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Effects of copper-phosphorous salt treatments on green colour protection and fastness of ma bamboo (*Dendrocalamus latiflorus*)

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Abstract

The objectives of this study were to find an appropriate method and process for treating ma bamboo (*Dendrocalamus latiflorus*) with a chromium-free copper-phosphorous salt (CuP). The effects of the constituent ratios and concentrations of CuP as well as treatment conditions on the green colour protection were investigated. In addition, accelerated UV light fastness tests, indoor exposure tests, and outdoor weathering tests were also carried out to evaluate the colour fastness and durability of bamboo culms. The results demonstrated that an attractive green colour was obtained by treating ma bamboo with 1% CuP at 60 °C for 3 h, using a 60:40 ratio of $CuSO_4$ to H_3PO_4 in an aqueous solution. Compared with untreated bamboo culms, the CuP-treated samples had the brighter greenish skin and also provided better green colour fastness. After 180 days of indoor exposure, the a^* value was slightly increased from −5.8 to −5.4.

Keywords: Green colour protection; Colour fastness; Bamboo; *Dendrocalamus latiflorus*; Copper-phosphorous salt (CuP)

1. Introduction

Bamboo, a perennial lignified plant, is one of the most important forest resources and grows more rapidly than any other woody plant on earth [1]. There are many genera of bamboos cultivated in Taiwan. Among them, ma bamboo (*Dendrocalamus latiflorus* Munro) is one of the most popular and valuable species because of its rapid growth rate, excellent flexibility, and easy machinability. It is widely used as an industrial material in pulping, furniture, and construction. Furthermore, bamboo fascinates people because of its green colour, which is due to abundant chlorophylls on its epidermis, making it different from other woody materials. But without any protective or stabilization treatment, bamboo culms are very susceptible to attack by fungi or insects, and hence discolour easily. Numerous studies have reported that light, oxygen [2–4], and enzymes [5] play important roles in chlorophyll degradation in vitro and in vivo. Similar to other green plants, bamboo loses its green colour as a result of the deterioration of chlorophyll when exposed in ambient conditions.

To overcome this problem and to encourage the bamboo industry to explore potential utilization and increase the economic value of bamboo products using green culms, green colour protection of bamboo has been widely studied during the last decade. In previous investigations, several inorganic salts, including chromates, nickel salts, and copper salts, were used as protectors [6–9]. In addition to these traditional chemicals, two arsenic-free preservatives, chromated copper phosphate (CCP) and chromated phosphate (CP), have been proven to be much more superior green-colour protectors than Boliden K-33 both for ma bamboo and moso bamboo (*Phyllostachys pubescens* Mazel) by these authors [10,11]. These two bamboo culms treated with CP or CCP also exhibited excellent colour fastness even in accelerated UV lightfastness or outdoor weathering exposure [12,13]. However, the potential toxicity of chromium and arsenic to humans, animals, and plants is well documented [14–16], thus chromium based preservatives such as CCA or arsenic-free CCP and CP might not be good for human health and damage the environment as well.

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In fact, chromium is a common contaminant in surface water and groundwater. It is the second most abundant inorganic groundwater contaminant at hazardous waste sites [17]. Chromium in the environment exists mainly as Cr(III) and Cr(VI). Under oxidizing conditions, chromium is highly soluble and mobile as Cr(VI) anions chromate (CrO_4^{2-}) and bichromate ($HCrO_4^-$). In contrast, under reducing conditions, Cr(VI) may convert to Cr(III), which is an insoluble precipitate and strongly adsorbed onto solid surfaces [18,19]. The oxidation states of Cr develop different biological activity. It is assumed that hexavalent chromium is about 100–1000 times more toxic than trivalent chromium [20]. Cr(VI) is a suspected carcinogen [21,22], whereas Cr(III) is an essential trace element for living beings, playing an important role in carbohydrate metabolism [23]. In our recent study, it revealed that CP oxidized the chemical components on the bamboo surface and consequently generated a large number of carbonyl groups and radicals [24]. Meanwhile, the oxidation states of Cr were converted from Cr(VI) to Cr(V) and then to Cr(III). Accordingly, most of the hexavalent chromium was reduced to Cr(III) on the CP treatment.

