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Green color protection of bamboo culms using one-step alkali pretreatment-free process

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Abstract This study evaluated the protection effectiveness of alcohol-borne reagents for the green color of ma bamboo (*Dendrocalamus latiflorus* Munro) and moso bamboo (*Phyllostachys pubescens* Mazel). The results show that the types and concentrations of alcohol-borne reagents, the kinds of solvent, and the conditions of treatment greatly affected the green color of these two bamboo species. Without alkali pretreatment, an excellent green color protection ($a^* = -14.5$) was obtained when the ma bamboo culms were treated with 0.5% methanol-borne copper chloride (CuCl_2) at 60°C for 30min. Similar results were also obtained when ma bamboo culms were treated with 0.5% methanol-borne copper nitrate [$\text{Cu}(\text{NO}_3)_2$] at 60°C for 2h ($a^* = -13.5$). For moso bamboo, an attractive green color in the bamboo culms was achieved by treating the specimens with 1% methanol-borne copper acetate [$\text{Cu}(\text{CH}_3\text{COO})_2$] at 60°C for 30min. The a^* value of treated specimens was -13.3 . In addition, results demonstrated that ultrasonic treatment was more effective on green color protection than conventional water bath treatment. When moso bamboo was treated with 1% copper acetate at 60°C in an ultrasonic bath for only 15min, a remarkable green color with an a^* value of -13.6 was obtained on the bamboo epidermis.

Key words Green color protection · Bamboo · Alkali pretreatment-free process · Alcohol-borne reagents · Ultrasonic treatment

Introduction

Bamboos, perennial lignified plants in the Bambusoideae family, are abundant in tropical and subtropical regions. In

Taiwan, the total bamboo land area is about 180 thousand hectares, and the total growth stock of bamboos is approximately 120 million poles.¹ There are nearly 58 species and four varieties occur all over the country, especially in Nan-Tou County.² Among them, ma bamboo (*Dendrocalamus latiflorus* Munro) and moso bamboo (*Phyllostachys pubescens* Mazel) are two of the popular and valuable species. The total share of both species is more than 53% of bamboo land area in Taiwan.³

Bamboo has become one of the most important nontimber forest products in Taiwan and other Asian countries. This is due mainly to its rapid growth rate, availability, renewable nature, high productivity, short maturity cycle, and multiple uses. However, apart from its susceptibility to attack by fungi and insects, the attractive green color of bamboo epidermis is liable to fade, consequently reducing the service life of bamboo products. Its durability depends on the climatic conditions and the environment, but untreated bamboo may have an average life of fewer than 1–3 years when it is exposed to atmosphere and soil.⁴ In previous investigations, several inorganic salts, including commercial chromates, nickel salts, and copper salts as well as custom made arsenic-free phosphorous-based reagents, e.g., chromated copper phosphate, chromated phosphate, and copper phosphorous salt, have all proven to be effective green color protectors for both ma bamboo and moso bamboo.^{5–9}

However, in all of the aforementioned treatments, alkali pretreatment was a necessary process for green color protection of bamboo culms. This process not only diminishes the protective effectiveness for bamboo culms against attack by organisms, but also increases the time and cost required.⁹ Thus, the objective of this study was to find an appropriate method and process, especially an alkali pretreatment-free process, for treating both ma and moso bamboos. Various alcohol-borne copper-based reagents including copper chloride, copper nitrate, and copper acetate were employed as bamboo green color protectors. Accordingly, the influences of various green color protection treatments and treatment conditions on the color of bamboo culms were investigated in this study, and their levels of

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green color effectiveness were evaluated by a color and color difference meter.

Materials and methods

Sample preparation

Three-year-old ma bamboo (*Dendrocalamus latiflorus* Munro) and moso bamboo (*Phyllostachys pubescens* Mazel) culms were obtained from the experimental forest of the National Taiwan University in Nan-Tou County. Both bamboo culms were cut into strips with dimensions of 50 mm (longitudinal) × 15 mm (tangential) × 4 mm (radial) and stored at 4°C in the dark before use.

