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Exposure testing of fasteners in preservative treated wood: Gravimetric corrosion rates and corrosion product analyses

Samuel L. Zelinka [a,*], Rebecca J. Sichel [b], Donald S. Stone [c]

[a] USDA Forest Products Laboratory, One Gifford Pinchot Drive, Madison, WI 53726, USA
[b] College of Engineering, University of Wisconsin, Madison, WI 53706, USA
[c] Department of Materials Science and Engineering, College of Engineering, University of Wisconsin, Madison, WI 53706, USA

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ABSTRACT

Research was conducted to determine the corrosion rates of metals in preservative treated wood and also understand the mechanism of metal corrosion in treated wood. Steel and hot-dip galvanized steel fasteners were embedded in wood treated with one of six preservative treatments and exposed to 27 °C at 100% relative humidity for 1 year. The corrosion rate was determined gravimetrically and the corrosion products were analyzed with scanning electron microscopy, energy dispersive X-ray spectroscopy, and X-ray diffraction. Although the accepted mechanism of corrosion in treated wood involves the reduction of cupric ions from the wood preservative, no reduced copper was found on the corrosion surfaces. The galvanized corrosion products contained sulfates, whereas the steel corrosion products consisted of iron oxides and hydroxides. The possible implications and limitations of this research on fasteners used in building applications are discussed.

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1. Introduction

The corrosiveness of wood treated with chromated copper arsenate (CCA), the major wood preservative of the past 30 years was studied in accelerated [1,2] and long term tests [3,4] in the 1980s and 1990s. These test results led to design recommendations for minimizing metal corrosion of metals in contact with CCA treated wood [5]. However, CCA was voluntarily withdrawn for use in residential applications in the United States on December 31, 2003; similar changes in regulation have taken place in Europe and Australasia. While alternatives to CCA are commercially available, there has only been limited work studying the corrosion of metals in contact with these alternatives.

Recent work has investigated corrosion of metals in contact with alternatives to CCA using accelerated test methods. The Simpson Strong Tie Corporation (Pleasanton, CA) published a technical bulletin [6] based upon over 6000 replicates using a high temperature high humidity test method [7] and reported relative corrosion rankings of hot-dip galvanized steel exposed to several wood preservatives. They found that alkaline copper quaternary (ACQ) and copper azole (CuAz) were roughly twice as corrosive as CCA. However, the high temperature high humidity test method has been shown to have poor correlation with in-service performance [8]. Both Zelinka et al. [9] and Kear et al. [10,11] examined corrosion in solutions of the preservative chemical as opposed to treated wood and found that the corrosion behaviour of metals in the treating solution was very different from behaviour in treated wood. The lack of success with these accelerated methods highlights the importance of long term tests; recently several papers have presented non-accelerated wood–metal corrosion data for newer wood preservatives.

Thorough work by Kear et al. [12] examined the behaviour of mild steel, hot-dip galvanized steel, and stainless steel (UNS S31600) exposed to CCA (type C), CuAz (type B), and ACQ (types B and C) treated at several retention levels. A unique aspect of this work was that both metal plates specified in the AWPA standard [7] as well as metal fasteners were tested. The wood–metal assemblies were exposed to either accelerated (49 °C, 90% RH for ~400 h) or non-accelerated conditions (21 °C, various RH for ~1 year). The data were presented as corrosion rate (mm year^{-1}). Similar to accelerated tests, Kear et al. found that CuAz and ACQ were more corrosive than CCA, and attributed this to differences in fixation between the preservative systems.

Zelinka and Rammer [13] reexamined a 14 year exposure study of CCA treated wood published by Baker [3], which is the basis for the International Building Code's recommendations for fasteners used in treated wood [5]. Using archived photographs, Baker's laboratory notebook, and a new surface area algorithm [14], Zelinka and Rammer were able to convert Baker's percent weight loss data into a corrosion rate (μm year^{-1}). Additional tests were conducted for 1 year in ACQ treated wood at the same temperature and relative

* Corresponding author. Tel.: +1 608 231 9277; fax: +1 608 231 9592.
E-mail address: szelinka@fs.fed.us (S.L. Zelinka).