However, to decrease the possible hazardous waste in the future and to dispel peoples' doubts, developing an environmentally-friendly green protector is necessary. Thus, this study sought appropriate chemical reagents without chromium for conserving the green colour of ma bamboo culms and compared the effectiveness of these reagents. In addition, three exposure tests, including accelerated UV lightfastness, outdoor weathering, and indoor exposure, were carried out to evaluate the colour fastness of treated ma bamboo.

2. Materials and methods

2.1. Sample preparation

Three-year-old ma bamboo (*Dendrocalamus latiflorus* Munro) culms were obtained from the experimental forest of National Taiwan University in Nantou County. The bamboo culms were cut into strips with a dimension of 50 (longitudinal)×15 (tangential)×40 mm (radial) and stored at 4 °C in the dark prior to use.

2.2. Pretreatment

Before treatment with chemical reagents, the bamboo specimens were pretreated at 80 °C with 4% potassium carbonate and 1% surfactant mixtures for 30 min, and then carefully rinsed with water. This pretreatment removes the waxes from the ma bamboo surface and ensures better penetration and reaction for the subsequent green-colour protection treatments [11].

2.3. Chemical treatment

The green-colour protection reagent used in this experiment was copper-phosphorous salt (CuP). To find the most appropriate composition of CuP on green-colour conservation, seven $CuSO_4/H_3PO_4$ (%, w/w) ratios, including 100:0, 80:20, 60:40, 50:50, 40:60, 20:80, and 0:100 were investigated. The concentrations of reagent were 0.5, 1, 2, 4 and 8%, respectively. Furthermore, to study the effect of various treatment times on the green colour protection of bamboo culm, alkali-pretreated specimens were treated with CuP in a 60 °C water bath for four durations (0.5, 1.0, 3.0 and 6.0 h) and then dried at 60 °C for 12 h.

2.4. Exposure test

For the colour fastness study, ma bamboo specimens were exposed to different conditions. One was exposure to an artificially accelerated lightfastness tester (Q-Panel Co.) using UVA-351 sunlamp (Philip Co.) as a light source, the temperature of the black panel being 60±2 °C. The second condition was an outdoor weathering exposure on a rack. The weathering rack was positioned at a 45° angle to the ground in the campus of National Taiwan University and facing south. The weathering period was from 1 October 2000 to 30 May 2001. The average temperature was 20.4 °C ranging from 8.7 to 33.8 °C and the average relative humidity was 78.5%. The total duration of sunshine was 516.4 h and the total precipitation duration was 972.2 h with a total precipitation of 1035.0 mm. In addition, an indoor exposure rack was also used in the same period to outdoor weathering exposure. The samples were exposed to these conditions for 32, 180 and 180 days, respectively.

2.5. Mildew resistance of CuP- treated bamboo epidermis

To compare the mildew resistance of various bamboo specimens during outdoor weathering exposure, the degree of surface disfigurement of the bamboo culms by microbial (or fungal) growth was evaluated following the ASTM standards (D3274- 82) [25]. Accordingly, infestation is represented in steps of eleven degrees from 0 to 10. A rating of 10 would indicate an epidermis totally free of disfigurement by microbial growth, whereas one with rating 0 has the heaviest infestation.

2.6. Measurement of surface colour

The colour of bamboo epidermis was measured by a colour and colour difference meter (Dr. Lange Co.) under a D_{65} light source. The tristimulus values X, Y, and Z of all specimens were obtained directly from the colorimeter. Based on these data, the L^* (value on the

white/black axis), a^* (value on the red/green axis), b^* (value on the blue/yellow axis), ΔE^* (the colour difference, $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$), ΔC^* (the chroma difference, $C^* = [(a^*)^2 + (b^*)^2]^{1/2}$), and ΔH^* (the hue difference, $\Delta H^* = [(\Delta E^*)^2 - (\Delta L^*)^2 - (\Delta C^*)^2]^{1/2}$) colour parameters were calculated as established by the Commission Internationale de Enluminure (CIE) in 1976 [10]. The a^* is the best parameter to show the red and green colours. A positive a^* value indicates the sample is red and a negative value is found for a green sample. The smaller the a^*, the greener is the sample.

