The Effect of Chemical Treatment on Green Color Preservation and Surface Characteristics of *Neosinocalamus affinis* Bamboo

Lin Chen,[#,a] Lu He,[#,a] Jiulong Xie,*[,a] Zhongpeng Qiu,[a] Jinqiu Qi,[a] Yongze Jiang,[a] Hui Xiao,[a] Yuzhu Chen,[a] Cornelis F. de Hoop,[b] and Chung-Yun Hse [c]

Bamboo culms or bamboo plywood possessing a native green surface color are highly valuable for use in interior decoration. However, the green surface color of bamboo can easily become discolored in ambient environments without any protection. In order to maintain the green surface color of *Neosinocalamus affinis* bamboo, this study used copper sulfate ($CuSO_4$), phosphoric acid (H_3PO_4), and acetic acid (CH_3COOH) solutions as protectors. The changes in surface color and characteristics of the bamboo samples before and after the treatment were evaluated. The results showed that the *N. affinis* bamboo samples treated with 2% $CuSO_4$ solution at 80 °C for 3 h exhibited an attractive green color, with a^* and b^* values of -7.61 and 5.13, respectively. The indoor durability test result indicated that bamboo samples treated with $CuSO_4$, $CuSO_4/H_3PO_4$, and $CuSO_4/CH_3COOH$ solutions all had excellent color durability. After the treatment, the relative copper content on the bamboo surface slightly increased, while the magnesium content decreased. The chlorophyll structure was changed, and reactions between chlorophyll and copper ions may have occurred. The silicon was almost completely removed, and the surface structure of the treated bamboo changed, resulting in good permeability for the treatment.

Keywords: Neosinocalamus affinis; Green color preservation; Chemical treatment; Surface characteristics

Contact information: a: College of Forestry, Sichuan Agricultural University, Chengdu, Sichuan 611130, PRC; b: School of Renewable Natural Resources, Louisiana State University Agricultural Center, Baton Rouge, Louisiana 70803, USA; c: Southern Research Station, USDA Forest Service, Pineville, Louisiana 71360, USA; [#] Authors contributed equally to this manuscript;
** Corresponding author: Jiulong Xie, jxie6@sicau.edu.cn*

INTRODUCTION

There are approximately 1,250 species of bamboo that are mainly found in Asian countries such as China. These bamboo species provide an annual output of 6 to 7 million tons (Liu *et al*. 2018a). As a green and renewable material, bamboo has attracted increasing attention due to its low cost and excellent performance (Sukmawan *et al*. 2016; Hao *et al*. 2018). Bamboo has been widely used in building materials and floor processing, composites, bioenergy, and other fields (Felisberto *et al*. 2018; Liu *et al*. 2018b). *Neosinocalamus affinis*, a cluster bamboo with a culm height of 8 m to 12 m, mainly grows in the Chinese provinces of Sichuan, Guizhou, Yunnan, Guangxi, Hunan, west Hubei, and south Shaanxi, where it is widely used in the daily lives of local residents. From an industry perspective, *N. affinis* bamboo is an important raw material for pulping, construction, furniture manufacturing, and other bamboo fiber-based materials (Wang *et al*. 2009; Sun *et al*. 2011). For example, the bamboo plywood made from *N. affinis* has excellent physical and mechanical properties (Qi *et al*. 2014). The bamboo fiber bundle reinforced composites prepared by *N. affinis* exhibited lower water absorption, higher structural stability, and higher mechanical properties (Xie *et al*. 2016). The scrimbers bamboo prepared by *N. affinis* is not only used in indoor building materials, but also in the fields of wind turbine blade, building structure material, container floor, and cement mold (Yu and Yu 2013).

