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ORIGINAL ARTICLE

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Environmentally benign methods for producing green culms of ma bamboo (*Dendrocalamus latiflorus*) and moso bamboo (*Phyllostachys pubescens*)

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Abstract The objective of this study was to find an effective method for treating ma bamboo (*Dendrocalamus latiflorus*) and moso bamboo (*Phyllostachys pubescens*) using new water-based reagents containing copper. The effects of green-color protection using various treatments on bamboo culms were examined in this study. Two methods were used: heating in a water bath and ultrasonic dipping. The results revealed that excellent green-color protection (a^* value of –6.2) was obtained when ma bamboo culms were treated with 0.25% ammoniacal copper quaternary compound-type B (ACQ-B) in a water bath at 100°C for 2 h. It was also found that the wettability of bamboo epidermis increased significantly after pretreatment in a mixture of 1% KOH and surfactant in a water bath at 100°C for 30 min. Furthermore, pretreated moso bamboo culms exhibited excellent green-color protection after they were treated with 0.25% ACQ-B at 100°C for 2 h (a^* value of –8.2). This novel treatment method definitely endows the bamboo culms with a fascinating green skin color and consequently could increase the economic value of bamboo products. No improvement in green-color protection was found when ultrasonic energy was added to the water bath at ambient temperature.

Key words Green-color protection · Bamboo · *Dendrocalamus latiflorus* · *Phyllostachys pubescens* · ACQ-B

Introduction

In Taiwan, from the coastline to 3000-m-high mountains, bamboo can be found in enormous quantities all over the island. Overall, Taiwan has over 60 species of bamboos, both native and imported.[1,2] Among them, ma bamboo (*Dendrocalamus latiflorus* Munro) and moso bamboo (*Phyllostachys pubescens* Mazel) are two of the most common and also the most valuable species. When combined, the acreage of these two species exceed 53% of the total in Taiwan.[3] In Taiwan, bamboo craftworks are extremely popular and widely used in daily life, and have been so since ancient times to the modern day because of the bamboo culms' high growth rate, availability, renewable nature, and short maturity cycle.[4] Among all of its characteristics, bamboo has fascinated people with its bright green color embedded in its surface. In view of this, bamboo has become one of the most important nontimber forest products in Taiwan and other Asian countries, and has been used as construction materials, composite materials, a handicraft resource, and other purposes.[5]

However, bamboo culm is susceptible to attack by fungi and insects, and the attractive green color of its epidermis tends to fade over time, consequently reducing the service life of bamboo products. The durability of bamboo products depends on the climatic conditions and the environment to which the products are exposed.[1] Untreated bamboo may only have an average life of less than 1–3 years when it is exposed to atmosphere and soil.[6] Because of this, green-color protection and preservation of bamboo has been widely studied in Taiwan over the past decade.[7–17] In previous investigations it has been reported that new water-based copper-containing reagents such as ammoniacal copper quaternary compound-type B (ACQ-B) have been proven to be effective green-color protectors for makino bamboo (*Phyllostachys makinoi* Hayata).[16] To meet the requirements of mass production without causing environmental pollution, it is imperative to keep suitable treatment process characteristics without using organic solvents. It is also important to keep the toxicity level of the reagents low. With all these considerations, the objective of this study was to find an effective and environmentally friendly reagent and suitable methods for the applications of protecting ma bamboo and moso bamboo culms and enhancement of the surface color. The effectiveness of green-color protection using two commercially available low-toxicity copper-

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based preservatives in different treatment conditions was evaluated.

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 National Taiwan University in Nan-Tou County. The bamboo culms were cut into strips with dimensions of 50 mm (longitudinal) × 15 mm (tangential) × 4 mm (radial) and stored at 4°C without exposure to light prior to use.

Chemical treatment

To study the effectiveness of protection of the green-color surface, two water-based preservatives, containing ammoniacal copper quaternary compound-type B (ACQ-B) and copper azole compound (CuAz), respectively, were used in the experiment.[16] For pretreatment, reagents including water-based surfactant and potassium hydroxide (KOH), were used in this study to prepare the samples before color treatment.[12] All reagents were purchased from Acros Organics (Geel, Belgium).