2.7. Analysis of variance

All results are expressed as mean ± S.D. ($n = 9$). The significance of difference was calculated by Duncan's test, and values < 0.05 were considered to be significant.

3. Results and discussion

3.1. Influence of treatment conditions on the colour of ma bamboo culms

Previous studies showed that ma bamboo or moso bamboo treated with CP had an excellent green colour protection and colour fastness [10–13], but 50% of chromate contained in CP might be recognized as a hazard to environment even to human health. Hence, developing chromium-free and low-toxic reagents is necessary. CuP (copper-phosphorous salt) was tested as a rudimentary green colour protector by the authors in 2000 [10]. After 2% CuP treatment, the CIE LAB colour parameters L^*, a^*, and b^* were 57.4, −5.0, and 23.7, respectively. This result revealed that CuP might be a suitable green colour protector. Hence, more detailed treatment conditions were investigated in this study. First, to understand the influence of $CuSO_4$/H_3PO_4 ratio on the colour of ma bamboo, various constituent ratios were investigated for finding the most appropriate composition of CuP. The results in Fig. 1 reveal that the a^* value decreased when we changed the $CuSO_4$/H_3PO_4 ratio from 100:0 to 0:100. In other words, increasing $CuSO_4$ proportion in CuP solutions enhanced the bamboo green colour performance. As for the L^* and b^* values, slight variations were observed among the ratios. These results demonstrated that the ratio of these two chemicals greatly affected the green colour of ma bamboo culms. Comparing with fresh bamboo which CIE LAB colour parameters L^*, a^*, and b^* were 32.4, −6.3, and 13.6, all the CuP-treated bamboo culms exhibited more lightness. However, when the $CuSO_4$/H_3PO_4 ratio was 60:40, the hue and chroma of the treated bamboo were almost the same as the fresh bamboo (Fig. 1), the chroma difference (ΔC^*) and the hue difference (ΔH^*) were only −0.1 and 0.7, respectively.

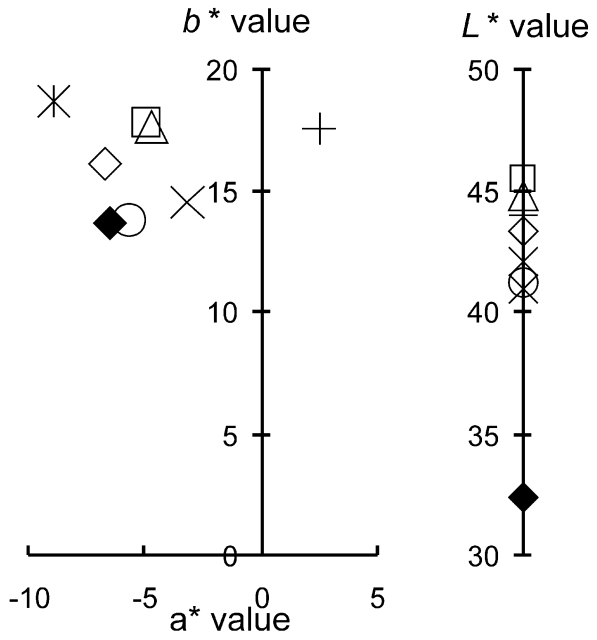

Fig. 1. Influence of $CuSO_4$/H_3PO_4 ratios in 2% CuP aqueous solution on the color of ma bamboo treated at 60 °C for 6 h (◆: fresh bamboo, ✳: 100:0, ◇: 80:20, ○: 60:40, □: 50:50, △: 40:60, ×: 20:80, +: 0:100).

This means that, at the ratio of 60:40, the CuP treated bamboo has an excellent green colour protection, since its epidermis colour was very close to that of the fresh bamboo culm.

