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Thermochemical Remediation of Preservative-Treated Wood

Ronald Sabo, Carol A Clausen, and Jerrold E Winandy

USDA Forest Service, Forest Products Laboratory,
One Gifford Pinchot Drive, Madison, Wisconsin 53726, U.S.A.

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Ronald Sabo, Carol A Clausen, and Jerrold E Winandy

USDA Forest Products Laboratory, One Gifford Pinchot Drive, Madison, Wisconsin 53726, U.S.A. rsabo@fs.fed.us

USDA Forest Products Laboratory, One Gifford Pinchot Drive, Madison, Wisconsin 53726, U.S.A.

cclausen@fs.fed.us

USDA Forest Products Laboratory, One Gifford Pinchot Drive, Madison, Wisconsin 53726, U.S.A.

jwinandy@fs.fed.us

ABSTRACT

Remediation methods continue to be developed and refined for CCA-treated wood in order to divert this material from landfills and create secondary products from the wood fiber. A two-step thermochemical remediation method was developed to assess the efficiency of metal removal from CCA-treated spruce flakes. Nearly all the metals (As, Cr, and Cu) were thermochemically extracted from chromated copper arsenate (CCA) treated Spruce using solutions of oxalic acid and sodium hydroxide. Oxalic acid was found to be highly effective for removing As and Cr, whereas mixtures of oxalic acid and sodium hydroxide were more effective for removing copper. Up to 100 % As and 97 % Cr were removed from treated flakes after one hour using a 1 % oxalic acid solution (pH 1.4) at 80 °C, but less half of the Cu was removed at pH 1.4 even after six hours. Nearly all the As, Cr, and Cu were extracted from the treated flakes after six hours using a 1 % oxalic acid solution adjusted to pH 3 with sodium hydroxide. However, the removal of As and Cr was slower with oxalic acid solutions that contained sodium hydroxide. A two-stage process in which the flakes were first extracted for one hour with oxalic acid (pH 1.4) then extracted for another hour after adjusting the solution to pH 3 with sodium hydroxide removed 99 % As, 96 % Cr, and 92 % Cu. The effects of temperature, pH, time, mixing, rinsing, and liquid to solid ratio on the removal of metals from CCA-treated Spruce flakes and particles is discussed.

Keywords: chromated copper arsenate (CCA), preservative, remediation, flakeboard, oxalic acid

1. INTRODUCTION

Millions of cubic meters of preservative-treated lumber are removed from service annually in the United States, and most of this spent wood is disposed into landfills or incinerated (Solo-Gabriele *et al.* 2000, Felton and De Groot 1996). This waste wood poses a potential environmental and health hazard since toxic metals are known to leach from wood treated with waterborne preservatives, such as chromated copper arsenate (CCA). Biobleaching from metal tolerant microbes in landfills may exacerbate leaching (Cole and Clausen 1996, Clausen 2000b). Furthermore, making use of such discarded lumber could provide some relief for the harvesting of worldwide forest resources.

Although numerous techniques have been shown to effectively remove metals from preservative-treated wood, most of the studies found throughout the literature either used economically infeasible processes or did not evaluate the efficacy of the remediated wood as a raw material for composite materials. Some of the methods reported for removing metals from preservative-treated wood include extraction with hydrogen peroxide (Cooper and Kazi 1999, Kazi and Cooper 2006), bacterial fermentation and/or extraction (Clausen 2000a, Clausen 2004a, Clausen 2004b, Clausen and Kartal 2001, Clausen *et al.* 2001, Clausen *et al.* 2006, Clausen and Kenealy

2004, Clausen and Smith 1998, Shiau *et al.* 2000), or chelating agents (Kamdem 1999, Kartal and Kose 2003), various acids (Cooper and Kazi 1999, Kazi and Cooper 2006), and recently by electro dialysis (Velizarova *et al.* 2002, Pedersen *et al.* 2005). Many of the reported techniques required days or weeks to effectively remove impregnated metals from treated wood, or they were prohibitively expensive. However, some methods have been reported in which virtually all the toxic metals can be extracted from preservative-treated wood in a matter of hours using dilute solutions. For example, extraction of CCA-treated wood in sodium oxalate (oxalic acid and sodium hydroxide) for three hours, following a one hour extraction in oxalic acid, was shown to remove 100 % of As and Cr and 96 % of Cu (Kakitani *et al.* 2006). Kakitani *et al.* (2007) reported nearly 100 % extraction of arsenic from treated wood after only one hour using oxalic acid at 75 °C. Thus, chemical extraction of metals from preservative-treated wood seems promising, but the feasibility of using such remediated wood to make quality composites has yet to be fully demonstrated.

Therefore, the primary aims of this study are to identify conditions favorable for initially detoxifying the flakes cut from preservative-treated wood and then producing composites from the remediated flakes. We demonstrate that 92 % - 99 % of As, Cr, and Cu can be removed from wood flakes after only two hours of chemical extraction, while greater removal levels can be achieved using longer extraction times. The optimal conditions, including chemical concentrations, temperature, and time, for extracting metals from treated lumber are being examined with the aim of producing durable composite products from the remediated wood.

