PRESERVATIVE TREATMENT OF BAMBOO CULM FOR PRODUCING GREEN COLOUR AND ENHANCING FUNGAL DURABILITY

Oh JJ[1,2], Ra JB[3] & Kim GH[1,*]

[1] *Division of Environmental Science & Ecological Engineering, College of Life Sciences & Biotechnology, Korea University, 145, Anam-ro, Seongbuk-gu, Seoul 02841, Korea*
[2] *Institute of Life Science and Natural Resources, Korea University, 145, Anam-ro, Seongbuk-gu, Seoul 02841, Korea*
[3] *Department of Interior Materials Engineering, Gyeongsang National University, Jinju 52725, Korea*

**lovewood@korea.ac.kr*

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This research was performed to investigate the feasibility of using copper-based wood preservative treatment for producing green colour on the bamboo surface and enhancing the fungal decay resistance of treated bamboo. Preliminary outdoor weathering tests were carried out to evaluate the green colour fastness of treated bamboo culms. In addition, fixation and leaching tests were also conducted to evaluate the safety of preservative-treated bamboo. A two-step treatment process was recommended to enhance bamboo green colour production, fungal decay resistance, production rate of treated bamboo and prevent sludge formation in treatment solution. In the treatment process, the bamboo was pressure-treated until refusal point with 4% chromated copper arsenate-3 to achieve required preservative retention and dipped into 4% chromated copper arsenate-3 for 2 days to produce green colour on the bamboo surface. The chromated copper arsenate-treated bamboo samples had outstanding colour fastness and mildew resistance after 24 months of outdoor weathering. The fixation and leaching tests demonstrated that the total amount of preservative components leached from the chromated copper arsenate-treated bamboo samples was high to raise safety issues and caused environmental problem during the use of treated bamboo in the field.

Keywords: Bamboo, chromated copper arsenate-3, green colour production, fungal decay resistance, colour fastness, safety

INTRODUCTION

The susceptibility of bamboo culm to fungus and insect attacks and the fading of its green colour over time was the major reason for bamboo products not to be preferred for exterior uses such as landscaping materials. The average service life of untreated bamboo was generally less than 3 years when it was used at ground-contact conditions (Liese 1980). Moreover, the attractive green colour of the bamboo epidermis gradually faded and discoloured into yellowish-brown and gray due to the deterioration of chlorophyll and weathering when exposed to ambient environments.

Various chemical treatments for green colour protection or conservation of bamboo were widely studied in Taiwan over the past decades (Chang & Wu 2000a, Chang & Wu 2000b, Chang & Yeh 2000, Chang & Yeh 2001, Chang et al. 2001, Chang et al. 2002, Chung et al. 2005, Chung et al. 2008, Chung et al. 2009, Chung et al. 2011, Wu et al. 2002, Wu et al. 2004, Wu et al. 2005). However, no studies on preservation of bamboo against decay from soft-rot fungi and green colour protection were conducted, even though there were a few studies on mildew resistance of chemically-treated bamboo during indoor and outdoor exposure (Chung et al. 2008, Wu et al. 2002, Tang et al. 2012). Treatment with wood preservative was important to protect bamboo products from decay and soft-rot fungi. The preservative must be absorbed into the treated bamboo products to achieve the required retention for preventing the attack of decay and soft-rot fungi. Since the high moisture content in fresh bamboo restricted the amount of preservative that could be absorbed, dried bamboo should be used for providing adequate protection against fungal biodeterioration. Preservative treatment with copper-based preservatives offered a useful solution for the problems as the green colour would be produced on the surface of treated bamboo and enhanced fungal resistance after treatment. In the case of wood, the surface colour of treated wood with copper-based preservatives such as chromated copper arsenate, alkaline copper quat, copper azole and bis-(N-cyclohexyl-diazeniumdioxy)-copper were generally greenish and became darker with the increase of preservative retention.

