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ORIGINAL ARTICLE

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Efficacy of wood preservatives formulated from okara with copper and/or boron salts

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Abstract As a substitute for high-cost copper azole (CuAz) and alkaline copper quaternary (ACQ) wood preservatives, alternative wood preservatives were formulated with okara, which is an organic waste from the production of tofu, and copper chloride and/or borax. Each preservative was used in treatment of wood blocks in a reduced-pressure method to measure its treatability. The treated wood blocks were placed in hot water for 3 days to examine the stability of the preservatives against hot-water leaching. The preservatives successfully penetrated into wood blocks, probably due to the use of ammonium hydroxide as a dissociating agent. However, the stability of okara-based preservatives dropped as the concentration of acid in the solutions used for hydrolysis of okara increased. The treatability and leachability of the preservatives were not affected by hydrolysis temperature but were negatively affected by the addition of borax. Leached wood blocks treated with okara-based preservatives and exposed to decay fungi *Gloeophyllum trabeum* and *Postia placenta* over 12 weeks showed good decay resistance. Okara-based wood preservatives can protect wood against fungal attack as effectively as CuAz, and have potential for use as environmentally friendly wood preservatives.

Key words Okara · Wood preservatives · Leachability · Treatability · Decay resistance

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Introduction

Chromated copper arsenate (CCA)-treated wood has been extensively used for over 60 years as a building material. In the United States, 65.3 million kilograms of CCA solution was used, and approximately 21 million cubic meters of CCA-treated lumber were produced annually in 1996.[1,2] However, several studies suggested that leaching of preservative components, such as chromium and arsenate, from CCA-treated wood may be harmful to human health and the environment.[3–6] The Environmental Protection Agency consequently prohibited the use of CCA-treated woods for residential purposes from 2004, but CCA is still widely used to protect wood for exterior uses.[7] More importantly, the disposal of CCA-treated waste wood will cause serious environmental problems. Humar et al.[2] reported that the volumes of CCA-treated wood disposed into landfills in the United States will reach 16 million cubic meters in 2020. Therefore, a viable nontoxic and nonpolluting alternative to CCA is needed as a wood preservative.

During the past 15 years, wood industries have been engaged in a considerable research and development program to make effective and environmentally more acceptable wood preservatives. As a result, alkyl ammonium compounds and azoles have been used as wood preservatives since the late 1990s.[8] However, due to the high costs of the preservatives when compared with CCA, several researchers have still sought new preservative systems. Recent approaches to obtain a wood preservative that is effective, environmentally benign, and cost-competitive have led to the use of renewable resources for the fixation of metal salts in a wood structure. For example, lignin,[9,10] tannin,[11] animal proteins such as egg albumin and milk casein,[12] and plant proteins such as soy flour and soy isolates[13,14] were used as fixatives of copper and/or boron salts in their new preservative system. These studies showed that the preservatives had very good decay resistance against fungal decay.

In Korea, renewable resources, such as soy, corn, lignin, and tannin, cannot be used as a fixative of the newly developed preservative system due to their high cost or limited supply. In our study, okara, the residue left in the manufacture of soymilk and tofu from ground soybeans by water extraction was chosen as a fixative because it is readily available with very low cost or no cost.[15] For instance, approximately 310 000 tons of okara, in Korea, was obtained from tofu production in 2004 (Han 2006; Personal communication), but most was dumped and burnt as waste. An additional reason for the suitability of okara is its chemical composition. Dry okara contains about 27% protein, 53% fiber and carbohydrates, 12% fats and oils, and 8% ash by weight. Proteins can be used for chelating boron and heavy metals such as copper and zinc to form water-insoluble complexes. The availability and protein content of okara are such that okara has a potential to be used as a fixative in wood preservative formulations.

Therefore, the aims of this study were: (1) to formulate okara-based wood preservatives containing copper and boron salts, (2) to entrain the effective components of the preservatives into wood blocks, (3) to measure the stability of wood blocks treated with the preservatives against leaching, and (4) to determine the efficacy of the preservatives against fungal decay.