2. EXPERIMENTAL METHODS

2.1 Wood Samples

Nominal 2- by 4-inch (38 mm x 89 mm) Spruce-Pine-Fir lumber was treated with chromated copper arsenate (CCA) Type C using a vacuum of 82 kPa followed by 0.8 MPa pressure for a total cycle time of about three hours, resulting in about 8.3 kg/m³ of preservative retention (Anderson *et al.* 1997). The treated lumber used for this study was naturally weathered for about ten years at the Forest Products Laboratory in Madison, WI. The Spruce lumber selected for this study was MSR grade 2250f/1.9E and had 660, 7 mm-deep incisions per square foot. The lumber was first cut into 150 mm sections and pressure-soaked with water for approximately one hour at approximately 0.8 MPa to facilitate flaking. The wood was then cut into flakes approximately 0.76 mm thick by 76 mm long using a disc flaker. The wood flakes were dried overnight at 105 °C in a tray drier. Additionally, wood particles were prepared for a limited number of supplemental extraction experiments by grinding flakes in a Wiley Mill to pass through 0.85 mm screen openings (20 mesh).

2.2 Chemicals

Oxalic acid dihydrate (VWR International) and sodium hydroxide (reagent grade, Sigma-Aldrich) were obtained in solid form and used for all treatments. Solutions of oxalic acid were prepared in deionized (DI) water unless otherwise explicitly stated as containing tap water. The oxalic acid weight concentrations were prepared on the basis of anhydrous oxalic acid. The pH of the oxalic acid solutions were adjusted by adding 10 % (wt) sodium hydroxide solution.

2.3 Metals Analysis

The concentration of arsenic, chromium, and copper in wood samples was measured by inductively coupled plasma (ICP) atomic emission spectrometry in accordance with the American Wood Preservers' Association (AWPA) standard A-21-00 (AWPA 2001). Approximately 2 g of 20-mesh ground wood samples were allotted for ICP analysis.

Approximately 40 mg of wood particles were digested, and elemental concentrations were determined using a Jobin Yvon Ultima emission spectrometer. The results are reported as a percentage relative to unremediated treated wood samples.

2.4 Bench Scale Extraction

In most cases, extractions were performed in a 3 L glass reaction vessel using 50 g (oven dry basis) of wood flakes and a liquid to solid ratio of 20:1. Extraction solutions were preheated to the appropriate temperature before the wood was added. Heat was removed from the vessel after the designated extraction duration, and the wood flakes were separated from the liquid by straining. A sample of the liquid extractate from each experiment was saved for future analysis. A portion of the flakes were thoroughly rinsed with deionized water, and the wood was dried overnight. A preliminary set of extractions using 1 % oxalic acid solutions were performed to examine the effect of time, temperature, and pH on the removal of arsenic, chromium, and copper from wood flakes, and a summary of these experimental conditions is shown in Table 1. Extractions using DI water without addition of acid or base are referred to as DI to avoid confusion with a pH-adjusted solution. Additional extraction conditions, including additional pH values and acid concentrations, as well as multistage extractions, were then chosen based on the results of these initial experiments.

Table 1: Variables used for initial extraction experiments

Time:	1, 2, & 6 hours
Temperature:	20 ^a , 50, and 80 °C
pH:	1.4, 1.8, 2.4, 3.0, & ~7 (DI water)
^a Due to poor performance, a limited number of extractions were done using acid solutions at 20 °C or using DI water.	

A supplemental set of experiments was also designed to determine if the amount of metals extracted from wood particles is affected by continuous mixing, extractant water source (deionized versus tap water), and liquid to solid ratio. Two levels were chosen for each of the five factors (Table 2), and a half-factorial design was constructed as shown in Table 3 for these experiments. These extractions were carried out using 15 g of wood particles and 150 g or 300 g of extractant solution in 400 mL glass beakers, which were partially submerged in a 50 °C water bath. Extractant solutions were made with 1 % (wt) oxalic acid in either tap or deionized water, and sodium hydroxide was used to adjust the solution pH from 1.4 to 2.4 when appropriate. After preheating the extractant solution, 15 g (oven dry basis) of wood particles were placed in the beaker, and the mixture was stirred until homogenous. For experiments requiring constant mixing, a motorized, propeller-type mixer was used at a speed just high enough to cause thorough mixing.

