Evaluation of the Dimensional Stability and Leaching Performance of ACQ/Wax Treated Southern Pine

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Southern pine (*Pinus* sp.) wood cubes were treated with ACQ solutions with wax modification and post-treated at 70 °C for 10 h with hot air circulation. The effects of wax concentrations in ACQ-treated southern pine on its dimensional stability and copper leaching performance were investigated. The ACQ/wax-treated wood exhibited improved water resistance during the water soak process. The testing of swelling and shrinkage performance of the treated wood showed that samples with a higher percentage of wax had higher resistance to water swelling and shrinkage. The samples with a lower percentage of wax addition had only a slight effect on the moisture swelling and shrinkage resistance. As a result, copper leaching from ACQ/wax-treated wood with a 2% wax concentration was reduced to a lower level compared to ACQ-treated wood. When the proportion of wax in ACQ preservative was less than 1%, a higher percentage of copper was leached from treated wood.

Keywords: Amine copper quaternary (ACQ); Wax; Dimensional stability; Copper leaching

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INTRODUCTION

In recent years, copper-amine has become an important constituent of several classical, novel, and proposed wood preservatives such as alkaline copper quat (ACQ) and copper azole (CA). Its importance is due to market restrictions on preservatives containing arsenic and chromium, for example, chromated copper arsenate (CCA), and the effectiveness of copper compounds against wood decay fungi (Humar *et al.* 2011). ACQ-treated wood has been more widely used in wooden construction, landscaping, decking, retaining walls, garden furniture, *etc*. However, there is a lack of the UV protection and water repellent constituents in the ACQ formula, leading to major drawbacks for exterior application. These drawbacks include poor dimensional stability and leaching resistance, which can severely affect the useful life of impregnated wood and increase maintenance cost during the service period.

Wood-based material under exterior conditions is prone to weathering caused by the deterioration of the impregnated wood surface. Deterioration is caused by a complex set of reactions induced by solar radiation, moisture, heat, oxygen, and environmental pollutants (Feist and Hon 1984). Moisture and the ultraviolet (UV) component of sunlight are the main agents for impregnated wood weathering (Chang *et al.* 1982; Temiz *et al.* 2006; Donath *et al.* 2007). The ultraviolet radiation depolymerizes lignin and cellulose (Derbyshire and Miller 1981; Evans *et al.* 1993), and water leaches the active ingredients of ACQ preservatives from treated wood during natural weathering exposure (Ruddick 2008; Cooper and Ung 2009).

Water also swells impregnated wood, and the subsequent loss of moisture results in shrinkage. During these cycles, visible cracks arise in the impregnated wood surface because of the growth of micro-cracks, photochemical reactions, or moisture-induced stress fields (Sandberg and Söderström 2006), which weaken the dimensional stability of wood products and increase copper leaching from treated wood.

The protection of preservative-treated wood from weathering is the focus of several research groups. One of the most important and effective methods is by application of pigmented stains, which reduce or limit impregnated wood weathering on the surface of wood (Nejad and Cooper 2010; Nejad *et al.* 2012). Humar *et al*. (2011) demonstrated that copper leaching is significantly reduced by the application of waterborne acrylic surface finishes on ethanolamine containing preservative-treated wood. Nejad and Cooper (2010) noted that the percentage of copper leaching from ACQ-treated wood coated with penetrating stains is significantly reduced during the first year of exterior exposure. However, a significant amount of quat remained available for leaching after 3 years of exposure because the coating deteriorates, and the quat leaching increases if coatings are not renewed on a regular basis (Nejad *et al*. 2012).