Freshly harvested *N. affinis* bamboo culms have been employed as materials for indoor and outdoor utilization in the Sichuan province of China, because of its abundance and excellent properties. However, due to the influences of heat, light, and moisture, the surface color of *N. affinis* bamboo gradually changes from green or dark green to gray or yellow without any treatment, resulting in the reduction in decoration ability and values (Wang and Ren 2008; Zhang *et al*. 2013a). Green paints have been applied to bamboo in order to restore the original color of fresh bamboo. However, existing epidermis and silicon on the bamboo surface has provided poor paint adhesion, resulting in uneven painting and cracks. The treatment of bamboo with wood preservatives such as chromated copper arsenate (CCA) and ammoniacal copper arsenate (ACA) could preserve the green color of bamboo for a certain period (Chang and Wu 2000; Chang and Yeh 2001). For example, the green color of *Dendrocalamus latiflorus* bamboo can be preserved after treatment with copper salt aqueous solution (Wu *et al*. 2002b). The use of quaternary ammonium copper and copper oxazole preservatives can also protect the green color of bamboo (Chung *et al*. 2005). Copper salt preservation treatment combined with chromium-based oxide and H_3PO_4 was found to better protect the green color of bamboo (Chang and Yeh 2001; Chang *et al*. 2002). However, since chromium and arsenic are toxic and are not environmentally friendly, their use has been gradually restricted (Yoshida *et al*. 2004; Deng and Chen 2014; Chen *et al*. 2018). Moreover, an alcohol-based solution was also explored to protect the bamboo color (Wu *et al*. 2004). Although this process was found to be efficient and environmentally friendly, its high processing cost and safety concerns limit its industrial application.

Although some bamboo species have been treated with chemicals for green color protection purposes, the properties of bamboo species differ from one another, so an efficient green color preservation process for *N. affinis* bamboo is still needed. Since arsenate is toxic, a treatment method avoiding using arsenate is preferable. Therefore, this study examined the color parameters and surface characteristics of the treated *N. affinis* bamboo using copper sulfate ($CuSO_4$) solution. This study investigated whether $CuSO_4$ solution with proper pretreatment was a good option for the green color preservation of *N. affinis* bamboo for interior decoration use.

EXPERIMENTAL

Materials

Three-year-old *N. affinis* bamboo samples with dimensions of 50 mm long × 40 mm wide × thickness were collected from Chengdu, China. The samples were stored in the dark at 4 °C before use. All chemicals were commercially purchased and used as received.

Treatment Process

The bamboo samples were first treated in a 1% potassium hydroxide solution maintained at 80 °C for 30 min in a water bath. The treated samples were thoroughly washed with distilled water until the solution reached a neutral pH. The alkali-treated samples were then treated with $CuSO_4$ solutions having different concentrations. The conditions for each treatment are listed in Table 1. The treated samples were oven-dried at 60 °C for 12 h.

Table 1. List of Conditions for Each Chemical Treatment on *N. affinis* bamboo

Solution Type	Concentration (%)	Time (h)	Temperature (°C)
$CuSO_4$ Aqueous Solution	1, 2, 4	1, 3, 5	40, 60, 80
$CuSO_4/H_3PO_4$ Solution (50:50)	0.5, 1, 2	3	80
$CuSO_4/CH_3COOH$ Solution (50:50)	0.5, 1, 2	3	80

Color Measurement

The color measurement was carried out immediately after the sample had been oven-dried. The color measurement was carried out by using a high-quality computer colorimeter (NR69CP; Shenzhen Threenh Technology Co., Ltd., Shenzhen, China) with a D65 light source and a measuring aperture of 3 mm. For each sample, 25 points were measured, and the mean values was taken. According to the TAPPI T524 om-79 (1979), in the CIELAB color system, L^* is the value on the white/black axis, a^* is the value on the red /green axis, and b^* is the value on the blue/yellow axis. The a^* value best represents the difference in green and red, where a smaller a^* value indicates a greener sample color.

Color Durability Test

Outdoor durability test

The treated samples were placed on a board for the outdoor durability test. To ensure that the samples could receive the maximum light energy, the board was placed on an inclined plane at an angle of 30° according to the local latitude (Fig. 1). The samples were maintained without any cover for 42 d (September 11, 2018 to October 23, 2018). The average precipitation, the pH value of the precipitation, and the average temperature during the test period were 102.6 mm, 7.43, and 18 °C, respectively.