Bamboo specimens were treated with 2% (w/w) preservatives in a water bath at 100°C for 2 h (alkali-pretreatment-free process) before the experiment. The main parameters of the experiment included the treatment methods (one-step and two-step), reagent concentrations (0%, 0.1%, 0.25%, 0.5%, 1%, 2%, and 4%), temperatures (25°, 40°, 60°, 80°, and 100°C), heating durations (0.25, 0.5, 1, 1.5, 2, 3, and 4 h), and soaking durations (0.5, 1, 1.5, 2, 4, and 6 days). It has been suggested that ultrasonic energy can be used to break up plant cell walls to facilitate the release of extractable compounds and to enhance absorption of solvent and chemical reagent. Therefore, an ultrasonic treatment method was also employed in this study to evaluate the influence of ultrasonic energy on the color of bamboo surface. In the ultrasonic experiments, the dipping treatment under ambient temperature was combined and carried out in an ultrasonic bath (Branson PC620, power 180 watts; output frequency 44 kHz).[14–16] After the treatment process was completed, 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 using a color difference meter (Dr. Lange, 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 the data, the L^* (value on the white/black axis), a^* (value on the red/green axis), b^* (value on the blue/yellow axis) color parameters were calculated using the method established by the Commission Internationale de l'Eclairage (CIE) in 1976.[7,8]

Characteristics of specimen epidermis

The bamboo surface morphology was examined and element mapping and line scanning were performed using a scanning electron microscope (SEM, Hitachi S-2400, Japan). Critical-point-dried specimens were taped onto a circular holder and coated with carbon. Carbon-coated samples were imaged at 15 keV.[14]

Wettability of specimen epidermis

The contact angle of a water bead 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.[9]

Analysis of variance

All results are expressed as mean ± standard deviation (SD) ($n = 9$). The significance of difference was calculated using the SAS Scheffe's test and values of $P < 0.05$ were considered significant.

Results and discussion

Color variations of bamboo treated with copper-based preservatives

Recently, some studies revealed that copper-based reagents are good candidates for green-color protectors of makino bamboo.[14,16] Hence, development of a suitable treatment process for low-toxicity reagents without organic solvent for the protection of the green color of ma and moso bamboo should also be considered. ACQ-B and CuAz are not only copper-based reagents, but are also commercially available wood preservatives.[18,19] Because ACQ-B and CuAz have been found to be effective color preservers for makino bamboo, they were selected as possible green-color protectors for ma bamboo and moso bamboo. Color variations in ma bamboo and moso bamboo culms treated with these two protectors were evaluated using the CIE LAB color specifications.[7–17]

In general practice, green color is evaluated in terms of the a^* value. For example, the color of untreated ma bamboo and moso bamboo culms fade easily after oven drying, and this is evidenced by the increased a^* value (from –9.2 and –4.4 to 6.9 and 7.4, respectively) (Table 1). Furthermore, the a^* values of ma bamboo treated with two types of 2% copper-based preservatives at 100°C for 2 h (without pretreatment process) showed that 2% ACQ-B ($a^* = –4.1$) was more effective than 2% CuAz ($a^* = –1.7$) (Table 1). This finding reveals that CuAz has no effect in green-color protection, but ACQ-B may be a suitable green-color protector. Possible reasons for these different results may be the different compositions of the treatment solutions, including different ingredients and their ratios in the formulations, and the different solubilizing agents (ammonia and ethanolamine, respectively) used for ACQ-B and CuAz copper-based reagents. When moso bamboo was treated using 2% ACQ-B, the CIE LAB color parameters *L**, *a**, and *b**, as shown in Table 1, changed from the initial values of 35.4, –4.4, and 14.8 (fresh bamboo) to 32.8, –3.1, and 14.3, respectively. This revealed that green-color protection was obtained when ma bamboo and moso bamboo culms were treated with 2% ACQ-B in a water bath. Similar results were obtained in the previous study, when it was found that green-color protection of makino bamboo culms was achieved after treatment with 2% ACQ-B.[16]

Effect of protector concentrations on the surface color of bamboo culms

With water-based ACQ-B found to be an effective green-color protector for ma bamboo and moso bamboo, more detailed studies on the treatment conditions were conducted. First, to understand the influence of ACQ-B concentrations on the color of ma bamboo and moso bamboo, six concentrations of 0.1%, 0.25%, 0.5%, 1%, 2%, and 4% were examined. Table 2 shows the changes of color parameters for ma bamboo and moso bamboo epidermis after treatment with various concentrations of ACQ-B at 100°C for 2 h. The *a** values of ma bamboo treated with 0%, 0.1%, 0.25%, 0.5%, 1%, 2%, and 4% ACQ-B were 6.4, –1.0, –6.2, –6.5, –5.8, –4.1, and –2.5, respectively. Among these, the 0.25%, 0.5%, and 1% ACQ-B treatments gave the best green color without statistically significant difference between them.

