brown as seen in Fig. 5a. The cumulative % change in total spectra (ΔE) for each group are indicated in Fig. 5b, at 15.8% for un-dyed BFC and 19.2 to 20.3% for dyed BFC at the end of only 90 days exposure in low-intensity (Winter) conditions. In previous work by Hu *et al.* (2015), color changes in dyed bamboo exposed to weathering were thought to be due to a combination of oxidation of bamboo components such as lignin and the decomposition of the dye. It should be noted that the phenolic resin coating the bamboo mat bundles is also reddish-brown and may have also undergone photo-degradation during weathering.

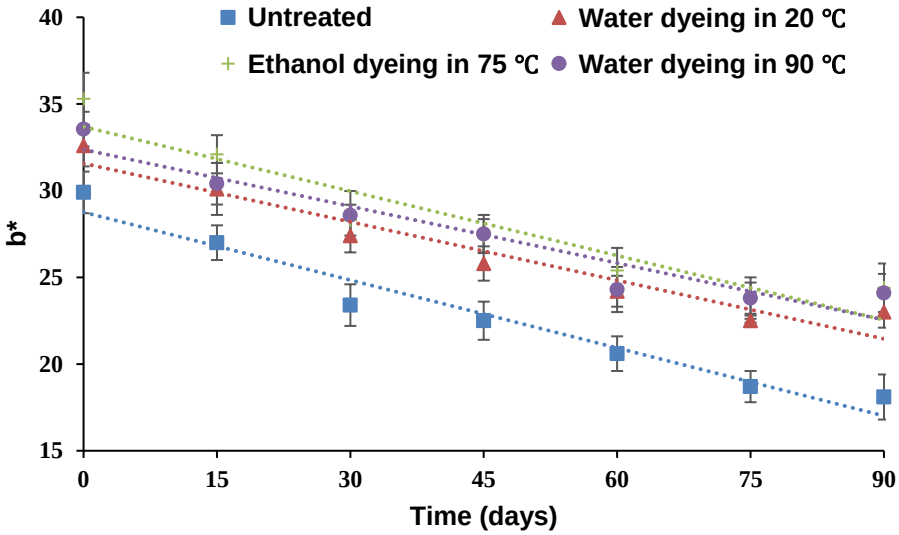
The change in color of the exposed surface is primarily due to UV induced photo-degradation of lignin. Light colored woods, including bamboos, darken in color initially and then become yellow or brown due to the accumulation of photodegraded oligomeric lignin constituents (Cui *et al.* 2004; Kishino and Nakano 2004b; Tolvaj and Faix 1995; Evans 2012). UV also destroys the luminescent groups of the polymer chain, and it alters the structure of other polymers, mainly lignin at surface, which in turn leads to degradation of brightness and reflectance of the red-green and yellow-blue spectra, and erosion of the degraded surface.



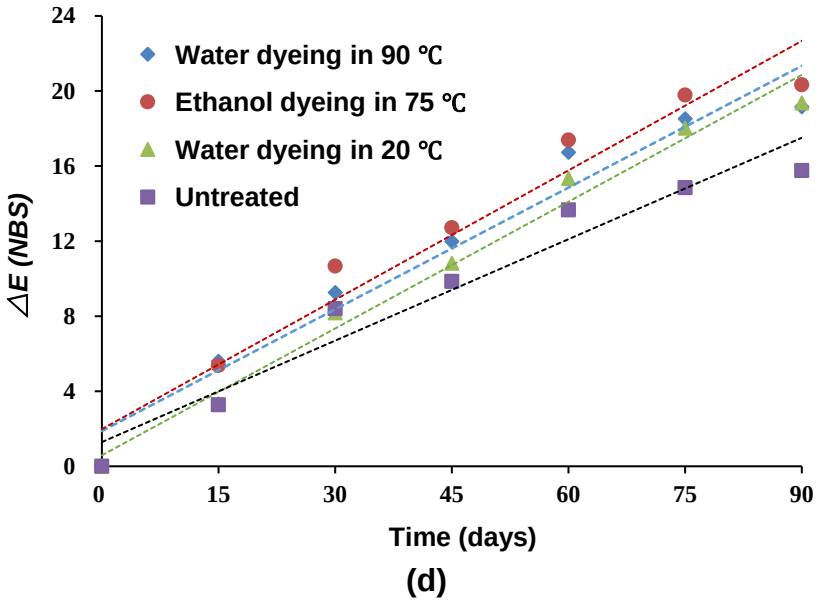
(a)