Materials and methods

Preservative formulations

Table 1 shows the type of preservative formulations prepared for this study. The preservative formulations were made with okara (OK), copper chloride ($CuCl_2$, CC) or/and sodium borate ($Na_2B_4O_7.10H_2O$, B). Okara was obtained from a tofu manufacturing company (CJ Food, Seoul), and was stored in a freezer at –4°C before use in preservative formulations.

The procedures for formulating preservative solutions are as follows. Okara was hydrolyzed in sulfuric acid solutions, before being reacted with metal salts. To examine the effects of hydrolysis conditions on the treatability and leachability of the okara-based preservatives, the acid concentration of the solution was adjusted to 0%, 1%, 2.5%, and 5% at two different temperatures (25°C and 80°C). Then metal salts were added into okara hydrolyzates to obtain a suspension of preservatives. In the suspension, the weight ratio of OK to CC on a dry weight basis was 1:1, and that of OK to B was 2:1. The target retentions of OK, CC, and B into wood blocks were 12, 12, and 6 kg/cm^3, respectively. Before wood blocks were treated by the suspension, 50 ml ammonium hydroxide (NH_4OH) was added into the suspension. To identify the efficacy of okara-based wood preservatives, copper azole (CuAz) preservatives were also used for this study. The CuAz-B type was obtained from Joong-Dong (Incheon, South Korea), and formulated to 2% solutions.

Treatment of wood blocks with wood preservatives

Wood blocks of dimensions 2.54 × 2.54 × 2.54 cm (L, R, T) from Scots pine sapwood (*Pinus sylvestris*) were prepared for this study. Wood blocks were impregnated with each 4% okara-based preservative solution under reduced pressure (500 mmHg) for 30 min, and subsequently treated by 1.177 MPa pressure for 30 min in a pressure cylinder (Joong-Dong, Incheon, South Korea). To measure the solution uptake of wood blocks in preservative formulations, treated wood blocks were conditioned for 24 h and then oven-dried

Table 1. Formulations of okara-based preservatives and their treatability and leachability

Hydrolysis conditions of okara		Metal salts	Treatability[a] (%)	Leachability[b] (%)
Acid concentration (%)	Temperature (°C)			
–	–	$CuCl_2$	116.33 (19.56)	3.18 (0.78)
0	25		96.91 (10.22)	2.36 (1.33)
1	25		133.70 (9.65)	4.10 (0.56)
2.5	25		138.23 (14.37)	2.72 (1.05)
5	25		177.85 (19.26)	5.55 (1.26)
0	80		81.51 (15.12)	2.74 (0.88)
1	80		138.01 (5.68)	2.59 (1.43)
2.5	80		148.08 (14.30)	4.56 (1.01)
5	80		154.89 (33.96)	6.23 (1.85)
–	–	$CuCl_2$ + borax	89.30 (10.48)	3.75 (0.49)
0	25		76.80 (13.55)	4.69 (0.73)
1	25		89.11 (18.36)	4.79 (0.88)
2.5	25		98.73 (27.24)	5.82 (1.38)
5	25		124.22 (12.41)	7.03 (0.69)
0	80		70.05 (16.02)	3.79 (0.72)
1	80		87.22 (10.00)	4.77 (0.48)
2.5	80		110.51 (20.45)	5.98 (1.18)
5	80		132.86 (37.69)	7.13 (1.19)
		CuAz[c]	114.23 (57.68)	6.18 (2.31)

Numbers in parentheses are standard deviations
[a] Percentage of actual retention relative to the target retention
[b] Percentage preservative leached from treated specimens
[c] CuAz-B (2%) is composed of copper (96.1%) and azole as tebuconazole (3.9%)