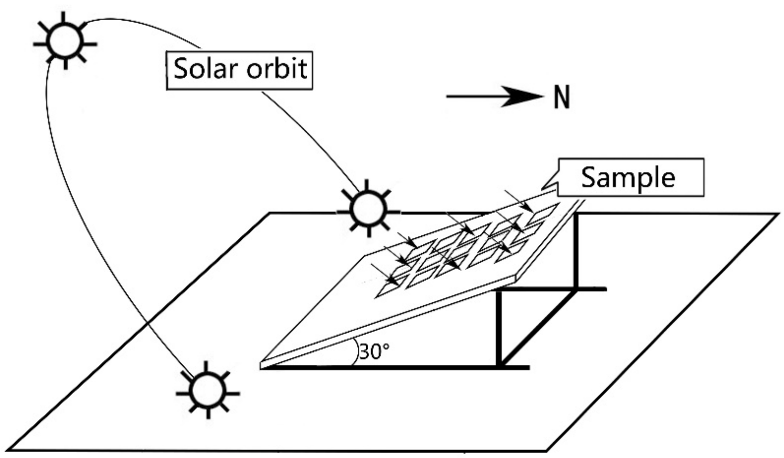

Fig. 1. Solar orbit and sample placement

Indoor durability test

For the indoor durability test, the samples were placed in an indoor environment with an average temperature of 23 °C and an average humidity of 80%. The test period was 42 d as well.

SEM Images

Scanning electron microscopy (SEM) energy-dispersive X-ray (EDX) (SU-3300; Hitachi High-Technologies Co., Ltd., Nake, Japan) analysis was used to observe the surface structure and detect the inorganic elements of the *N. affinis* bamboo surface samples before and after the treatment. The green bamboo on the surface was peeled off, and samples that were 5 mm × 5 mm × 1 mm in size were prepared. The samples were oven dried at 103 °C until a constant weight was reached. After drying, a conductive carbon double-sided adhesive tape was used to bind the sample on a metal support. Gold was sputtered and coated with a thickness of 3 nm to 6 nm on the sample surface prior to the test.

Chlorophyll Analysis

For the chlorophyll structure analysis, a referenced method was used (Chang *et al*. 1998). Samples were scraped from the green surface of the bamboo, and a 2 g sample was weighed and mixed with 95% ethanol. Silica and calcium carbonate were added for grinding. When the mixture reached a white viscous state, the mixture was filtered and 15 mL of 95% alcohol was added to the filtrate. The absorbance of the liquid was measured using an ultraviolet (UV) spectrophotometer (UV-4802; UNICO, Dayton, NJ, USA).

RESULTS AND DISCUSSION

Chromaticity Changes

Figure 2 shows the effect of the $CuSO_4$ concentration on the CIE $L^*a^*b^*$ values of the samples.

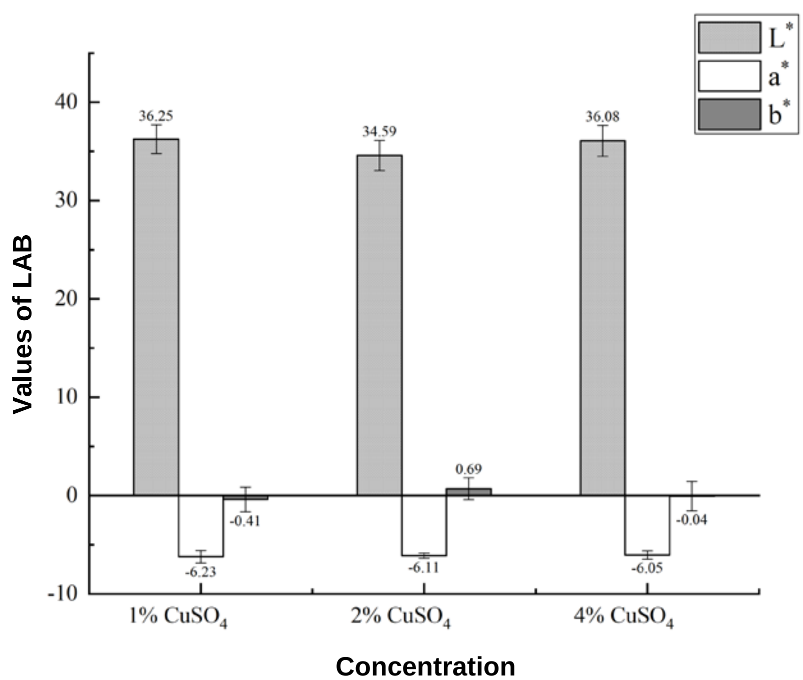

Fig. 2. Effect of $CuSO_4$ concentration on the color parameters of bamboo samples treated at 60 °C for 3 h

The results showed that the a^* values of the samples treated with different concentrations were all smaller than -6.0, and the a^* values slightly decreased as $CuSO_4$ concentration increased. The influence of $CuSO_4$ concentration on the b^* values of the samples was not obvious. The b^* values of the samples treated with 2% $CuSO_4$ solution were near 0.69, which means that the treatment had no obvious yellow tendency. The b^* values of the samples treated with 1% and 4% concentrations of $CuSO_4$ were below 0. By comparing the a^* values of the samples, the treatment with 2% $CuSO_4$ was more suitable.