This study was conducted to investigate the feasibility of using copper-based wood preservative treatment for both green colour production and bamboo preservation against fungal decay. An appropriate preservative including optimum preservative concentration for green colour production and decay resistance was selected and the proper treatment method was carefully chosen to consider mass production and to avoid sludge formation. Green colour fastness was evaluated through 24-month outdoor weathering and the safety of preservative-treated bamboo was also investigated through fixation and leaching tests.

MATERIALS AND METHODS

Sample preparation

Three-year-old giant timber bamboo (*Phyllostachys bambusoides*), hachiku bamboo (*Phyllostachys nigra* var. *henosis*) and moso bamboo (*Phyllostachys pubescens*) were obtained from the planted bamboo forest in the southern part of Korea. The fresh bamboo culms were cut into strips with dimensions of 50 mm (longitudinal) × 20 mm (tangential) × 5 mm (radial) for green colour production tests and cut into 20 mm (longitudinal) × 20 mm (tangential) × 5 mm (radial) samples for decay and fixation/leaching tests. All the samples were stored at 20 °C until their moisture content reached about 12%.

Pretreatment for wax removal

The wax layer on bamboo culm was removed with the optimised treatment conditions determined in the previous study by Ra et al. (2020). Air-dried bamboo samples were treated with a mixture of 5% potassium hydroxide and 3.5% sodium dodecyl sulfate at 90 °C for 1 hour. The bamboo samples were immersed in distilled water for 24 hours to remove the residual chemicals and air-dried at room temperature until their moisture content reached about 12%.

Preservative treatments

The air-dried bamboo samples were dip-treated for 10 days and pressure-treated with 5% preservative solutions to investigate the effects of preservative types and treatment methods on green colour production. Four water-based copper-based wood preservatives such as chromated copper arsenate-3 (47.5% CrO_3, 18.5% CuO and 34.0% As_2O_5), alkaline copper quat-2 (66.7% CuO and 33.3% didecyldimethylammonium chloride), copper azole-1 (49.0% CuO, 49.0% H_3BO_3 and 2.0% tebuconazole) and bis-(N-cyclohexyl-diazeniumdioxy)-copper-1 (61.5% CuO, 24.5% H_3BO_3, and 14.0% Bis-(N-cyclohexyldiazeniumdioxy)-copper) were tested in this study. The pressure treatments were performed by the full-cell process where an initial vacuum of 760 mmHg was applied for 30 minutes and the bamboo samples were later treated under the pressure of 14 kg cm^{-2} until reaching the refusal point. After the treatment, all specimens were allowed to drip for 10 minutes and conditioned without drying at 21 °C for 2 weeks for appropriate fixation of metal components. The samples were then air-dried at room temperature for 2 weeks before measurement of surface colour.

Colour measurements

The surface colour of fresh and preservative-treated bamboo samples in the L*a*b* colour space was measured with a colourimeter. In the colour space, the chromaticity coordinate L* indicated lightness the value on the white/black axis, a* indicated the value on the red/green axis and b* indicated the value on the blue/yellow axis. The green colour of the bamboo surface was evaluated in terms of a* value. A positive a* value indicated the sample was red and a negative value was found for a green sample. Thus, a smaller value of a* indicated a greener sample.

Decay tests

Among the four treatments, only chromated copper arsenate-3 dip treatment produced the green colour on the bamboo surface. Thus, decay tests were performed to determine the minimum treatment concentration of chromated copper arsenate-3 needed to prevent fungal attack by decay and soft-rot fungus. The samples for decay tests were pressure-treated with various concentrations of chromated copper arsenate-3 solutions by the full-cell process.