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Table 1
Composition and retention of the preservatives tested.

Treatment	Nominal composition[a]	Retention (kg m^{-3})	
		Nominal	Actual
CCA *(type C)* Chromated copper arsenate	47.5 wt.% CrO_3 18.5 wt.% CuO 34.0 wt.% As_2O_5	4	1.6
ACQ *(type D)* Alkaline copper quaternary	66.7 wt.% CuO 33.3 wt.% DDAC[b]	4	2.9
CuAz *(type C)* Copper azole	96.8 wt.% CuO 1.6 wt.% Tebuconazole 1.6 wt.% Propiconazole	1	0.7
MCQ Micronized copper quaternary	66.7 wt.% CuO 33.3 wt.% DDAC	4	5.0
DDAC *(carbonate)* Alkyl ammonium compound	100.0 wt.% DDAC	1.3	N A [c]

[a] Nominal compositions from AWPA standard P5 [27], with the exception of MCQ, which comes from ICC-ES evaluation report ESR-1980.
[b] DDAC, didecyldimethylammonium carbonate.
[c] This preservative could not be analyzed with ICP-AES.

humidity conditions that Baker tested (27 °C, 100% RH). The authors found that ACQ treated wood was more corrosive than CCA treated wood and that the relative corrosiveness of the wood treatments depended on the metal tested.

In addition to these long term laboratory tests, Li [15] has published results of outdoor field exposure tests of steel, galvanized steel, and stainless steel fasteners embedded in ACQ, CuAz, and CCA treated wood.

The work by these authors has quantified the difference in corrosion rates between CCA, the historical baseline, and new wood preservatives such as ACQ and CuAz. These measured corrosion rates have practical implications for designing treated wood structures. The current work expands upon these earlier works with the goal of better understanding the mechanism of corrosion in treated wood. In addition to gravimetric tests, the current work examines the corrosion products with several techniques to find correlations between the preservative chemistry and corrosion products in both treated and untreated wood.

2. Experimental

Southern pine (*Pinus* spp.) boards, 38 mm by 140 mm by 2.5 m from the same plantation were split into 5 groups for treatment with either chromated copper arsenate (CCA–C), alkaline copper quaternary – with a carbonate formulation (ACQ–D), micronized (<1000 nm) copper quaternary (MCQ), didecyl dimethyl ammonium carbonate (DDAC), or left untreated as a control. The newer, carbonate, formulations of ACQ–D use didecyl dimethyl ammonium carbonate instead of didecyl dimethyl ammonium chloride; it is believed that these formulations are less corrosive than the chloride formulations. Lumber treated with copper azole (CuAz) was also tested in this study, but the lumber came from a different plantation, and had starting dimensions of 38 mm by 89 mm by 1.2 m. The retention of the preservatives was verified using inductively coupled plasma atomic emission spectroscopy (ICP-AES). The ACQ and MCQ formulations tested in this study are similar in that they both contain the same nominal amount of copper and DDAC, however, they differ in how the copper was applied to the wood structure. ACQ is comprised of soluble copper in a waterborne solution whereas MCQ is comprised of sub-micron sized particles of insoluble copper compounds. More information on the differences between the preservatives and a comprehensive review of the biological and wood treatment aspects of these wood preservatives has recently been published by Freeman and McIntyre [16]. Details on the composition, specified, and actual treatment retentions for the preservatives used in this study are given in Table 1.