Moreover, for moso bamboo, Table 2 also shows the changes of *a** values for bamboo epidermis after treatment with various concentrations of ACQ-B. The *a** values of moso bamboo treated with 0%, 0.1%, 0.25%, 0.5%, 1%, 2%, and 4% ACQ-B were 3.6, –1.2, –4.9, –1.4, –0.6, –3.1, and 4.2, respectively. Of these, the 0.25% ACQ-B treatment exhibited the best green-color performance (*a** values for bamboos treated with concentrations higher than 0.25% were not significantly different according to Scheffe's test). In summary, it is clear that ma bamboo and moso bamboo epidermis are afforded effective green-color protection after treatment with 0.25% ACQ-B at 100°C for 2 h.

Influence of treatment methods on the surface color of bamboo culms

To illustrate the effects of treatments on green-color protection, scanning electron microscopy (SEM) was also used to investigate the morphological changes of moso bamboo

Table 1. Surface color of ma bamboo and moso bamboo culms treated with 2% copper-based preservative

Chemical	CIE LAB					
	Ma bamboo			Moso bamboo		
	*L**	*a**	*b**	*L**	*a**	*b**
Control[a]	33.6 ± 0.5	–9.2 ± 0.8	9.9 ± 1.0	35.4 ± 0.7	–4.4 ± 1.2	14.8 ± 1.2
Untreated	64.3 ± 0.4	6.9 ± 1.1	30.7 ± 0.8	58.3 ± 0.6	7.4 ± 1.4	26.4 ± 1.0
CuAz	46.6 ± 1.0	–1.7 ± 1.3	26.9 ± 1.1	43.6 ± 0.2	–1.2 ± 0.2	17.0 ± 0.7
ACQ-B	37.0 ± 1.3	–4.1 ± 0.7	19.3 ± 1.4	32.8 ± 1.5	–3.1 ± 0.3	14.3 ± 1.3

CIE LAB, Color specifications of Commission Internationale de l'Eclairage; CuAz, copper azole; ACQ-B, ammoniacal copper quaternary compound-type B
[a] Fresh bamboo

Table 2. Changes in the surface color of ma bamboo and moso bamboo culms treated with various concentrations of waterborne ACQ-B at 100°C for 2 h

ACQ-B concentration (%)	CIE LAB					
	Ma bamboo			Moso bamboo		
	*L**	*a**	*b**	*L**	*a**	*b**
Control[a]	33.6 ± 0.5	–9.2 ± 0.8^{F}	9.9 ± 1.0	35.4 ± 0.7	–4.4 ± 1.2^{D}	14.8 ± 1.2
0	47.5 ± 0.2	6.4 ± 0.3^{A}	32.3 ± 0.4	54.2 ± 0.7	3.6 ± 0.4^{A}	18.3 ± 0.9
0.1	51.3 ± 0.2	–1.0 ± 0.7^{B}	30.8 ± 0.3	46.2 ± 1.2	–1.2 ± 1.0^{B}	22.7 ± 1.6
0.25	50.8 ± 0.4	–6.2 ± 0.8^{E}	25.2 ± 0.5	35.3 ± 0.5	–4.9 ± 1.0^{D}	14.2 ± 0.6
0.5	49.2 ± 1.1	–6.5 ± 0.2^{E}	27.9 ± 0.1	30.8 ± 1.4	–1.4 ± 0.5^{B}	15.0 ± 0.4
1	45.1 ± 0.2	–5.8 ± 0.3^{DE}	26.6 ± 0.4	35.8 ± 1.0	–0.6 ± 0.7^{B}	15.1 ± 0.7
2	37.0 ± 1.3	–4.1 ± 0.7^{D}	19.3 ± 1.4	32.8 ± 1.5	–3.1 ± 0.3^{C}	14.3 ± 1.3
4	37.2 ± 1.0	–2.5 ± 0.9^{C}	22.8 ± 1.3	27.4 ± 0.3	4.2 ± 0.6^{A}	7.6 ± 0.3

*a** values marked with different letters are significantly different at the level of $P < 0.05$ according to Scheffe's test
[a] Fresh bamboo

epidermis in this study. Figure 1 shows that the whitish sedimentary mass on fresh moso bamboo culms is a layer of wax, and we also found that moso bamboo has a thicker layer of wax than ma bamboo. We have previously found that wax layers inhibit the penetration and reaction of green-color protectors, and that the combination of alkali pretreatment followed by a green-color protector treatment was able to improve the green bamboo epidermis.[9] In light of this, we also evaluated the influence of treatment methods, including one-step and two-step methods, on color of moso bamboo culms.