Combinations of active substances such as water repellents and/or UV light protectants protect the wood preservatives from weathering (Evans *et al.* 2003; Mai and Militz 2004; Schulte *et al.* 2004; Chen *et al.* 2009). Water repellents such as hot oil appear to reduce the long-term leaching potential of the copper component and improve dimensional stability of preservative treated wood (Treu *et al.* 2003, 2011). The prevention of liquid water uptake from rain and dew leads to higher resistance against surface erosion and leaching of wood components. In addition, water repellent treatment lowers the moisture level of wood in water submersion tests (Donath *et al.* 2007), and thus decreases the tendency of crack formation along with the risk of biological degradation. Archer and Cui (1997) evaluated the performance of commercial preservative/water repellent systems and reported that a good correlation exists between surface checking and swellometer results for pressure treated southern pine.

Among these different types of water repellents, wax emulsion is a safe, reliable, predictable, inexpensive, and non-corrosive repellent. It can be used to lower the moisture content so that decaying processes are no longer possible (Goethals and Stevens 1994). Montan wax emulsion and boric acid act synergistically against wood decay fungi, and they decrease boron leaching from impregnated specimens from 50% to 20% (Lesar *et al.* 2009).

In related research, Lesar and Humar (2011) evaluated five water emulsions with various concentrations and found that the treated specimens were more resistant to wood decay fungi, especially if treated with polyethylene and oxidized polyethylene wax, and also that the sorption of water was reduced significantly. Wax emulsion can also be used with other modification methods to improve the comprehensive properties of treated wood, such as combination with thermal modification (Wang *et al.* 2014), silane compound emulsion, and borate pre-treatment (Wang *et al.* 2015a).

To improve the dimensional stability and the copper leaching resistance of ACQ-treated wood, wax emulsions with different concentrations were added to the ACQ solution to determine the appropriate formula for this study.

EXPERIMENTAL

Material and Methods

Samples and treatment

The pure sapwood of kiln-dried southern pine (*Pinus* sp.) was prepared from defect free and clear straight grain lumber. The density was about 0.54g/cm^3. The growth ring width of 3 to 5 cm was selected and cut into small cubes with dimensions of 19.0 ± 0.2 mm (longitudinal) by 19.0 ± 0.2 mm (radial) by 19.0 ± 0.2 mm (tangential) and stored in a conditioning room to reach an equilibrium moisture content of between 9 and 10%. The cubes were weighed, and those with similar weight were selected as test samples. Two sets of solutions were used for impregnation. The first set was an aqueous solution of wax emulsion with three concentrations, and the second set contained 0.6% ACQ (33.3% Dodecyl Dimethyl Benzyl ammonium Chloride, 66.7% copper oxide) preservative and 0, 0.5%, 1.0%, or 2.0% paraffin wax (melting point: 58 to 60 °C, purity: 40%) emulsion. Both the concentrations of ACQ and wax were the concentrations in the final formulation of the mixture preservatives.

Samples were vacuum-treated as follows. The vacuum was set to -0.1 MPa for 30 min, and solution was admitted into the samples. The vacuum was released, and the beaker from the vacuum tank was removed. The beaker was covered with plastic film to minimize evaporation, and the blocks remained in solution for another hour. The treated samples were post-treated at 70 °C for 10 h with hot air circulation. After post-treatment, the leaching test and the dimensional stability tests were performed with two sets of samples, as shown in Table 1.

Table 1. Treatment and Post-Treatment Conditions for Samples Used in Different Experiments

<table>
<tr><th>Experiment</th><th>Copper Leaching</th><th>Dimensional Stability</th><th>Replicates</th></tr>
<tr><td>Untreated</td><td rowspan="4">N</td><td rowspan="8">Y</td><td rowspan="8">6</td></tr>
<tr><td>0.5% wax</td></tr>
<tr><td>1.0% wax</td></tr>
<tr><td>2.0% wax</td></tr>
<tr><td>0.6% ACQ</td><td rowspan="4">Y</td></tr>
<tr><td>0.6% ACQ + 0.5% wax</td></tr>
<tr><td>0.6% ACQ + 1.0% wax</td></tr>
<tr><td>0.6% ACQ + 2.0% wax</td></tr>
</table>

