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Chemical remediation of wood treated with micronised, nano or soluble copper preservatives[1)]

Abstract: The potential for extraction of copper from wood treated with micronised, nano or soluble forms of copper has been evaluated in view of chemical remediation. In focus were EDTA, oxalic acid, bioxalate, and D-gluconic acid for extraction of Cu from treated wood. Bioxalate extractions for 24 h resulted in Cu removal over 95% for all tested materials, and the effectiveness of oxalic acid extraction was very similar to that of nano-CuO-treated wood. Bioxalate was more effective than oxalic acid in removing Cu from ACQ-D, MCQ, MCA, CA-C and Cu-ethanolamine treated wood. D-gluconic acid extractions resulted in the lowest Cu removal for nano-CuO even though D-gluconic acid was effective for all other materials. As the pH of D-gluconic acid decreased, Cu removal was improved except for nano-CuO. There is no distinctive difference in Cu removal from wood treated with ACQ-D, MCQ, CA-C, MCA and Cu-ethanolamine.

Keywords: bioxalate, CCA, copper azole, copper quat, EDTA, gluconic acid, micronised Cu, nano-Cu, oxalic acid, remediation

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Introduction

Numerous studies have focused on remediation of treated wood since the 1990s reflecting public and scientific awareness about release of toxic metals from treated waste wood disposed of in landfills or by burning or composting. Substantial progress has been made in remediation of preservative-treated waste wood by chemical extraction with mineral and organic acids and by bioremediation with fungi and bacteria. Kartal and Imamura (2003) and Clausen and Lebow (2011) have reviewed this progress. Remediation of chromated copper arsenate (CCA) -treated wood was preferably studied because considerable amounts of this type of wood were in service and in the waste wood stream (Kemiha et al. 2011; Hse et al. 2013). In recent years, new Cu-based preservative systems without Cr and As were applied for wood protection due to restrictions of CCA type wood preservatives for most residential applications (Lebow et al. 2004). Alkaline Cu-quat (ACQ), and copper-azole (CA), the two major copper systems in North America, are based on ethanolamine for complexation of soluble copper ions. In addition to these "water-soluble Cu" formulations, micronised-copper based systems have been introduced into the North American and European market and nano-copper has also been evaluated as a potential wood preservative (Kartal et al. 2009; Xue et al. 2012; Lee et al. 2014).

The performance and environmental impact of treated wood are greatly affected by fixation and leaching properties of preservative components. Matsunaga et al. (2009) stated that in preservatives containing micronised copper particles, the micro-distribution of the particles is different from that of wood treated with conventional aqueous preservatives. In conventional Cu systems, fixation is a result of a number of chemical reactions such as chelate formation and ion exchange resulting in insoluble complexes in treated wood. On the other hand, fixation in micronised Cu systems is believed to occur primarily through simple deposition in pit chambers, and on tertiary cell wall layers rather than via chemical reactions within the cell wall (Cooper 1991; Freeman and McIntyre 2008). Matsunaga et al. (2011) reported on the distribution of nano-Cu particles in the membrane and border of bordered pits.

A study of Kartal et al. (2009) demonstrated that nano-Cu is leach-resistant compared to soluble Cu oxides. Nanometer size particles smaller than the diameter of a wood window pit (<10 000 nm) or the opening of the bordered pit (400–600 nm) may penetrate deeply and distributed evenly (Freeman and McIntyre 2008). The leaching behavior of Cu from micronised Cu treated wood

in service and removal of Cu by remediation processes were expected to be different, due to other fixation mechanisms and distribution in wood, when compared to water-soluble conventional systems.