To further understand the influence of CuP concentrations on the colour of ma bamboo, in addition to 2% of CuP, four other concentrations, 0.5, 1, 4 and 8%, were examined. Table 1 shows the changes of colour parameters on ma bamboo epidermis after treatment with various concentrations of CuP. The a^* values of ma bamboo treated with 0, 0.5, 1, 2, 4 and 8% CuP were 0.2, −3.8, −5.5, −5.7, −4.5, and −4.7, respectively. Among them, 1 and 2% of CuP-treated bamboo exhibited the best green colour performance (both bamboos have no statistically significant variation by Duncan's test). Accordingly, it is clear that the epidermis of ma bamboo could achieve an effective green colour protection after treatment with only 1% CuP at 60 °C for 6 h.

Table 1
Changes in color parameters of ma bamboo culms after treatment with CuP of different concentrations at 60 °C for 6 h

Concentrations (%)	CIE LAB		
	L^*	a^*	b^*
0	45.6 ± 2.5	0.2 ± 0.9	18.4 ± 1.6
0.5	44.8 ± 2.3	−3.8 ± 0.7	20.0 ± 1.3
1	46.8 ± 2.4	−5.5 ± 0.6	17.7 ± 1.8
2	41.2 ± 3.0	−5.7 ± 0.4	13.8 ± 2.1
4	50.1 ± 1.9	−4.5 ± 0.6	18.1 ± 1.6
8	48.1 ± 2.4	−4.7 ± 0.6	17.2 ± 1.6

Furthermore, the influence of treatment temperature during green colour protective process was also investigated. The results in Fig. 2 reveal that the a^* value decreased when the temperature was raised from room temperature to 60 °C, the a^* values were −0.7 (25 °C), −2.0 (40 °C), and −5.5 (60 °C), whereas it increased when the treatment temperature was more than 60 °C. The a^* value changed from −5.5 to −4.5 when the temperature was raised to 80 °C. In other words, among the various temperatures used, the best green colour performance was obtained when the bamboo culm was processed at 60 °C.

Considering the practical application in the manufacturing of green bamboo products, it is also important to reduce the treatment time during the manufacturing process. Therefore, five different treatment times, including 1, 2, 3, 6, and 9 h, were examined for colour protection and the results were shown in Fig. 3. The a^* values of ma bamboo culms treated with 1% CuP at 60 °C for 1, 2, and 3 h were −4.0, −4.8, and −5.8, respectively. However, the differences in a^* values were verified to be not statistically significant when the treatment time was more than 3 h. With treatment for 3, 6 and 9 h, the a^* values range between −5.5 and −6.1.

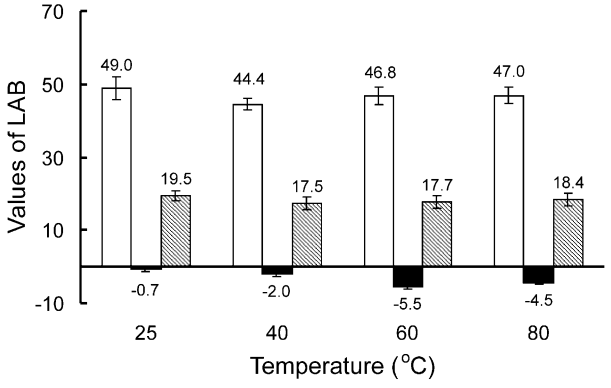

Fig. 2. Changes in color parameters of ma bamboo culms after treatment with 1% CuP at different temperatures for 6 h (white bars: L^*, black bars: a^*, striped bars: b^*).

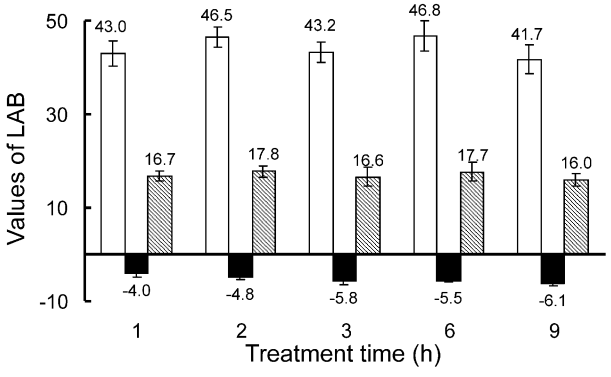

Fig. 3. Changes in color parameters of ma bamboo culms after treatment with 1% CuP at 60 °C for different times (white bars: L^*, black bars: a^*, striped bars: b^*).