Chemical treatment

The green color protection reagents used in this experiment were three alcohol-borne copper-based salts: copper acetate [Cu(CH₃COO)₂], copper nitrate [Cu(NO₃)₂], and copper chloride (CuCl₂). All reagents were purchased from Acros Organics (Geel, Belgium) and the treatment solutions were formulated in methanol or ethanol.

Bamboo specimens were treated with 1% (w/v) chemical reagents in a 60°C water bath for 2 h to evaluate the effectiveness of green color protection by these reagents. Furthermore, to find the most appropriate method and process for treating ma and moso bamboos with alcohol-borne green color protectors, the effects of various treatment conditions, e.g., reagent concentrations (0.1%, 0.5%, 1%, 2%, and 4%), treatment temperatures (25°, 40°, and 60°C), treatment durations (0.25, 0.5, 1, and 2 h), and solvent types (methanol and ethanol), on the green color protection of bamboo culms were investigated. Additionally, it has been suggested that ultrasound can be used to disrupt plant cell walls to facilitate the release of extractable compounds and enhance mass transport of solvent (or chemical reagent) from the continuous phase into plant cells.¹⁰ Thus, an ultrasonic treatment method was also employed in this study to evaluate the influence of ultrasonic treatment on the color of bamboo surface. Hence, during the treating process, a

water bath was replaced by an ultrasonic bath (Branson PC620, power 180 watts; output frequency 44 kHz). After treatment, all samples were dried at 60°C for 12 h before measurement of surface color and other properties.

Measurement of surface color

The color of bamboo epidermis was measured by a color and color difference meter (Dr. Lange, Germany) under a D₆₅ light source with a test-window diameter of 5 mm. 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), and b* (value on the blue/yellow axis) color parameters were calculated, as established by the Commission Internationale de l'Eclairage (CIE) in 1976.⁷

Wettability of specimen epidermis

The contact angle of water on the treated surface at ambient conditions was used as the index of wettability. A CA-A type contact angle meter (Kyowa Kaimenkagaku, Japan) was employed for this measurement.

Analysis of variance

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

Results and discussion

Color variations of bamboo treated with methanolic copper-based reagents

Color variations of ma or moso bamboo treated with 1% methanolic copper-based reagents at 60°C for 2 h were evaluated using the CIE LAB color specifications. As shown in Table 1, the CIE LAB color parameters L*, a*,

Table 1. Color parameters and contact angles of ma bamboo and moso bamboo culms treated with 1% methanol-borne green color protectors at 60°C for 2 h

Species	Chemicals	CIE LAB			Contact angle (°)
		L*	a*	b*	
Ma bamboo	– ^a	33.7 ± 0.5	–9.2 ± 0.8 ^A	9.9 ± 1.0	80.1
	Cu(NO ₃) ₂	49.4 ± 0.4	–15.0 ± 1.1 ^B	23.9 ± 0.7	65.2
	Cu(CH ₃ COO) ₂	47.9 ± 0.6	–8.7 ± 0.9 ^A	24.1 ± 0.9	68.4
	CuCl ₂	42.5 ± 0.5	–14.9 ± 0.8 ^B	16.6 ± 1.0	63.8
Moso bamboo	– ^a	40.9 ± 0.7	–4.4 ± 1.2 ^A	17.6 ± 1.2	83.6
	Cu(NO ₃) ₂	42.8 ± 0.9	–2.7 ± 1.0 ^A	22.3 ± 0.7	62.1
	Cu(CH ₃ COO) ₂	53.1 ± 0.6	–14.1 ± 1.1 ^B	27.5 ± 0.9	68.6
	CuCl ₂	29.2 ± 0.7	11.0 ± 0.7 ^C	12.2 ± 1.1	70.2

a* values marked by different letters are significantly different at the level of *P* < 0.05 according to the Scheffe test

^aFresh bamboo

and b^* of fresh ma bamboo culm were 33.7, -9.2 , and 9.9 , respectively. The a^* value is a color parameter on the red/green axis; a positive a^* value represents red color, whereas a negative a^* value represents green color. In fact, the a^* value has been used successfully as the main physical parameter for characterizing the extent of green color degradation in peas.¹¹ In addition, it is relatively easy to evaluate the effectiveness of green color development by looking at the a^* value.¹² Hence, comparison of the a^* values of ma bamboo treated with different methanolic copper-based reagents showed that copper nitrate and copper chloride exhibited effective green color protection because they gave high negative a^* values. The a^* values for these two reagents for treated ma bamboo were -15.0 and -14.9 , respectively. In addition, the L^* values improved from 33.7 (fresh bamboo) to 49.4 and 42.5 , respectively. These results indicated that both treated color tones were not only greener but also brighter.

