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Rapid microwave-assisted acid extraction of southern pine waste wood to remove metals from chromated copper arsenate (CCA) treatment

Abstract: Recovery of metals from chromated copper arsenate (CCA)-treated southern pine wood particles was investigated by extraction in a microwave reactor with binary combinations of acetic acid (AA), oxalic acid (OxA), and phosphoric acid (PhA). Use of OxA was not successful, as insoluble copper oxalate complexes impeded copper removal. The combination of OxA and AA also had adverse effects on copper extraction. In contrast, the combination of AA and PhA enhanced the chromium recovery rate. The highest recovery rate of metals could be achieved with a mixture of 2.75% PhA and 0.5% AA at 130°C for 10 min in the microwave oven. The total recovery rate approached 100% for arsenic, 96.7% for chromium, and 98.6% for copper in a one-step process.

Keywords: binary acid solution, CCA-treated wood, microwave extraction

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Introduction

Preservative-treated wood products have a long service life, but sooner or later they fail. It is estimated that approximately 12×10^6 m^3 $year^{-1}$ of spent chromated copper arsenate (CCA)-treated wood products in the United States and Canada will be removed from service in the next 20 years (Kazi and Cooper 2006). Because of their toxic arsenic and chromium contents, which are partly water soluble, their proper disposal is problematic (Townsend et al. 2005; Moghaddam and Mulligan 2008). Landfilling is becoming costly or even impractical owing to strict regulatory requirements and liability. The literature describes many alternatives of disposal, such as by pulping with organic acids (Stephan et al. 1996), liquefaction and precipitation of the metals (Lin and Hse 2005), hydrothermal treatment (Catallo and Shupe 2008), and pyrolysis (Govaerts et al. 2011; Kemiha et al. 2011), just to mention a few. All these suggested approaches have some disadvantages. The burning of CCA-treated wood is also dubious for reasons of health care and environmental protection. New techniques are needed for recycling and disposal of decommissioned CCA-treated wood.

Exposure of CCA-treated wood to an acid solution leads to the reoxidation of the chromium and to the conversion of the CCA elements into their water-soluble form (Shiau et al. 2000; Clausen 2004; Humer et al. 2004; Gezer et al. 2006; Kazi and Cooper 2006; Kakitani et al. 2007; Janin et al. 2009). In the quoted studies, various organic acids were tested for this purpose, such as oxalic, acetic, citric, and formic acid. These studies indicated that the acid extraction process is slow. However, acid exaction aided by microwave energy accelerates the process (Yu et al. 2009). This study demonstrated that 1) microwave-assisted acid extractions with oxalic acid (OxA), acetic acid (AA), and phosphoric acid (PhA) reduce the reaction times from hours to minutes compared to the conventional methods; 2) OxA extraction effectively removes arsenic (As) and chromium (Cr) but not copper (Cu); 3) AA extraction is highly effective for the removal of As and Cu but not of Cr; and 4) extraction with PhA was less effective than that with either OxA or AA. Extraction with two acids has advantages and can be performed in a one-step process with a combination of two acids or by means of a two-step process consisting of consecutive acid extraction steps.

In the present study, two acids were mixed, and the extraction potential of the binary mixtures of OxA, AA, and PhA was studied. The objective of the present study was to develop a cost-effective, microwave-based, double-acid extraction system to maximize the removal of CCA elements from spent CCA-treated wood. This task was addressed by optimizing acid combinations

and concentrations, extraction times, and the reaction temperature to minimize any environmental impact.

Materials and methods

CCA-treated southern pine (*Pinus* sp.) guard rail posts were obtained from Arnold Forest Products Co. (Shreveport, LA, USA). The posts were reduced to sawdust on a table saw. The sawdust was screened to collect sawdust that passed through a 40-mesh sieve and retained on a 60-mesh sieve and then dried to a constant weight in an oven maintained at 50°C. The dried sawdust was stored in a heavy-duty zip-lock polyethylene bag and investigated without further treatment. All acids for extraction were of reagent grade and obtained from commercial sources. The acids were diluted with deionized water.