In the current study, chemical remediation by oxalic acid (OA), bioxalate (BO, a solution of oxalic acid containing sufficient NaOH to adjust the pH to 3.2), D-gluconic acid (GlA) or ethylene di-amine tetra-acetic acid (EDTA) as chelating agents was followed by Cu recovery from treated wood. D-glutonic adid, a non-toxic and easily biodegradable sugar acid, is a good sequestering agent for heavy metals and it is as a possible extracting agent for metal-polluted soils due to its ability to chelate earth alkaline elements and heavy metals (Fischer and Bipp 2002). Di-amine tetra-acetic acid forms chelates with both transition-metal ions and main-group ions and it is successful for leaching and remediation of heavy metals from contaminated soils. Kartal (2003) reported that EDTA may also be an agent for the improved removal of Cu from CCA-treated wood waste. Chemical remediation by EDTA may be one way to extract Cu for remediation of wood treated with Cu-based preservatives. Oxalic acid is useful as a reducing agent for bleaching and ink removal. It is known that the removal of Cu, Cr, and As from CCA-treated wood waste increased significantly after OA extraction (Clausen and Smith 1998; Clausen 2000; Clausen et al. 2000, 2001; Kartal and Clausen 2001a,b; Kartal and Köse 2003; Kakitani et al. 2007, 2009). Bio-oxilate (BO) produced from OA via NaOH additions, on the other hand, increases significantly extraction efficiency of CCA elements over OA (Kakitani et al. 2007, 2009). The objective of this study was to determine optimum release conditions of modified woods with micronised Cu, nano-Cu, and soluble Cu treatments via chemical extraction with the substances indicated above.

Materials and methods

Commercial ACQ-D (alkaline Cu-quat – type D) (Viance, Incorporated, Charlotte, NC), MCQ (micronised Cu-quat) (Osmose, Griffin, GA), MCA (micronised Cu-azole), CA-C (Cu-azole – type C) (Lonza, Atlanta, GA), Cu-Et (Cu-ethanolamine) (Viance, Incorporated, Charlotte, NC), CCA-C (chromated copper arsenate – type C) and nano-CuO (Cu oxide-40 nm) solutions were investigated. With the exception of CCA-C, preservative solutions were adjusted in order to reach a consistent CuO retention level of 0.64 kg m^{-3} in treated wood blocks for each preservative treatment. The chromated copper arsenate – type C (CCA-C)-treated wood in this study has been pressure-treated to a target retention of 1.4 kg m^{-3} CuO as part of an earlier research project.

Wood blocks (19×19×19 mm^3) were cut from sapwood portions of southern pine lumber. The wood blocks were free of knots and visible concentrations of resins and were without visible evidence of infection from mould, stain, or wood-degrading fungi, and had 2–4 growth rings per cm in cross sections. The blocks were conditioned to a moisture content (MC) of 10–12% in a conditioning room at 27°C and 70% relative humidity (RH) for 2 weeks before treatment. Groups of 50 pre-weighed wood blocks were vacuum-treated for 40 min at -172 kPa with each treatment according to AWPA E10 (2012). After treatment the blocks were dry-blotted and re-weighed to determine the uptake of treatment solution. The treated blocks were conditioned for 2 weeks at room temperature (r.t.) to ensure that fixation was complete and then equilibrated to stable weight at 27°C and 70% RH for 2 weeks. The blocks were then ground to pass a US Standard 40-mesh screen (420 mm) by a Grindomix GM200 (Retsch, Incorporated Newtown, PA) and the ground wood was conditioned at 27°C and 70% RH for 2 weeks before extractions. CCA-C treated sawdust was obtained from previously treated SP stakes (19×19×450 mm^3) with CCA-C Q2 solution at 1.2% concentration. The stakes were treated with CCA-C by means of a full cell process with an initial vacuum of -75 kPa (gauge) for 30 min followed by pressure maintained at 1.03 MPa (gauge) for 1 h.

Oxalic acid, BO, EDTA, or D-gluconic acid (GlA) (49–53% in water by wt) (Sigma, St. Louis, MO, USA) were the chelating agents (Figure 1). Bioxalate solution was prepared from 0.125 mol of OA with deionised water (DI-water), and the pH of the solution was then adjusted to 3.2 by adding NaOH solution under monitoring pH changes. pH of other GlA solutions was also adjusted by adding NaOH solution and pH was monitored by a digital pH-meter. DI-water extractions served as control. Each ground sawdust sample of 4 g was placed in a separate 100 ml-volumetric flask along with 80 ml of chelating agent solution for each extraction process.