Table 2: Five factors for the supplemental extraction experiments

Variable	Level 1 (-)	Level 2 (+)
1. Water	DI	tap
2. Mixing	no	yes
3. pH	1.4	2.4
4. Time [h]	1	6
5. Liquid to solid ratio	10:1	20:1

Table 3: Conditions for supplemental experiments

Experiment ID	Water	Mixing	pH	Extraction Time [h]	Liquid to Solid Ratio
A	tap	no	1.4	1	10:1
B	DI	yes	1.4	1	10:1
C	DI	no	2.4	1	10:1
D	tap	yes	2.4	1	10:1
E	DI	no	1.4	6	10:1
F	tap	yes	1.4	6	10:1
G	tap	no	2.4	6	10:1
H	DI	yes	2.4	6	10:1
I	DI	no	1.4	1	20:1
J	tap	yes	1.4	1	20:1
K	tap	no	2.4	1	20:1
L	DI	yes	2.4	1	20:1
M	tap	no	1.4	6	20:1
N	DI	yes	1.4	6	20:1
O	DI	no	2.4	6	20:1
P	tap	yes	2.4	6	20:1

2.5 Pilot Scale Extraction

Pilot-scale extraction experiments were performed using the preservative treatment facilities at the Forest Products Laboratory. Approximately 6 kg of flakes were enveloped in cheesecloth, placed in polypropylene mesh bags, and placed inside a 220 L treatment cylinder. About 270 L of extracting solution was preheated to 80 °C in a prep tank before being circulated through the treatment cylinder containing the flakes. The solution was returned to the prep tank after the first extraction stage so that sodium hydroxide could be added and flake samples could be taken prior to the second stage. After the extractions were complete, the flakes were rinsed with water, dried in a tray drier, and stored for making flakeboards.

3. RESULTS AND DISCUSSION

Initial laboratory-scale extraction experiments were conducted to serve as a base-line for the removal of metals from preservative-treated wood and to aid in the selection of conditions for pilot-scale experiments, in which composites will be made. These results and their implications for future laboratory and pilot-scale are reported here along with some pertinent results from the literature.

3.1 Time, Temperature, and pH

The amount of metals extracted from preservative-treated wood was significantly impacted by pH, temperature, and extraction time. As expected, longer extractions and higher temperatures both facilitated the removal of As, Cr, and Cu, although Cu removal appears to be less impacted by time or temperature as shown in Fig. 1 and Fig. 2. Typically, the removal levels of As and Cr are similarly affected by extraction conditions, while the amount of copper extracted is affected differently. For example, as pH is increased from 1.4 to 3.6, the amount of As and Cr remaining in the wood increases, whereas the amount of copper decreases (Fig. 1). In other words, the extraction of As and Cr is more effective at pH 1.4 than at pH 3.6, whereas the extraction of Cu is more effective at pH 3.6.