The acid extractions were carried out in a Milestone MEGA 1200 laboratory microwave oven. Unless otherwise stated, 1 g of wood meal was weighted into a 100-ml Teflon reaction vessel with a magnetic stirring bar. The concentration of acids in solutions is given as percent by weight per volume of deionized water (g/v). Twenty milliliters of acid combinations of AA/OxA, AA/PhA, or PhA/OxA was then added to the reaction vessel and thoroughly mixed. The reaction vessel was then placed on the rotor tray inside the microwave cavity. The temperature was monitored by an automatic fiber optic temperature control system. Based on the monitored temperature, the output power was auto-adjusted during acid extraction. In this study, the temperature was increased from room temperature to 160°C at a heating rate of 32.5°C min^{-1} and then was kept constant for 30 min, unless otherwise indicated. After a cooling period of 30 min at the end of acid extraction, the extracted liquids were vacuum filtered through Whatman No. 4 filter paper and then diluted to 100 ml in a volumetric flask for the determination of the CCA elements. The solid wood residue retained on the filter paper was oven dried and then subjected to acid digestion prior to the determination of chromium, arsenic, and copper. Samples were digested in accordance with American Wood Protection Association Standard A7-93 (AWPA 2008). The procedure requires that solid residues be accurately weighed into 100-ml test tubes. For each gram of solid residue, 15 ml of nitric acid was added. A digestion blank along with the samples was also prepared. The test tubes were placed into an aluminum heating block, and the temperature was increased and maintained at 120°C until a transparent liquid was obtained. The transparent liquid was cooled to room temperature and 5 ml of H_2O_2 was dropwise added. If the solution was not clear after this treatment, the temperature was increased and another 5 ml of hydrogen peroxide was added. The sample was continually heated until approximately 1 ml of the sample solution remained in the test tube. The sample was carefully transferred into a 25-ml volumetric flask and then diluted with distilled water to a 25-ml solution for the determination of the Cr, As, and Cu in the residue by means of inductive coupled plasma atomic emission spectroscopy (ICP-AES). The recovery rate (RR, %) of the elements in acid extraction was then determined with the following formula: $RR\ (\%)=100\times Ws/(Ws+Wr)$, where Ws is the weight of the CCA elements in the acid-extracted solution and Wr is the weight of the CCA elements in the wood residue.

Data were evaluated by analysis of variance (ANOVA) with SAS 9.0 software (SAS 2008). The significant differences between mean values were determined by Duncan's multiple range test.

Results and discussion

Experiments with OxA/AA mixtures

CCA recovery rate (RR_{CCA}) with binary acid solutions consisting of oxalic (OxA) and acetic acid (AA) are summarized in Table 1. In all acid combinations, >98% and 99% of As and Cr could be recovered, respectively. However, the average Cu recovery was surprisingly low (30.1–34.6%) in view of the previous finding that 97.5% Cu removal can be attained with only AA extraction in a microwave reactor (Yu et al. 2009). Probably, insoluble complexes between OxA and copper occurred (Pizzi 1982, 1990; Bull 2001; Humer et al. 2004; Kakitani et al. 2009). Kirsch and Maurer (1998) reported on the formation of mixed acid complexes containing AA and OxA in the organic phase. In addition, in the presence of anhydrous AA at high temperatures, OxA can decompose quickly (Pszonicki and Tkacz 1976). This is the reason why mixtures of AA/OxA are not suitable for CCA extraction.

