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Electrochemical corrosion testing of fasteners in extracts of treated wood

Samuel L. Zelinka [a,*], Douglas R. Rammer [a], Donald S. Stone [b]

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

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Abstract

A recent change in wood preservatives has highlighted the need for a rapid, quantitative test to measure the corrosion rates of metals in contact with treated wood that could be used to evaluate new fasteners or new wood preservatives. A new method was developed where polarisation resistance tests were conducted on fasteners exposed to a water extract of wood treated with alkaline copper quaternary (ACQ). Good correlation was found between the corrosion rates using this new method and previous one year exposure data for carbon steel, hot-dip galvanized and electroplated galvanized fasteners. These data suggest that polarisation tests run in wood extract may be an effective, rapid test method to evaluate new fasteners or wood preservatives.
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1. Introduction

Waterborne preservatives are often used to extend the service life of wood used in exterior applications. These waterborne preservatives are usually comprised of metallic or organometallic salts, which protect the wood by inhibiting rot, decay, and attack by insects. However, waterborne preservatives have been shown to cause increased corrosion for metals embedded in or in contact with the treated wood [1] because the metallic ions in the preservative can act as an added oxidizer [2,3]. While the corrosiveness of waterborne preservatives has been documented in the literature for well over 80 years [4], it has not generally been regarded as a problem because chromated copper arsenate (CCA) [5], the major wood preservative of the past 50 years, did not greatly accelerate corrosion of metals in contact with wood [2].

With the voluntary withdrawal of wood treated with CCA for use in residential construction in January 2004, the corrosiveness of treated wood has become a concern. Preliminary research [3,6] has shown CCA replacements, such as Copper Azole (CuAz) [5] and alkaline copper quaternary (ACQ) [5], are more corrosive than CCA. However, quantifying the corrosion rate of metals in contact with treated wood presents many challenges [7] due to the complexity and inhomogeneity of treated wood. A rapid test method which would enable quantification of the performance of metals in contact with treated wood would be of great value in the wood industry.

The mechanism of corrosion of metals in contact with treated wood is not well understood. The corrosiveness of CCA-treated wood has generally been attributed to chemically active cupric ions in the wood preservative [8]. However, Simm and Button [9], who examined fasteners exposed to CCA-treated wood with energy dispersive X-ray spectroscopy (EDX), found no copper on the fasteners, which suggests that the reduction of copper was not the cathodic reaction. Additionally, if corrosion were due solely to the cupric ions in the preservative, the corrosion

* Corresponding author. Tel.: +1 608 231 9277; fax: +1 608 231 9508.
E-mail address: szelinka@fs.fed.us (S.L. Zelinka).

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of metals in contact with the preservative in a virgin state (without coming in contact with wood) would be higher than corrosion of metals in treated wood because none of the cupric ions would have reacted with the wood. However, the corrosion rate of metals in contact with wood preservatives was found to be much lower than the corrosion rates of metal in contact with treated wood [10]. It is likely preservative acts synergistically with chemicals in the wood to create a unique corrosive environment.

Despite a fundamental lack of understanding of corrosion of metals in wood, there have been several attempts to create a rapid, quantitative electrochemical test to measure the corrosion of metals in contact with wood [10–13]. Several of these tests [11–13] involve placing an electrode in the treated wood and running either a polarisation resistance or a full polarisation test. These tests are complicated by the high electrolyte resistance of wood [11] and require intricate, time consuming machining for each specimen. Additional complications are present with the inherent inhomogeneities in wood, such as moisture content gradients or the uniformity of the preservative treatment. Therefore, a different method [10] was attempted by the authors where by direct current electrochemical tests were conducted in dilute solutions of the wood preservatives, where the concentration was adjusted to match the concentration of preservatives in treated wood. It was found that the measured corrosion rates from dilute preservative solutions are orders of magnitude lower than corrosion rates found from exposure tests in treated wood.