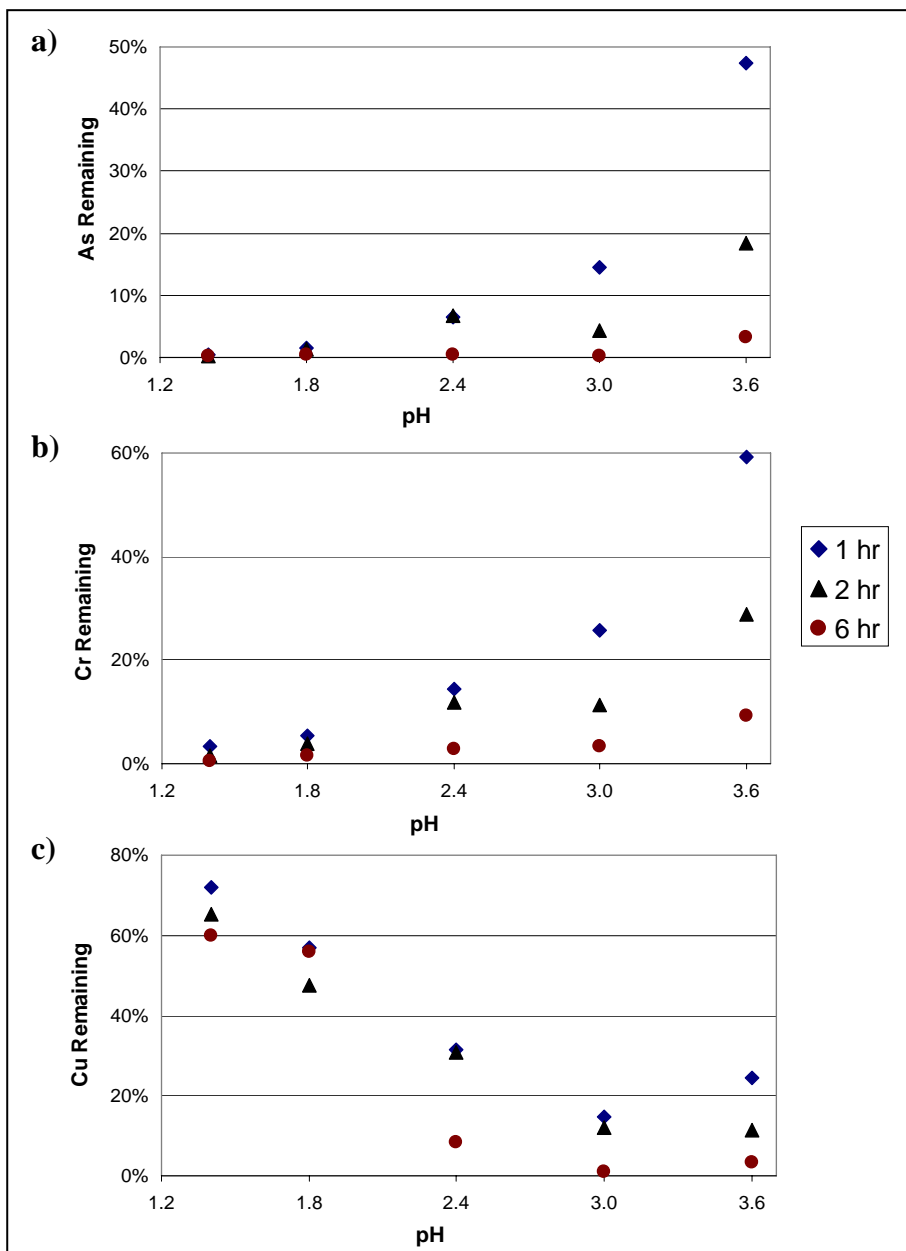


Figure 1: Extraction of wood flakes with 1% oxalic acid at 80 °C after rinsing

Kazi and Cooper (1998) reported similar results in which arsenic and chromium removals followed similar trends. However, they reported results different from those obtained in this study with respect to copper removal. They showed that for extractions using 2.5 % oxalic acid, the removal of As and Cr from Southern Yellow Pine particles increased with reaction temperature, whereas the removal of Cu actually decreased with increasing temperature over the range from 30 °C to 90 °C. Extractions using oxalic acid resulted in greater removals of arsenic and chromium than phosphoric acid, formic acid, citric acid, or sodium hydroxide at similar weight concentrations. However, all of these acids removed more copper than oxalic acid, which appeared to peak in effectiveness for copper at a concentration of one percent weight (Kazi and Cooper 1998). Recently, Kakitani *et al.* (2007) showed that dilute solutions (0.125 mol/L) of oxalic acid at elevated temperature were effective in extracting arsenic and chromium, whereas solutions adjusted to pH values between 2.2 and 5.2 with sodium hydroxide were much more effective at extracting copper from treated wood. Also, the removal of copper from treated wood

with oxalic acid/sodium hydroxide mixtures was shown to be fairly unaffected by temperature, whereas the removal of arsenic and chromium was temperature-dependent (Kakitani *et al.* 2007).

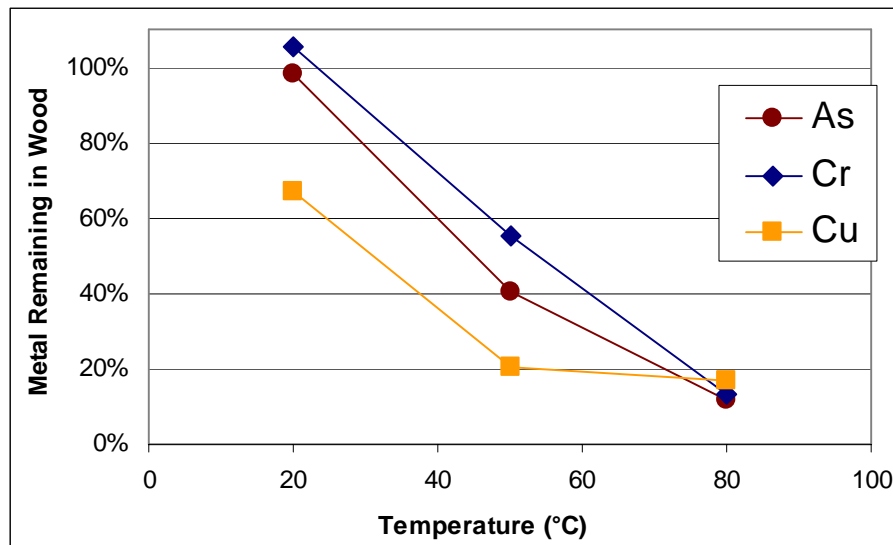


Figure 2: Extraction at pH 2.4 for 6 hours, without rinsing

Also, it is noteworthy that extractions done at either room temperature, regardless of pH level, or with pH-neutral deionized water, regardless of temperature, were not effective at removing metals from flakes of treated wood. Fig. 3 shows that most of these experiments resulted in less than 20 % of the metals being removed after six hours, and none resulted in more than 52 % removal.

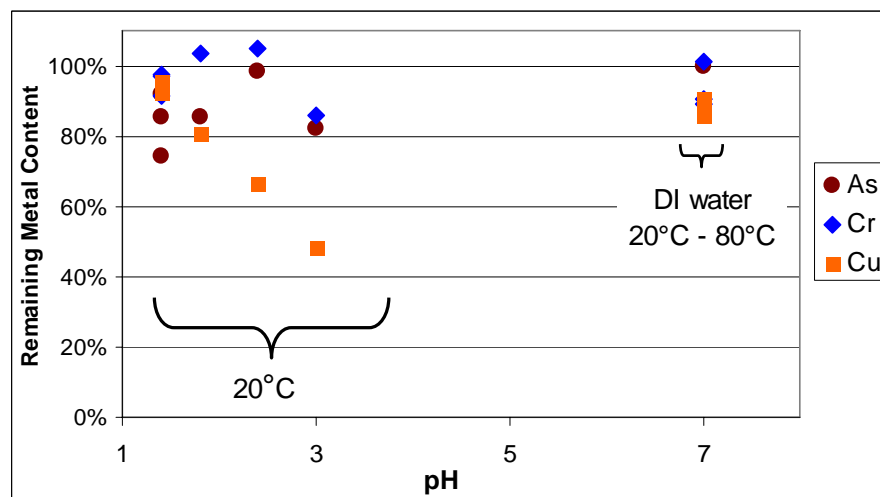


Figure 3: Extractions using acid solutions at 20 °C or those using only water (i.e., no added chemicals) are ineffective at removing metals from wood flakes