Our previous studies found that alkali pretreatment was necessary for the green color protection of bamboo epidermis and removed the wax layer on their outer surfaces.^{13,14} Thus, various alkali chemicals including potassium carbonate, sodium carbonate, and even potassium hydrate have been used in the past decade.^{15,16} Unfortunately, alkali pretreatment not only diminishes the protection for bamboo culms against attack by organisms, but it also increases the time and cost. In contrast, the alkali pretreatment was unnecessary when bamboo specimens were treated with the alcohol-borne green color protectors used in this study. Based on the observations of morphological changes of bamboo epidermis, it was noted that the waxes on the surface of all bamboo treated with methanol-borne copper-based reagents were effectively removed, except for the capes of silica cells of the cuticular layer (observed by scanning electron microscopy, not shown). Additionally, Table 1 shows that the contact angle of the bamboo surface decreased from an initial value of 80.1° to 65.2° and 63.8° after copper nitrate and copper chloride treatment, respectively. Thus, alcohol-borne reagents do not reduce the self-defense layer, and also provide better wettability or penetration for subsequent treatments, e.g., coating and preservative treatments, etc.

Moso bamboo could not obtain satisfactory green color protection under the aforementioned copper nitrate or copper chloride treatments. The a^* values of moso bamboo culms treated with these two reagents were -2.7 and 11.0 , respectively. However, an excellent green color protection could be obtained by copper acetate treatment. The L^* , a^* , and b^* color parameters of treated bamboo changed from the initial values of 40.9 , -4.4 , and 17.6 (fresh bamboo) to 53.1 , -14.1 , and 27.5 , respectively. In addition, the contact angle decreased from an initial value of 83.6° to 68.6° after copper acetate treatment (Table 1). These results demonstrated that both ma bamboo and moso bamboo could achieve a premium color protection by using appropriate alcohol-borne protectors.

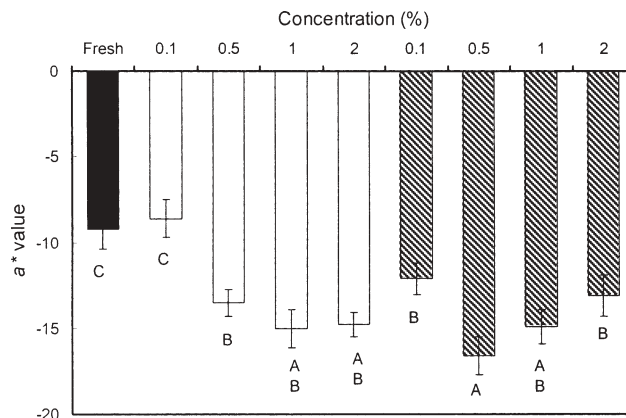


Fig. 1. Changes in the a^* value of ma bamboo culms after treatment with various concentrations of methanol-borne copper nitrate and copper chloride at 60°C for 2h. *Black bar*, fresh bamboo; *white bars*, copper nitrate treatments; *striped bars*, copper chloride treatments. a^* values marked by different letters are significantly different at the level of probability $P < 0.05$ according to the Scheffe test

Influence of protector concentrations on the color of bamboo culms

According to aforementioned results, methanol-borne copper nitrate and copper chloride are two effective green color protectors for ma bamboo. To understand the influence of concentrations of both copper nitrate and copper chloride on the color of ma bamboo, in addition to 1% reagent, three other concentrations of 0.1%, 0.5%, and 2% were examined. Figure 1 shows the changes in a^* values on ma bamboo epidermis after treatment with various concentrations of copper nitrate and copper chloride at 60°C for 2h. Accordingly, no statistically significant difference was observed among the specimens treated with 0.5%, 1%, and 2% copper nitrate (the a^* values were -13.5 , -15.0 , and -14.8 , respectively). This result reveals that ma bamboo treated with 0.5% copper nitrate gives superior green color performance ($a^* = -13.5$). Similarly, 0.5% copper chloride-treated bamboo ($a^* = -16.6$) also showed the best green color performance among various treatment concentrations. According to Scheffe's test, however, the color of copper chloride-treated bamboo culm is significantly greener than that treated with copper nitrate at the concentration of 0.5%.