1% OA (pH: 1.4), 1% BO (pH: 3.2), 1% EDTA (pH: 2.2) or GlA at pH 9.0, 7.0, 2.5, 2.2, and 2.1 were the additives at 1, 5, and 10% concentration levels to extract copper from sawdust samples obtained by grinding treated wood blocks (Table 2, Figure 2). Extraction occurred at r.t. for 6 h and 24 h in an orbital shaker at 120 rpm. After extraction the solutions in the flasks were filtered through Whatman no. 4 filter papers (vacuum pump), and rinsed three times with 50 ml of DI-water each time. Rinsed sawdust was oven-dried at 60°C for 24 h and conditioned at 23°C and 65% RH.

Unextracted and extracted sawdust samples were digested and analysed for residual Cu, Cr and As by inductively coupled plasma (ICP) emission spectroscopy (ICP-ES) based on the AWPA A21 standard method (AWPA 2012) to determine percentage Cu, Cr and As released during extractions.

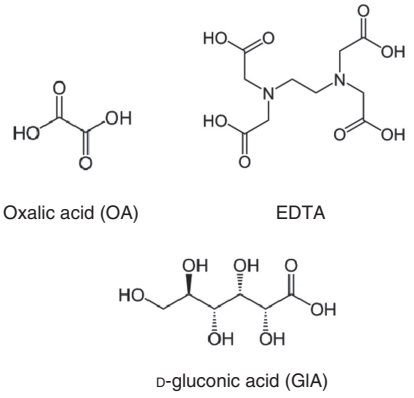

Figure 1 Chelating agents.

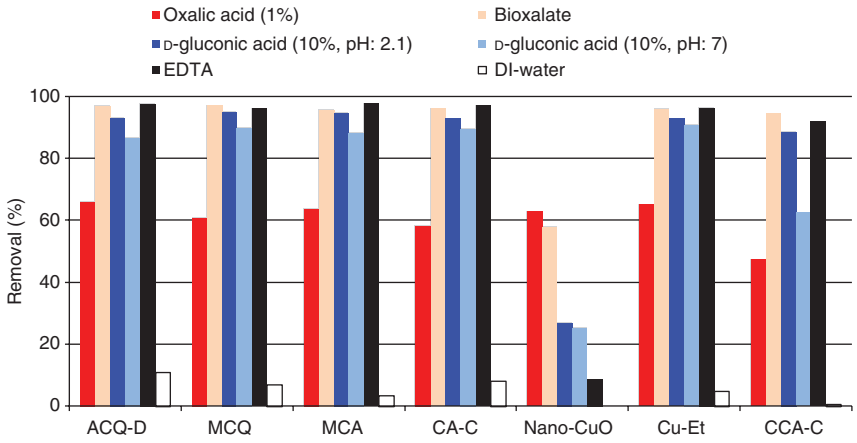

Figure 2 Cu removal from treated wood after a 6 h extraction. Values represent the average of duplicate experiments.

Results and discussion

Table 1 shows uptake retention of CuO in the treated wood for each wood preservative and the Cu content analysis (mg g^{1}) by ICP-ES in the treated wood prior to extractions.