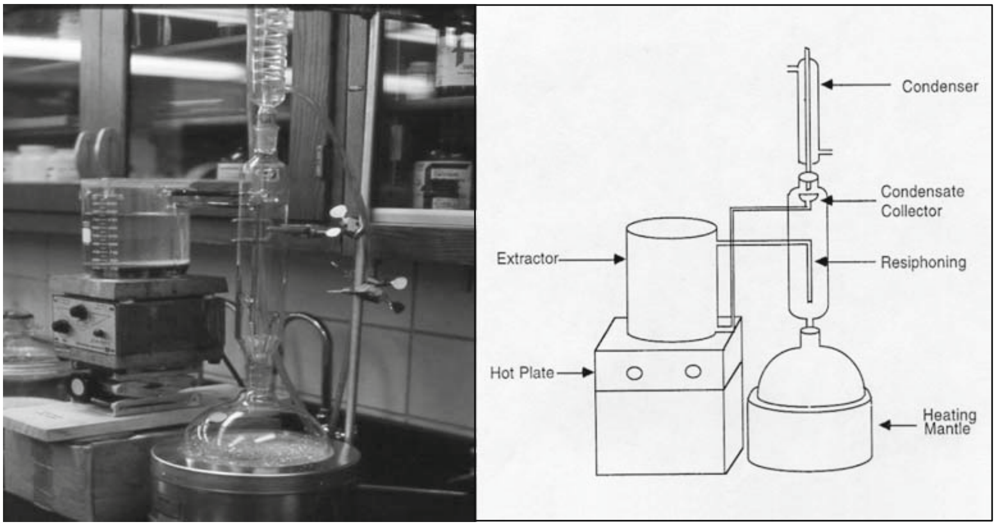

Fig. 1. The image (*left*) and schematics (*right*) of the extraction apparatus for studying leachability of wood preservatives

at 75°C for 24 h. The difference between the original dry weight of wood blocks and its final dry weight after treatment was regarded as the treatability of each preservative formulation.

Leaching procedure

Prior to exposure to decay fungi, treated wood blocks were subjected to hot-water leaching to evaluate the stability of each preservative formulation. As shown in Fig. 1, saturated wood blocks were put in a 3-l extractor and leached in hot water (70°C) for 72 h.[12] The hot water was replaced with fresh condensate at a rate of approximately 350 ml/h. After leaching, wood blocks were air-dried for 24 h, oven-dried at 80°C overnight, and weighed. Leachability of each preservative formulation is expressed as a percent weight loss of the wood blocks treated by a preservative formulation due to hot-water leaching.

Decay resistance of leached wood blocks

Brown-rot fungi *Postia placenta* (POP, KCTC No. 6671) and *Gloeophyllum trabeum* (GLT, KUC No. 8013) were used in decay trials with leached wood blocks. POP and GLT were obtained from the Korean Forest Research Institute and Korea University, respectively. Decay resistance of the leached wood blocks exposed to fungi, as well as control blocks, was determined in compliance with ASTM Standard D 1413-05b.[16]

Prior to the decay trial, soil culture bottles inserted into a decay chamber were sterilized for 30 min. Fungus cultured on potato dextrose agar was inoculated on the surface of soil in the culture bottles. After the fungal mycelia covered the surface, sterilized wood blocks were placed onto the surface, three blocks per bottle. The soil–block culture was incubated at 26° ± 1°C and 75% relative humidity for 12 weeks. At conclusion of the incubation, wood blocks were removed from the culture bottles, and the mycelium was removed from the surface of each block. The cleansed wood blocks were dried overnight at 80°C in an oven and weighed to determine weight loss.

After weight loss was determined, matchstick-like samples with dimensions of 2.54 × 0.2 × 0.2 cm (L, R, T) were cut from the blocks for chemical analysis. Two grams of the sample was put in a thimble and leached with 100 ml hot distilled water (98° ± 1°C) in an extractor for 2 h. Then the leachates were analyzed for copper content by atomic absorption spectrophotometry (AAS; SpectrAA-200HT, Varian, USA).