3.2 Two-Stage Extractions

Because oxalic acid was more effective at removing As and Cr than Cu from treated wood and because oxalic acid/sodium hydroxide mixtures were more effective at removing Cu than As and Cr, a two-stage extraction process was expected to result in better overall removals of As, Cr, and Cu. Therefore, extractions were performed in two stages, one with and one without sodium

hydroxide added to the 1 % oxalic acid solution. Table 4 shows the relative amount of metals remaining in the flakes after the first and second extraction steps. Extractions with the first stage

Table 4: Extraction results for two-stage process at two pH levels, each stage lasting for one hour at 80 °C

First Stage				Second Stage			
pH	Remaining Metals			pH	Remaining Metals		
	As	Cr	Cu		As	Cr	Cu
1.4	5%	11%	80%	3.6	1%	4%	9%
1.4	6%	13%	88%	3.8	1%	4%	8%
3.0	37%	47%	34%	1.4	1%	7%	31%
3.6	58%	68%	29%	1.4	2%	11%	21%

at pH 1.4 followed by a second stage at pH 3.6 – pH 3.8 were more effective than those with the pH shift reversed from pH 3.0-3.6 to pH 1.4. This effect is probably because the lower pH solution is more effective at removing As and Cr, leaving primarily Cu to be removed by the extraction using a oxalic acid/sodium hydroxide solution. Conversely, if the oxalic acid/sodium hydroxide solution is used in the first stage, the extraction of Cu competes with that of As and Cr, resulting in significant levels of Cu that cannot be removed in subsequent extractions using oxalic acid without sodium hydroxide.

Kakitani *et al.* (2006) reported similar results using a two-stage process to extract metals from wood powder, but they found longer second-stage extractions were required to achieve removals similar to those reported here. Following a one-hour extraction using oxalic acid, they reported that extraction for an additional hour at pH 3.2 using an oxalic acid/sodium hydroxide mixture resulted in less than 70 % removal of Cu and less than 90 % removals of As and Cr; however, after three hours at pH 3.2, they report 100 % removal of As and Cr and 96 % removal of Cu (Kakitani *et al.* 2006). Also noteworthy is that using a two-stage extraction process results in removal levels in two hours similar to those after six hours with a one-stage extraction using an oxalic acid/sodium hydroxide mixture. We also expect that process optimization will lead to more effective removals at shorter times, thereby making such a remediation process more economically attractive.

3.3 Rinsing Extracted Flakes

After the extractions were complete, the wood flakes had a significant amount of remaining metals that could be readily rinsed with water. Fig. 4 compares the amount of metals remaining on wood flakes before and after thoroughly rinsing with water following extractions with 1 % oxalic acid at 80 °C for one hour. For example, at pH 1.8, 22 % of the initial As remained on the wood immediately following the extraction, but only 2 % remained after rinsing. Therefore, steps should be taken to remove the residual water-soluble metal complexes following extractions.