Untreated
Water dyeing in 20 ℃
Ethanol dyeing in 75 ℃
Water dyeing in 90 ℃
a*
24
20
16
12
8
4
0
15
30
45
60
75
90
Time (days)

(b)

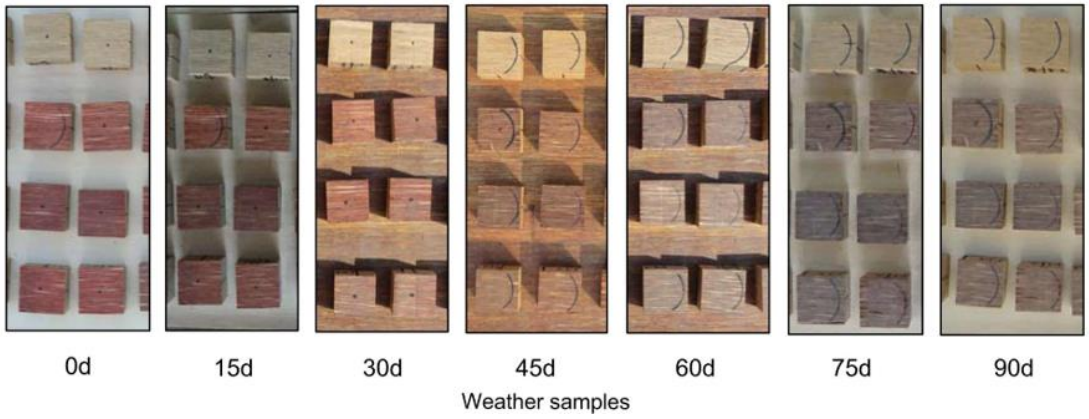

Untreated
Water dyeing in 20 ℃
Ethanol dyeing in 75 ℃
Water dyeing in 90 ℃
b*
40
35
30
25
20
15
0
15
30
45
60
75
90
Time (days)

(c)

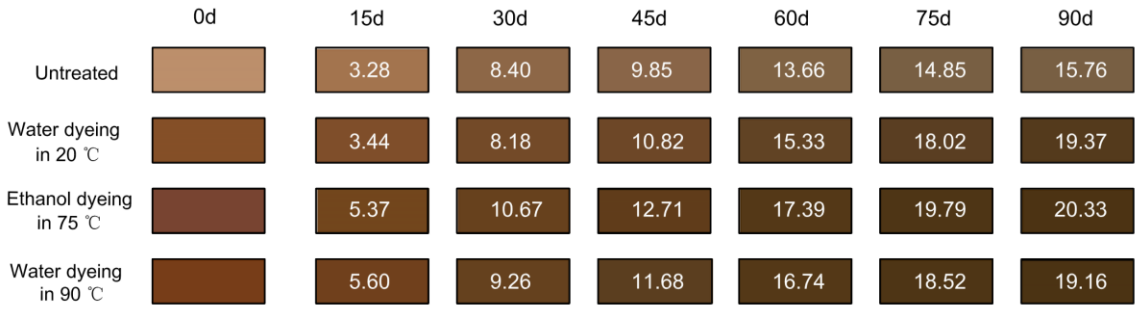


(d)

Fig. 4. Changes of color parameters of BFC samples during the period of 90 days of outdoor exposure: (a) brightness, *L**; (b) red-green spectrum, *a**; (c) yellow-blue spectrum, *b**; (d) Total color difference (△*E**).



(a)



(b)

Fig. 5. (a) visual color change in weathering specimens, (b) average cumulative change in surface color (Δ*E*)

Wood based bio-composites exposed to outdoor weathering for 6 or 12 months became grey as photodegraded lignins are leached from the wood, resulting in surface layers that are rich in light colored cellulose (Arndt and Willeitner 1969).

Change in Mass and Thickness

The % mass and thickness loss values for the weathering specimens are shown in Fig. 6.