Hence, taking the production cost into consideration, the 3 h would be the better choice for producing bamboo with green colour.

3.2. Green colour fastness of ma bamboo culms

According to the above-mentioned results, treating ma bamboo with 1% CuP (the ratio of $CuSO_4$:H_3PO_4 is 60:40) at 60 °C for 3 h was a good treatment process for green colour protection. To further evaluate the green colour fastness and durability of the treated ma bamboo, three exposure tests, including the accelerated UV light fastness test, the indoor exposure test, and the outdoor weathering test, were carried out in the study. The results of accelerated UV light fastness are displayed in Fig. 4. After 32 days of light fastness test, the a^* values of untreated (fresh bamboo) and alkali-pretreated ma bamboo increased from −6.8 and −6.7 to 9.0 and 11.1, respectively. In other words, the colour of the specimens changed from green to yellow during the 32-day light fastness test. In contrast, the a^* values of 1% CuP-treated ma bamboo culms were negative during the exposure test. After 32-day exposure, the a^* value of CuP-treated specimens changed from −5.8 to 0.0. In our previous study on the correlation between softwood discolouration induced by accelerated lightfastness testing and by indoor exposure, it was demonstrated that discolouration in the accelerated lightfastness test is ca. 250 times more severe than that in the indoor exposure test [26]. Accordingly, it implies that the specimen exposed to an artificially accelerated lightfastness tester equipped with UVA-351 lamps for 32 days is approximately equal to that exposed to indoor environments for 22 years. Hence, comparing with the colour changes of untreated bamboos, it demonstrated clearly that the green colour fastness of CuP-treated ma bamboo culms was reasonably good. The cause for the decrease in the a^* value in the first two days after irradiation may be the photoactivation of the

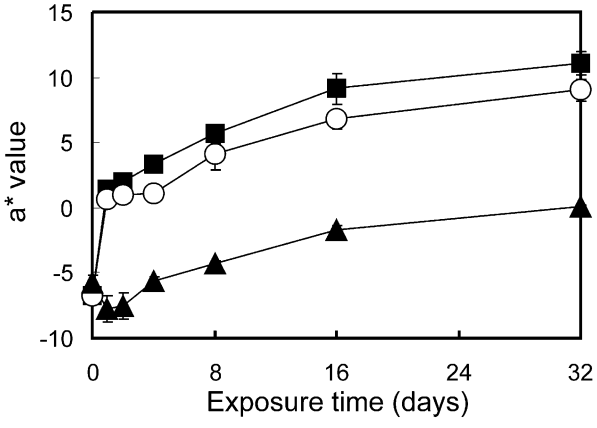

Fig. 4. Changes in a^* of bamboo culms after exposure to an accelerated UV lightfastness test for 32 days (○: fresh bamboo, ■: alkali-pretreated bamboo, ▲: CuP-treated bamboo).

reagents before the fixation, subsequently followed by the migration and precipitation of chemical products to the surface of specimens. Similar results have been reported on CCA-treated wood [27] as well as CP- and CrO_3-treated ma bamboo [12].

The variations in the a^* values of the specimens after exposure to outdoor environment for 180 days are shown in Table 2. Similar to the results obtained from the accelerated UV-light fastness test, untreated ma bamboo culms varied from green colour to yellow. The a^* values of the specimens exposed to outdoor weathering for 180 days were 2.2 and 1.6 for untreated and alkali-pretreated bamboos. Similarly, the a^* value of CuP-treated ma bamboo culm was also increased from initial −5.8 to 1.0 after 180-day weathering exposure. However, the mildew resistance of CuP-treated bamboo was higher than that of untreated ones. After outdoor weathering exposure, the mildew resistance degrees were rated with 2 and 4 for untreated bamboo and CuP-treated bamboo, respectively. These results demonstrated that the green colour retention ability of CuP-treated bamboo was similar to that of the untreated control, but it had a higher mildew resistance than the untreated one after 180-day weathering exposure.