Figure 3 shows the effect of the treatment time on the CIE $L^*a^*b^*$ values of the samples. The a^* values for the samples treated for 1 h, 3 h, and 5 h were -6.91, -7.61, and -6.89, respectively. The a^* values of the samples treated for 3 h were the lowest. By prolonging the treatment time, the a^* values first increased and then decreased, while the b^* values first decreased and then increased. The b^* values for the samples treated for 3 h was 5.13, which was higher than those treated for 1 h and 5 h. The above results showed that the samples with 3 h treatment can obtain a relative low a^* values and a high b^* values.

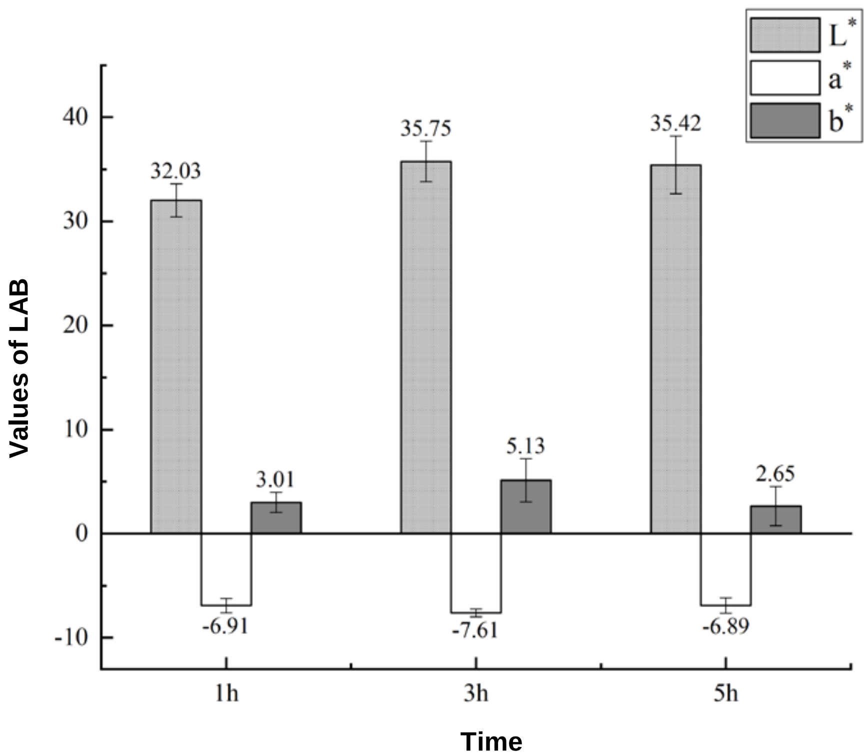

Fig. 3. Effect of treatment time on the color parameters of bamboo samples treated with 2% $CuSO_4$ at 60 °C

Figure 4 shows the CIE $L^*a^*b^*$ values of the samples with respect to the treatment temperatures. By increasing the treatment temperature, the a^* values of the samples first increased and then decreased. When the treatment temperature was maintained at 80 °C, the sample had an a^* value of -7.61 and a b^* value of 5.13. These values were larger than the samples treated at 40 °C and 60 °C, which indicated that the samples treated at 80 °C were much greener and brighter. A higher temperature may also have a negative effect on the mechanical properties of bamboo for constructional utilizations, so a treatment temperature higher than 80 °C should be avoided in this process (Zhang *et al*. 2013b).