Data analysis

Twelve wood blocks were treated with each formulation. For decay trials, half of the wood blocks, randomly assigned, were exposed to POP and the other half to GLT. Effects of each variable, such as the type of metal salts and the hydrolysis conditions of okara used for formulating preservative solutions, were examined by the general linear model procedure with the Statistical Analysis System programming package. A 95% confidence level was used in all statistical tests. Significant effects with $P < 0.05$ were further characterized by the least significant difference test between means.

Results and discussion

Effect of metal salts on the treatability and leachability

Table 1 shows the actual percent retention of okara-based preservative formulations in wood blocks. Measured retentions of wood blocks treated with CC/OK and CC/B/OK were 133.65% and 98.69%, respectively. Actual retentions of the okara-based preservatives were very close to or higher than target retention. The results indicate that, although the molecular size of complexes formed by okara,

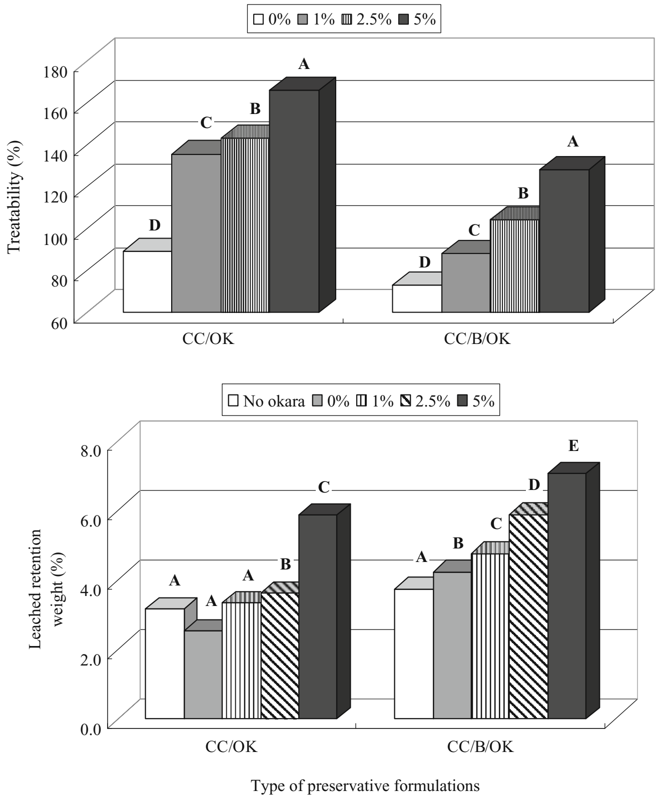

Fig. 2. Effect of acid concentration used for okara hydrolysis on the treatability (*top*) and leachability (*bottom*) of okara-based preservatives. *OK*, okara hydrolyzates; *B*, borax; *CC*, copper chloride. Different capital letters over columns indicate significant difference at $P = 0.05$ (least significance difference test)

copper, and/or boron is too large to penetrate into sample wood blocks, the use of ammonium hydroxide as a dissociating agent in the preservative formulations might alleviate the penetration problem. In addition, the addition of B in CC/OK formulations decreased the treatabilities of the preservative formulations (Fig. 2). As suggested by Thevennon et al.,[12] the molecular size of the complexes formed by boron, protein in okara, and copper might be larger than that of okara-copper complexes, and thus the treatability of CC/B/OK is lower than that of CC/OK.

Okara-based preservatives are very stable against hot-water leaching. For instance, the leachabilities of CC/OK and CC/B/OK were 3.86% and 5.50%, respectively (Table 1). However, as shown in Table 1 and Fig. 2, the leachabilities of CC (3.18%) and CC/B (3.75%) were lower than those of CC/OK and CC/B/OK. In other words, the addition of okara, used for fixing metal salts in wood blocks, did not improve the stability of okara-based preservatives against leaching. The higher leachability of CC/OK and CC/B/OK might be attributed to leaching of soluble substances and degradation products of okara that did not chelate copper and boron salts, and chelated complexes existed on the surface of wood blocks. The addition of B in CC/OK formulations negatively influenced its leachabilities ($P = 0.01$), probably due to the high water solubility of boron (Fig. 2).