Percentage removal of Cu from treated sawdust by the chelating agents and DI-water is given in Table 2. As expected, chelate extractions were more effective than that with DI-water. Cu removal was not detectable when nano-CuO-treated sawdust (W_{NCuO}) was extracted with DI-water. Highest Cu removal was obtained in the extractions for W_{ACQ-D}. After the 6 h extraction there was a distinct difference in the Cu removal by OA and BO extraction for all samples except NCuO. Copper removal increased from around 60 to over 95% when BO was employed for 6 h and 24 h extraction times. In extractions of W_{NCuO} for 24 h, OA was able to remove 95% of Cu, but BO removed slightly less Cu. 24 h extraction time resulted in only slight increases in Cu removal for all treatments (compared to 6 h) except NCuO, where substantially greater releases were observed after 24 h. Under all conditions BO was more effective than OA in removing Cu from W_{ACQ-D}, W_{MCQ}, W_{MCA}, W_{CA-C}, and W_{Cu-Et} because of its strong complexing properties. Additionally, BO and EDTA were effective at solubilising Cu complexed by the chromium component of CCA-C. BO has been shown to be more effective for Cu removal from CCA-C treated wood than OA (Kakitani et al. 2006, 2009). Di-amine tetra-acetic acid at 1% concentration was very effective in Cu removal for all preservatives except NCuO. A previous study by Kartal (2003) reported 93% Cu removal from W_{CCA-C} by 1% EDTA extraction for 24 h. Kartal and Köse (2003) also found similar Cu removal by 1% EDTA extraction. Even though EDTA is very effective at removing Cu, it was unable to chelate 40 nm particles of NCuO in this study. Possible reasons for this may be the strong affinity of NCuO to wood components, the inability of EDTA to chelate agglomerated nanoparticles, or improved penetration (uniform distribution) of Cu in treated wood. Unlike hammer-milled micronised particles of Cu (MCu), uniform nanoparticles of Cu by pyrolysis agglomerate readily without dispersing agents and at high concentrations, i.e., under conditions following vacuum treatment of wood (Clausen et al. 2010). Kartal et al. (2009) found no measurable amounts of Cu in leachates from W_{NCuO}, when the treated wood blocks were subjected to a 14-day-leaching course based on the AWPA E11-06 standard test (AWPA 2012). The results can be interpreted that the charges were modified and the Van der Waals forces might have accounted for the low leaching of NCuO. For MCu-containing wood preservatives, Cu removal by EDTA and DI-water were considerably higher than seen in the NCuO treatment. Freeman and McIntyre (2008) have reported that MCu particles are physically deposited into the wood structure while Cu is chemically

Table 1 Retention of CuO and Cu content in treated wood.

Preservatives	CuO (kg m^{-3})[a]	Cu (mg g^{-1})[b]
ACQ-D	0.66 (0.01)	1.19
MCQ	0.64 (0.01)	1.22
MCA	0.63 (0.01)	1.29
CA-C	0.63 (0.01)	1.29
NCuO	0.63 (0.01)	0.76
Cu-Et	0.62 (0.01)	1.31
CCA-C	1.44 (0.05)	1.66

[a]Each value represents average of 50 wood specimens for each treatment. Values in parentheses are standard deviations.
[b]Cu content in wood before extraction by ICP.

Table 2 Percentage removal of copper from treated sawdust during extraction (duplicate experiments). (NCuO: nano-CuO with 40 nm particle size. For other abbreviations see text.)

Cu removal from	Extr. time (h)	OA pH 1.4, 1%	BO pH 3.2, 1%	EDTA pH 2.2, 1%						D-gluconic acid	DI-water
					pH 9.0, 10%	pH 9.0, 5%	pH 9.0, 1%	pH 2.1, 10%	pH 2.2, 5%	pH 2.5, 1%	
ACQ-D	6	65.9	96.8	97.6	82.8	75.9	54.4	93.2	88.5	70.4	11.0
	24	69.1	98.0	98.2	84.8	80.4	64.8	93.3	89.3	72.0	12.0
MCQ	6	60.7	97.0	96.2	75.8	67.1	31.8	95.1	88.8	74.7	7.1
	24	62.5	97.9	97.6	82.7	73.0	38.2	95.5	90.0	74.5	7.3
MCA	6	63.8	95.8	97.9	75.8	67.0	34.5	94.7	87.4	69.1	3.6
	24	65.3	97.5	99.0	80.6	71.2	37.2	93.9	87.7	72.1	5.0
CA-C	6	58.2	96.1	97.0	83.7	78.2	39.8	92.8	88.9	73.8	8.3
	24	60.7	96.8	98.2	85.6	80.1	43.9	93.1	88.3	72.4	9.9
NCuO	6	62.8	58.0	8.8	22.5	20.9	15.5	27.0	17.5	17.1	0.0
	24	95.1	90.0	12.1	45.2	33.8	21.9	51.2	31.8	20.4	0.0
Cu-Et	6	65.2	95.9	96.3	76.2	40.9	38.7	93.0	87.3	72.3	4.8
	24	66.4	97.0	95.1	86.3	79.1	45.9	94.1	88.4	72.9	5.9
CCA-C	6	46.8	94.1	91.9	58.6	50.9	33.2	88.6	84.0	67.8	0.1
	24	47.3	94.6	97.0	67.6	57.7	39.1	93.0	88.9	72.8	0.6