Note: Y, perform the test; N, not perform the test

Dimensional Stability Test

Water absorption measurement

All the treated and untreated specimens were conditioned in an oven set at 60 ± 2 °C for 24 h and cooled over anhydrous cupric sulfate in a desiccator before water absorption measurements were taken according to standard GB/T 1934.1 (2009). Moisture content (MC) of the specimens was measured using the oven-dry method. According to GB/T 1934.1 (2009), samples in the beaker were immersed below the surface of the deionized water at least 50 mm and measured after 6 h, 24 h, 48 h, 96 h, 192 h, ...960 h. At that time, the difference between two adjacent sampling MC of most samples was less than 5%, except for the untreated and 0.5% wax-treated groups. To obtain the maximum water absorption, all the groups of samples were in a vacuum during impregnation with the deionized water after the 960-h water absorption test. Before each measurement, the water on the surface of the sample was absorbed by a piece of blotting paper. The deionized water in the beaker was changed every 4 to 5 days to keep it clean. The percent of water absorption (WA) by the samples was computed using Eq. 1,

$$WA = \frac{W_2 - W_1}{W_1} \times 100\% \qquad (1)$$

where W_1 and W_2 are the weights of each specimen before and after the water absorption measurement.

Wood swelling measurement

According to standard GB/T 1934.2 (2009), after the wood was conditioned and cooled, the tangential and radial dimensions of the samples were measured in micrometers to an accuracy of ± 0.01 mm (TS_1, RS_1). Samples began moisture absorption in the conditions of relative humidity (65 ± 3%) and at a temperature of 20 ± 2 °C until the dimensions were not changed at all. During the moisture absorption process, 3 samples were chosen, and the changes in their tangential dimensions were recorded every 6 h until the difference between the adjacent measurements was no more than 0.02 mm. At that time, the tangential and radial dimensions of all the samples were recorded to an accuracy of ± 0.01 mm (TS_2, RS_2). The percentages of the tangential and radial moisture absorption swelling (*MTS* and *MRS*) by the samples were computed using Eqs. 2 and 3,

$$MTS = \frac{TS_2 - TS_1}{TS_1} \times 100\% \qquad (2)$$

where TS_1 and TS_2 are the tangential dimensions of each specimen before and after the moisture absorption swelling measurement.

$$MRS = \frac{RS_2 - RS_1}{RS_1} \times 100\% \qquad (3)$$

In Eq. 3, RS_1 and RS_3 are the radial dimensions of each specimen before and after the water moisture swelling measurement.

After moisture absorption measurements, the samples were placed in a beaker of deionized water, and the water absorption swelling of the samples was measured. After immersion for 20 days, 3 samples were chosen, and the tangential dimensions were recorded every 3 days until the difference between the adjacent measurements was no more than 0.02 mm. The tangential and radial dimensions of all the samples were recorded to an accuracy of ± 0.01 mm (TS_3, RS_3), and the percentages of the tangential and radial water absorption swelling (*WTS* and *WRS*) of the samples were found using Eqs. 4 and 5,

$$WTS = \frac{TS_3 - TS_1}{TS_1} \times 100\% \qquad (4)$$

where TS_1 and TS_3 are the tangential dimensions of each specimen before and after the water absorption swelling measurement.

$$WRS = \frac{RS_3 - RS_1}{RS_1} \times 100\% \quad (5)$$

In Eq. 5, WS_1 and WS_3 are the radial dimensions of each specimen before and after the water absorption swelling measurement.