After the preservative treatment, the samples were allowed to drip for 10 minutes, conditioned without drying at 60 °C for 24 hours and then air-dried at room temperature for 2 weeks. The white-rot fungus *Trametes versicolour* and the soft-rot fungus *Arthrinium phaeospermum* were used for the decay test. The reason for selecting *T. versicolour* and *A. phaeospermum* as test fungus was their high isolating frequency from bamboo used outdoors in Korea and their decay capabilities (Kim et al. 2011). The decay tests for white-rot fungus were performed according to the Japanese Industrial Standard (JIS) K 1571 (JIS 2010) while the tests for soft-rot fungus were performed according to a vermiculite burial method (Nilsson 1973).

Outdoor exposure test

In the colour fastness study, air-dried bamboo samples with dimensions of 100 mm (longitudinal) × 50 mm (tangential) × 5 mm (radial) were pretreated for wax removal and followed by a two-step process in which the bamboo samples were first pressure-impregnated until refusal point with 4% chromated copper arsenate-3 and then dipped into 4% chromated copper arsenate-3 for 2 days. After the preservative treatments, the samples were allowed to drip for 10 minutes, conditioned without drying at 21 °C for 2 weeks, and then air-dried at room temperature for 2 weeks. The treated bamboo samples were exposed outdoor for 24 months beginning from 1st May 2016 to 30th April 2018. The weathering rack was positioned facing south at an angle of 45 °C to the ground in the campus of Korea University in Seoul, Korea. The surface colour of weathered chromated copper arsenate-treated bamboo samples in the L*a*b* colour space was measured by using a colourimeter.

Fixation and leaching tests

Air-dried bamboo samples with dimensions of 20 mm (longitudinal) × 20 mm (tangential) × 5 mm (radial) were pretreated for wax removal and later treated by a two-step process consisting of the pressure impregnation of bamboo samples until reaching refusal point with 4% chromated copper arsenate-3 followed by 2 days dipping into 4% chromated copper arsenate-3. The treated samples were immediately wrapped in aluminum foil and underwent fixation without drying at 21 °C for different periods of time. The rates for fixation in the treated bamboo samples were evaluated using the expressate method (McNamara 1989). The expressed solutions were analysed for its hexavalent chromium content using the diphenylcarbazide colourimetric method (ASTM 2007). The rate of fixation at any given time was evaluated by comparing the hexavalent chromium concentration of the expressed solution with the initial treating solution.

The leaching tests were performed according to the AWPA standard E11–15 with some modifications (AWPA 2015a). After a 14-day leaching period, the combined leachate samples from the nine sampling times were analysed using inductively coupled plasma-optical emission spectrometry to determine their chromated copper arsenate components according to the procedure given in the AWPA standard A21-14 (AWPA, 2015b). The total leaching was expressed as the percent loss of each chromated copper arsenate component relative to the amount initially retained in the treated samples.

RESULTS AND DISCUSSION

Selection of the preservative type and treatment method for green colour production

The feasibility of using the wood preservative treatment for the production of green colour on the dried bamboo surface was evaluated by comparing the a* values of dip-treated and pressure-treated bamboo samples with 5% solutions of chromated copper arsenate-3, alkaline copper quat-2, copper azole-1 and bis-(N-cyclohexyl-diazeniumdioxy)-copper-1 with those of fresh bamboo. The a* values of bamboo samples dip-treated with alkaline copper quat-2, copper azole-1 and bis-(N-cyclohexyl-diazeniumdioxy)-copper-1 showed positive values regardless of bamboo species, suggested that those preservatives had no effect on the production of green colour (Table 1). On the contrary, the giant timber bamboo, hachiku bamboo, and moso bamboo samples treated with chromated copper arsenate-3 had the a* values of -14.95, -16.70 and -14.78, respectively. The results indicated that chromated copper arsenate-3 could be used for green colour production.

In the pressure treatment, no clear green colour could be produced on the bamboo surface for all the tested preservatives. The colour of the bamboo surface after the pressure treatment turned from greenish brown to light brown, which was different from the original colour of the fresh bamboo surface. A possible reason for result might be the pressure treatment could not provide sufficient time for the copper ions to adhere to the bamboo surface.