As shown in Table 3, the two-step ACQ-B-treated specimens (a^* = –8.2) had better green-color performance than the one-step ACQ-B-treated (a^* = –5.2) specimens. This is because the pretreatment of moso bamboo with 1% KOH–surfactant mixture is an efficient and reliable method for removing the siliceous wax layer. In addition, it was observed that the contact angle decreased from an initial value of 83.6° to 60.3° after the two-step treatment, indicating the enhanced hydrophilic property of the epidermis, which promotes reactions between water-based protectors and the bamboo epidermis. From the aforesaid results, it was also realized that a thicker wax layer on the bamboo epidermis will inhibit the penetration of water-soluble reagents and reaction of green color. These results demonstrated that moso bamboo could achieve premium color protection by using a two-step treatment with waterborne copper-based preservative.

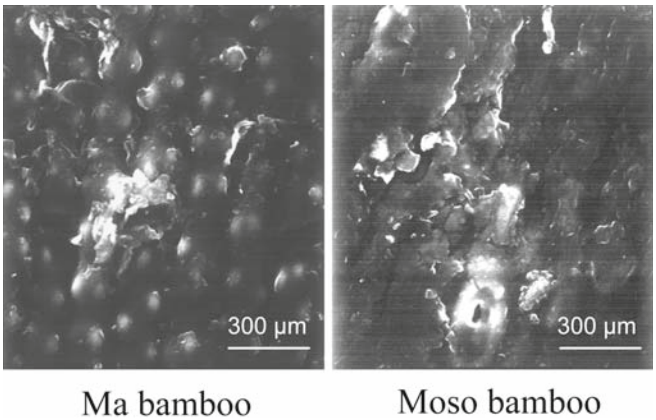

Fig. 1. Scanning electron micrographs of surface structure of ma bamboo (*left*) and moso bamboo (*right*) culms

Effects of treatment temperature on green-color protection

To understand the influence of treatment temperature on green-color protection, five temperatures of 25° (room temperature), 40°, 60°, 80°, and 100°C were used in this study. For ma bamboo treated with 0.25% ACQ-B, the results in Table 4 reveal that the a^* value decreased when the temperature was raised from room temperature to 100°C; the a^* values were 4.5 (25°C), 2.7 (40°C), 1.6 (60°C), –4.2 (80°C), –6.2 (100°C). In other words, among the temperatures examined, the best green-color performance was obtained when the ma bamboo culms were processed at 100°C. Similarly, moso bamboo treated with 0.25% ACQ-B showed similar results. After treatment with 0.25% ACQ-B at 100°C for 2 h, the a^* value of moso bamboo was –8.2

Table 3. Effects of two different treatment methods on the color parameters of moso bamboo surface

Treatment process	L^*	a^*	b^*	Contact angle (°)
Fresh	35.4 ± 0.7	–4.4 ± 1.2	14.8 ± 1.2	83.6
One-step[a]	53.1 ± 0.7	–5.2 ± 0.6	28.8 ± 0.6	70.2
Two-step[b]	35.1 ± 0.5	–8.2 ± 1.0	16.7 ± 0.6	60.3

[a] Treated with 0.25% ACQ-B solution at 100°C for 2 h
[b] Pretreatment: mixture of 1% KOH and 1% surfactant at 100°C for 30 min in water bath. ACQ-B treatment: 0.25% ACQ-B solution at 100°C for 2 h

Table 4. Influence of treatment temperature on the color parameters of ma bamboo and moso bamboo culms treated with 0.25% ACQ-B solution