Two types of box nails used for siding (8d) were tested in this study, a plain carbon steel nail and a hot-dip galvanized nail. Both nails came from the same batch; the plain carbon steel nails were culled from the production line just prior to galvanizing. The nails had a nominal length of 64 mm and a nominal head diameter of 7 mm; the galvanized nails had a nominal shank diameter of 3 mm, with the carbon steel nails being slightly smaller. Each fastener was digitally imaged, the surface area for each fastener was determined from the digital image using an algorithm developed previously. Alloy compositions were analyzed with a Niton XL3[1] X-ray fluorescence (XRF) analyzer (Table 2). Galvanized coating thicknesses were measured on several fasteners with eddy-current thickness gages; thicknesses ranged between 10–25 μm.

Prior to exposure the lumber was sectioned into blocks 50 mm by 38 mm by 90 mm. A pilot hole (1.5 mm in diameter) was drilled into the centre of the 50 mm by 38 mm face of the block. These blocks were then allowed to equilibrate over several weeks in a room that was kept at 27 °C, 90% relative humidity (RH).

Immediately prior to exposure, the fasteners were cleaned, degreased, and weighed. The fasteners were cleaned in a bath with soap and water that was ultrasonically agitated for 5 min. Afterwards, the fasteners were rinsed and placed in a bath of distilled water that was ultrasonically agitated for 5 min. Finally, the fasteners were degreased with acetone and allowed to dry. Once dry, the fasteners were weighed to the nearest 0.1 mg and hand-driven through the pilot hole into a block of wood. The wood/fastener assemblies were then placed above a reservoir of high purity distilled water inside a sealed desiccator which was kept in a room held at 27 °C, 90% RH; the resulting environment inside the desiccator was 27 °C, 100% RH as dictated by the Gibbs phase rule. The average wood moisture content in this environment, determined gravimetrically from untreated specimens, was 24.6 ± 0.6% (standard deviation). The blocks were removed after 1 year of exposure (366 days) for specimens analyzed gravimetrically and after approximately 7 months for the qualitative specimens.

Fasteners were removed from the wood in a way that minimized damage to the corrosion products. Notches were sawn in the wood block, and the block was then placed into a vise. As pressure was applied, the wood split apart and the fastener could be removed by hand.

For the gravimetric specimens, the corrosion products were removed with a combination of ultrasonic agitation and chemical solvents. Fasteners were placed in a 50:50 (volume ratio) mixture of water and Evapo-Rust™ (Orison Marketing, Abilene TX, USA). Ultrasonic agitation was then applied for 60 min. The fasteners

[1] Trade name, Thermo Scientific, Billerica, MA, USA

Table 2
Elemental composition of the fasteners tested.

	Carbon steel		Hot-dip galvanized	
	wt (%)	±2σ	wt (%)	±2σ
Bi	0.004	0.001	1.67	0.060
Co	–	–	0.123	0.020
Cr	0.103	0.014	0.060	0.007
Cu	0.273	0.026	–	–
Fe	Balance		3.800	0.070
Mo	0.013	0.002	–	–
Mn	0.729	0.039	–	–
Ni	0.145	0.030	–	–
Pb	–	–	0.135	0.028
Si	0.162	0.078	–	–
Ti	–	–	0.012	0.004
Zn	0.035	0.010	Balance	
Zr	–	–	0.009	0.004

were then wiped with a lint-free paper towel and allowed to dry before being weighed to the nearest 0.1 mg. The same process and solvents were used on both the galvanized and steel fasteners; this process was able to successfully remove both red and white rusts. The amount of base metal removed by the cleaning processes, m_c, was measured by cleaning uncorroded specimens; m_c was -2.9 ± 1.4 (standard deviation) mg for the galvanized fasteners and -0.9 ± 0.9 mg for the steel fasteners.

To analyze the corrosion products, corroded fasteners were examined in a Zeiss (Oberkochen, Germany) EVO 40 scanning electron microscope (SEM) with a Thermo Noran Vantage (Waltham, MA, USA) energy dispersive X-ray spectroscopy (EDS) analyzer. The surfaces of the corroded fasteners were examined with SEM and EDS after being removed from the treated wood with no further cleaning. For cross sectional analysis, corroded fasteners were mounted in conductive phenolic resin, rough polished to 1200 grit, and final polished with a 1.0 μm alumina slurry.