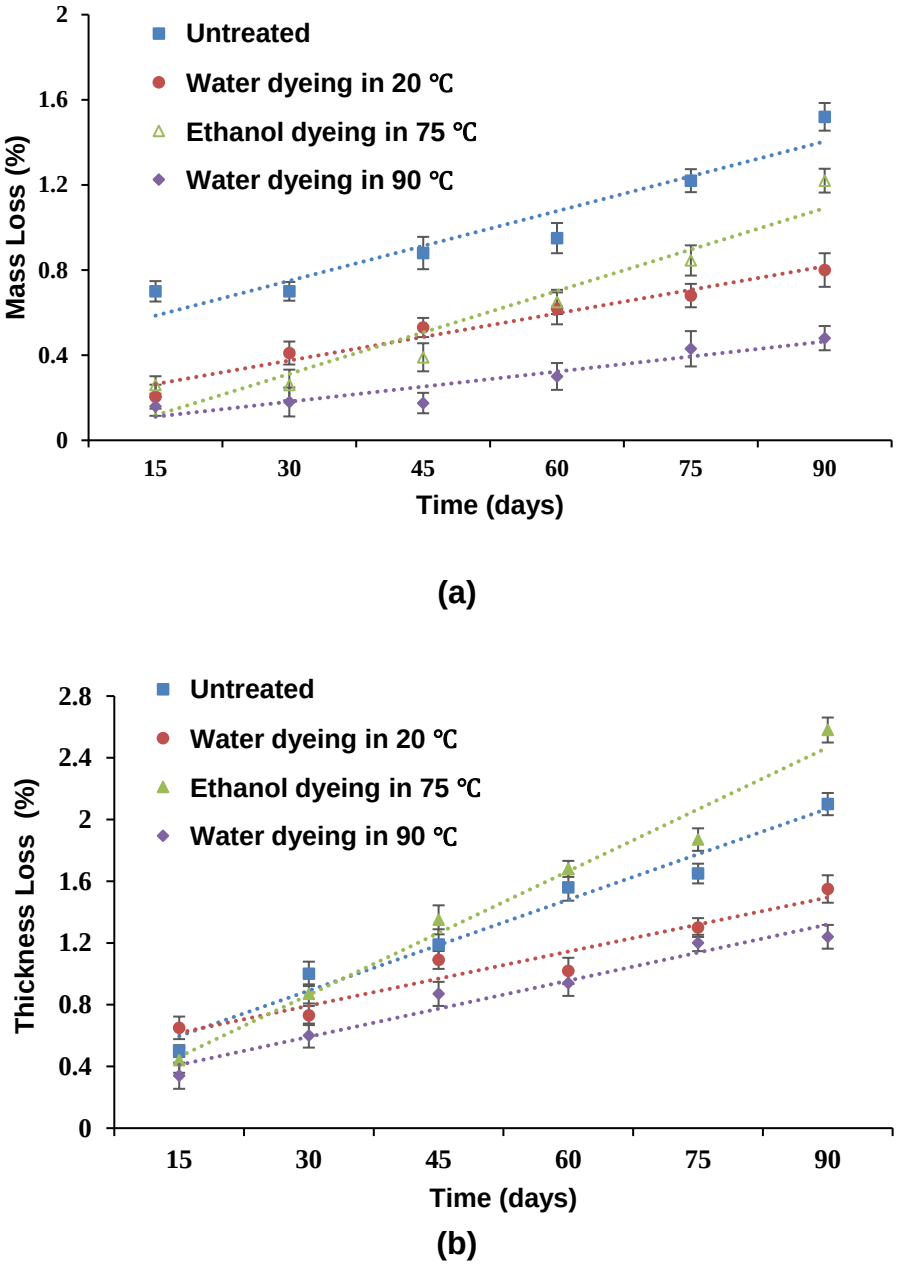


Fig. 6. (a) % mass loss; (b) % thickness loss in BFC weathering specimens over 90 days

From Fig. 6a, the % mass loss was not quite linear but gradually increased with weathering time. Mass and thickness losses were greater if the BFC was made from

ethanol-dyed bamboo. These suffered even greater thickness loss than the undyed bamboo, whereas the specimens of water-dyed BFC were more resistant to reduction in mass and thickness during weathering. Ethanol not only allows the dye to penetrate more effectively into the bamboo tissue but it also creates a partial pulping effect through the chemical degradation of lignin (Botello *et al.* 1999; Hamzah *et al.* 2020), which likely caused it to degrade faster under UV light, contributing to greater material loss. The presence of degraded lignin in the ethanol-dyed BFC also likely explains its more rapid color degradation, with the lignin undergoing faster photodegradation and loss from the surface. The dye only coats the insides of cells and is weakly bonded to the bamboo tissue compared with its natural pigments, and more easily lost over the course of weathering, further contributing to fading and weight loss during the period. With the few rainy days during the weathering period, samples would have had little chance to absorb moisture, and the mass and thickness losses were believed to be primarily caused by erosion and drying shrinkage.

