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Evaluation of the effectiveness of alcohol-borne reagents on the green colour protection of makino bamboo (*Phyllostachys makinoi*)

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Abstract

In the past, a complicated two-step process including alkali pretreatment was necessary for the protection of green bamboo surface colour. However, that process not only diminished the protection effectiveness for bamboo culms against attack by organisms, but also increased the time and cost required. Also, to date, no suitable reagents and treatment processes have been found to achieve the green colour protection of makino bamboo culms. Thus, the objectives of this study were to find an appropriate method and process, especially an alkali-pretreatment-free process, for treating makino bamboo with alcohol-borne copper based reagents. This study investigates influences of various green colour protection treatments and treatment conditions on the colour of bamboo culms. In addition to heating in a water bath, the ultrasonic heating method was examined. The results reveal that, without alkali-pretreatment, an excellent green colour protection was obtained when the makino bamboo culms were treated with 2% methanol-borne copper chloride ($CuCl_2$) in a 60 °C water bath for 2 h. However, compared with conventional heating in a water bath, improvement on the green colour protection cannot be achieved by using ultrasonic heating.
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Keywords: Green colour protection; Bamboo; *Phyllostachys makinoi*; Alcohol-borne; Copper chloride

1. Introduction

Bamboos occur in tropical, subtropical, and temperate zones, with widely distributed genera and species. Worldwide, there are about 75 genera and more than 1250 known species of bamboo [1], most of which are distributed in Asia with some in Africa, South America and Australia. In Taiwan, bamboo plants constitute an important forest resource; nearly 58 species and four varieties occur all over the country, especially in Nan-Tou County [2]. Among them, makino bamboo (*Phyllostachys makinoi* Hayata), moso bamboo (*Phyllostachys pubescens* Mazel), and ma bamboo (*Dendrocalamus latiflorus* Munro) are the three of the most common and valuable species.

Since bamboo is abundantly available over Asia, it is widely used as a construction material and for other purposes. However, in spite of its many excellent properties, the attractive green colour of its epidermis is liable to fade, also reducing the service life of bamboo products. In previous investigations, several inorganic salts, including commercial chromates, nickel salts, and copper salts as well as custom-made arsenic-free chromated copper phosphate (CCP), chromated phosphate (CP), and chromium-free copper-phosphorous salt (CuP) have all proven to be effective green colour protectors for both ma bamboo and moso bamboo [3–7].

However, to date it is very difficult to obtain a treatment for the attractive green of makino bamboo by using any of these protectors. This bamboo belongs to the Poaceae (Gramineae), has no secondary growth, and culms made of about 50% parenchyma cells and 40% supporting tissue of fibers [8]. 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 [9] but also strengthens the outer epidermis of plants, preventing external invasions [10,11]. Previously, alkali pretreatment was necessary to remove the siliceous wax layers from bamboo surfaces to provide better penetration and reaction for the green-colour protection treatment [12,13]. However, this process

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diminished the protection effectiveness for bamboo culms against the attack of organisms [7].

Accordingly, it is likely that alkali pretreatment was not a substantial process, so it would be preferable if the pretreatment could be avoided. Moreover, the time and cost for the production of green bamboo culms would be reduced if the tedious two-step treatment process could be replaced by a simple one-step treatment. In this study, without alkali-pretreatment, various alcohol-borne copper based reagents were employed as bamboo green colour protectors, and their levels of green colour effectiveness were evaluated by a colour and colour difference meter. In addition, the surface morphologies and silicon distribution of treated bamboo were also examined by a scanning electron microscope equipped with an energy dispersive X-ray spectrometer.

2. Materials and methods

2.1. Sample preparation

Three-year-old makino bamboo (*Phyllostachys makinoi* Hayata) culms were obtained from the experimental forest of National Taiwan University in Nan-Tou County. The bamboo culms were cut into strips with a dimension of 50 mm (longitudinal) ×15 mm (tangential)×4 mm (radial) and stored at 4 °C in the dark before use.

2.2. Chemical treatment

The green-colour protection reagents used in this experiment were five solvent-borne copper based salts, including copper sulphate ($CuSO_4$), copper acetate [$Cu(CH_3COO)_2$], copper nitrate [$Cu(NO_3)_2$], copper chloride ($CuCl_2$), and copper naphthenate (10% copper contained). All reagents were purchased from Acros Organics Co. (Geel, Belgium) and these treatment solutions were formulated in methanol, except copper naphthenate (Wako Pure Chemical Industries Co., Japan), for which xylene/ethanol (1:1, v/v) was used.