It may not be surprising, *a posteriori*, that the corrosion rates from tests run in solutions of the wood preservatives do not match corrosion rates from exposure tests in treated wood. Direct exposure to the (as produced) wood preservative as a model for exposure for treated wood is based on several assumptions: (1) the wood preservative does not react with the wood, or this reaction does not affect the corrosiveness of the preservative, (2) corrosion of metals embedded in wood is caused by aqueous components of the free water in the wood cell wall, (3) the chemical constituents of wood are not corrosive, or at least much less corrosive than the wood preservatives. It was known *a priori* that assumption (3) is poor because corrosion rates of metals in contact with untreated wood are finite [1], and the organic extractives in wood pulp have an effect on corrosion [14]. However, approximation (2) is most likely valid, as this mechanism of corrosion is mentioned in the literature [8] and the corrosion rates of metals in contact with wood drop sharply when the wood moisture content is below fibre saturation where there is no free water in the wood [11]. The original experiment [10] revealed that assumption (1) is also poor, and that corrosion rates in dilute solutions of preservative-treated wood are not correlated with corrosion rates of metals in treated wood.

Because of the poor correlation between tests run in dilute solutions of wood preservatives and exposure tests, we decided to modify the original test method by changing the solution to make it more representative of the treated wood environment. We modified the original test by creating an extract of treated wood by placing the sawdust of treated wood in contact with water and running the corrosion test in this extract. Thus the extract is comprised of the water soluble components of treated wood. If corrosion of metals embedded in wood is truly an aqueous mechanism, it is likely that this extract contains nearly the same corrosive components as the free water within the cell walls, and this should give better correlation than the original test in a dilute preservative solution.

This work reports on polarisation resistance tests run in an extract of ACQ treated wood, and its correlation to exposure tests [3]. The objective of our work is to prove that there is a correlation between exposure test data and electrochemical tests run in the extract derived from treated wood. Closer examinations of the chemical composition of this extract and the mechanism of the corrosion reaction in the extract, as well as extracts made from other preservatives, are currently being examined by the authors.

2. Materials and methods

The treated wood extract corrosion test was designed to be both simple and easily scalable. In short, the procedure consisted of collecting wood sawdust from ACQ treated Southern Pine (*Pinus* spp.), placing this sawdust in contact with high purity distilled water for a specified length of time at room temperature, and filtering off the sawdust at the end.

2.1. Extraction techniques

At the beginning of the study, different extraction techniques were examined to see which techniques worked best. The effect of the sawdust to water ratio and length of extraction time were examined by running a polarisation resistance test in the solution. The polarisation resistance results were then compared with the results of an exposure test [3], which had been conducted on fasteners embedded in wood exposed to 100% relative humidity at 26.7 °C. Most of the major exposure tests of fasteners exposed to treated wood have been run under these conditions [1,2,9]. And while a full chemical analysis of the extracts would have been prohibitively expensive at this early stage of development, the extract was partially quantified by measuring the concentration of copper using an inductively coupled plasma mass spectrometer (ICP). The pH of the extract was also measured.

From these preliminary tests, the conditions chosen for extraction were a ratio of 1:10 sawdust to water (weight basis) and an extraction time of one week. One week was chosen for the extraction because the concentration of copper in the extract remained constant at times greater than one week, which suggested that most of the available copper had been extracted. Although the components of the extract may have leached at different rates into the extract, copper was chosen as a basis because it was believed to

have the largest effect on corrosion rate [8]. Additionally, the results of polarisation resistance tests, in the one week extract, agreed with the exposure test data.

It was brought to our attention that the high temperatures associated with sawing the lumber may chemically alter the wood or the preservative and therefore change the corrosiveness of the extract. To test this, small wood chips were created with a hand planer. There was no measurable difference in copper concentration or polarisation resistance between the planed wood and the sawdust extracts, so the sawdust method was used on the remainder of the tests because it was less labour intensive.

There were problems with mould growing in the original extract, which was created from the same lumber as the original exposure study [3]. Mould, even when filtered off before the corrosion tests, seemed to have a large effect on measured polarisation resistance, which we attributed to consumption of oxygen and/or other organic compounds by the mould within the solution. A fresh solution was created using material from the same commercial wood supplier and same treatment retention as the exposure study reported previously [3]. To prevent mould, the solutions were stored at low temperatures (1 °C) and rapidly brought to room temperature using microwave radiation prior to testing. It was assumed that microwave heating did not alter the chemistry of the solution. If mould was observed in any container of extract, that extract was thrown out and not tested. To reduce variability, the extract was made as one large batch in a 55 gallon drum and transferred to smaller containers before storage and testing. The copper concentration of the extract used for all tests was 19.85 mg L^{-1}; the extract pH was 6.6.