fixed in wood preservative systems based on soluble Cu chemicals. The quoted authors pointed out that the binding mechanisms for a part of MCu and soluble Cu in corresponding preservatives may be similar based on ion exchange, but the majority of MCu fixation is a simple deposition. Clausen et al. (2010) have stated that nano-metals may improve penetrability into wood. With particle sizes of nano-metals smaller than the diameter of pits in the wood, complete penetration and uniform distribution can be expected. Matsunaga et al. (2007) observed particle deposits of MCu in ray tracheids and pit lumens.

Other than EDTA and DI-water, the lowest Cu removal from NCuO was obtained following GlA extractions. As the pH of GlA decreased from 9.0 to around 2, the Cu removal increased except for NCuO indicating the importance of acidity of GlA with this regard. Expectedly, the higher concentrations yielded more Cu removal. Even though the effect of extraction time on Cu removal was distinctive for extractions at pH 9.0, this trend disappeared for extractions at pH 2. Taylor et al. (2001) stated that organic acids such as GlA can remove CCA components because of its chelating ability with most metals. D-gluconic acid splits one hydrogen atom and the active gluconate anion GlA^- forms readily water soluble metal complexes in the course of dissolving oxides, hydroxides, and carbonates of polyvalent cations (Bipp et al. 1998; Survila et al. 2010). The stability of these complexes and the chelating properties of sugar acids are pH dependent. D-gluconic acid is also been well known for a long time for chelating heavy metals from contaminated soils (Peters 1999; Fischer and Bipp 2002). Xue et al. (2010) described the traditional utilisation of organic acids such as GlA for recovering metals from municipal solid waste incineration fly ash.

Figure 2 shows the Cu removal by 10% GlA at the pH levels of 2.1 and 7.0 for 6 h compared with the removal by OA, BO, EDTA and DI-water. The lowest Cu removal was obtained from W_{NCuO}, where Cu extraction by GlA at both pH levels was much lower than those by all extraction methods excluding EDTA and DI-water. As seen before, less Cu was removed at elevated pHs of GlA. Except for BO and EDTA, Cu removal in $W_{ACQ\text{-}D}$, W_{MCQ}, W_{MCA}, and $W_{Cu\text{-}Et}$ were greater than those in $W_{CCA\text{-}C}$, i.e., Cu fixed in $W_{CCA\text{-}C}$ has a higher resistance to extraction with OA, and GlA when extracted for 6 h.

Overall pH effects of GlA and OA extraction on Cu removal are presented in Figures 3 and 4, respectively. As seen in Figure 3, acidic GlA is much more favourable than alkaline conditions for Cu removal. The pH effect of GlA on $W_{CCA\text{-}C}$ was much more apparent when its acidic pH changed to neutral pH in comparison with the other Cu-preservatives. Changing the pH of OA from 1.4 to

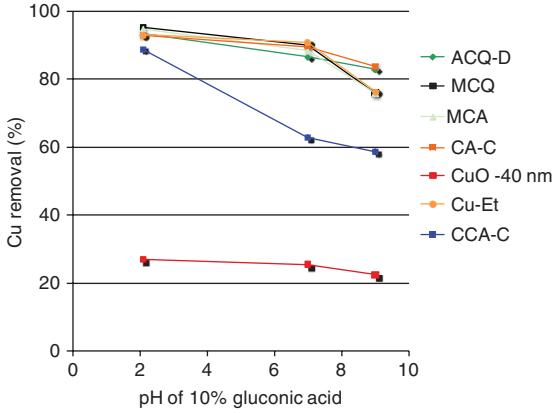

Figure 3 Effect of pH of 10% GlA solution on Cu removal from treated sawdust (6 h extraction). Values represent the average of duplicate experiments.