Bamboo specimens were treated with various chemical reagents (2 and 4%) in a 60 °C water bath for 2 h to evaluate the effectiveness of green colour protection by these reagents. Furthermore, to find the most appropriate method and process for treating makino bamboo with alcohol-borne green colour protectors, the effects of various treatment conditions, e.g. reagent concentrations (0, 0.25, 0.5, 1, 2 and 4%), treating temperatures (25, 40, and 60 °C), and treating durations (0.5, 1, 2, 4, and 6 h), on the green colour protection of bamboo culms were investigated. Additionally, an ultrasonic treatment method was also used in this study to evaluate the influence of ultrasonic treatment on the colour of bamboo surface. Hence, during the treating process, a water bath was replaced by an ultrasonic bath (Branson PC620, USA; power 180 W; output frequency 44 kHz). After treatment, all samples were dried at 60 °C for 12 h before measurement of surface colour and other properties.

2.3. Measurement of surface colour

The colour of bamboo epidermis was measured by a colour and colour difference meter (Dr. Lange Co., Germany) 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 l'Eclairage (CIE) in 1976 [5].

2.4. Wettability of specimen epidermis

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

2.5. SEM-EDX spectroscopy analysis

The bamboo surface morphologies and silicon mapping were examined by a scanning electron microscope equipped with an energy dispersive X-ray spectrometer (SEM-EDX, Hitachi S-2400, Japan). The critical point-dried specimens (5×3×0.5 mm) were taped on circular holders and coated with carbon for SEM-EDX analysis. Carbon-coated samples were imaged at 15 kV.

2.6. Analysis of variance

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

3. Results and discussion

3.1. Colour variations of makino bamboo treated with alcohol-borne copper based reagents

Colour variations of makino bamboo treated with alcohol-borne copper based reagents were evaluated using the CIE LAB colour specifications. The CIE LAB colour parameters L^*, a^*, and b^* of fresh makino bam-

boo culm were 36.0, −5.7, and 19.6, respectively. The a^* value is a colour parameter on the red/green axis. Positive a^* value represents red colour, whereas negative a^* value represents green colour. In general, a more negative a^* value means a deeper green colour. Hence, as described in our previous paper [14], it is relatively easy to evaluate the effectiveness of green colour development by looking at the a^* value. Comparison of the a^* values of specimens treated with two different concentrations of $CuSO_4$ (2% and 4%) showed that 2% $CuSO_4$ was more effective in green colour protection than a 4% solution, and similar results were also obtained from other reagents (Table 1). In addition, among all of the reagents employed, the best green colour protection result was obtained by 2% $CuCl_2$ treatment. Although, as shown in Table 1, the a^* value of 2% $Cu(CH_3COO)_2$-treated bamboo was the same as fresh bamboo ($a^* = -5.7$), however the colour difference ($\Delta E^* = 22.8$) was significantly more than that of 2% $CuCl_2$ treated one ($\Delta E^* = 16.8$). The L^*, a^*, and b^* colour parameters of 2% $CuCl_2$-treated makino bamboo culm were 50.3, −9.4, and 27.6, respectively. Differences in the colour parameters between 2% $CuCl_2$-treated bamboo and fresh specimen were 14.3 (ΔL^*), −3.7 (Δa^*), and 8.0 (Δb^*). This indicates that the treated colour tones were not only greener but also brighter and bluer.

Copper naphthenate, a solvent-borne reagent, was first tested as a rudimentary green colour protector by the authors in 2000 [5]. Those results demonstrated that ma bamboo (*Dendrocalamus latiflorus* Munro) treated with copper naphthenate had an acceptable green colour protection. But, it is known that different bamboo species showed the varied green colour performance, even using the same process and reagent [5,6]. In practice, after 2% copper naphthenate treatment, makino bamboo culms exhibit a good green colour protection. As shown in Table 2, their L^*, a^*, and b^* colour parameters were changed from initial 36.0, −5.7, and 19.6 (fresh bamboo) to 57.6, −6.5, and 27.5, respectively. However, in contrast with copper chloride treatment, copper naphthenate treated bamboo specimens had less effective green colour protection. The a^* values of copper chloride and copper naphthenate treated bamboo culms were −9.4 and −6.5, respectively.