Wood shrinkage measurement

All the treated and untreated specimens were immersed into 20 ± 2 °C deionized water to reach stable dimensions before air drying shrinkage and oven drying shrinkage measurements were recorded according to standard GB/T 1932 (2009). To determine sample dimension changes, the tangential dimensions of 3 chosen samples were recorded every 3 days until the difference between the adjacent measurements was 0.02 mm at most. The tangential and radial dimensions of all the samples were noted to an accuracy of ± 0.01 mm (TL_1, RL_1). Samples were kept in a wet condition throughout the process. After measurements, the samples were placed in the conditions of relative humidity (6 ± 3%) and at a temperature of 20 ± 2 °C for air drying shrinkage. During air drying shrinkage, 3 samples were chosen, and the tangential dimensions were measured and recorded every 6 h until the difference between the adjacent measurements was less than or equal to 0.02 mm. The tangential and radial dimensions of all samples were recorded to an accuracy of ± 0.01 mm (TL_2, RL_2). The percentages of tangential and radial air drying shrinkage (ATS and ARS) by the samples were computed using Eqs. 6 and 7,

$$ATS = \frac{TL_1 - TL_2}{TL_1} \times 100\% \quad (6)$$

where TL_1 and TL_2 are the tangential dimensions of each specimen before and after air drying shrinkage measurement.

$$ARS = \frac{RL_1 - RL_2}{RL_1} \times 100\% \quad (7)$$

In Eq. 7, TL_1 and TL_2 are the radial dimensions of each specimen before and after air drying shrinkage measurement.

After the air drying shrinkage measurements were recorded, the samples were placed in an oven set at 60 ± 2 °C for 6 h. The temperature was increased to 103 ± 2 °C for 8 h, and the samples were cooled over anhydrous cupric sulfate in a desiccator before wood shrinkage measurements were taken. After the cooling process, the tangential and radial dimensions of all the samples were recorded to an accuracy of ± 0.01 mm. Samples with serious distortions were thrown out and supplemented with new ones. The tangential and radial wood shrinkage (OTS and ORS) was computed by Eqs. 8 and 9, respectively,

$$OTS = \frac{TL_1 - TL_3}{TL_1} \times 100\% \quad (8)$$

where TL_1 and TL_3 are the tangential dimensions of each specimen before and after oven drying shrinkage measurement.

$$ORS = \frac{RL_1 - RL_3}{RL_1} \times 100\% \tag{9}$$

In Eq. 9, RL_1 and RL_3 are the radial dimensions of each specimen before and after oven drying shrinkage measurement.

Statistical analysis

The effects of wax addition on the dimensional results of the treated wood was evaluated by one-way analysis of variance and S-N-K(S) test calculated in the SPSS software. In the variance analysis, the significant value P was calculated and compared with the critical value 0.05. The relationship $P < 0.05$ means that the factor has a significant effect on the experimental result.

Leaching test

To evaluate the effect of wax addition on copper leaching from ACQ-treated southern pine, leaching tests were performed on samples with different ACQ treatments after hot air post-treatments following the AWPA E11 standard (2006) as shown in Table 1. Nine replicates in the same treatment condition were selected, and among them, six samples were placed in one beaker to perform the leaching test, and the other 3 samples were used to determine the original copper content in this treatment condition. The leachate was exchanged at prescribed intervals. The first interval was 6 h and then after 24 h, 48 h, and thereafter at 48 h intervals. The leaching test lasted for a total of 14 days. After the leaching test, the blocks were air-dried, milled to powder, and dried at 103 ± 2 °C for 24 h. At this time, 0.15 g of wood powder from each sample was weighed and digested with an acid mixture of nitric acid and perchloric acid. The copper content was analyzed using an atomic absorption spectroscopy (AAS) equipment produced by Thermo Fisher Scientific Corporation of America. Percentage of leached copper was calculated according to the amount of copper impregnated into the specimens without leaching test and the copper amount remaining in the specimens after leaching tests.