Effects of hydrolysis conditions used for hydrolysis of okara

To reveal the effect of acid concentration in hydrolysis of okara on the treatability of okara-based preservatives, okara was hydrolyzed in aqueous solution (OK-0) and in 1%, 2.5%, and 5% sulfuric acid solutions (OK-1, OK-2.5, and OK-5). Retentions of okara-based preservatives in wood blocks increased with increasing acid concentration (Fig. 2). These results mean that the molecular size of okara was decreased with increasing acid concentration, and consequently complexes formed with hydrolyzed okara and metal salts might be small enough to penetrate freely into the wood structure.

In the CC/OK formulations, no significant difference was found between the leachabilities of CC/OK-0 and CC/OK-1 ($P = 0.08$). However, the leachabilities consecutively increased when the acid concentrations increased from 1% to 2.5% ($P = 0.04$) and from 2.5% to 5% ($P = 0.01$). The leachabilities of CC/B/OK formulations steadily increased with increasing acid concentrations. Such results indicate that, at a high acid concentration, nonchelated okara with a reduced molecular weight might be easily leached from wood blocks, and thus the leachabilities become higher.

Temperature (25°C and 80°C) for the hydrolysis of okara did not statistically affect the treatability and leachability of okara-based preservatives, as shown in Fig. 3. It became apparent that acid concentration is more effective than temperature for reducing the molecular weight of okara, and severe hydrolysis of okara might adversely influence the leachability of okara-based preservatives.

Decay resistance of okara-based preservatives

Decay resistance of leached wood blocks treated with okara-based preservatives against GLT and POP is shown in Tables 2 and 3. Weight losses of control wood blocks against GLT and POP were 13.08% and 20.38%, respectively. Very little or no decay against copper-intolerant GOP was observed in wood blocks treated with CC and CC/OK. On the other hand, leached wood blocks treated with CC/OK did not show any decay or measured less than 1% weight loss against POP, but those with CC showed about 4% weight loss. The results indicate that complexes chelated with okara and copper salts might not be leached from wood blocks during leaching, and thus CC/OK shows higher decay resistance than CC. Baechler and Roth[17] reported that wood blocks should contain at least 14 kg m^{-3} copper to effectively protect wood against decaying fungi such as POP. From the results of our study, we might conclude that leached wood blocks treated with CC/OK contain sufficient copper salts to protect wood from decay.

Leached wood blocks treated with CC/B and CC/B/OK formulations showed excellent decay resistance against POP and GLT. For instance, no weight loss against the decay fungi was found in wood blocks treated with CC/B/OK and CC/B (Tables 2 and 3). In addition, CC/B/OK showed better decay resistance than CC/OK. The results might be due to boron salts imparting excellent decay resistance to the wood. Johnson and Gutzmer[18] showed that boron salt in ammoniacal copper borate (ACB) had good decay resistance against brown-rot fungi, but wood specimens treated with ACB failed to protect the wood against decay after exposing them in the ground for 6 years due to gradual leaching of boron into the soil. Therefore, okara-based preservatives might be used for aboveground protection of wood against brown-rot decay, but their effectiveness in ground-contact applications needs to be evaluated in field trials.