Table 1
Colour variation of makino bamboo after treatment with various alcohol-borne copper based reagents at 60 °C for 2 h

Specimens	CIE LAB		
	L^*	a^*	b^*
Fresh bamboo	36.0±1.2	−5.7±0.5 C	19.6±1.8
2% $CuSO_4$-treated	61.4±3.6	−4.0±2.2 E	37.6±4.7
4% $CuSO_4$-treated	63.1±2.7	−2.5±1.1 F	37.8±3.5
2% $CuCl_2$-treated	50.3±2.5	−9.4±1.1 A	27.6±1.2
4% $CuCl_2$-treated	44.9±2.5	−5.1±2.1 C,D	23.4±1.9
2% $Cu(CH_3COO)_2$-treated	57.7±2.1	−5.7±1.1 C	26.7±3.4
4% $Cu(CH_3COO)_2$-treated	58.0±2.5	−4.7±0.6 D	25.0±1.3
2% $Cu(NO_3)_2$-treated	58.4±2.3	−6.6±0.8 B	29.1±2.5
4% $Cu(NO_3)_2$-treated	51.1±4.5	−4.9±1.2 D	23.7±1.3

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

Previously, a complicated process, usually alkali-pretreatment, was necessary for the green colour protection of bamboo surface. For this, various alkali chemicals including potassium carbonate, sodium carbonate, and even potassium hydrate were used [12,15]. These studies showed that the combination of an alkali-pretreatment followed by a green colour protector treatment was able to retain the green bamboo epidermis. 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, that process was unnecessary when bamboo specimens were treated with alcohol-borne green colour protectors. Fig. 1 shows the SEM and SEM-EDX Si mapping of 2% alcohol-borne $CuCl_2$-treated makino bamboo epidermis. Based on observations of morphological changes of bamboo epidermis, it was noted that the waxes on the bamboo surface were effectively removed, except for the capes of silica. This is demonstrated in Table 2, which shows that the contact angle of the bamboo surface was decreased from an initial value of 84.8 to 68.0 after $CuCl_2$ treatment. 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 treatment, etc.

3.2. Influence of treatment conditions on the colour of makino bamboo culms

Because these results indicate that alcohol-borne copper chloride may be an effective green colour protector for makino bamboo, more detailed treatment conditions were further investigated. First, to understand the influence of $CuCl_2$ concentrations on the colour of makino bamboo, in addition to 2% of $CuCl_2$, four other concentrations, 0.25, 0.5, 1, and 4% were examined. Table 3 shows the changes of colour parameters on makino bamboo epidermis after treatment with various concentrations of $CuCl_2$ at 60 °C for 2 h. The a^* values of makino bamboo treated with 0, 0.25, 0.5, 1, 2 and 4% $CuCl_2$ were 2.7, −5.7, −8.3, −9.5, −9.4, and −5.1, respectively. Of these, 1 and 2% of $CuCl_2$-treated bamboo exhibited the best green colour performance (both bamboos have no statistically significant variation by Scheffe's test). However, comparison of the colour difference ΔE^* and the chroma difference ΔC^* (based on fresh bamboo) of bamboo specimens treated with 1% and 2% $CuCl_2$ showed that both values of 2% $CuCl_2$ treatment were smaller than that of 1%. Accordingly, it is clear that the epidermis of makino

Table 2
Changes in colour parameters and contact angle of makino bamboo culms after treatment with 2% alcohol-borne $CuCl_2$ and copper naphthenate

Specimens	CIE LAB			Contact angle (°)
	L^*	a^*	b^*	
Fresh bamboo	36.0±1.2	−5.7±0.5 B	19.6±1.8	84.8±1.2
$CuCl_2$-treated	50.3±2.5	−9.4±1.1 A	27.6±1.2	68.0±1.9
Copper naphthenate-treated	57.6±0.7	−6.5±0.8 B	27.5±0.7	65.7±1.7

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

Table 3
Changes in colour parameters of makino bamboo culms after treatment with $CuCl_2$ of different concentrations at 60 °C for 2 h