Dip treatments were performed by immersing the dried bamboo samples into each of 1%, 3% and 5% chromated copper arsenate-3 solutions for 3, 6, 12, 24, 48, 120 and 240 hours. The a* values decreased progressively with the increase of dipping time and a green colour similar to the fresh bamboo colour was developed on the treated bamboo surface after about 2 days of the dip treatment, except for the giant timber treated with 1% chromated copper arsenate-3 and moso bamboo treated with 1% chromated copper arsenate-3 (Figure 1). The a* values of fresh hachiku bamboo were at -5.53 and could be achieved by the dip treatment with 1% chromated copper arsenate-3. However, the production of green colour similar to fresh giant timber bamboo and moso bamboo could not be achieved even after 10 days dip treatment with 1% chromated copper arsenate-3. In common practical application, a short treatment time that could produce colour was more desirable. Thus, dip treatment with 3% chromated copper arsenate-3 for 2 days was recommended to produce green colour on the dried bamboo surface even though the a* values of giant timber bamboo treated with 3% chromated copper arsenate-3 for 2 days were lower than that of fresh giant timber bamboo.

The chromated copper arsenate-3 treatment for producing green colour and simultaneously enhancied fungal durability can only be used in countries where the chromated copper arsenate usage was still applicable. Non-chromated copper arsenate preservatives such as alkaline copper quat

Table 1 The values of L* a*, and b* of giant timber bamboo, hachiku bamboo and moso bamboo dip-treated with 5% copper-based wood preservatives for 10 days

Treatment types	Giant timber bamboo			Hachiku bamboo			Moso bamboo		
	L*	a*	b*	L*	a*	b*	L*	a*	b*
Untreated bamboo	33.40 (1.58)	-7.52 (0.71)	11.69 (1.72)	44.52 (4.99)	-4.98 (0.66)	9.08 (1.80)	34.79 (2.25)	-5.47 (1.04)	7.56 (2.44)
CCA-3	52.60 (0.61)	-14.95 (0.85)	21.22 (1.30)	50.37 (1.40)	-16.70 (0.39)	17.83 (0.33)	48.90 (0.53)	-14.78 (0.18)	20.29 (0.50)
ACQ-2	28.95 (3.12)	1.81 (0.20)	9.63 (3.49)	25.51 (2.78)	2.98 (0.80)	10.56 (1.68)	29.86 (4.10)	2.81 (0.58)	12.77 (3.66)
CUAZ-1	30.73 (1.21)	2.19 (0.44)	12.27 (1.51)	28.34 (1.64)	3.21 (0.65)	15.44 (2.01)	33.84 (1.78)	3.56 (0.43)	14.87 (2.11)
CuHDO-1	36.83 (0.66)	3.09 (0.54)	17.17 (0.90)	37.20 (1.32)	3.27 (0.65)	17.37 (2.08)	35.83 (0.78)	3.57 (0.33)	15.98 (2.00)

Values represented the mean of three replicates, values in a parenthesis represented standard deviation;
CCA-3 = chromated copper arsenate-3, ACQ-2 = alkaline copper quat-2,
CUAZ-1 = copper azole-1, CuHDO-1 = bis-(N-cyclohexyl-diazeniumdioxy)-copper-1;
L* = value on the white/black axis, a* = value on the red/green axis, b* = value on the blue/yellow axis