For moso bamboo, Table 2 shows the changes of a^* values on bamboo epidermis after treatment with various concentrations of copper acetate (an appropriate alcohol-borne protector for moso bamboo). The a^* values of moso bamboo treated with 0.1%, 0.5%, 1%, 2%, and 4% copper acetate were -9.6 , -9.9 , -14.1 , -14.9 , and -15.2 , respectively. Of these, the concentrations higher than 1% exhibited the best green color performance (the a^* values of bamboos for concentrations higher than 1% are not statistically significant by Scheffe's test). Accordingly, it is clear that the epidermis of moso bamboo achieves effective green color protection after treatment with 1% copper acetate at 60°C for 2h.

Table 2. Changes in color parameters of moso bamboo culms after treatment with various concentrations of methanol-borne copper acetate at 60°C for 2h

Concentration (%)	CIE LAB		
	L^*	a^*	b^*
Control ^a	40.9 ± 0.7	-4.4 ± 1.2 ^A	17.6 ± 1.2
0.1	53.4 ± 1.1	-9.6 ± 0.8 ^B	23.4 ± 0.6
0.5	52.5 ± 1.2	-9.9 ± 0.7 ^B	24.1 ± 0.9
1	53.1 ± 0.9	-14.1 ± 0.4 ^C	27.5 ± 1.1
2	55.7 ± 1.4	-14.9 ± 0.8 ^C	37.0 ± 0.5
4	60.2 ± 1.1	-15.2 ± 0.4 ^C	35.2 ± 0.8

a^* values marked by different letters are significantly different at the level of $P < 0.05$ according to the Scheffe test

^aFresh moso bamboo

Table 3. Changes in color parameters of ma bamboo culms after treatment with 0.5% methanol-borne copper chloride at different temperatures for 2h

Temperature (°C)	CIE LAB		
	L^*	a^*	b^*
25	40.9 ± 0.5	6.7 ± 1.3 ^A	37.6 ± 1.1
40	36.1 ± 0.8	-2.8 ± 0.7 ^B	27.5 ± 1.3
60	42.5 ± 0.7	-16.6 ± 0.9 ^C	16.6 ± 1.0

a^* values marked by different letters are significantly different at the level of $P < 0.05$ according to the Scheffe test

Influence of treatment temperature on green color protection

To understand the influence of treatment temperature on green color protection, the three temperatures of 25°C (room temperature), 40°C, and 60°C were examined in this study. For ma bamboo treated with 0.5% methanol-borne copper chloride, the results in Table 3 reveal that the a^* value decreased when the temperature was raised from room temperature to 60°C; the a^* values were 6.7 (25°C), -2.8 (40°C), and -16.6 (60°C). In other words, among the various temperatures examined, the best green color performance was obtained when the ma bamboo culm was processed at 60°C. Similarly, copper acetate-treated moso bamboo showed the same variation under various treatment temperatures. After treatment with 1% copper acetate at 60°C for 2h, the a^* value of moso bamboo was -14.0 (Table 4); in contrast, the values were -4.0 and -7.5 when moso bamboo was processed at 25°C and 40°C, respectively.