X-ray powder diffraction was used to qualitatively identify phases in the corrosion products. Powder samples were prepared by removing the corrosion products with a razor blade. An Inel (Artenay, France) CPS 120 wide angle diffractometer with a Cu K-α source was used to collect the powder X-ray diffraction patterns.

3. Results

The corrosion rate (R in μm year^{-1}) was calculated by

$$R = K\frac{m_f - m_i + m_c}{A\rho(t_f - t_i)} \quad (1)$$

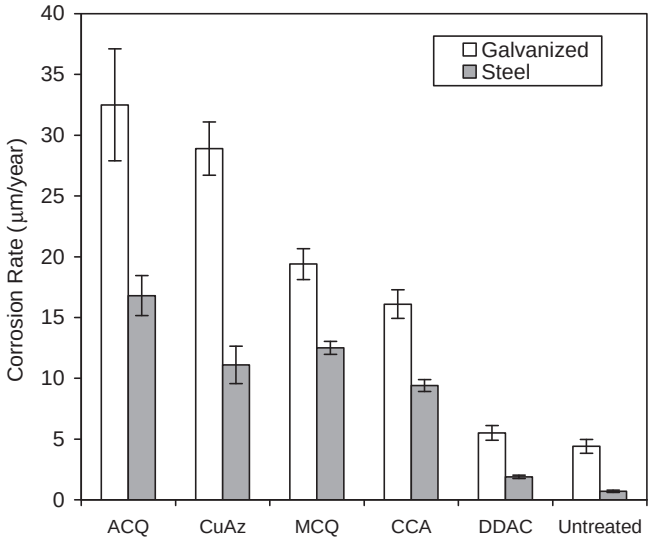

Fig. 1. Corrosion rate measured in the 1-year, 27 °C, 100% RH exposure test for hot-dip galvanized (white) and steel (gray) fasteners for different wood preservative treatments. Error bars represent the uncertainty in the mean (the standard error).

where m is the mass (g), t is the time (h), A is the surface area (cm^2) calculated from the digital image, ρ is the density as tabulated in ASTM G1[17], and K is a constant for unit analysis (87 600 000 μm cm^{-1} h year^{-1}). The subscripts i and f refer to initial, and final, respectively and m_c was defined earlier. The 1-year corrosion rate for hot-dip galvanized and steel fasteners is shown in Fig. 1.

The surfaces of the corrosion products on the nail head, which was exposed directly to the 27 °C, 100% RH environment as well as the nail shank, which was embedded in the wood were analyzed with SEM and EDS. The morphology and elemental composition of the corrosion products were similar for the head and shank and were also similar across preservative treatments for both the steel and galvanized nails. Characteristic SEM images of the corrosion products are given for steel and galvanized steel in Fig. 2. Cross sectional images for steel and galvanized steel are shown in Fig. 3 and Fig. 4, respectively. The micrographs in most cases represent the areas with the largest corrosion product. Pitting was observed in the cross sectional image of the steel fastener exposed to untreated wood.

The most important result from the EDS testing was that copper was not detected on any of the corroded fasteners. Carbon, iron, and oxygen were detected on all of the corroded steel fasteners.