Experiments with PhA/OxA or PhA/AA

Table 2 summarizes the average RR of CCA elements with combinations of PhA with either OxA or AA. In the mixed PhA and OxA solution, the RR of Cr and As approached 100%. However, the highest RR_{Cu} was 52%. For comparison, in a one-step OxA extraction (microwave reactor) the yield was 39% and in a PhA extraction it was 79% (Yu et al. 2009). Accordingly, the efficiency of PhA in the binary solution is not satisfactory for copper recovery. In a two-step process beginning with 1 h of OxA extraction followed by a 3-h PhA extraction, Kakitani et al. (2006) found that the two acids alone were ineffective for remediation of CCA-treated wood.

Concentration of acids		Recovery rate of elements		
AA (%)	OxA (%)	As (%)[a]	Cr (%)[a]	Cu (%)[a]
1.5	1	99.65±0.24	99.88±0.49	34.57±1.02
1	1	99.67±0.45	99.89±1.03	32.93±1.18
0.5	1	99.59±0.27	99.65±0.96	31.50±0.85
1.5	0.5	99.56±1.45	99.43±1.73	31.47±0.91
1	0.5	99.21±0.65	99.37±0.83	30.60±0.79
0.5	0.5	98.78±0.79	99.38±1.28	30.11±1.32

Table 1 Recovery rate of elements by extraction with mixtures of acetic acid (AA) and oxalic acid (OxA) with the concentrations indicated. Conditions: microwave reactor at 160°C for 30 min.
[a]Each value is an average of three sample replications.

Concentration of acids (%)		Recovery rate of elements		
PhA (%)	Other acids (%)	As (%)[a]	Cr (%)[a]	Cu (%)[a]
0.5	OxA 1	99.52±0.23	97.12±0.21	33.82±1.15
1.25	OxA 1	99.12±0.13	97.39±0.49	36.49±1.05
2.0	OxA 1	98.90±0.55	97.67±0.49	41.30±2.01
3.5	OxA 1	99.58±1.05	98.77±1.03	52.14±1.49
0.5	AA 1	94.05±2.28	23.52±1.72	95.38±2.42
1.25	AA 1	96.66±1.38	50.03±2.09	97.38±1.02
2.0	AA 1	99.01±0.60	72.18±1.45	97.46±0.67
3.5	AA 1	99.52±0.91	98.10±1.37	98.60±0.93

Table 2 Recovery rate of elements by extraction with mixtures of phosphoric acid (PhA), oxalic acid (OxA), and acetic acid (AA) with the concentrations indicated. Conditions: microwave reactor at 160°C for 30 min.
[a]Each value is an average value of three sample replications.

As demonstrated above, combinations of PhA and AA are effective for removal of As and Cu, but the challenge is the chromium recovery. In this context, it is remarkable that the addition of 1.0% AA into a series of PhA solutions enhanced the RR_{Cr} in linear proportion to the PhA concentration, whereas the PhA concentration of 3.5% was most effective with a RR_{Cr} of 98.1% (Tables 2 and 3).

Concentration of acids		Recovery rate of elements		
PhA (%)	AA (%)	As (%)[a]	Cr (%)[a]	Cu (%)[a]
3.50	2	99.52	99.30	99.02
2.75	2	99.48	99.21	99.01
2.00	2	98.89	74.32	98.23
1.25	2	96.77	51.77	97.99
0.50	2	95.50	25.79	96.87
3.50	1.5	99.52	99.21	98.97
2.75	1.5	99.24	97.98	98.65
2.00	1.5	98.66	73.33	97.98
1.25	1.5	97.79	49.36	97.56
0.50	1.5	95.01	24.57	96.22
3.50	1	99.52	98.10	98.60
2.75	1	99.06	96.37	98.56
2.00	1	99.01	72.18	97.46
1.25	1	96.66	50.03	97.38
0.50	1	94.05	23.52	95.38
3.50	0.5	99.61	95.35	98.01
2.75	0.5	99.01	96.23	98.07
2.00	0.5	98.54	70.37	97.36
1.25	0.5	96.03	48.95	96.85
0.50	0.5	93.57	23.06	95.12

Table 3 Recovery rate of elements by extraction with mixtures of PhA and AA with the concentrations indicated. Conditions: microwave reactor at 160°C for 30 min.
[a]Each value is an average value of three sample replications.