Temperature (°C)	CIE LAB					
	Ma bamboo[a]			Moso bamboo[b]		
	L^*	a^*	b^*	L^*	a^*	b^*
Control[c]	33.6 ± 0.5	–9.2 ± 0.8	9.9 ± 1.0	35.4 ± 0.7	–4.4 ± 1.2	14.8 ± 1.2
25	45.2 ± 0.3	4.5 ± 0.8	26.4 ± 0.4	52.2 ± 0.9	–0.4 ± 0.5	24.6 ± 1.1
40	47.9 ± 0.4	2.7 ± 0.4	24.6 ± 0.2	46.5 ± 0.8	–0.3 ± 0.8	25.6 ± 0.5
60	45.4 ± 1.9	1.6 ± 1.6	26.2 ± 1.3	45.2 ± 0.4	–0.3 ± 0.5	24.3 ± 0.1
80	44.6 ± 0.9	–4.2 ± 1.3	27.1 ± 0.1	44.4 ± 0.3	–3.7 ± 0.8	23.7 ± 0.2
100	50.8 ± 0.4	–6.2 ± 0.8	25.2 ± 0.5	35.1 ± 0.5	–8.2 ± 1.0	16.7 ± 0.6

[a] Green-color protection were treated with 0.25% ACQ-B solution for 2 h
[b] Pretreatment: mixture of 1% KOH and 1% surfactant at 100°C for 30 min in water bath. Green-color protection were treated with 0.25% ACQ-B solution for 2 h
[c] Fresh bamboo

(Table 4), the lowest among the temperatures employed in the experiment. It is speculated that the high temperature not only melted down wax layers easily and quickly but also promoted the reactions between the protector and bamboo epidermis, consequently enhancing the effectiveness of green-color protection.

Effects of treatment time on green-color protection

A short treatment time is desirable for practical applications in the manufacturing of green bamboo products.[14,15] Therefore, seven different treatment times (0.25, 0.5, 1, 1.5, 2, 3, and 4 h) were examined for green-color protection. The results are shown in Fig. 2. First, the lowest *a** value of ma bamboo culms treated with 0.25% ACQ-B at 100°C for 2 h was –6.2. The *a** values decreased progressively when the treatment time was increased up to 2 h. The *a** values of moso bamboo culms treated with 0.25% ACQ-B at 100°C for 1.5 and 2 h were –6.3 and –8.2, respectively. However, the *a** values started to increase when the treatment time exceeded 2 h. With treatment for 3 and 4 h, the *a** values increased to –4.3 and 0.3, respectively. Hence, taking production cost into consideration, a treatment time of 2 h would be the best choice for producing ma bamboo and moso bamboo culms with an excellent green color.

Effects of dipping treatment on green-color protection

To find the best conditions for dipping treatment at room temperature (about 25°C), ma bamboo and moso bamboo culms were treated in 0.25%, 0.5%, and 1% ACQ-B for six different treatment durations of 0.5, 1, 1.5, 2, 4, and 6 days. Results of dipping treatments under ambient conditions are shown in Fig. 3. No green-color protection was detected when ma bamboo was treated with 0.25%, 0.5%, or 1% ACQ-B aqueous solutions. The *a** value reached a minimum reading of –2.7 when moso bamboo specimens were immersed in an aqueous solution of 0.25% ACQ-B for 2 days. However, *a** values increased when the dipping time exceeded 2 days. With treatment for 4 and 6 days, *a** values changed to –2.2 and –2.6, respectively. Observations also revealed that the epidermis of treated bamboo remained shiny, indicating that the wax layer cannot be removed completely, resulting in lower wettability and permeability between protectors and bamboo epidermis.

In our previous studies, results clearly revealed that heat treatment with ultrasonic energy, which has been widely used in many fields over the past decade, was a feasible and reliable method for protection of bamboo color.[14–17] However, the treatment effect of ultrasonic energy on the color protection of bamboo under ambient conditions has not been investigated prior to the present study. We used ultrasonic treatment at ambient temperature to investigate the color changes of ma bamboo and moso bamboo epidermis. As discussed above, moso bamboo is not afforded green-color protection when specimens are treated with ACQ-B at ambient temperature (about 25°C). Thus, to further understand the influence of ultrasonic treatment on the color of ma bamboo and moso bamboo culms, the same treatment conditions were employed except that the water bath was replaced by an ultrasonic bath. Samples treated with five different ACQ-B concentrations of 0.25%, 0.5%, 1%, 2%, and 4% for 2 h using the ultrasonic treatment were examined. The results in Fig. 4 demonstrate that the *a**