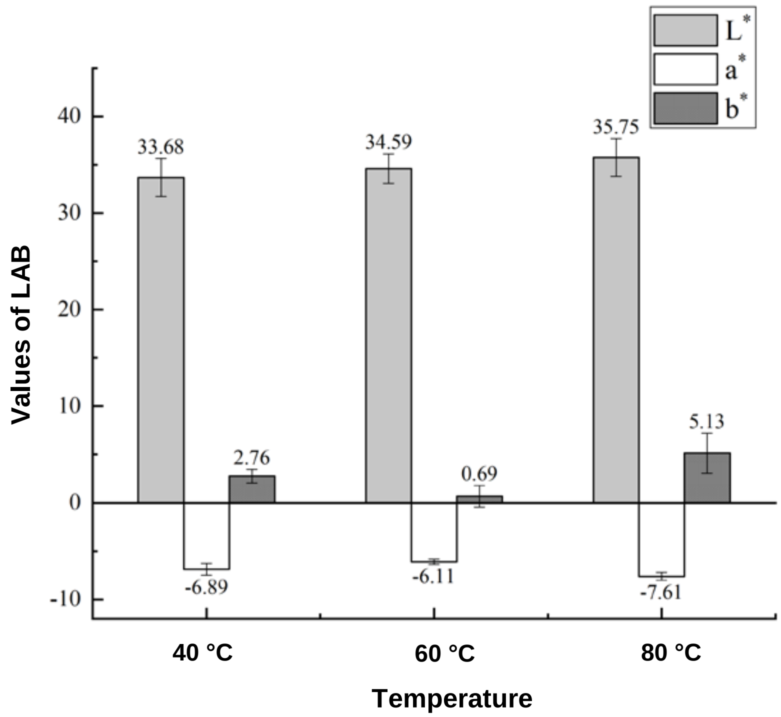

Fig. 4. Effect of temperature on the color parameters of bamboo samples treated with 2% $CuSO_4$ for 3 h

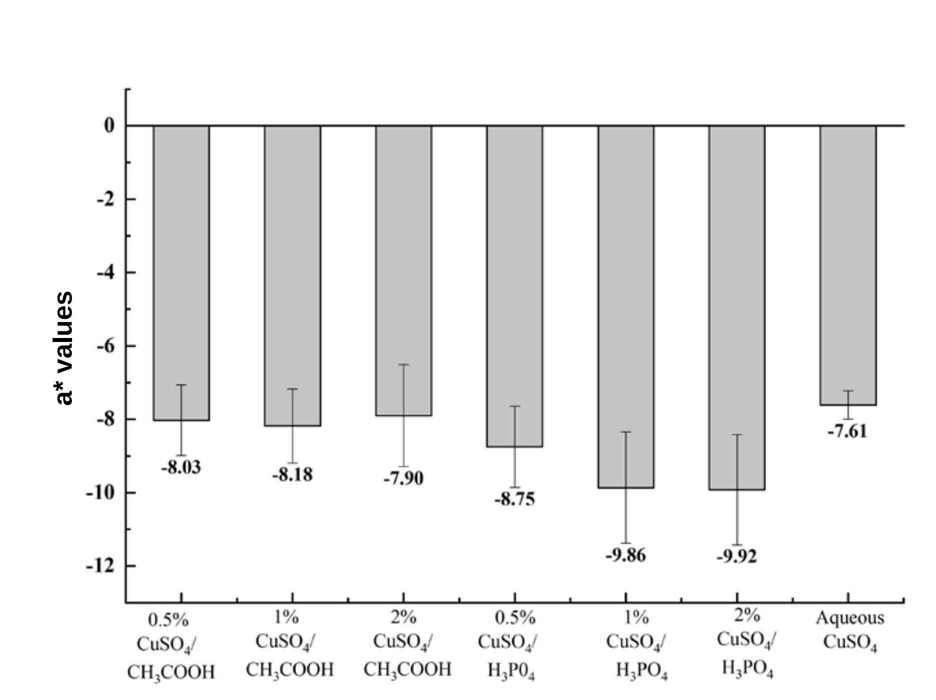

Fig. 5. Effect of solution type on the color parameters of bamboo samples treated at 80 °C for 3 h

Figure 5 shows the a^* values of the bamboo samples treated in different $CuSO_4$ solutions at 80 °C for 3 h. The samples treated in the $CuSO_4/H_3PO_4$ and $CuSO_4/CH_3COOH$ solutions had less negative a^* values than those treated in the $CuSO_4$ solution. The a^* values for the samples treated in the $CuSO_4/H_3PO_4$ solution were less negative than those treated in $CuSO_4/CH_3COOH$ solution. The changes in the concentration of the binary solutions had no obvious influence on the a^* values.