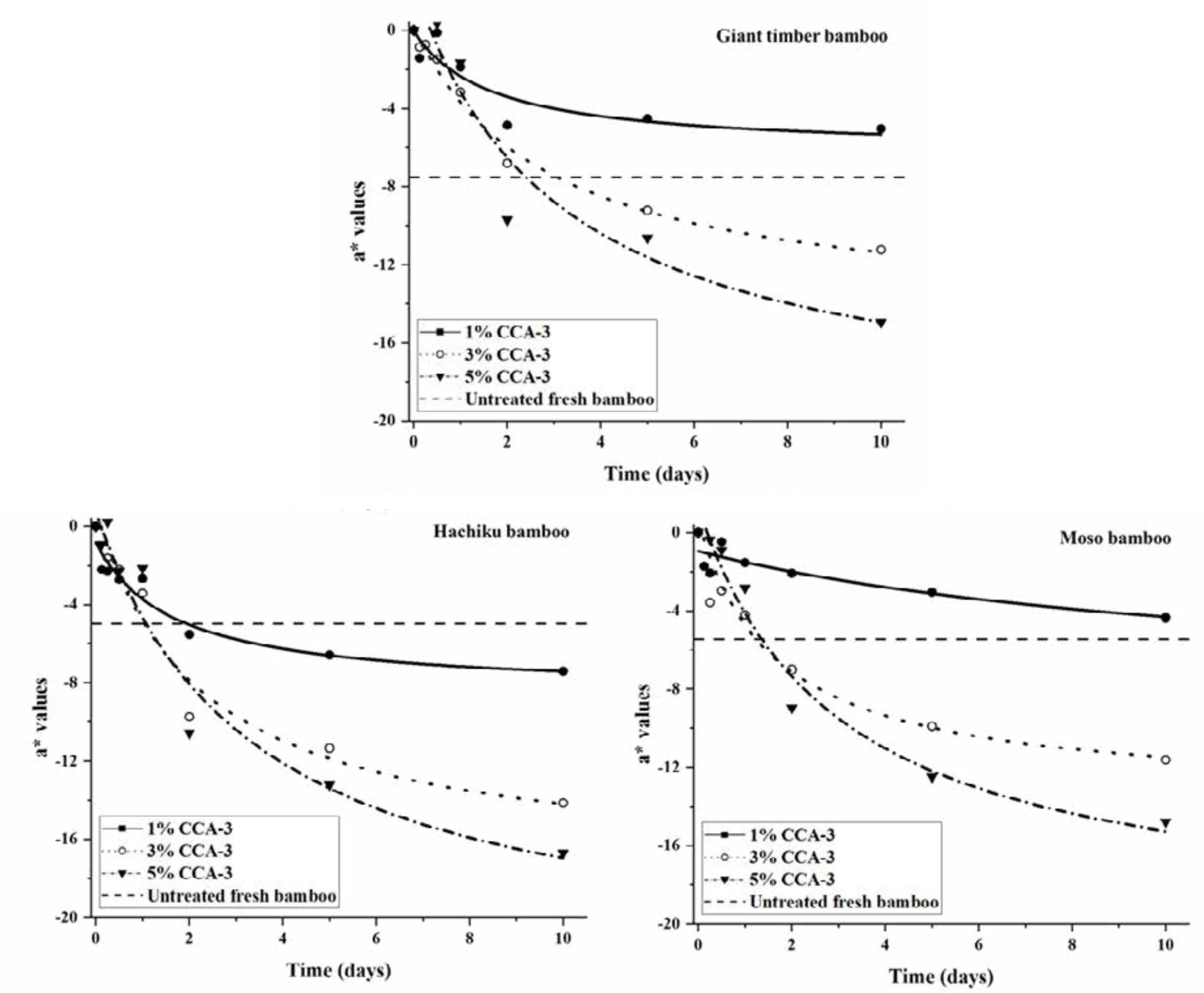

Figure 1 Changes of a* values of the dip-treated dried bamboo samples with 1, 3 and 5% chromated copper arsenate-3 solutions for various dipping times

was applied for the same purposes to replace chromated copper arsenate because its use was banned in many countries due to human health and environmental safety concerns. Such usages of the non-chromated copper arsenate preservatives alone were not sufficient for green colour production as shown in Table 1. Thus, further investigation such as including green colourants as additives in those preservatives should be explored.