Influence of treatment time on green color protection

Reducing the treatment time is an important factor for practical applications in the manufacturing process of green bamboo products. Therefore, four different treatment durations of 0.25, 0.5, 1, and 2h were examined for color protection. As shown in Fig. 2, the a^* values of ma bamboo culms treated with 0.5% copper chloride at 60°C for 0.25, 0.5, 1,

Table 4. Changes in color parameters of moso bamboo culms after treatment with 1% methanol-borne copper acetate at different temperatures for 2h

Temperature (°C)	CIE LAB		
	L^*	a^*	b^*
25	47.5 ± 0.9	-4.0 ± 1.2 ^A	19.2 ± 0.8
40	46.9 ± 0.8	-7.5 ± 0.9 ^B	19.2 ± 0.9
60	56.2 ± 0.4	-14.0 ± 0.5 ^C	35.2 ± 0.6

a^* values marked by different letters are significantly different at the level of $P < 0.05$ according to the Scheffe test

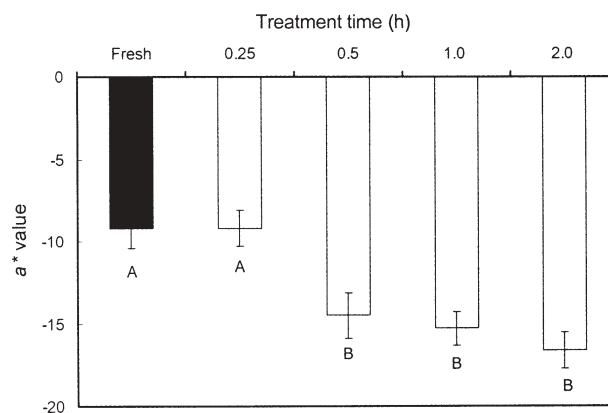


Fig. 2. Changes in the a^* value of ma bamboo culms after treatment with 0.5% methanol-borne copper chloride at 60°C for different times. Values marked by different letters are significantly different at the level of $P < 0.05$ according to the Scheffe test

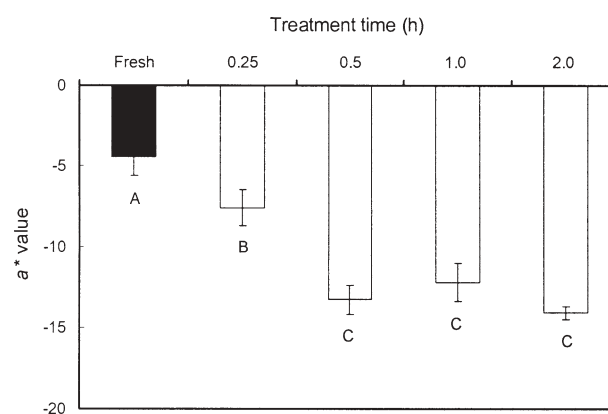


Fig. 3. Changes in the a^* value of moso bamboo culms after treatment with 1% methanol-borne copper acetate at 60°C for different times. Values marked by different letters are significantly different at the level of $P < 0.05$ according to the Scheffe test

and 2h were -9.2, -14.5, -15.3, and -16.6, respectively. Accordingly, when the treatment time was more than 0.5h, the a^* values of those treatment bamboos have no statistically significant variation by Scheffe's test. As for the moso bamboo, Fig. 3 shows that the a^* values of moso bamboo culms treated with 1% copper acetate at 60°C for 0.25, 0.5, 1, and 2h were -7.6, -13.3, -12.2, and -14.1, respectively. Similarly, these values have no statistically significant varia-

Table 5. Changes in the a^* value of ma bamboo and moso bamboo culms after treatment with 2% methanol-borne or ethanol-borne green color protectors at 60°C for 2 h

Species	CuCl ₂		Cu(CH ₃ COO) ₂		Cu(NO ₃) ₂	
	Methanol borne	Ethanol borne	Methanol borne	Ethanol borne	Methanol borne	Ethanol borne
Ma bamboo	-13.1 ^A	-10.4 ^B	-9.5 ^A	-9.7 ^A	-14.8 ^A	-12.1 ^B
Moso bamboo	14.0 ^B	11.0 ^A	-14.9 ^A	-17.1 ^A	-3.8 ^B	-16.3 ^A

The values of each protector treatment marked by different letters are significantly different at the level of $P < 0.05$ according to the Scheffe test

tion when the treatment time is more than 0.5 h. Hence, taking the production cost into consideration, a treatment time of 0.5 h would be the best choice for producing bamboo with an excellent green color.