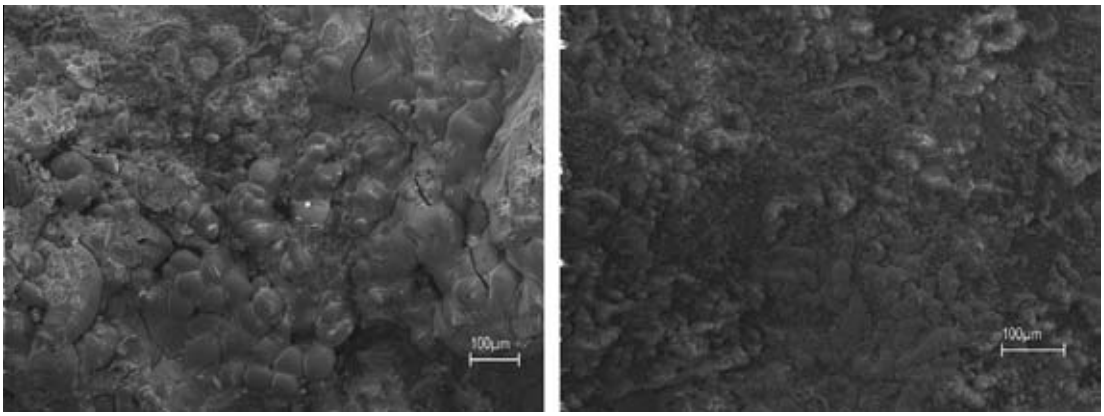

Fig. 2. Surface morphology of the corrosion products for steel (left) and galvanized steel (right) exposed to ACQ treated wood (27 °C, 100% RH for 7 months). Morphologies for other treatments were similar and had similar compositions. The scale marker represents 100 μm.

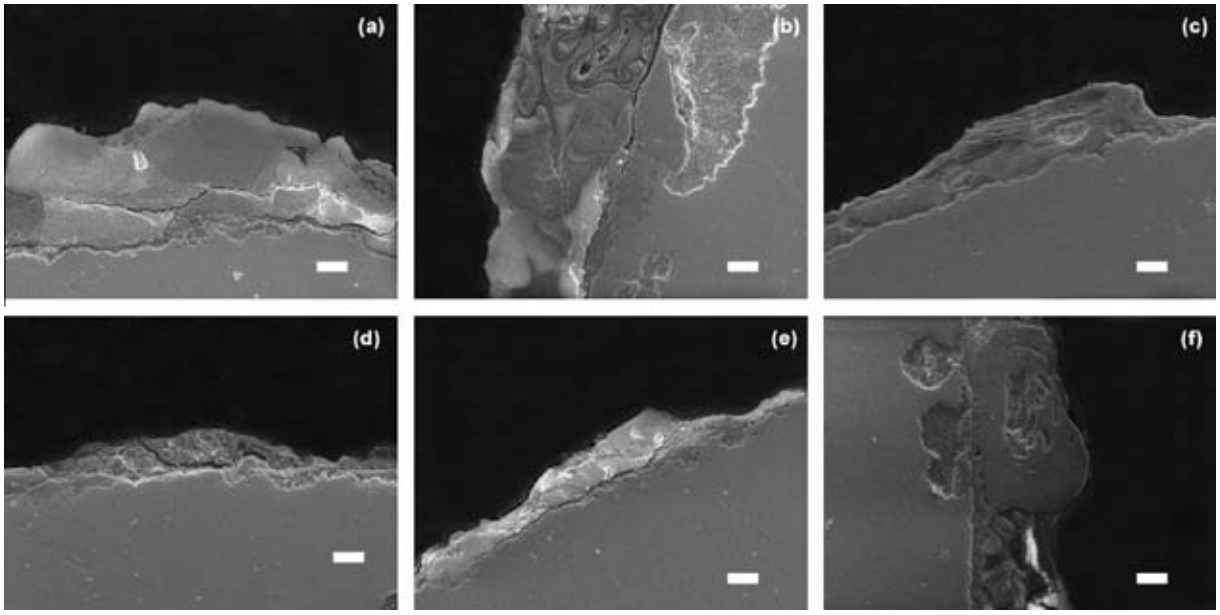

Fig. 3. Cross section SEM images of the steel fasteners exposed to ACQ (a) CuAz (b) MCQ (c) CCA (d) DDAC (e) and untreated (f) wood (27 °C, 100% RH for 7 months). The scale marker represents 20 μm.