Conc. (%)	CIE LAB			ΔC^*	ΔH^*	ΔE^*
	L^*	a^*	b^*			
Fresh bamboo	36.0±1.2	−5.7±0.5 C	19.6±1.8	–	–	–
0	64.0±1.5	2.7±1.0 D	41.7±3.2	21.4	160.3	36.7
0.25	56.4±2.7	−5.7±1.5 C	37.2±2.3	16.6	−7.6	26.9
0.5	57.0±2.8	−8.3±1.7 B	33.1±1.8	13.5	−2.2	25.1
1	55.6±2.8	−9.5±1.5 A	28.9±1.8	9.9	1.8	22.1
2	50.3±2.5	−9.4±1.1 A	27.6±1.2	8.7	3.1	16.8
4	44.9±2.5	−5.1±2.1 C	23.4±1.9	3.5	−4.4	9.8

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

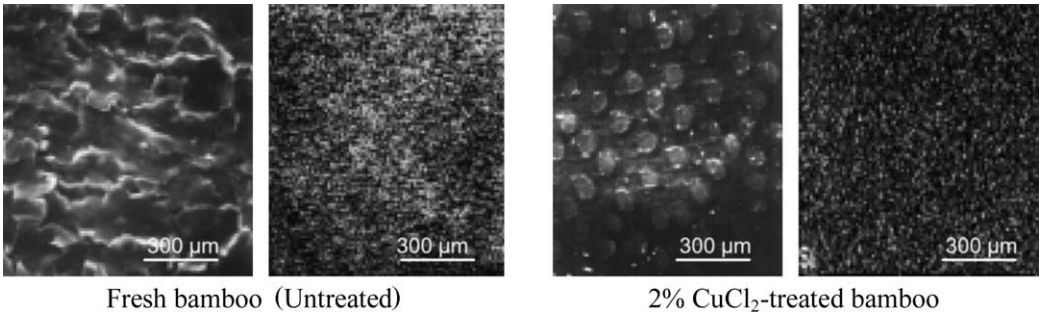

Fig. 1. SEM and SEM-EDX Si mapping of makino bamboo epidermis before and after 2% $CuCl_2$ treatment.

bamboo achieves an effective green colour protection after treatment with 2% $CuCl_2$ at 60 °C for 2 h.

Furthermore, to understand the influence of treatment temperature on green colour protection, three temperatures including 25 °C (room temperature), 40 °C, and 60 °C were also examined in this study. 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 6.7 (25 °C), 1.8 (40 °C), and −9.4 (60 °C). In other words, of the various temperatures used, the best green colour performance was obtained when the bamboo culm was processed at 60 °C. However, at ambient temperature, a green bamboo culm can be observed when the dipping time was increased from 2 h to more than 12 h (Fig. 3). The a^* values of makino bamboo treated with 2% $CuCl_2$ at room temperature for 0.5, 1, 2, 4, and 6 days were −6.3, −14.4, −12.4, −12.9, and −12.0, respectively. Of them, the best green colour protection was obtained for dipping 1 day because by extending the dipping time more than 1 day no enhanced effect could be observed. Furthermore, even by increasing the protector concentration from 2% to 4%, green colour performance was not improved by dipping treatment at ambient temperature.

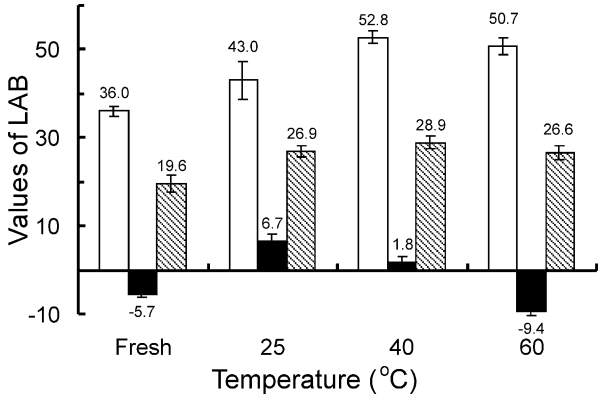

Fig. 2. Changes in colour parameters of makino bamboo culms after treatment with 2% $CuCl_2$ at different temperatures for 2 h (white bars: L^*, black bars: a^*, stripped bars: b^*).