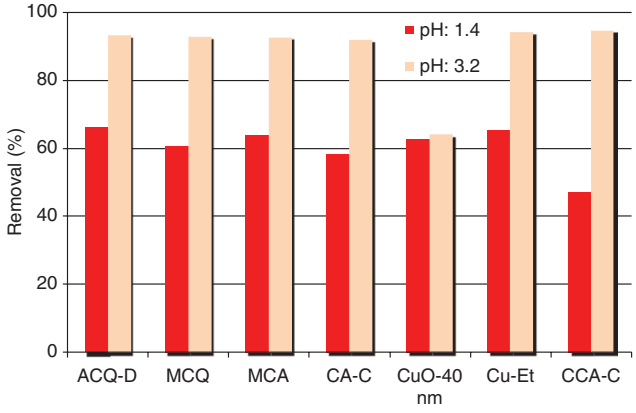

Figure 4 Effect of pH of 1% oxalic acid (pH 1.4) or 1% bioxalate (pH 3.2) solution on Cu removal from treated sawdust (6 h extraction). Values represent the average of duplicate experiments.

3.2 (BO form) increased the removal of Cu from woods treated with all other Cu preservatives except for W_{NCuO} (Figure 4).

Figure 5 shows As, Cr, and Cu removal from W_{CCA-C} after 6 h extraction. Oxalic acid at pH 1.4 was more effective in removing As and Cr than Cu; however, when the pH was elevated to 3.2 (BO form), the removal of these elements decreased. For GlA at both pH 2.1 and 7.0, the same amount of Cr was removed from W_{CCA-C}, while increased pH resulted in less As removal. Di-amine tetra-acetic acid, on the other hand, was the least effective chelating agent for removal of As and Cr in this case.

Conclusions

There is no distinctive difference in Cu removal between treatments with ACQ-D/MCQ, CA-C/MCA and Cu-Et even though the fixation mechanism in micronised systems (MCQ, MCA) is known to be different from the chemistry in water-soluble systems (ACQ-D, CA-C). Nano-Cu was resistant to EDTA extraction, whereas Cu removal was over 95% in sawdust treated with other Cu-based wood preservatives. Copper removal in nano-CuO treated wood by OA and BO after 6 h extractions was much lower than those after 24 h. BO was very effective in Cu removal for all wood treatments at both extraction times and for nano-CuO after 24 h. As a weak, noncorrosive, nonvolatile, nontoxic, and easily-biodegradable organic acid, GlA might be a cheaper alternative to commercial EDTA and BO in chemical remediation of Cu-treated waste wood.

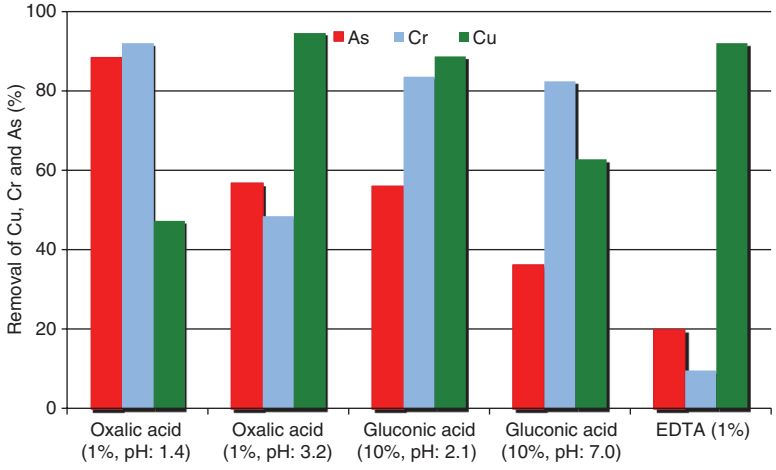

Figure 5 Removal of As, Cr, and Cu from CCA-C-treated wood sawdust by the chelating agents indicated (6 h extraction). Values represent the average of duplicate experiments.

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