RESULTS AND DISCUSSION

Water Absorption

The percentage of water absorption as a function of water soaking duration is presented in Fig. 1. The water absorption test was carried out for about 960 h. The absorption curves can be divided into three major zones: from 0 to 24 h corresponding to the fast filling of water in the cell wall, the moisture content reached 25 to 40%, near the fiber saturation point; from 24 to 200 h corresponding to the fast filling of water in the cell lumen and the intercellular space; and after 200 h, the percentages of water absorption leveled off and tended to reach plateau with lowest absorption rates after 960-h soaking. Compared to the untreated wood, the ACQ-treated wood had more water resistance due to the decrease of hydrophilic functional groups, which is caused by the fast stabilization reactions between the copper and the hydroxyl group in the treated wood (Zhang 2000; Ruddick 2003). Wax addition into wood was key for improving water resistance of the treated wood and provided better water resistance performance in both the wax treated wood and the ACQ/wax-treated wood with a higher wax addition from Fig. 1. During water absorption, the percentage of absorbed water in the ACQ/wax-treated wood was clearly lower than in the ACQ-treated wood. In the initial stage of water absorption, the percentage of water in ACQ-treated wood was nearly 50%; however, groups with 2.0% wax and ACQ treatment reached 20% absorption. From the final stage after vacuum impregnation, the percentage of water absorption was less than 100% for the groups with 2.0% wax and ACQ treatment. However, it went up to 130% for the ACQ-treated wood. This result was consistent with other research in the impregnation of wood with an emulsion of montan wax to reduce water uptake (Lesar and Humar *et al.* 2011).

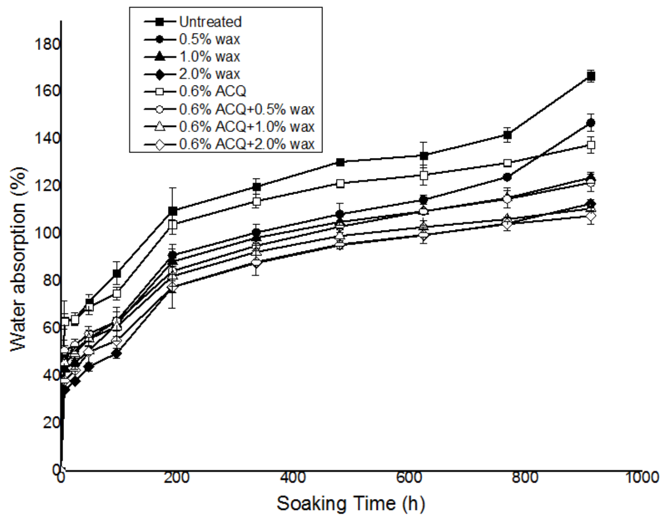

Fig. 1. Water absorption of treated and untreated southern pine

Swelling and Shrinkage

Percentages of tangential/radial moisture and water swelling of treated and untreated southern pine are presented in Table 2. Higher percentages of wax addition in wood improved the resistance to moisture swelling. Addition of 2% wax to ACQ solution reduced average tangential swelling (MTS) from 1.07 % to 0.73 %; for radial swelling, the reduction was from 0.99 % to 0.81 %.

Table 2. Percentages of Tangential/Radial Moisture and Water Swelling of Treated and Untreated Southern Pine

Treatment	Wax (%)	ACQ (%)	Moisture Swelling		Water Swelling	
			MTS	*MRS*	*WTS*	*WRS*
Untreated	-	-	1.21(0.22)	1.15(0.41)	7.24(0.30)	5.78(0.82)
0.5% wax	0.5	-	1.24(0.38)	0.77(0.67)	7.64(0.67)	5.86(0.56)
1.0% wax	1.0	-	0.89(0.34)	1.10(0.86)	7.47(0.12)	6.33(0.34)
2.0% wax	2.0	-	0.84(0.08)	0.63(0.20)	7.61(0.16)	6.46(0.29)
0.6% ACQ	-	0.6	1.07(0.49)	0.99(0.68)	7.19(0.40)	5.48(0.45)
0.6% ACQ +0.5% wax	0.5	0.5	0.96(0.30)	1.08(0.45)	7.16(0.27)	5.52(1.01)
0.6% ACQ +1.0% wax	1.0	1.0	1.06(0.37)	0.95(0.89)	7.27(0.39)	6.27(0.74)
0.6% ACQ+2.0% wax	2.0	2.0	0.73(0.15)	0.81(0.85)	7.15(0.36)	5.11(0.50)

The results indicated that a lower percentage of wax addition had only a slight effect on the moisture swelling improvement, just as it was shown in the groups with 0.5% and 1.0% wax addition. For water swelling performance, wax addition had a negligible impact on the improvement on the swelling resistance. Only the treated wood with 2.0% wax slightly decreased the water swelling ratio.

