Factors Affecting the Use of Increment Cores to Assess Fixation

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Abstract: As part of an effort to ensure that treated wood products have minimal environmental and handling concerns, an American Wood Preservers Association task force is considering the development of a test to assess the degree of fixation of waterborne wood preservatives. The proposed test involves removal and leaching of increment cores. This paper describes a laboratory study to determine how parameters such as extraction time, water volume, water temperature; depth of assay zone, and between sample variability might affect the results of such a test for chromated copper arsenate (CCA) treated wood. The results suggest that an extraction time of 4 hours would be sufficient if chromium is the element of interest. Water volume did not appear to be a concern for chromium or copper, but may be for arsenic. Leaching was affected by water temperature, suggesting that the test should incorporate temperature limitations. Depth of assay zone and location of core removal along the board did not have a great effect, especially for chromium, but this test only used cores that were fully penetrated with preservative. Because the amount of chromium leached underwent the greatest change during fixation, and because chromium leaching appears to be the CCA element that is best suited for a fixation test. The low overall variability between samples indicates that this type of test could be conducted with the removal of relatively few increment cores.

Key Words: chromated copper arsenate, CCA, fixation, test, increment core, leaching, chromium, copper, arsenic

Introduction

Members of the treating industry and the American Wood Preservers Association (AWPA) have long recognized the need to produce treated wood with minimal handling and environmental concerns. In recent years, the AWPA Executive Committee has sought to provide more detailed guidance on environmentally responsible treatment practices within the AWPA standards. Various AWPA subcommittees have been directed to establish “Migration/Fixation/Depletion” task forces to consider additional processing or inspection recommendations for the standards under their jurisdiction. Within Subcommittee P-4, Inorganic Preservative Systems, the Migration/Fixation/Depletion task force is considering recommendations for processing or inspection of wood treated with chromated copper arsenate (CCA) and other waterborne preservative systems. These waterborne preservatives depend on chemical “fixation” reactions to stabilize the preservative components within the wood. Accordingly, one of the focuses of the P-4 task force is to develop a method to assess the degree of fixation of treated wood before it leaves the treating plant. The task force is currently considering a method in which increment cores are removed from the treated wood, leached in distilled water, and the water is then analyzed to determine how much preservative has leached from the wood. As part of this effort, a laboratory study was conducted to determine how various parameters, such as extraction time, water volume, water temperature, depth of assay zone, and within and between board variability might affect the results.

Materials and Methods

Treatment -- Twenty-four Southern pine sapwood 2 x 4’s (38 by 89 mm) were cut to 36 inch (914 mm) length, and conditioned to approximately 9-12% moisture content. Eight of the specimens were end-coated with a neoprene sealer prior to treatment. All of the specimens were treated in a single charge using a 1.1%
CCA-C solution (112 hour vacuum followed by 1 hour pressure at 150 psi (1034 kpa)). The rigorous treating schedule was used to ensure full penetration. Retention, based on uptake, varied from 0.41 to 0.47 lbs/ft$^3$ (6.6 to 7.5 kg/m$^3$), with an average retention of 0.44 lbs/ft$^3$ (7.0 kg/m$^3$). Immediately after treatment, the boards were wet-stacked on a cart with 0.125 inch (3.2 mm) stickers between each course. A weighted sheet of plywood was placed on top the stack to retard drying of the top boards. The boards were stacked so that the eight end-sealed specimens and four other boards that had not been end-sealed were exposed. The boards were then moved to a conditioning room maintained at 74 F (23 C) and 65% relative humidity.

Core Sample Removal -- Cores were removed from the outer edges of the outer boards using five increment borers with equivalent diameters. Borers were alternated to insure that they remained cool, and flushed with distilled water after each use.

Boards Without End Seal: These boards were used to assess the effect of sample position with respect to the end of the board. The boards were divided into 3 zones: 1) Outer, or within 6 inches (152 mm) of the end, 2) middle, within 6-12 inches (152 - 305 mm) of the end, and 3) the inner 12 inches (305 mm). Cores were removed from the three zones of the boards after 2, 7 and 14 days of fixation. At each sampling time, two 0.5 inch (13 mm) cores were removed from the outer, middle and inner sampling zones and placed in 10 mls of room temperature distilled water in a scintillation vial. The cores were allowed to extract for 4 hours (without agitation), and then removed from the water.

End-sealed Boards: These boards were used to assess the effect of extraction time, water volume, water temperature and core length on leaching. Because they were end-sealed, coring locations were randomized along the length of the boards without regards to distance from the end. Each leaching condition was replicated once on each of the eight boards. All of the cores were removed after 7 days of fixation. No agitation was used.

Table 1. Leaching conditions for cores removed from end-sealed boards. All cores were removed after 7 days of fixation.