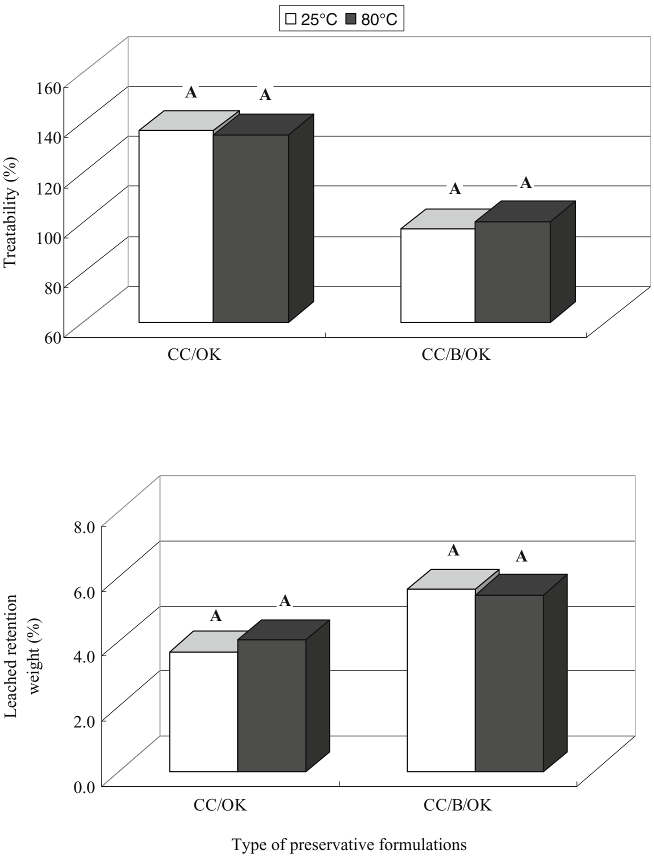

Fig. 3. Effect of hydrolysis temperature on the treatability (*top*) and leachability (*bottom*) of okara-based preservatives. Different capital letters over columns indicate significant difference at $P = 0.05$ (least significance difference test)

After determination of the decay resistance of leached wood blocks treated with okara-based preservatives against POP, quantitative analysis of copper in decayed wood blocks was conducted (Table 4). The copper contents of CC and CC/B were lower than those of CC/OK and CC/B/OK. The results indicate that okara in CC/OK and CC/B/OK formulations might play an important role in fixing copper in the wood structure, and consequently higher copper content was observed in wood samples from CC/OK and CC/B/OK formulations after the decay trial. In comparison of copper contents between okara-based preservatives, decayed wood blocks treated with CC/B/OK showed lower copper content than those treated with CC/OK. One of the possible explanations for the lower copper content of CC/B/OK might be the leaching of copper–boron complexes. For instance, in CC/B/OK formulations, it is possible to form protein–copper, protein–boron, and copper–boron complexes. During leaching, the copper–boron complexes in wood blocks treated with CC/B/OK are leached out due to high water solubility, and thus the wood blocks contain less copper than wood blocks treated with CC/OK. To confirm such speculation, quantitative analysis of boron salts left in decayed wood blocks is required.

Comparison of okara-based preservatives and CuAz

Table 5 shows the comparative results of the treatabilities and leachabilities of wood blocks treated with okara-based preservatives and CuAz. The treatabilities of most okara-based preservatives did not differ from that of CuAz, and furthermore the treatability of CC/OK-5, regardless of hydrolysis temperature, was statistically higher than that of CuAz. In comparison of leachabilities, there was no significant difference between CuAz and okara-based preservatives. Interestingly, okara-based preservatives formulated with OK-0 and OK-1 had higher stabilities against hot-water leaching than CuAz. From the comparative analysis, it can be concluded that okara-based preservatives might protect wood against decay fungi as effectively as CuAz.

Table 2. Decay resistance of leached wood specimens treated with okara-based preservatives and CuAz against brown-rot fungus *Gloeophyllum trabeum*

Hydrolysis conditions		Preservative treatment					
Temperature (°C)	Sulfuric acid (%)	CC	CC/OK	CC/B	CC/B/OK	CuAz[a]	Control
25	0	0	0.02	0	0	0	13.08
25	1	–	0	–	0	–	–
25	2.5	–	1.29	–	0	–	–
25	5	–	0.09	–	0	–	–
80	0	–	0.03	–	0	–	–
80	1	–	0.18	–	0	–	–
80	2.5	–	0	–	0	–	–
80	5	–	0	–	0	–	–