Likewise, carbon, oxygen, and zinc were detected on all of the corroded galvanized fasteners. For galvanized fastener that had clearly visible red rust (ACQ and CuAz) iron was also detected in the EDS. Finally, arsenic was detected on both the steel and galvanized steel fastener exposed to CCA treated wood. The arsenic likely came from the arsenate in the preservative; it is believed that the arsenates may play a role as a corrosion inhibitor in the CCA preservative system [9].

For the steel nails, the predominant corrosion products across all treatments were goethite (α-FeOOH) and magnetite (Fe_3O_4). The galvanized corrosion products depended on the treatment to which the fasteners were exposed (Fig. 5). For fasteners in CuAz treated wood, namuwite $\{Zn_2(SO_4)(OH)_6{\cdot}4H_2O\}$, and hydrozincite $\{Zn_5(CO_3)_2(OH)_6\}$ were clearly identifiable in the XRD pattern, although it is possible that smithsonite ($ZnCO_3$) and simonkolleite $\{Zn_5(OH)_8C_{l2}{\cdot}(H_2O)\}$were also present. For the fasteners exposed to other treatments, namuwite and hydrozincite were the clearly identifiable phases, with possibly smithsonite and zincite (ZnO).

4. Discussion

It is instructive to compare ACQ and MCQ to DDAC, as these preservatives all contain the same organic biocide and differences in these treatments are due to the presence and type of copper used in the formulations. For both steel and galvanized steel, the DDAC treated wood was only slightly more corrosive than the untreated wood. This suggests that the corrosiveness of these wood preservatives comes from the copper rather than the organic biocides and is

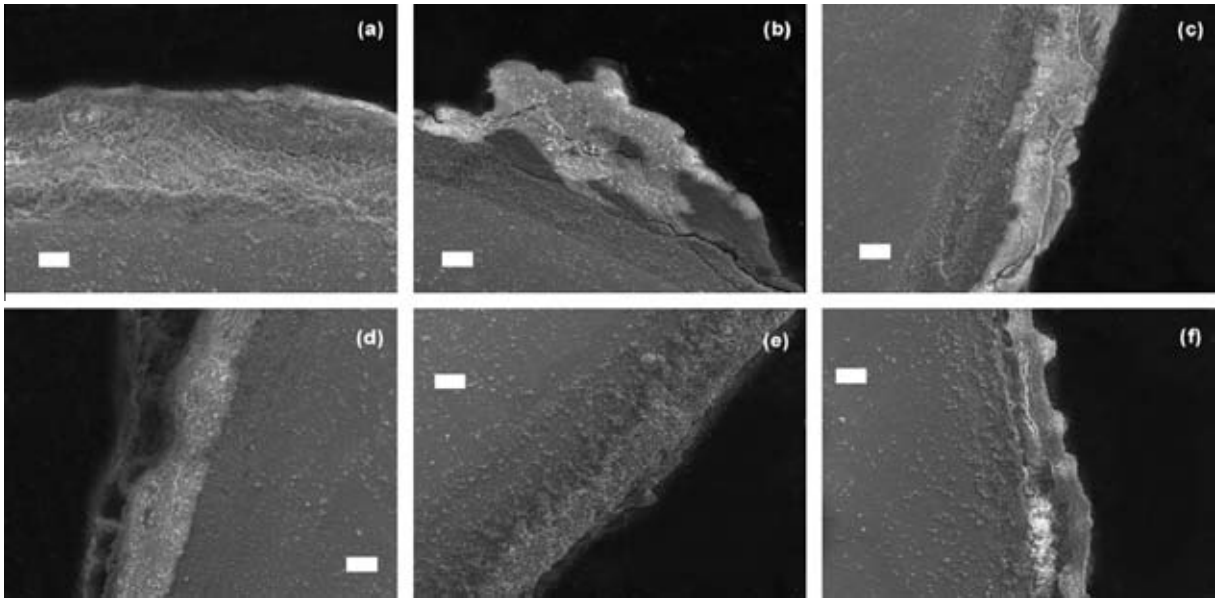

Fig. 4. Cross section SEM images of the galvanized fasteners exposed to ACQ (a) CuAz (b) MCQ (c) CCA (d) DDAC (e) and untreated (f) wood (27 °C, 100% RH for 7 months). The scale marker represents 20 μm.