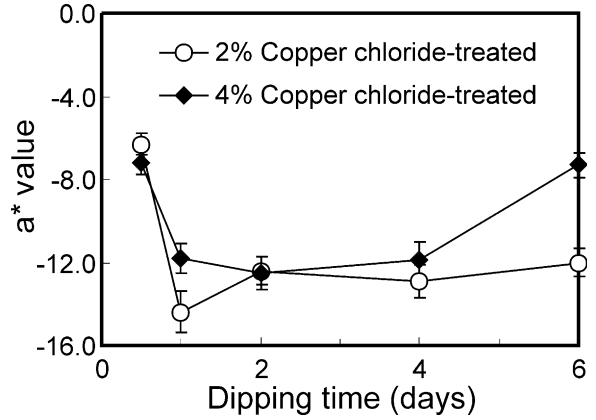

Fig. 3. Changes in a^* value of makino bamboo culms after treatment with 2 or 4% $CuCl_2$ at ambient temperature for different times.

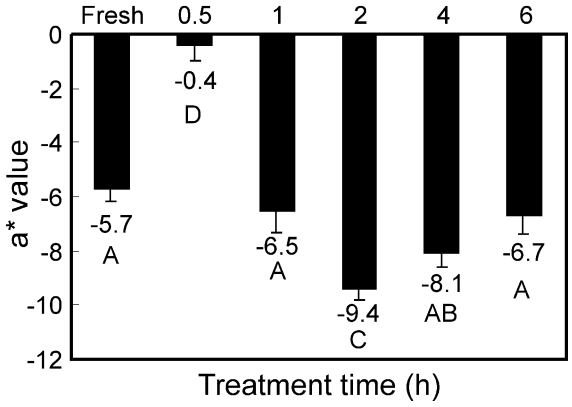

Fig. 4. Changes in a^* value of makino bamboo culms after treatment with 2% $CuCl_2$ at 60 °C for different times (marked by different letters are significantly different at the level of $P<0.05$ according to the Scheffe test).

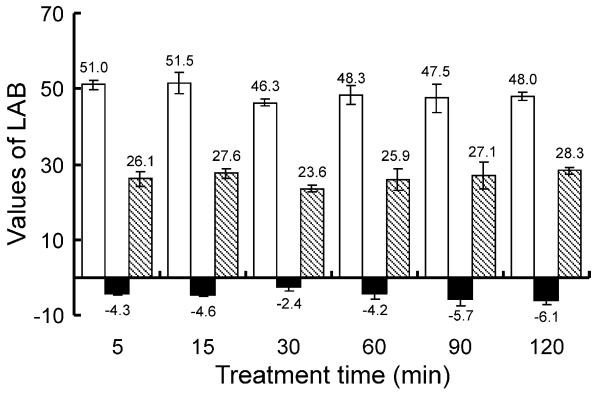

Fig. 5. Changes in colour parameters of makino bamboo culms after treatment with 2% $CuCl_2$ at 60 °C by using ultrasonic bath for different times (white bars: L^*, black bars: a^*, stripped bars: b^*) (the a^* value marked by different letters are significantly different at the level of $P<0.05$ according to the Scheffe test).

Reducing the treatment time is an important factor for practical applications in the manufacturing process of green bamboo products. Therefore, five different treatment times, including 0.5, 1, 2, 4, and 6 h, were examined for colour protection, with results as shown in Fig. 4. The a^* values of makino bamboo culms treated with 1% $CuCl_2$ at 60 °C for 0.5, 1, and 2 h were −0.4, −6.5, and −9.4, respectively. However, the a^* values increased when the treatment time was more than 2 h. With treatment for 4 and 6 h, the a^* values were changed to −8.1 and −6.7, respectively. Hence, taking the production cost into consideration, a treatment time of 2 h would be the best choice for producing bamboo with an excellent green colour.