Percentages of tangential/radial air and oven drying shrinkage of treated and untreated southern pine are presented in Table 3. During air drying, the percent of tangential/radial shrinkage of treated and untreated southern pine was very small because the evaporation was mainly from free water in the cell lumen and intercellular space. During oven drying, the drying shrinkage ratio was higher than in air drying, which was attributed to the bound water evaporation during this stage. Compared with ACQ-treated wood, a small improvement of the tangential drying shrinkage resistant was only found in the ACQ and 2.0% wax-treated wood. However, the percent of radial oven drying shrinkage decreased more obviously in the treated wood with wax addition.

Table 3. Percentages of Tangential/Radial Air and Oven Drying Shrinkage of Treated and Untreated Southern Pine

Treatment	Wax (%)	ACQ (%)	Air Drying Shrinkage		Oven Drying Shrinkage	
			ATL	*ARL*	*OTL*	*ORL*
Untreated	-	-	0.09(0.05)	0.12(0.14)	6.27(0.52)	5.62(0.50)
0.5% wax	0.5	-	0.16(0.15)	0.20(0.18)	6.14(0.32)	5.81(0.60)
1.0% wax	1.0	-	0.11(0.16)	0.06(0.14)	6.28(0.14)	6.13(0.47)
2.0% wax	2.0	-	0.17(0.07)	0.06(0.16)	6.26(0.39)	5.55(0.30)
0.6% ACQ	-	0.6	0.02(0.03)	0.55(0.32)	6.47(0.47)	6.00(0.58)
0.6% ACQ+0.5% wax	0.5	0.5	0.15(0.07)	0.14(0.06)	6.45(0.19)	5.29(0.16)
0.6% ACQ+1.0% wax	1.0	1.0	0.06(0.03)	0.05(0.05)	6.88(0.33)	5.10(0.22)
0.6% ACQ+2.0% wax	2.0	2.0	0.08(0.12)	0.32(0.48)	6.42(0.39)	5.22(0.69)

The one-way analysis of variance for the treated southern pine in different ACQ/wax treatments was shown in Table 4. It was noted that wax addition played significant effects on the water absorption and tangential moisture swelling. The results have proved that southern pine treated with ACQ/wax modified preservatives was necessary for its safe outdoor application.

Table 4. One-way Analysis of Variance for the Treated Southern Pine in Different ACQ/wax Treatments by SPSS

Quadratic Sum		df	F	Significant
Water Absorption	2058.67	11	26.23	0.00
Tangential Moisture Swelling	0.68	11	4.57	0.04
Radial Moisture Swelling	1.23	11	0.46	0.72
Tangential Water Swelling	1.02	11	0.07	0.98
Radial Water Swelling	5.80	11	1.48	0.29
Tangential Air Drying Shrinkage	0.05	11	2.65	0.12
Radial Air Drying Shrinkage	0.93	11	2.62	0.12
Tangential Oven Drying Shrinkage	1.36	11	0.99	0.44
Radial Oven Drying Shrinkage	4.96	11	4.11	0.05

Leaching Performance Analysis

The copper leaching from ACQ and different ACQ/wax-treated southern pine are shown in Fig. 1. When the proportion of wax in the ACQ preservative was increased to 2%, the copper leaching from ACQ/wax-treated was reduced to a lower level compared with ACQ-treated wood. It was obvious that as a water repellent, a higher proportion of wax in the ACQ preservative influenced the rate of water uptake and thus reduced copper leaching. The result was consistent with some previous studies (Lesar *et al.* 2009; Wang 2015b), which indicated that the higher leaching resistance of preservative was attributable to the hydrophobic film formed by the wax in the treated wood during hot temperature treatment.