Durability Test

Figure 6 shows the a^* values of the bamboo samples before and after the indoor and outdoor exposure tests. After 42 d of indoor exposure, the increments in the a^* values of the samples treated with $CuSO_4$ aqueous solution, $CuSO_4/CH_3COOH$ solution, and $CuSO_4/H_3PO_4$ solution were 0.32, 1.42, and 0.14, respectively. While the change in the a^* values for the indoor exposure samples was not obvious, the change in the a^* values for the samples after 42 d of outdoor exposure was obvious. The increments in the a^* values of the outdoor exposure treated with $CuSO_4$ aqueous solution, $CuSO_4/CH_3COOH$ solution, and $CuSO_4/H_3PO_4$ solution were 6.37, 7.43, and 11.75, respectively. This result revealed that although lower a^* values can be achieved in $CuSO_4/H_3PO_4$ or $CuSO_4/CH_3COOH$ solution, after the exposure test, all the samples had higher a^* values than those treated with $CuSO_4$ aqueous solution. This may be since acidity could slightly damage the original bamboo structure, resulting in a lower resistance ability to aging.

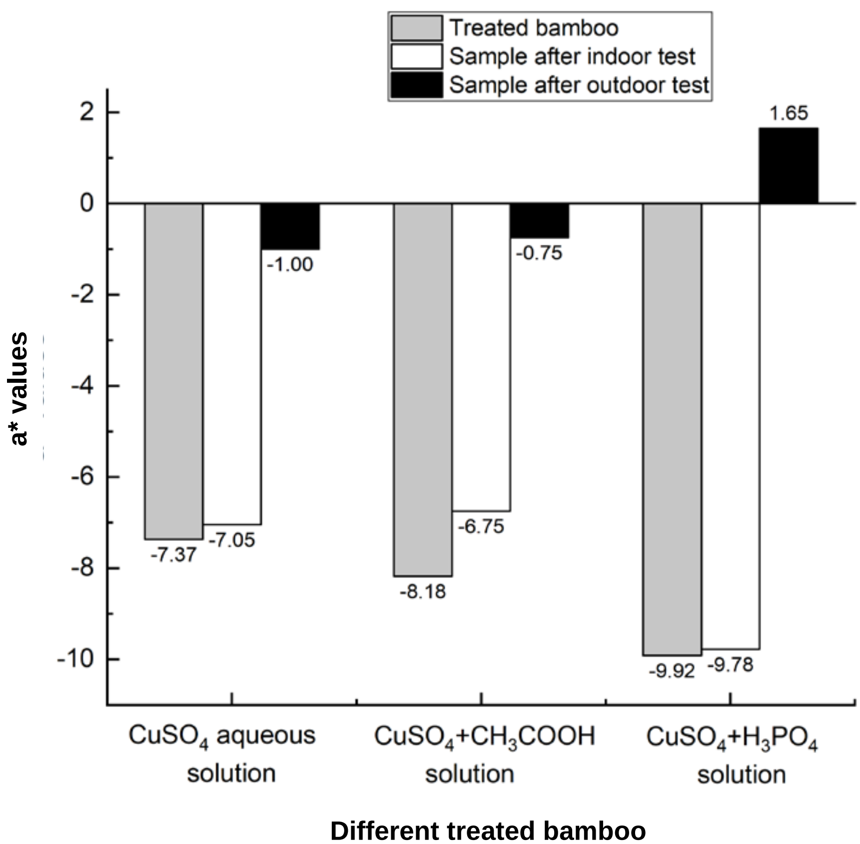

Fig. 6. Changes in *a** values of the bamboo samples before and after the exposure test for 42 d

From the above results, it was concluded that the *N. affinis* samples treated in $CuSO_4$ aqueous solution had higher a^* values after the indoor exposure test. The samples treated with $CuSO_4$ aqueous and $CuSO_4/H_3PO_4$ solution had minimal changes in their a^* values, indicating that these samples had great potential in interior decoration utilization. However, after the outdoor exposure test, only the bamboo samples treated with $CuSO_4$

aqueous solution had negative a^* values, revealing that the *N. affinis* bamboo samples treated with $CuSO_4$ aqueous solution could be used for a considerable period in an outdoor environment without any cover or protection. However, the greater a^* values of the outdoor test sample than that of the indoor test sample may be because that chlorophyll is degraded and discolored by sunlight and rain (Cubas *et al.* 2008).