Minimum chromated copper arsenate concentration for fungal decay resistance

Decay tests were conducted to determine the minimum concentration of chromated copper arsenate-3 solution effective in preventing biological deterioration caused by decay and soft-rot fungus using the AWPA standard soil-block test. The mean percent mass lost of the chromated copper arsenate-treated bamboo samples at different concentration levels were shown in Figure 2. Based on the JIS K 1571 Standard, the preservative concentration which caused mass loss 3% after a 12-week fungal exposure was the minimum concentration for effective protection against fungal deterioration. The test results showed the minimum concentrations of chromated copper arsenate-3 solution required which prevented the attack of the *T. versicolour* and *A. phaeospermum* were 1.5% and 4.0% or equivalent to 6.60 kg m^{-3} and 17.60 kg m^{-3}, respectively.

Therefore, the performance requirement of JIS K 1571 was fulfilled by pressure treatment with at least 4% chromated copper arsenate-3. The prevention against soft-rot

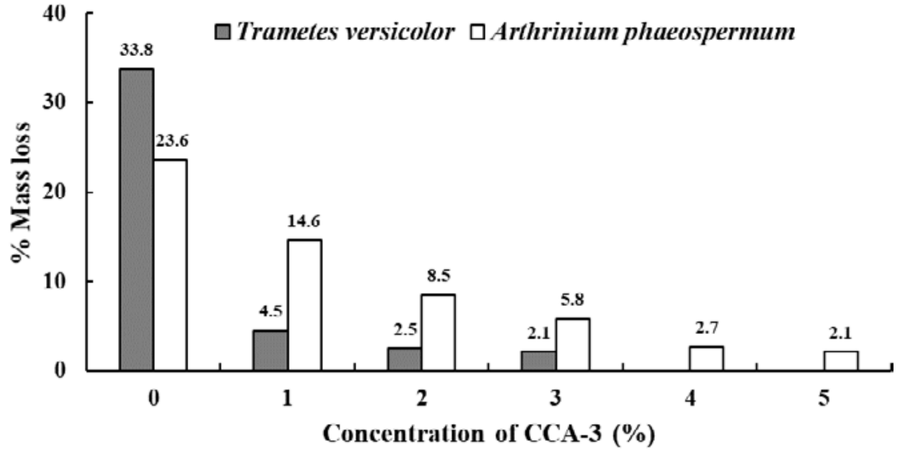

Figure 2 Effect of the concentration of chromated copper arsenate-3 on percent mass loss of treated bamboo against *Trametes versicolour* (white rot fungus) and *Arthrinium phaeospermum* (soft rot fungus)

fungi attack required higher retention of copper-containing preservatives (Daniel & Nilsson 1988). Previous studies showed the preservative uptake of the bamboo samples was lower than expected at 55 to 60% (Lee et al. 2001) and bamboo material was relatively challenging to treat than wood material (Möller & Mild 2019)

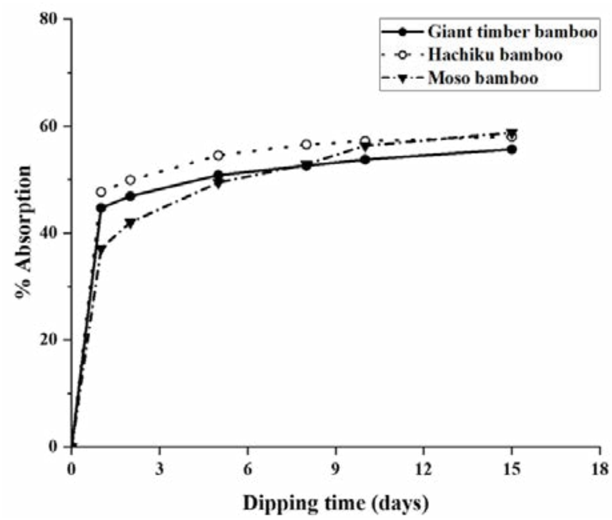

Figure 3 Changes of chromated copper arsenate-3 absorption of dip-treated bamboo samples according to dipping time