However, in this study, it was found that if the proportion of wax in ACQ preservative was less than 1%, a higher percentage of copper was leached out from treated wood, as observed from Fig. 2. Although this result conflicted with our hypothesis, it was established that a reasonable proportion of wax in ACQ-modified preservative was very critical for the stability of the solution, which would affect the copper leaching performance.

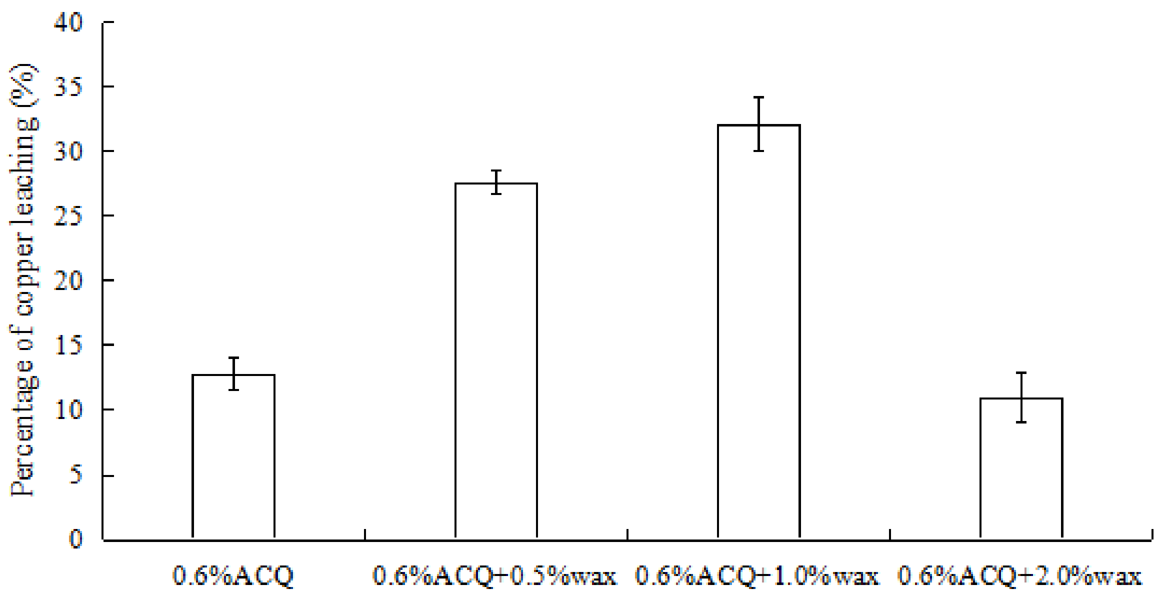

Fig. 2. Copper leaching of ACQ and a variety of ACQ/wax treated southern pine

CONCLUSIONS

1. During the whole process of water absorption, the percentage of water absorption in the amine copper quaternary (ACQ)/wax-treated wood was clearly lower than that of the ACQ-treated wood. At the initial stage of water absorption, the percentage of water absorption in ACQ-treated wood was nearly 50%. However, it was about 20% for the groups with 2.0% wax and ACQ treatment.

2. The highest moisture swelling and shrinkage resistance of the treated wood were found in samples with a higher percentage of wax addition (2%), while the samples with a lower percentage of wax addition had only a slight effect on the moisture swelling and shrinkage improvement.

3. When the proportion of wax in ACQ preservative was increased to 2%, copper leaching from ACQ/wax-treated wood was reduced to a lower level than the ACQ-treated wood with a lower proportion of wax, which can be attributed to the poor stability of the solution, which would affect the copper leaching performance.

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