Decay resistance is expressed as percent weight loss after exposing specimens to the brown-rot fungus, *Gloeophyllum trabeum*, for 12 weeks
CC, copper chloride; OK, okara hydrolyzates; B, borax
[a]Wood blocks were treated with CuAz-B type (2%)

Table 3. Decay resistance of leached wood specimens treated with okara-based preservatives and CuAz against brown-rot fungus *Postia placenta*

Hydrolysis conditions		Preservative treatment					
Temperature (°C)	Sulfuric acid (%)	CC	CC/OK	CC/B	CC/B/OK	CuAz[a]	Control
25	0	4.06	0.36	0	0	0	20.38
25	1	–	0	–	0	–	–
25	2.5	–	0.40	–	0	–	–
25	5	–	0.79	–	0	–	–
80	0	–	0	–	0.18	–	–
80	1	–	0	–	0	–	–
80	2.5	–	0	–	0	–	–
80	5	–	0	–	0	–	–

Decay resistance is expressed as percent weight loss after exposing specimens to the brown-rot fungus, *Postia placenta*, for 12 weeks
[a]Wood blocks were treated with CuAz-B type (2%)

Table 4. Residual amounts of copper in leached wood blocks

Sulfuric acid[a] (%)	CC	CC/OK	CC/B	CC/B/OK	CuAz[b]
0	38.59	41.80	10.79	12.64	16.90
1	–	30.62	–	21.64	–
2.5	–	43.95	–	13.71	–
5	–	74.10	–	13.50	–

Data given in units of parts per million (ppm)
[a]Concentration of sulfuric acid used for hydrolysis of okara
[b]Wood blocks were treated with CuAz-B type (2%)

Table 5. Comparison of treatability and leachability between okara-based preservatives and CuAz

Hydrolysis conditions		Treatability				Leachability			
Temperature (°C)	Sulfuric acid (%)	CC	CC/OK	CC/B	CC/B/OK	CC	CC/OK	CC/B	CC/B/OK
25	0	NS (0.091)	NS (0.61)	NS (0.17)	NS (0.06)	– (0.01)	– (0.01)	– (0.01)	NS (0.08)
25	1	–	NS (0.27)	–	NS (0.11)	–	– (0.03)	–	– (0.04)
25	2.5	–	NS (0.21)	–	NS (0.49)	–	– (0.01)	–	NS (0.47)
25	5	–	+ (0.01)	–	NS (0.60)	–	NS (0.51)	–	NS (0.30)
80	0	–	NS (0.07)	–	– (0.03)	–	– (0.01)	–	– (0.01)
80	1	–	NS (0.18)	–	NS (0.13)	–	– (0.01)	–	NS (0.06)
80	2.5	–	NS (0.09)	–	NS (0.83)	–	NS (0.12)	–	NS (0.93)
80	5	–	+ (0.04)	–	NS (0.36)	–	NS (0.75)	–	NS (0.22)

For CuAz treatment, blocks were treated with CuAz-B type (2%). P values are shown in parentheses
NS, No significant difference from value for CuAz-based preservatives at $P = 0.05$ [least significance difference (LSD) test]; –, significantly less than value for CuAz-based preservatives at $P = 0.05$ (LSD test); +, significantly more than value for CuAz-based preservatives at $P = 0.05$ (LSD test)

Conclusions

Although the molecular weights of okara–copper and okara–copper–boron complexes might be large, most measured retention of wood blocks treated with okara-based preservatives was very close to the target retention, probably due to the use of hydrolyzed okara and ammonium hydroxide as a dissociating agent. Okara-based preservatives are very stable against hot-water leaching. Very little or no decay against both POP and GLT was observed in the wood blocks treated with CC/B/OK. From the results of our study, it can be concluded that okara-based preservatives might be easily treated into wood, be quite stable against leaching, and effectively protect wood against decay fungi. We have shown that the okara-based preservatives might be as effective and environmentally friendly as CuAz, and, furthermore, offer ways of cutting the production cost as compared with CuAz. However, the okara-based preservatives should be evaluated by long-term ground-contact testing to identify its applicability.

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