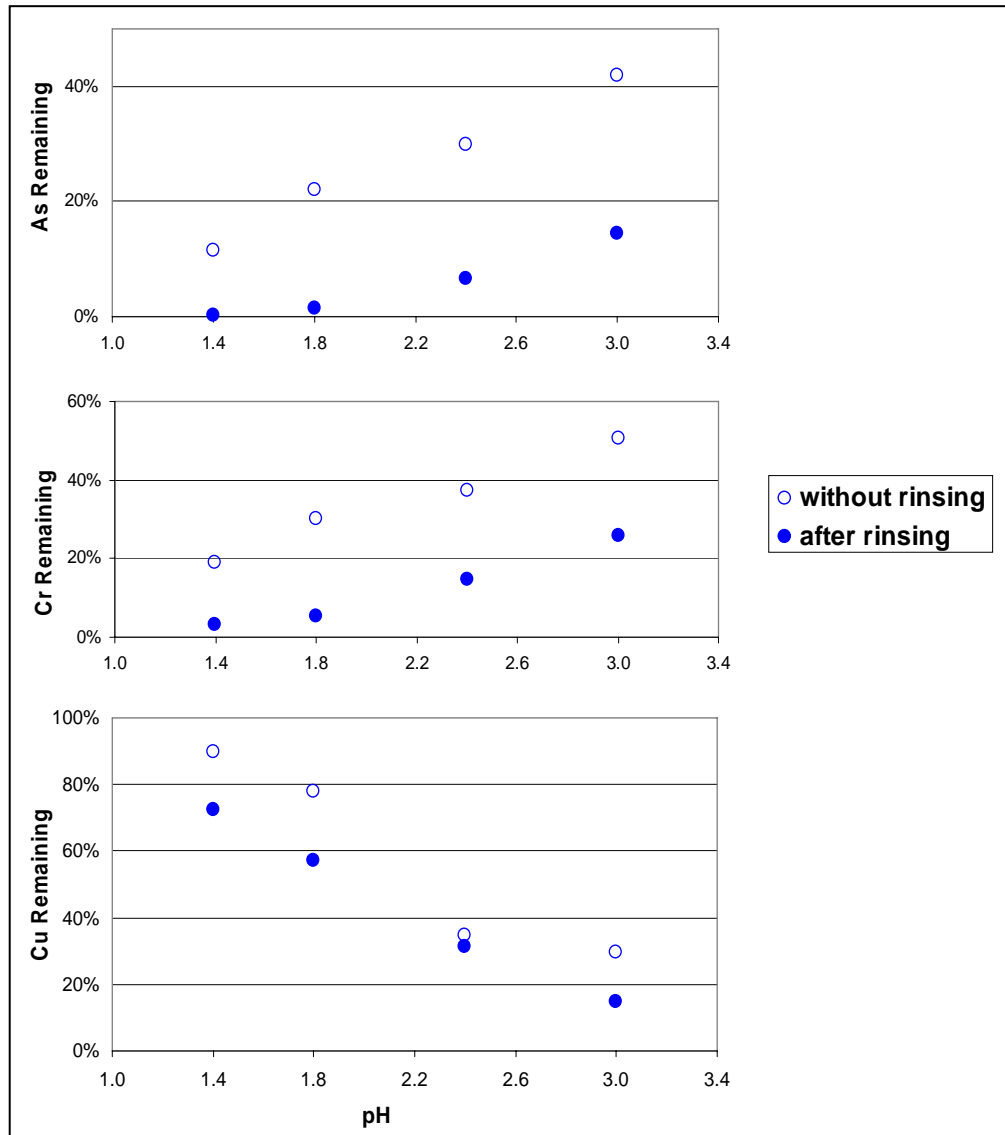


Figure 4: Rinsing wood flakes following extraction removes significant amount of metals (The data shown are for 1 hour extraction using 1 % oxalic acid at 80 °C)

3.4 Continuous Mixing, Water Source, and Liquid to Solid Ratio

Supplemental experiments were performed to determine how the extraction of metals from preservative-treated wood particles was affected by continuous mixing, municipal water compared to deionized water, and the solid to liquid ratio. The results from the two-level, five variable half-factorial design are shown in Table 5. Effects for the five variables and two-factor interactions were calculated using Yates's algorithm, and then ranked and plotted on custom-generated probability paper as shown in Fig. 5. Observations that deviate from a linear fit of the effects close to zero are unlikely to be random, and these factors can be considered to significantly affect the extraction levels. Therefore, according to Fig. 5, pH and time (variables 3 and 4, respectively), as well as their interactions have the most significant effect on the removal of As and Cr. The removal of Cu, however, appears to be predominately affected by pH, at least under the range of conditions for these experiments.

Table 5: Results for supplemental experiments

Experiment ID	Water	Mixing	pH	Time [h]	Liquid to Solid Ratio	Metals Remaining		
						As	Cr	Cu
A	tap	no	1.4	1	10:1	21%	40%	67%
B	DI	yes	1.4	1	10:1	13%	29%	67%
C	DI	no	2.4	1	10:1	62%	85%	7%
D	tap	yes	2.4	1	10:1	46%	65%	6%
E	DI	no	1.4	6	10:1	1%	9%	76%
F	tap	yes	1.4	6	10:1	1%	6%	43%
G	tap	no	2.4	6	10:1	20%	35%	5%
H	DI	yes	2.4	6	10:1	7%	20%	2%
I	DI	no	1.4	1	20:1	13%	31%	59%
J	tap	yes	1.4	1	20:1	14%	29%	35%
K	tap	no	2.4	1	20:1	48%	67%	5%
L	DI	yes	2.4	1	20:1	44%	65%	4%
M	tap	no	1.4	6	20:1	2%	12%	60%
N	DI	yes	1.4	6	20:1	1%	8%	66%
O	DI	no	2.4	6	20:1	13%	29%	3%
P	tap	yes	2.4	6	20:1	11%	26%	4%

A large amount of precipitated minerals were visible after adding oxalic acid to municipal tap water, so the source of water was expected to impact the amount of metals extracted from the treated wood. However, the amount of As, Cr, and Cu extracted was similar using 1 % (wt) oxalic acid solutions in either deionized or tap water, as shown in Fig. 6. For the most part, extractions performed using a smaller liquid to solid mass ratio of ten without mixing resulted in less As and Cr being removed from the wood than other combinations. The results for arsenic are shown in Fig. 7. However, this effect of interaction between mixing and liquid to solid ratio appear to be borderline significant. Kakitani *et al.* (2007) extracted metals from treated wood using oxalic acid adjusted to pH 3.2 with sodium hydroxide at liquid to solid ratios of 10, 15, and 20, and the affect of liquid to solid ratio was modest at best, especially at short extraction times.