3.3. Influence of ultrasonic treatment on the colour of makino bamboo culms

Ultrasonic treatment is widely applied on many fields, e.g. natural product extraction [16–20], chemical reactions [21], and even food preservation [22,23] etc., the results obtained clearly revealed that it was a feasible and reliable method. However, ultrasonic treatment has never been used for protection of bamboo colour. As discussed above, the best green colour protection was obtained when makino bamboo treated with 2% $CuCl_2$ in a 60 °C water bath. Thus, in order to further understand the influence of ultrasonic treatment on the colour of makino bamboo culms, these same conditions were employed in this of the study, except that the water bath was replaced by the ultrasonic bath. The results in Fig. 5 demonstrated that, with treatment for 5 min, a green bamboo culm ($a^*=-4.3$) was obtained by using ultrasonic treatment, although bamboos treated this way had the problem of non-uniform colour distribution on the surface. However, that problem was solved and a better green colour performance was observed when the treatment time was more than 1 h. After 1 h, 1.5 h, and 2 h treatment, the a^* values of bamboo epidermis were −4.2, −5.7, and −6.1, respectively. Thus, compared with water bath treatment, ultrasonic treatment shows no further improvement on the green colour protection.

As discussed above, the results demonstrate that the best treatment procedure for green colour protection was achieved by treating makino bamboo with 2% alcohol-borne $CuCl_2$ at 60 °C for 2 h. Compared with conventional water-borne $CuCl_2$, as shown in Table 4, the alcohol-borne $CuCl_2$-treated makino bamboo culms exhibit an excellent green colour protection, whereas the water-borne $CuCl_2$-treated specimens show poor green colour protection on the surface, even using the ultrasonic treatment. Thus, an attractive green colour of makino bamboo can be obtained successfully by using a one-step treatment with alcohol-borne reagents. As for the colourfastness of these treated bamboos, our preliminary results show that they exhibit an excellent green colourfastness under indoor conditions. Besides, the on-going experiments also reveal that this novel one-step treatment using alcohol-borne reagents can be

Table 4
Changes in colour parameters of makino bamboo culms after treatment with 2% $CuCl_2$ at 60 °C by using water bath or ultrasonic bath for 2 h

Specimens	Treatment process	CIE LAB		
		L^*	a^*	b^*
2% Alcohol-borne $CuCl_2$	Water-bath	50.7±1.9	−9.4±1.0	26.6±1.5
2% Water-borne $CuCl_2$		51.3±3.0	3.3±2.9	26.2±1.9
2% Alcohol-borne $CuCl_2$	Ultrasonic bath	53.5±1.0	−9.7±0.9	25.1±1.0
2% Water-borne $CuCl_2$		52.9±2.9	0.6±2.7	27.5±1.1

successively applied on the other bamboo species. These studies are in progress and the complete results will be addressed in the near future. Furthermore, to provide a more realistic elucidation, the detail trials, including chemical leaching ability, quantitative analysis of bamboo surface silica, and mechanisms of green colour protection, etc., are worthwhile investigating.

4. Conclusion

Similar to other green plants, bamboo loses its green colour as a result of the deterioration of chlorophyll when exposed in ambient conditions. To inhibit the discolouration and extend the service life of bamboo, topics regarding to the green colour protection of bamboo culms have been extensively investigated in the past decade. As a result, many reagents, including CCP, CP and CuP, have been proven to be effective green colour protectors for ma bamboo and moso bamboo using a two-step treatment. However, the greenish appearance cannot be achieved by treating makino bamboo culms with the same above-mentioned reagents and processes. Thus, finding a suitable reagent and a treatment process is imperative to protect the attractive green colour of makino bamboo culms. Accordingly, an appropriate green colour protector, alcohol-borne copper chloride, and a new one-step treatment process were developed successfully in this study. Without alkali-pretreatment, an excellent green colour protection was obtained when the bamboo culms were treated with 2% methanol-borne $CuCl_2$ in a 60 °C water bath for 2 h. Furthermore, after treatment, the waxes on the bamboo surface were effectively removed, but the capes of silica were still located on the bamboo epidermis. These results demonstrate that an effective and efficient treatment process was carried out for the green colour protection of makino bamboo culm.