Chlorophyll Structure Analysis

Figure 7 shows the UV-visible absorption spectra of the chlorophyll solution extracted from the fresh bamboo and the samples treated with $CuSO_4$ aqueous solution. There were two absorption peaks near 437 nm and 665 nm in the spectra of the ethanol extract from the fresh bamboo, which represent the typical structure of chlorophyll. No absorption peaks were observed in the range of 350 nm to 750 nm band in the spectrum of the air-dried bamboo (Sartory and Grobbelaar 1984), indicating that the chlorophyll in the air-dried bamboo samples was destroyed (Qudsieh *et al*. 2002). This result confirmed that the existing chlorophyll on the bamboo surface was an essential condition for exhibiting the green color of bamboo. There were also two absorption peaks in the ethanol extract of *N. affinis* bamboo samples treated with $CuSO_4$ aqueous solution. Compared to the peak positions on the spectrum of the fresh bamboo, the peaks of the treated bamboo shifted to lower band ranges, indicating that the chemical structure of the chlorophyll was different between the fresh green bamboo samples and the treated bamboo samples (Wu *et al.* 2002a). Usually, chlorophyll color changes from green to yellowish brown by losing the magnesium ions under light oxidation, temperature changes, and enzymes (Cubas *et al.* 2008). A hypothesis could be made that the chlorophyll was first changed by losing the magnesium ions under the treatment temperature of 80 °C, then copper ions reacted with the chlorophyll forming the copper chlorophyll (Guzmán *et al*. 2002). Since the copper chlorophyll had better color stability to acid and light (Del Giovine and Fabietti 2005; Tumolo and Lanfer-Marquez 2012), the green color of the bamboo was retained.

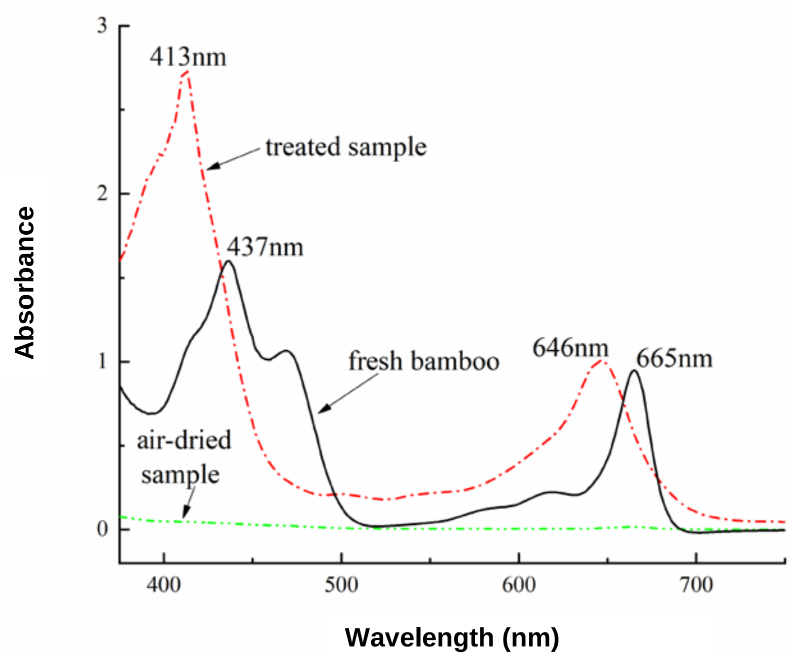

Fig. 7. The UV-visible spectra of the chlorophyll extracted from the fresh, air-dried, and $CuSO_4$ treated *N. affinis* bamboo samples

Surface Chemical Analysis

To further test possible reactions between the chlorophyll and the copper ions, the changes of elements on the green bamboo surface before and after the treatment was elucidated by EDX analysis. The results are shown in Fig. 8. The relative magnesium content slightly decreased from 0.12% to 0.10%, which showed the loss of magnesium ions during the drying process of bamboo. The copper content increased from 0.66% to 2.14%, revealing that the combination of copper and chlorophyll may occur during the treatment process. This result was consistent with the peaks shift in the UV-visible spectra of the chlorophyll. One interesting result was that the silicon element content dramatically decreased from 14.4% to 1.04% after the treatment. The removal of the silicon on the bamboo surface may provide good conditions for the treatment reactions with increasing the permeability (Yuan *et al*. 2016).