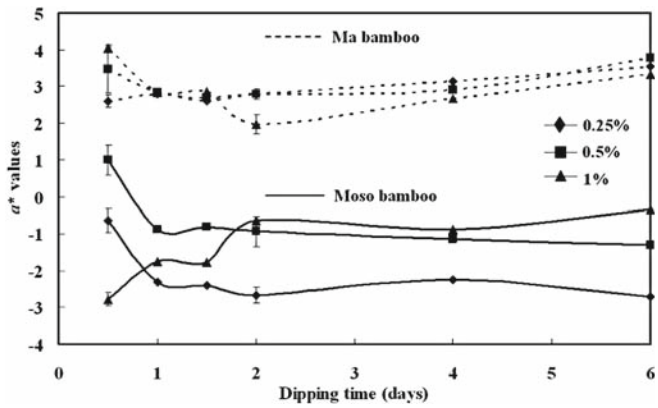

Fig. 3. Changes in *a** value of ma bamboo and moso bamboo treated with 0.25%, 0.5%, and 1% ACQ-B solution under ambient conditions for 6 days

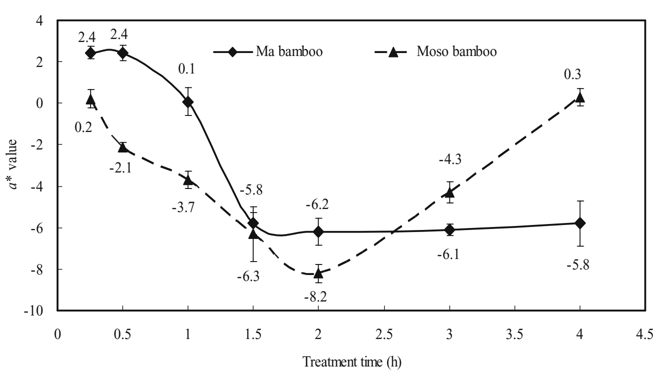

Fig. 2. Changes in *a** value for ma bamboo and moso bamboo culms after treatment with 0.25% ammoniacal copper quaternary compound-type B (ACQ-B) at 100°C for different times

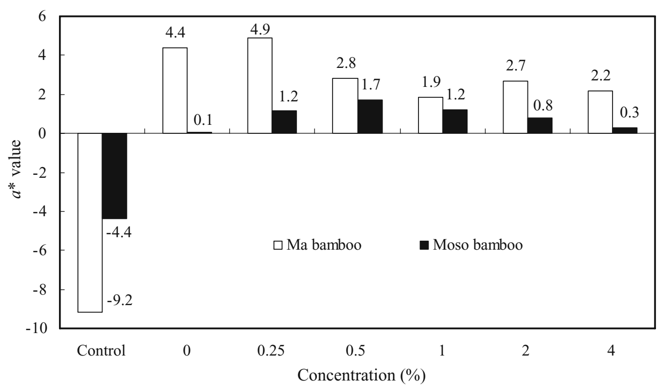

Fig. 4. Changes in *a** value of ma bamboo and moso bamboo treated with different concentrations of ACQ-B solution for 2 h using ultrasonic treatment

values were all above zero when ma bamboo and moso bamboo culms were treated with different concentrations of ACQ-B for 2 h. These results contrast with those from a previous study that used ultrasonic treatment and heating; the *a** value of makino bamboo culms after treatment with 2% ACQ-B at 60°C in an ultrasonic bath for 15 min was –10.0.[16] These results indicate that without heating, green-color protection of ma bamboo and moso bamboo cannot be achieved by using ultrasonic treatment at ambient temperature.

Conclusions

New water-based, copper-containing preservatives were used in this study to investigate their effects on green-color protection and surface properties of ma bamboo and moso bamboo culms. The results showed that the best treatment procedure for green-color protection with the *a** value of –6.2 was achieved by treating ma bamboo in 0.25% ACQ-B solution at 100°C for 2 h. For moso bamboo, the results showed that pretreated culms (mixture of 1% KOH and surfactant at 100°C in a water bath for 30 min) showed excellent green-color protection after treatment with 0.25% ACQ-B at 100°C for 2 h with an *a** value of –8.2. Green-color protection was not able to be enhanced by ultrasonic treatment. These results will expand the potential utilization of ACQ-B preservative and increase the economic value of bamboo products because this novel treatment method endows the bamboo culms with a fascinating green skin color.

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