<table>
<thead>
<tr>
<th>Water Volume (mls)</th>
<th>Extraction Time (hrs)</th>
<th>Water Temp (F/C)</th>
<th>Core Length (in/mm)</th>
<th>Number of Cores in Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>0.25</td>
<td>74/23</td>
<td>0.5/13</td>
<td>2</td>
</tr>
<tr>
<td>10</td>
<td>1</td>
<td>74/23</td>
<td>0.5/13</td>
<td>2</td>
</tr>
<tr>
<td>10</td>
<td>4</td>
<td>74/23</td>
<td>0.5/13</td>
<td>2</td>
</tr>
<tr>
<td>10</td>
<td>24</td>
<td>74/23</td>
<td>0.5/13</td>
<td>2</td>
</tr>
<tr>
<td>20</td>
<td>4</td>
<td>74/23</td>
<td>0.5/13</td>
<td>2</td>
</tr>
<tr>
<td>10</td>
<td>4</td>
<td>36/2</td>
<td>0.5/13</td>
<td>2</td>
</tr>
<tr>
<td>10</td>
<td>4</td>
<td>74/23</td>
<td>0.25/6</td>
<td>4</td>
</tr>
</tbody>
</table>

Leachate Analysis -- The leachates were analyzed for chromium, copper, and arsenic using Atomic Absorption Spectroscopy.

Results and Discussion

End-sealed Boards -- The various leaching conditions are shown in Table 1. All of these samples were removed after 7 days of fixation.

Effect of extraction time: As shown in Figure 1, extraction time did affect the amount of leaching from the cores. Concentrations of copper and arsenic detected in the extract increased at each time interval, although the rate of release appeared to be substantially slower after 4 hours. Chromium was least sensitive to extrac-
tion time, with little leaching difference between 4 and 24 hours. If chromium is the element of interest, there appears to be little need to extract the cores for more than 4 hours. Perhaps the more important finding was that the variability in leaching between replicates tended to be lower at the shorter extraction times. For the purposes of a fixation test, reproducibility may be more important than achieving maximum leaching, and thus one of the shorter leaching times might suffice. However, there is the concern that with a very short leaching period the tendency of the inspector to error by a few minutes would have a greater effect.

**Effect of Water Volume:** One set of samples was leached in 20 mls of water instead of the usual 10 mls to determine if the amounts of CCA components detected would be cut in half. As seen in Figure 2 this was the case for copper and chromium. However, the amount of arsenic detected at 20 mls appears to be slightly more one-half of that detected at 10 mls, indicating that there may be a concentration effect. For the purposes of a test, the water:wood ratio will need to be great enough to prevent a high concentration in the extract from hindering further leaching.

**Effect of Water Temperature:** Leaching cores in 36 F (2 C) water for 4 hours produced slightly lower levels of all three CCA components than did leaching cores in 74 F (23 C) water. One can only speculate whether this difference would increase or decrease with shorter extraction times or in wood that had been allowed to fix for a shorter time. The difference is not great, but since there does appear to be some effect, it might be wise to suggest that cores are collected in water that is at or near room temperature. One could also argue that the water temperature should be the same as that of the wood, or that cold water should always be used, based on the concern that cores removed from wood at 36 F and placed in water at 70 F may undergo fixation during leaching and thus the amount of leaching could be underestimated.

**Effect of Assay Zone:** The amount of leaching from 4, 0.25 inch (6 mm) cores was slightly less than that from 2, 0.5 inch (13 mm) cores for copper and arsenic. This may reflect more complete fixation in the outer portions of the wood; these differences might be expected to be greater earlier in the fixation process. Assay zone had little effect for chromium, but it still appears that a single assay zone should be used for the test. It will also be important that only cores with 100% penetration of the assay zone be evaluated. Because of this a smaller assay zone may be more desirable, especially in western species.

**Boards Without End-seal**

**Effect of Fixation Period:** As expected, the length of the fixation period did affect leaching, especially for chromium (Figure 3). Copper release was least sensitive to fixation periods, suggesting that copper may not be an ideal indicator for degree of fixation because the test would need to detect relatively small changes. Instead, chromium appears to be the logical choice since it has the greatest decrease in leaching over time.

**Effect of Distance from End of Board:** As shown in Figure 4, the distance that core samples were removed from the end of the board had little effect on leaching of copper, chromium or arsenic, especially at longer fixation periods. This is an encouraging finding, since it suggests that we do not need to be greatly concerned about the position along the board from which the cores are removed.

**Overall Variability:** The overall variability in leaching from cores removed from different boards was low, especially as fixation progressed (Figure 3). Although variability will be greater in a commercial charge, it appears that it may be possible to estimate the fixation condition of a charge with relatively few cores.

**Conclusions**

The results of this trial suggest that it may be practical to develop a CCA fixation test that involves leaching of increment cores. Several of the parameters evaluated did affect the results of the test, but it should be possible to minimize these effects by prescribing the conditions to be used in the test. It appears that of the three CCA components, chromium may be best-suited for monitoring in a fixation test. Chromium undergoes the greatest change in leaching during the fixation, but appears to be least affected by parameters such as depth.
of assay zone and location of core removal. Although extraction time strongly affected the amount leached for all three CCA components, the amount of chromium leached stabilized after about 4 hours. The low variability between replicate samples suggests that a test could provide meaningful results with the removal of relatively few increment cores.

**Figure 1.** Effect of extraction time on the amount of chromium, copper, and arsenic leached from increment cores after 7 days of fixation.
Figure 2. Effect of water temperature, water volume, and depth of assay zone on the amount of chromium, copper and arsenic leached from increment cores after 7 days of fixation. Control is room temperature water, 0.5 inch (13 mm) assay zone, and 2 cores placed in 10 mls of water.
Figure 3 Effect of fixation period on the amount of chromium, copper and arsenic leached from the increment cores. Replicate samples extracted for 4 hours.
Figure 4. Effect of location along the board on the amount of chromium, copper and arsenic leached from the increment cores. Average of samples extracted for 4 hours.