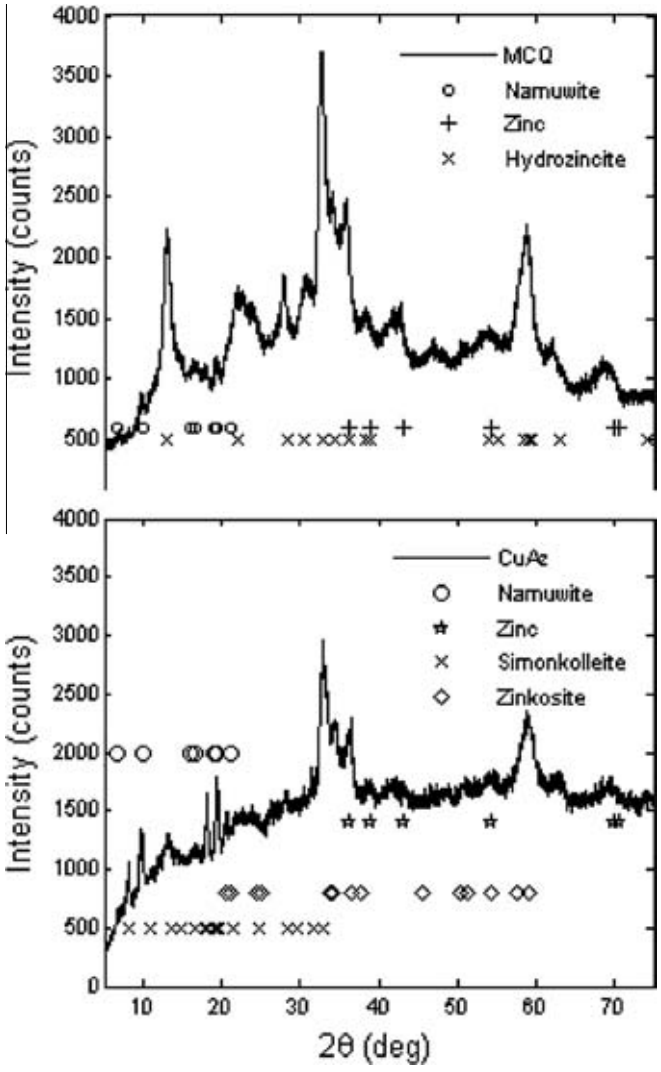

Fig. 5. X-ray powder diffraction pattern for galvanized fasteners exposed to MCQ treated wood (top) and CuAz treated wood (bottom). Specimens exposed to other treatments had similar diffraction patterns to MCQ.

congruent with previous theories [18] that the cathodic corrosion reaction is the reduction of cupric ions. The MCQ and ACQ both have nominally the same amount of copper. In ACQ, the treatment solution consisted of copper solubilised in an ammonia or ethanolamine, whereas the MCQ solution contains a suspension of insoluble sub-micron-sized copper compounds [19]. The fact that the MCQ treated wood is more corrosive than DDAC treated wood suggests that after treatment, some of the copper becomes soluble and participates in the corrosion reaction. ACQ treated wood was the most corrosive. It has been shown that the corrosion rate can depend on the amount of copper in the wood [12]. Even though in these tests the MCQ treated wood had more total copper than the ACQ treated wood, it is possible that the ACQ treated wood was more corrosive than the MCQ treated wood because there was more copper available to participate in the corrosion reaction. This comparison of the relative corrosiveness of MCQ and ACQ is limited to the specific conditions of this study, and does not necessarily reflect the relative corrosivity under a range of in-service exposure conditions.