Influence of solvent type and ultrasonic treatment on the color of bamboo culms

To understand the influence of alcoholic solvents on green color protection, methanol-borne and ethanol-borne protectors were investigated in this study. Table 5 shows that ma bamboo exhibits a satisfactory green color after treatment with 2% copper chloride or copper nitrate. The a^* values of ma bamboo culms treated with copper chloride in methanol and in ethanol were -13.1 and -10.4, respectively. This result shows that the color of methanol-borne treatments was greener than that of ethanol-borne treatments. Copper nitrate-treated ma bamboo culms showed the same tendency. The a^* values of bamboo culms were -14.8 and -12.1 by using methanol and ethanol as solvents, respectively.

For moso bamboo, the results of Table 5 reveal that moso bamboo shows good green color protection after treatment with 2% alcohol (methanol and ethanol) borne copper acetate. The a^* values of bamboo culms treated with copper acetate in methanol and in ethanol were -14.9 and -17.1, respectively. However, the difference between the a^* values of these two alcohol-borne copper acetate-treated bamboo culms is not statistically significant. Moreover, an excellent green color protection ($a^* = -16.3$) can be achieved by treating moso bamboo with 2% ethanol-borne copper nitrate, but no green color effectiveness ($a^* = -3.8$) was observed by using methanol as a solvent with its a^* value being more positive than that of fresh bamboo.

Regarding ultrasonics, this treatment has been widely applied in many fields over the past decade, e.g., in natural product extraction,¹⁷⁻¹⁹ chemical reactions,²⁰ and even in food preservation.²¹ The results obtained in these applications clearly revealed that it was a feasible and reliable method. However, the application of this technique as a protective treatment for bamboo and other woody materials is rare. As discussed above, moso bamboo can obtain excellent green color protection after treatment with methanol-borne copper acetate. Thus, to understand the influence of ultrasonic treatment on the color of bamboo culms, the color parameters of moso bamboo culms treated with 1% copper acetate in a 60°C ultrasonic bath were evaluated. As shown in Table 6, after treatment with copper

Table 6. Changes in color parameters of moso bamboo culms after treatment with 1% methanol-borne copper acetate at 60°C in an ultrasonic bath different with treatment times

Treatment time (min)	CIE LAB		
	L^*	a^*	b^*
Control ^a	40.9 ± 0.7	-4.4 ± 1.2 ^A	17.6 ± 1.2
15	52.8 ± 1.2	-13.6 ± 0.8 ^B	30.9 ± 0.6
30	54.3 ± 0.8	-13.3 ± 0.5 ^B	33.8 ± 0.9
60	55.1 ± 0.5	-13.9 ± 0.9 ^B	34.2 ± 0.8
120	60.0 ± 0.9	-14.4 ± 1.3 ^B	32.9 ± 0.5

a^* values marked by different letters are significantly different at the level of $P < 0.05$ according to the Scheffe test

^aFresh moso bamboo

acetate for 15 min, the L^* , a^* , and b^* color parameters of bamboo culms were changed from the initial values of 40.9, -4.4, and 17.6 (fresh bamboo) to 52.8, -13.6, and 30.9, respectively. When the treatment time extended to 30, 60, and 120 min, the a^* values of treated specimens were -13.3, -13.9, and -14.4, respectively. It is clear that no significant color variation was observed by increasing the treatment duration. In other words, ultrasonic treatment is more effective on green color protection than water bath treatment. With treatment with 1% copper acetate in a 60°C ultrasonic bath for 15 min, the moso bamboo could achieve an effective green color protection.

Conclusions

In the past, without alkali pretreatment, no suitable reagents and treatment processes could be found to achieve green color protection of bamboo culms. Thus, a complicated two-step treatment, especially alkali pretreatment, has been necessary for green color protection of bamboo culms to date. Fortunately, appropriate green color protectors, alcohol-borne copper-based reagents, and a novel one-step treatment process were developed successfully in this study. Without alkali pretreatment, excellent green color protection was obtained when ma bamboo and moso bamboo culms were treated with 0.5% copper chloride and 1% copper acetate in methanol in a 60°C water bath for 30 min, respectively. Furthermore, with the use of an ultrasonic bath instead of a water bath, the treatment duration could be decreased markedly. These results demonstrated that an effective and efficient treatment process was achieved for the green color protection of bamboo culms.

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