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References

[1] Sharma YML. Bamboos in the Asia-Pacific region. In: Lessard G, Chouinard A, editors. Bamboo research in Asia. Ottawa CA: IDRC; 1980. p. 99–120.
[2] Wu SC. Research and development of the production and utilization of bamboo in Taiwan. For Prod Ind 1982;1:18–24.
[3] Chang ST, Lee HL. Protection of the green colour of moso bamboo (*Phyllostachys edulis*) culms and its colourfastness after treatment. Mokuzai Gakkaishi 1996;42:392–6.
[4] Chang ST. Comparison of the green colour fastness of ma bamboo (*Dendrocalamus* spp.) culms treated with inorganic salts. Mokuzai Gakkaishi 1997;43:487–92.
[5] Chang ST, Wu JH. Green colour conservation of ma bamboo (*Dendrocalamus* latiflorus) treated with chromium based reagents. J Wood Sci 2000;46:40–4.
[6] Chang ST, Yeh TF. Protection and fastness of green colour of moso bamboo (*Phyllostachys pubescens* Mazel) treated with chromium based reagents. J Wood Sci 2001;47:228–32.
[7] Wu JH, Wu SY, Hsieh TH, Chang ST. Effects of copper-phosphorous salt treatment on green colour protection and fastness of ma bamboo (*Dendrocalamus latiflorus*). Polym Degrad Stab 2002; 78:379–84.
[8] Wu KT. The effect of high-temperature drying on the antisplitting properties of makino bamboo culm (*Phyllostachys makinoi* Hay.). Wood Sci Technol 1992;26:271–7.
[9] Abasolo WP, Yoshida M, Yamamoto H, Okuyama T. Silica in several rattan species. Holzforschung 2001;55:595–600.
[10] Samuels AL, Glass ADM, Ehret DL, Menzies JG. The effects of silicon supplementation on cucumber fruit: Changes in surface characteristics. Ann Bot 1993;72:433–40.
[11] Yamauchi M, Winslow MV. Effect of silica and magnesium on yield of upland rice in the humid tropics. Plant Soil 1989;113: 265–70.
[12] Chang ST, Yeh TF. Effect of alkali pretreatment on surface properties and green colour conservation of moso bamboo (*Phyllostacys pubescens* Mazel). Holzforschung 2000;54:487–91.
[13] Chang ST, Wu JH, Yeh TF. Effects of chromated-phosphate treatment process on the green colour protection of ma bamboo (*Dendrocalamus latiflorus*). J Wood Sci 2002;48:227–31.
[14] Chang ST, Wu JH. Stabilizing effect of chromated salt treatment on the green colour of ma bamboo (*Dendrocalamus latiflorus*). Holzforschung 2000;54:327–30.
[15] Chang ST, Yeh TF, Wu JH, Hon DNS. Reaction characteristics on the green surface of moso bamboo (*Phyllostachys pubescens* Mazel) treated with chromated phosphate. Holzforschung 2002; 56:130–4.

[16] Chang ST, Wang SY, Wu JH. Rapid extraction of epidermis chlorophyll of moso bamboo (*Phyllostachys pubescens*) culm using ultrasonics. J Wood Sci 1998;44:78–80.
[17] Wu JH, Wang SY, Chang ST. Extraction and determination of chlorophylls from moso bamboo (*Phyllostachys pubescens*) culm. J Bamboo Rattan 2002;1:171–80.
[18] Mecozzi M, Amici M, Romanelli G, Pietrantonio E, Deluca A. Ultrasound extraction and thin layer chromatography-flame ionization detection analysis of the lipid fraction I marine mucilage samples. J Chromatogr 2002;963:363–73.
[19] Bruni R, Guerrini A, Scalia S, Romagnoli C, Sacchetti G. Rapid techniques for the extraction of vitamin E isomers from *Amaranthus caudatus* seeds: ultrasonic and supercritical fluid extraction. Phytochem anal 2002;13:257–61.
[20] Rosales-Conrado N, Leon-Gonzalez ME, Perez-Arribas LV, Polo-Diez LM. Determination of chlorophenoxy acid herbicides and their esters in soil by capillary high performance liquid chromatography with ultraviolet detection, using large volume injection and temperature gradient. Anal Chim Acta 2002;470: 147–54.
[21] Anthony EJ, Jia L, Cyr L, Smith B, Burwell S. The enhancement of hydration of fluidized bed combustion ash by sonication. Environ Sci Technol 2002;36:4447–53.
[22] Loske-Mehling AM, Hernandez-Galicia C, Armenta-Ruiz E, Prieto-Calderon FE. Shock wave potential as a food preservation method. Tip Revista Especializada en Ciencias Quimico-Biologicas 2000;3:23–30.
[23] De Gennaro L, Cavella S, Romano R, Masi P. The use of ultrasound in food technology I: Inactivation of peroxidase by thermosonication. J Food Eng 1999;39:401–7.