BFC made from natural un-dyed bamboo was also higher in mass and thickness losses during weathering, suggesting the correct treatment procedure (solvent and temperature) with water-based dye can be effective at reducing the susceptibility of BFC products such as outdoor decking to weathering, particularly if the bath temperature is higher, *i.e.* 90 °C. The dyeing process in the hot water bath for three days could be also considered to be a heat treatment, which is used to increase the dimensional stability of wood products (Zhang *et al.* 2013a). Previous studies (Hu *et al.* 2014; Liu *et al.* 2020) have shown that dyeing at higher temperatures increases the color fastness of the material. Higher temperatures can increase the reactivity and bonding between dye molecules and free radicals on the material surface (Hu *et al.* 2014).

Micro Structural Change

SEM micrographs of bamboo fiber material before and after weathering are shown in Fig. 7a for un-dyed and 7b for dyed fiber before aging. Higher magnification views of the inside surface of cell lumens are shown Figure 7c and Fig. 7d for un-dyed and dyed tissue. Basic brown G dye is suitable for dying bamboo tissue with very small pores (Kim *et al.* 2016); however, weathering can still be detrimental to the surface of dyed wood tissue (De Lima *et al.* 2020). As shown in Fig. 7a, c, the surface of the untreated bamboo was relatively smooth, whereas a large number of closely spaced spherical dye particles can be seen covering the cell lumen surfaces of the dyed tissue. The dye particles were mostly less than 1μm (Fig. 7d). This indicates it is possible for the dye particles to penetrate transversely into the cell lumens via any open pit pores connecting the cells, which from Fig. 7c are approx. 2 μm across.

The appearance under SEM of un-dyed and dyed tissue after 90 days of weathering is shown in Fig. 8a and b, respectively. From Fig. 8b, it can be seen that the exposed inner lumen wall with the dye lining had become wrinkled from weathering and fungal hyphae and spores (white particles and fine filaments) are evident on the surface. It was not clear in the absence of surface leaching tests how much of the dye was lost from the exposed surface after weathering. Fungal hyphae, which can be seen in the weathered, dyed BFC specimens in Fig. 8b, on the surface can also contribute to surface discoloration (Feist 1990a).