On the other hand, only the portion of the light with long wavelengths ($\lambda > 300$ nm) is transmitted through a window glass filter and irradiates specimen surfaces. Therefore, the discolouration was less significant when the specimens were exposed indoors. After 180 days of indoor exposure, the a^* values of untreated, alkali-pretreated, and CuP-treated bamboo culms changed from −6.8, −6.7 and −5.8 to 8.3, 7.7 and −5.4, respectively (as shown in Fig. 5). Among these specimens, only the a^* value of CuP-treated ma bamboo decreased at the incipient stage and remained its green colour ($\Delta a^* = 0.4$) even after 180-day indoor exposure. This leads to the conclusion that ma bamboo culms treated with CuP solution possessed excellent green colour fastness under indoor conditions.

Table 2
Changes in color parameters and mildew resistance of CuP-treated ma bamboo culms after 180-day outdoor weathering exposure

Specimens	Exposure time (days)	a^*	Δa^*	ΔE^*	Mildew resistance[a]
Fresh bamboo	0	−6.8	–	–	10
	180	2.2	9.0	11.4	2
Alkali-pretreated bamboo	0	−6.7	–	–	10
	180	1.6	8.3	15.5	2
CuP-treated bamboo	0	−5.8	–	–	10
	180	1.0	6.8	14.2	4

[a] A rating of 10 would indicate an epidermis totally absent of disfigurement by microbial growth, whereas with 0 having the heaviest infestation.

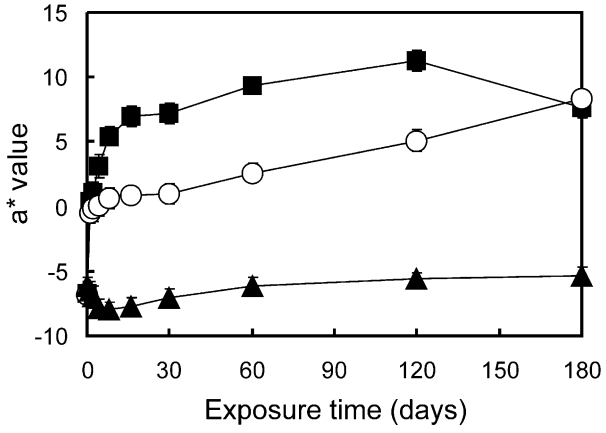

Fig. 5. Changes in a^* of bamboo culms after indoor exposure for 180 days (○: fresh bamboo, ■: alkali-pretreated bamboo, ▲: CuP-treated bamboo).

In addition, Fig. 5 also shows that the colour fastness of ma bamboo was significantly decreased after alkali pretreatment. It is known that there are many capes of silica cells in the cuticular layer of bamboo culm. Previous studies showed that silica not only influences the cuticular transpiration and CO_2 uptake of plants [28] but also strengthens the outer epidermis of plants and prevent external invasions [29–31]. Although, alkali pretreatment can remove the siliceous wax layers from bamboo surfaces and provide better penetration and reaction for the subsequent green-colour protection treatment [10,13], this process diminishes the protection effectiveness for bamboo culms against discolouration (Figs. 4 and 5) and the attack of other organisms. Accordingly, it is likely that alkali pretreatment was not a substantial process; hence it would be appreciated if the pretreatment can be avoided. Further studies on the treatment process using alkali-pretreatment-free and a low-toxic reagent to obtain the green colour protection of bamboo are in progress and the results will be reported in the near future.

4. Conclusions

Due to the chromium component, conventional green colour protectors including Boliden K-33, CCP and CP etc. can cause harmful effects to human beings and animals during processing. Therefore, a chromium-free and low toxic protector, CuP, was developed. The concentration, treatment time, temperature, and ratio of $CuSO_4$ to H_3PO_4 in CuP solutions greatly affected the green colour of ma bamboo culms. Our results showed that the green colour of ma bamboo culms can be appropriately protected when samples were treated using 1% CuP at 60 C for 3 h, with a 60:40 ratio of $CuSO_4$ to H_3PO_4 in an aqueous solution. The CuP-treated ma bamboo had higher colour fastness and durability than

untreated bamboo after exposure to both accelerated UV light fastness and outdoor weathering. In addition, the CuP-treated bamboo culm also exhibited good colour fastness, the Δa^* value was 0.4 after 180-day indoor exposure.

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