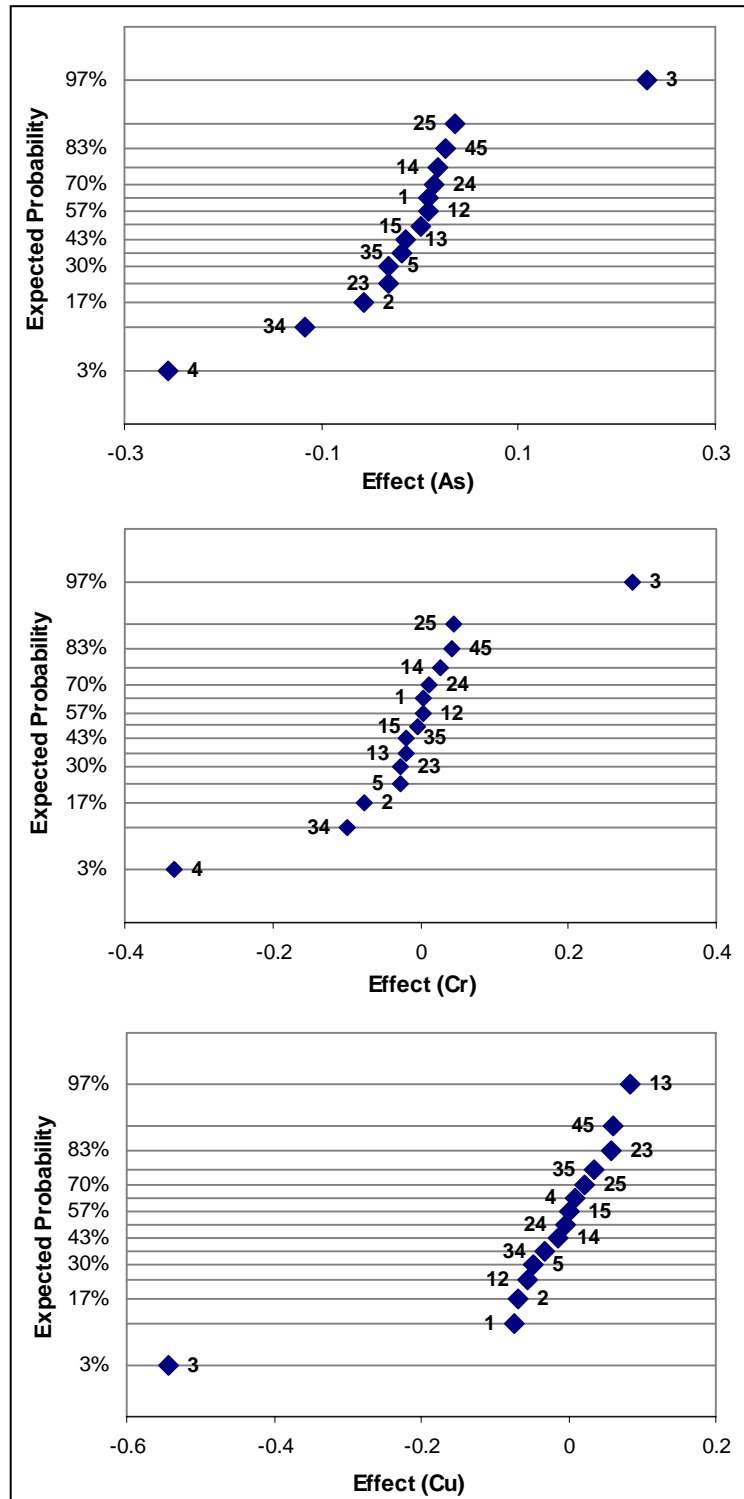


Figure 5: Probability plot of effects from supplemental experiments. The effects are as follows: 1) water source; 2) mixing; 3) pH; 4) time; 5) liquid to solid ratio