Experiments with PhA/AA systems

Table 3 summarizes the RR of CCA metals obtained by the combination of five PhA and four AA concentrations in microwave extraction at 160°C for 30 min. ANOVA indicates that PhA concentration has a significant effect on the RR_{CCA}, but the effect of the AA concentration is not significant within the concentration range evaluated. The results of the Duncan's multiple range tests for significant means are summarized in Table 4. The average RRs were 99.3% (As), 97.5% (Cr), and 98.4% (Cu), when the concentration of PhA was 2.75% regardless of the concentration of AA. Obviously, the average RR_{Cr} cannot be improved significantly by raising the PhA concentration above 2.75%; however, the RR_{Cr} decreased significantly with PhA concentration decrements below 2.75%. Therefore, a PhA concentration of 2.75% was chosen for additional process optimization experiments.

Although the effect of AA concentration was statistically not significant, a RR above 99% was attained for all CCA elements when AA concentration increased to 2.0%. The concentration of PhA is more important as stated above in terms of RR_{Cr} (Table 4). Chromium has the highest resistance to extraction because of its strong bonding capacity with lignin (Pizzi 1990). However, it is not fully clear how the presence of AA in PhA improves the chromium recovery, and the following literature overview may help with speculation about the reasons.

For the preparation of activated carbon (Jagtoyen and Derbyshire 1998; Zuo et al. 2009) and flame-retardant

		Recovery rate of elements		
Concentration of acids	n[a]	As (%) mean[b]	Cr (%) mean[b]	Cu (%) mean[b]
PhA (%)				
0.50	12	94.53 D	23.78 D	95.90 D
1.25	12	96.82 C	50.03 C	97.22 C
2.00	12	98.78 B	73.55 B	97.80 B
2.75	12	99.26 A B	97.47 A	98.44 A
3.5	12	99.52 A	97.99 A	98.71 A
AA (%)				
0.50	15	97.42 A	66.67 A	97.29 A
1.00	15	97.66 A	68.5 A	97.47 A
1.50	15	98.01 A	68.67 A	97.62 A
2.0	15	98.03 A	70.08 A	98.06 A

Table 4 Summary of the Duncan's multiple range tests for significant means on the effect of concentrations of PhA and AA on the CCA recovery rate.
[a]*n* indicates number of samples.
[b]Different letters indicate significantly different.

products (Inagaki et al. 1976) from various lignocellulosic materials, PhA was already successfully applied. PhA-treated lignocellulosic materials begin to lose carbonyl and methyl groups in hemicelluloses at 50°C; lignin starts to change at 100°C, and substantial oxidation of cellulose occurs and ketones are formed at 150°C (Jagtoyen and Derbyshire 1998). Polymer chains are split around 150°C (Solum et al. 1995). The treatment of cellulose with PhA resulted in reduced substrate crystallinity and increased accessibility (Saito et al. 1994; Nilsson et al. 1995). Interestingly, Dobele et al. (1999) found that PhA pretreatment of cellulose enhances essentially the yield of levoglucosenone in a subsequent pyrolysis step, probably by promoting the dehydration of the anhydroglucose units. Thus, it is possible that these characteristic reactions of PhA may each contribute to chromium recovery. For instance, the composition and structural changes in lignin induced by PhA treatment may weaken the bond binding the metal-lignin complexes to increase the chromium removal. Furthermore, decreased crystallinity and cellulose surface exposure by solubilization of hemicelluloses by the PhA treatment may result in increasing access of AA to enhance the metal recovery rate.