The most important results from the SEM and EDS data is the absence of deposited copper in the corrosion products since the currently accepted mechanism of corrosion in treated wood [20] involves the reduction of cupric ions in the preservative. The absence of copper on the corroded fasteners could have two explanations: either cupric ions are not reduced as part of the corrosion reaction or cupric ions are reduced in the reaction but are not deposited on the surface. We believe, it is the latter because several key experiments [12,21,22] have shown the importance of cupric ions on wood/metal corrosion. For instance, Kear et al. [12] found a weak correlation between the corrosiveness of the wood preservative and the amount of copper in the preservative. When polarization measurements were made in liquid extracts of treated wood containing cupric ions, reduced copper was detected with EDS on the corroded fasteners [21]. Furthermore, the extract tests have good correlation to solid wood exposure when the preservatives contain cupric ions [22], but do not correlate for preservatives that do not contain cupric ions. The present work suggests that the mechanism of corrosion in both the extract and the solid wood is the reduction of cupric ions; however, this raises the question that if the cupric ions are being reduced, why are they not being deposited on the corroded surface. It is possible that the cupric ions are reduced near the metal surface, but instead of depositing on the fastener, the reduced copper remains in the wood.

In all cases, the galvanized fasteners had a higher corrosion rate than the steel fasteners. This has practical applications due to the widespread use of galvanized fasteners. Although zinc coatings cathodically protect the underlying steel at coating defects, the main protection mechanism of the zinc coatings is barrier protection due to the fact that in many environments zinc corrodes more slowly than steel [23]. In atmospheric corrosion, zinc usually forms hydrozincite and smithsonite which passivate the surface and cause the zinc to have a lower corrosion rate than steel. However, when exposed to certain conditions, such as air at 100% relative humidity [24], and in closed containers with wood vapours containing volatile acetic and formic acids [25], zinc corrodes more rapidly than steel. Several researchers have observed that zinc fasteners have corroded more rapidly than carbon steel fasteners in treated wood although the zinc corrosion products were not identified [4,26]. We found that, in addition to hydrozincite, namuwite also formed for fasteners embedded in treated wood. The presence of non-passivating compounds in the zinc corrosion products exposed to treated wood may explain why the galvanized fasteners corroded more rapidly than the steel fasteners.

Other work, using different exposure conditions, has found that galvanized fasteners corroded more slowly than steel fasteners in treated wood, although the corrosion products were not identified in that study [12]. It is possible that different corrosion products formed under the two, different conditions, which may explain the relative differences between steel and galvanized steel in these two studies. Comparing between these two studies, it seems that the type and morphology of the zinc corrosion products play a large role in the corrosion of metals in wood products. Furthermore, the corrosion products are sensitive to the test method. Future work should compare corrosion products from field exposure studies to those formed under different laboratory test protocols to optimize laboratory test methods. Until a better understanding of the relationship between laboratory and field measurements can be developed, caution should be used when extrapolating laboratory corrosion rates.

5. Conclusions

Corrosion rates were measured for steel and galvanized steel in southern pine treated with several wood preservatives. Corrosion rates over all treatments were small in absolute terms (1–35 μm year^{-1}). For each treatment, the steel fasteners had lower corrosion rates than the galvanized fasteners. The zinc corrosion products contained namuwite, and hydrozincite. The type and

morphology of the corrosion products may explain why zinc corrosion rates were greater than steel corrosion rates. Future work should focus on identifying the phases in the corrosion products for both field and laboratory measurements. Until more is understood about the type of corrosion products that form in wood used in in-service conditions, care should be taken in directly applying these results to construction practices.

Copper was not detected in any of the corrosion products. However, we believe that the corrosion mechanism in treated wood involves the reduction of cupric ions, but the reduced copper does not deposit on the corroding fastener.

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