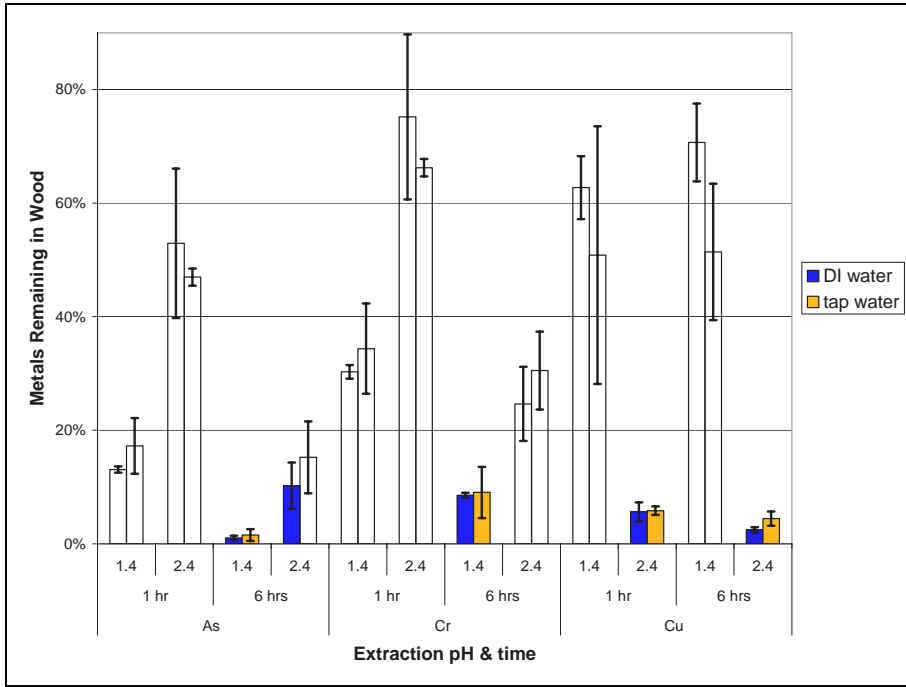


Figure 6: Effect of water source on metals extraction

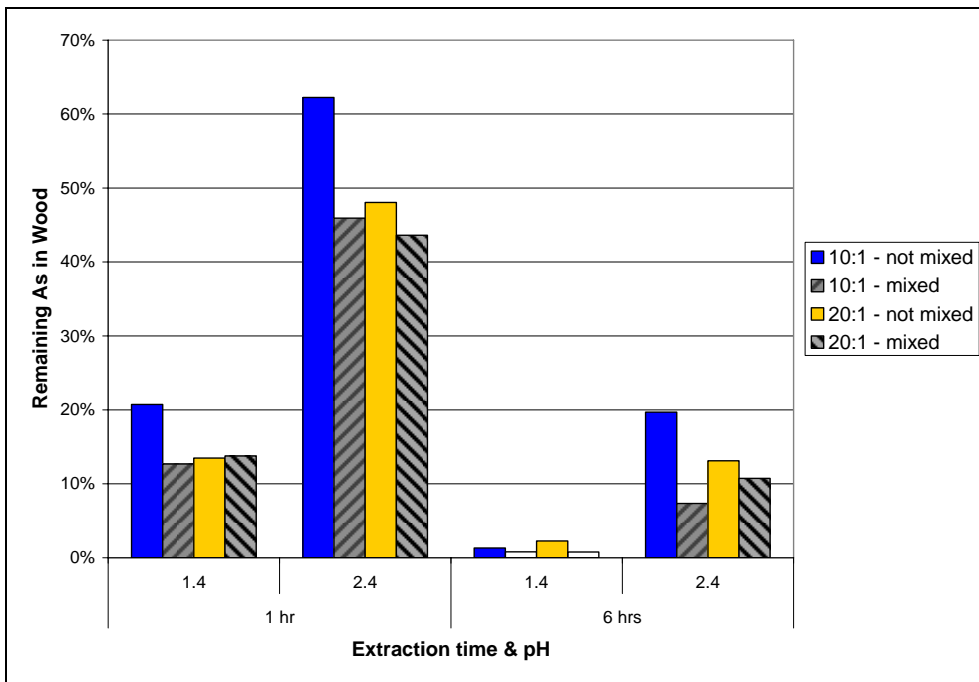


Figure 7: Effects of liquid to solid ratio and mixing on extraction of arsenic

4. CONCLUSIONS

Nearly all the metals impregnated into wood samples by treatment with chromated copper arsenate (CCA) were readily extracted using one-percent oxalic acid solutions that were pH adjusted with sodium hydroxide. Increased temperatures facilitated the removal of arsenic, chromium and copper, although the effect of temperature on copper removal appeared less pronounced than on arsenic and chromium. Oxalic acid alone was rather ineffective at extracting copper but was quite effective at extracting arsenic and chromium. The addition of sodium hydroxide to oxalic acid resulted in increased copper removals but decreased the amount of

arsenic and chromium removed. Two-stage extractions in which most of the As and Cr were first removed using oxalic acid, followed by a second extraction using an oxalic acid/sodium hydroxide mixture resulted in more rapid removal of metals. Furthermore, the extracted wood flakes contained a significant amount of residual water-soluble metal compounds that could be removed by rinsing with water. Although a significant amount of dissolved minerals in local tap water were precipitated by oxalic acid, solutions prepared from tap water were equally effective as those prepared from deionized water. While the effects of continuous mixing and liquid to solid ratio were not deemed statistically significant, extractions carried out at the lower liquid to solid ratio of 10 with no mixing resulted in lower metals removals than other extractions.

Based on these preliminary laboratory experiments, pilot-scale experiments are being planned in which composite panels will be made from remediated preservative-treated wood. A limited number of additional laboratory experiments are currently being performed to determine optimal extraction conditions that are most likely to lead to the lowest amount of residual metals while retaining favorable physical and mechanical properties of composites produced from the remediated wood. For example, a two-stage extraction is being investigated in which virtually all arsenic and chromium is removed in the first stage within one hour and the remaining copper is removed using a solution at a higher pH in the second stage. Once these conditions have been determined, pilot-scale extraction experiments will be conducted, and the remediated wood flakes will be used to make boards that will be tested for physical properties and mechanical performance.

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