Optimization of the microwave reactor system

The RR_{CCA} for different reaction times and temperatures is summarized in Table 5. All extractions were carried out at fixed acid concentrations of 2.75% (PhA) and 0.5% (AA). ANOVA indicates that reaction temperature has a significant effect on the RR_{CCA}, whereas reaction time is not relevant in this regard. The results of the Duncan's multiple range tests for significant means are summarized in Table 6. Average maximum RR was attained for As and Cu at 100°C; but, as expected, Cr required 130°C to reach the maximum RR. The RR_{Cr} was consistently lower than that of As and Cu even at higher temperatures. The effects of PhA concentration (Table 4) and those of temperature (Table 6) show the importance of process optimization. A concentration of 0.5% AA and 2.75% PhA and an extraction time of 10 min at 130°C were the best optimum for chromium dissolution. In a microwave reactor, the extraction is more rapid than that obtainable with conventional heating.

For comparison, Kazi and Copper (1998) found in a sequential extraction with formic acid and OxA (2 h, 50°C, 10% acid concentration) that 95% Cr, 100% Cu, and 88% As can be removed. Kakitani et al. (2006) found in a two-step extraction process at 75°C with citric acid (1 h, 6.4%) followed by PhA (3 h, 3.26%) that 100% As, 99.1% Cu, and 95.7% Cr can be eliminated from treated wood.

The found optimum in the present study (0.5% AA and 2.75% PhA in microwave extraction) is substantially more convenient than the acid extraction with 10% acid concentration (Kazi and Copper 1998) and the two-step method of 6.4% citric acid and 3.26% PhA extraction (Kakitani et al. 2006) with conventional heating.

		Recovery rate of elements		
Temperature (°C)	Time (min)	As (%)	Cr (%)	Cu (%)
60	10.00	67.84	33.06	81.89
60	20.00	77.34	40.98	91.34
60	30.00	83.80	51.12	92.94
70	10.00	84.05	47.35	91.61
70	20.00	91.02	60.92	94.61
70	30.00	93.40	65.21	94.98
80	10.00	92.82	64.99	92.41
80	20.00	94.20	70.76	96.37
80	30.00	95.55	77.23	96.94
100	10.00	98.16	78.76	97.27
100	20.00	98.83	85.14	97.35
100	30.00	99.10	88.26	97.90
130	10.00	99.57	93.39	98.50
130	20.00	99.60	95.20	98.63
130	30.00	99.80	96.70	98.70
160	10.00	99.56	95.78	98.50
160	20.00	99.62	96.20	98.70
160	30.00	99.81	98.10	98.75

Table 5 Effects of microwave extraction temperature and time on the recovery rate of elements with fixed concentrations of 0.5% AA and 2.75% PhA.

	n[a]	As (%) mean[b]	Cr (%) mean[b]	Cu (%) mean[b]
Temperature (°C)				
60	9	76.33 D	41.72 E	88.73 C
70	9	89.49 C	57.88 D	93.71 B
80	9	94.19 B	70.99 C	95.51 B
100	9	98.70 A	84.40 B	97.44 A B
130	9	99.51 A	95.10 A	98.61 A
160	9	99.66 A	96.69 A	98.64 A
Time (min)				
10	18	90.33 A	68.91 A	93.32 A
20	18	93.42 A	74.87 A	96.30 A
30	18	95.19 A	79.60 A	96.70 A

Table 6 Summary of the Duncan's multiple range tests for significant means on the effect of temperature and time on the CCA recovery rate.
[a]*n* indicates number of samples. [b]Different letters indicate significantly different.

Conclusions

The results show that diluted PhA mixed with AA is very effective to recover CCA elements in a microwave reactor within a short period. A binary acid solution consisting of 2.75% PhA and 0.5% AA (10-min reaction time at 130°C) is optimal to achieve the maximum recovery rate of all CCA elements. However, larger-sized wood chips should be extracted to simulate the industrial praxis. Further research concerning AA concentration, reaction time and temperature, along with how to best utilize metal-free wood is needed to maximize economic viability.

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