Treatment method on bamboo for green colour production and fungal resistant

Based on the above findings on the chromated copper arsenate-3 concentrations for green colour production and fungal decay resistance, bamboo samples should be treated with a 4% chromated copper arsenate-3 solution. Subsequently, green colour production could be achieved by a 2-day dip treatment with 3% chromated copper arsenate-3 and two days dipping with 4% chromated copper arsenate-3 was enough to produce green colour on the bamboo surface. However, two days dipping in 4% chromated copper arsenate-3 was impossible to achieve preservative retention in the bamboo necessary for fungal decay resistance. Therefore, an additional study was conducted to determine the optimal dipping time to achieve the same uptake of chromated copper arsenate-3 with pressure treatment until refusal point at 55.1% for giant timber bamboo, 60.8% for hachiku bamboo and 59.5% for moso bamboo. The result showed 15 days dipping in 4% chromated copper arsenate-3 solution was required (Figure 3).

Dipping treatment for 15 days resulted in a* values higher than those of fresh bamboo, indicating that 15 days was enough to produce green colour (Table 2). However, such dipping treatment was not suitable for mass production of sludge-free treated bamboo because of longer dipping time and insoluble sludge formation. The additional study demonstrated that pale green sludges were formed after 3-5 days of dip treatment. The sludge was caused by the reaction of starches in bamboo and the remaining potassium hydroxide in bamboo after the wax layer removing process with chromated copper arsenate components.

Table 2 Changes in a* values of bamboo samples after dip treatment for 15 days and two-step treatment

Bamboo species	Untreated bamboo	Dip-treated bamboo	Pressure treated and then dip-treated bamboo
Giant timber bamboo	-7.52 (0.71)	-11.07 (1.61)	-6.75 (0.35)
Hachiku bamboo	-4.98 (0.66)	9.53 (1.51)	-5.69 (0.97)
Moso bamboo	-5.47 (1.04)	11.81 (0.39)	-6.87 (0.82)

Values represented the mean of three replicates, values in parenthesis represented standard deviation

Therefore, a two-step treatment process was recommended to shorten the treatment time and to prevent sludge. Bamboo was pressure saturated until refusal point with chromated copper arsenate-3 to achieve required preservative retention and then dipped into preservative solution to produce green colour on the bamboo surface. As the result of further experiments, the chromated copper arsenate absorption reached 60% and green colour was produced on the bamboo surface after the pressure-treated bamboo samples were immersed in 4% chromated copper arsenate-3 for 2 days. There was no significant difference in the a* values between pressure-treated and 2-days dipped bamboo and fresh bamboo (Table 2).

Green colour fastness of chromated copper arsenate-treated bamboo

Variations in the a* values of the bamboo samples during outdoor weathering for 24 months were shown in Figure 4. The a* values of all the bamboo species remained relatively the same, which showed that the green colour of the chromated copper arsenate-treated bamboo samples was maintained close to fresh bamboo samples after the 24-month exposure to outdoor environment. In addition, no evidence of fungal decay and mold growth on the sample surface.

Safety of chromated copper arsenate-treated bamboo

There was a slight difference in chromium reduction between the bamboo species but the fixation time of all chromated copper arsenate-treated bamboo samples was for 12 days (Figure 5). The fixation time for chromated copper arsenate in treated bamboo was shorter than the previously known time in treated wood. The amount of chromated copper arsenate components fixed in treated bamboo was lower than treated wood due to low absorption rate