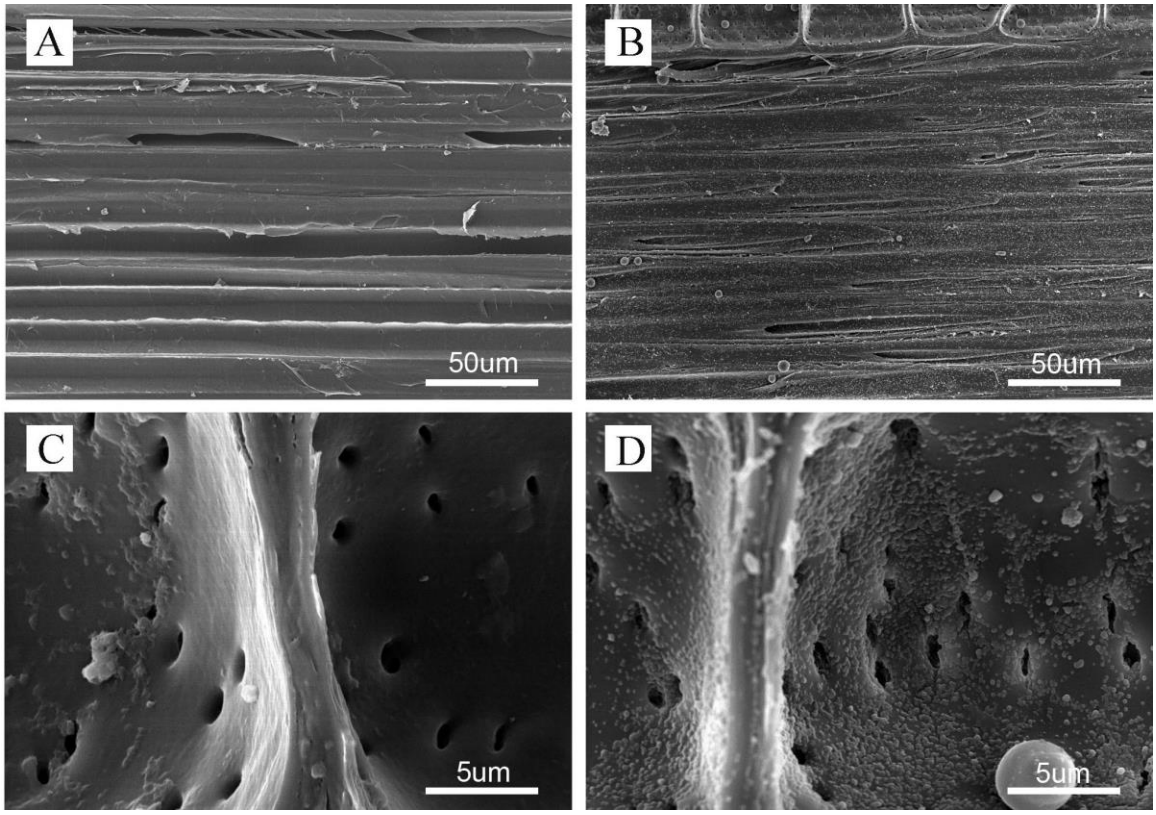


Fig. 7. Scanning electron microscopy images of the untreated and dye-treated bamboo fibers

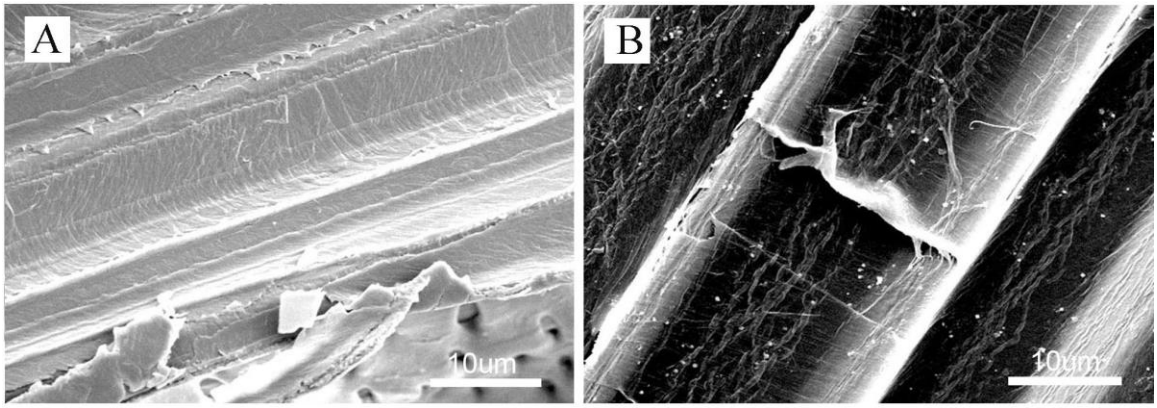


Fig. 8. Scanning electron microscopy images of the untreated (a) and dye-treated BFC (b) after 90 days weathering

CONCLUSIONS

Crushed, flattened bamboo fiber bundles were dyed with basic brown G wood dye dissolved in water at 20 °C or 90 °C or in ethanol at 75 °C before drying, coating with PF resin, and hot pressing into BFC decking material. Bamboo, being less permeable, required a three times longer dying period (3 days) compared with wood; however SEM images indicated that the dye particles were small enough to move through the narrow pit pores of bamboo tissue and coat the insides the lumens. Weathering trials in Autumn/Winter in

Beijing over 90 days showed that the dyed product was darker to start with but experienced faster color degradation than BFC made from un-dyed bamboo, which remained a lighter but more consistent in color over the weathering period. Color degradation and mass and thickness loss were greatest in the ethanol-dyed BFC, which was attributed to the chemical degradation of the lignin component in the bamboo caused by ethanol. The higher temperature water dying treatment (90 °C) gave the greatest resistance to mass and thickness loss during outdoor exposure and may have benefitted from the longer period of 'heat treatment' of the bamboo tissue. Further work is needed to quantify dye leaching from treated bamboo tissue and to develop more water-tight and fade-resistant dyes for exterior bamboo products. Weathering tests should also be extended to include harsher summer conditions with higher UV index, and over multiple years.

ACKNOWLEDGMENTS

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