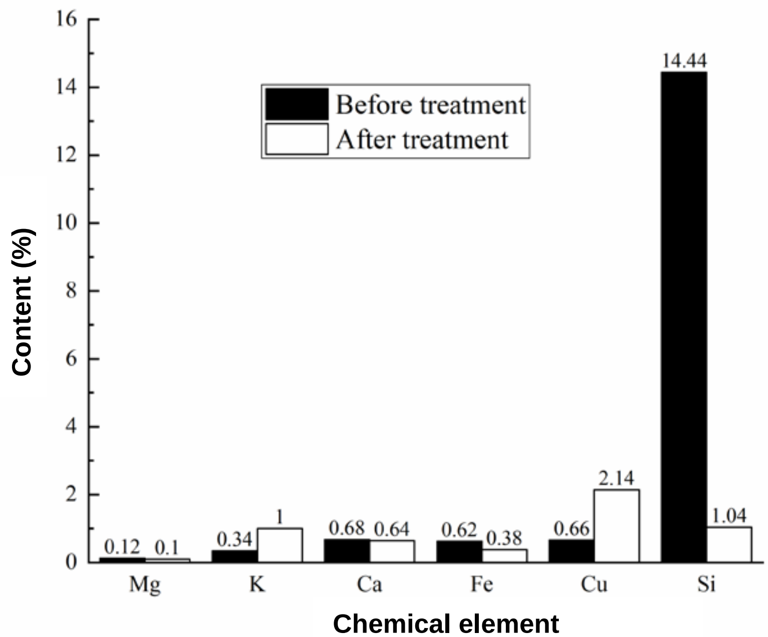

Fig. 8. Changes in the relative element content of bamboo epidermis before and after $CuSO_4$ aqueous solution treatment

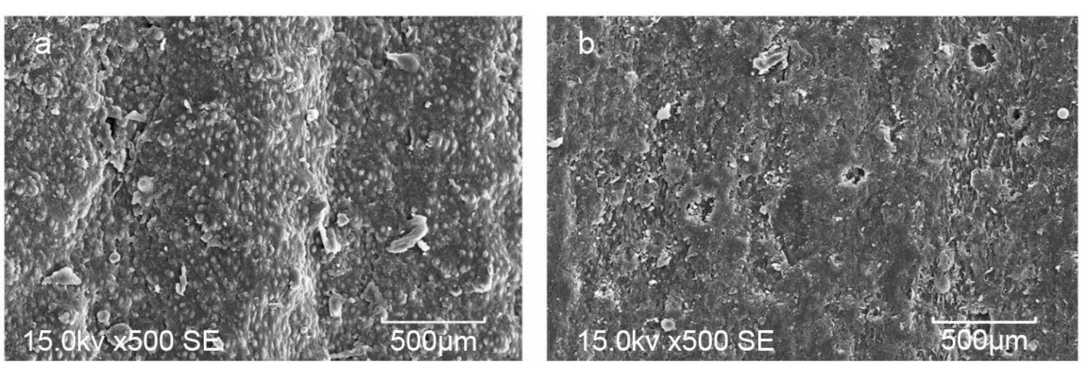

Fig. 9. The SEM images of (a) the original bamboo epidermis sample and (b) the treated sample

Surface Morphology Analysis

Figure 9 shows the SEM images of the *N. affinis* bamboo surface and the treated samples. The *N. affinis* had a rough surface with spherical substances. The spherical substances were not visible on the surface of the $CuSO_4$ treated samples, providing a much looser structure. The structural changes during the treatment process provided favorable conditions for the permeability of the reagents and promoted the reactions.

CONCLUSIONS

1. The *Neosinocalamus affinis* bamboo samples treated by the $CuSO_4$ aqueous solution at 80 °C for 3 h were able to exhibit excellent green color, indicated by low a^* values.
2. The *N. affinis* samples treated in the $CuSO_4/H_3PO_4$ and $CuSO_4/CH_3COOH$ solutions had less negative a^* values compared to those treated in the $CuSO_4$ aqueous solution. The $CuSO_4/CH_3COOH$ solution had the best efficiency on the green color preservation indicating by the less negative a^* values.
3. The durability test showed that the treated *N. affinis* samples exhibited almost the same color performance to the fresh bamboo after the indoor durability test, indicating that these samples had great potential in interior decoration utilization.
4. The chlorophyll structure changed after the treatment, and a reaction may take place between the chlorophyll and the copper ions during the treatment. The removal of silicon provided favorable conditions for the reactions.

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