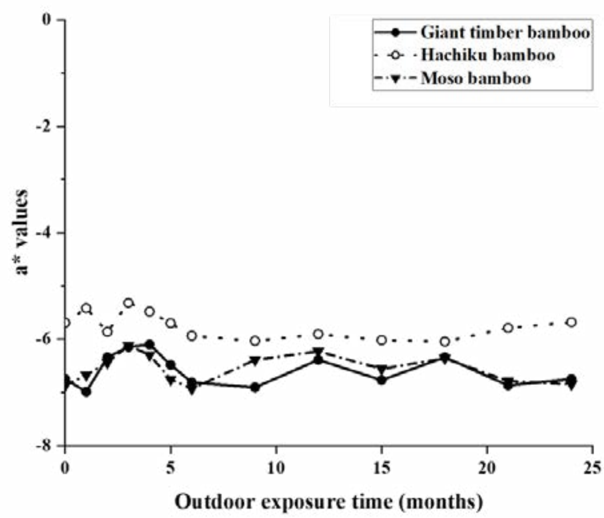

Figure 4 Changes in a* values of three bamboo species after outdoor exposure for 24 months

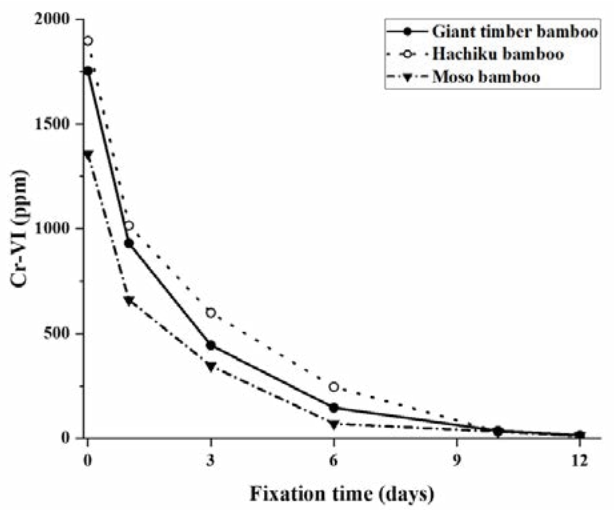

Figure 5 Comparison of fixation characteristics among three bamboo species during non-drying fixation at 21 °C

Table 3 The degree of chromated copper arsenate components leached from fully fixed chromated copper arsenate-treated bamboo

Bamboo species	Total amount of chromated copper arsenate components in leachate (ppm)		
	Chromium	Copper	Arsenic
Giant timber bamboo	0.04	0.12	0.21
Hachiku bamboo	0.03	0.09	0.22
Moso bamboo	0.04	0.11	0.19

of bamboo.

The total amount of chromium, copper and arsenic components leached from the chromated copper arsenate-treated bamboo samples after the appropriate fixation time of 12 days were shown in Table 3. No significant difference in leaching amount was observed between the bamboo species and the amount was not high to raise public concern about the risk of environmental pollution and hazard to human health. The cumulative leaching amount of chromated copper arsenate components was in the order of arsenic > copper > chromium and the same trend was observed in chromated copper arsenate treated wood.

CONCLUSIONS

Dip treatment for dried bamboo culm with 3% chromated copper arsenate-3 at ambient temperature for 2 days produced green colour regardless of bamboo species. Decay tests showed that at least 4% chromated copper arsenate-3 should be used for the pressure treatment to obtain enough retention for protection against fungal decay attack. Based on the findings, dried bamboo culm should be treated with 4% chromated copper arsenate-3 solution for the green colour production and fungal resistant. A two-step treatment process in which bamboo samples were pressure treated until refusal point with 4% chromated copper arsenate-3 to achieve required preservative retention and then dipped into 4% chromated copper arsenate-3 for 2 days to produce green colour on the bamboo surface was recommended. The recommendation managed to reduce the 15 days dip treatment and helped in the mass production of sludge-free chromated copper arsenate-treated bamboo. The chromated copper arsenate-treated samples exhibited excellent green colour fastness, resistance to fungal decay and discolouration after 24-month exposure to outdoor weathering. The leaching amount of preservative components from chromated copper arsenate-treated bamboo were not high to raise potential public health and ecological risks after the complete fixation of treated bamboo.

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