2.2. Metals tested

Four different types of fasteners were tested: a bright carbon steel 8d nail, an 8d hot-dip zinc galvanized carbon nail, a 4d aluminium alloy (UNS AA5056) nail, and a 64 mm long electroplated zinc galvanized screw. These fasteners were the same make and from the same company as the fasteners used in the exposure tests [3]. The coating thickness on the electroplated screw was 8 μm and the coating thickness of the hot-dip galvanized fastener was 66 μm. The compositions of the different galvanized coatings are listed in Table 1. To fit in the standard test flask [15], the fasteners were machined to a length of 41 mm, after which a hole was drilled and tapped so that the fastener could be incorporated with the same gasket system used in the ASTM standard test [15]. The bare metal exposed from machining the top of the fastener was covered by a gasket during testing. During fabrication, fasteners were held with a specially designed polytetrafluoroethylene fixture to minimize surface damage. Prior to polarisation resistance testing, the fasteners were imaged with a high resolution camera, and the surface areas were calculated by an algorithm developed by the authors [16].

Table 1
Composition of coatings of the two types of galvanized fasteners tested

	Hot-dip (wt%)	Electroplated (wt%)
Aluminium	0.003	0.37
Bismuth	0.0009	–
Boron	–	0.015
Calcium	–	0.031
Chromium	–	0.074
Copper	–	0.016
Iron	0.14	–
Magnesium	0.001	0.008
Manganese	0.002	–
Silicon	0.014	–
Zinc	99.8	99.3

2.3. Test conditions

In the original electrochemical experiments [10], we decided to make the tests as repeatable as possible by deaerating the solution, using standard metals, and giving the specimens a standard surface finish and geometry as defined in ASTM G-59 [15]. Because of the poor correlation to exposure tests in simple dilute preservative solutions, we decided to run the electrochemical test on real unpolished fasteners, from the same batch that were used for the exposure tests. This would allow for direct comparison of exposure and electrochemical results.

However, the amount of dissolved oxygen that should be supplied in these aqueous tests is a more difficult question because the amount of oxygen available for corrosion in the cellular structure of wood is unknown, and would depend on the depth of penetration of the fastener [9]. It was decided to neither aerate or deaerate the solutions in the majority of tests because in real exposures, there is no source of oxygen inside of the wood. Additionally, the solution was not stirred because in preliminary tests where stirring was attempted with a magnetic stir bar, the stir bar would strike the fastener which resulted in odd polarisation data.

Because the partial pressure of oxygen was not fixed by a gas purge or stirring, the amount of dissolved oxygen in the extract in the local vicinity of the fastener may change (due to the corrosion reaction) during the polarisation tests. To examine this issue, additional tests were run on carbon steel nails to investigate the effect of dissolved oxygen. In these additional tests, the solution was bubbled at a flow rate of 120 cc/min with either nitrogen (to deaerate) or compressed air (to aerate) for 60 min prior to testing. This bubbling was continued throughout the test to maintain a constant level of dissolved oxygen near the fastener. Five replicates were run for each condition.

2.4. Procedure

Immediately prior to electrochemical testing the fasteners were cleaned using the same three-step process used in the exposure test [3]. The fasteners were first placed in an ultrasonic cleaner with a soap solution for 5 min. The

fasteners were then rinsed under flowing high purity distilled water (18 MΩ) before being placed in a high purity distilled water bath that was ultrasonically agitated for 5 min. The fasteners were then rinsed with acetone and rinsed again with high purity distilled water.

The experimental parameters of the polarisation resistance test are closely related to ASTM G-59 [15]. After the metal was immersed in the extract solution, the open circuit potential was measured for 60 min. At the end of that period, the potential was increased at a constant scan rate of 0.166 mV/s over a range of 60 mV starting 30 mV below the open circuit potential. The test was then stopped. The potential was measured against a saturated calomel electrode (SCE) with a Gamry PC14-300 potentiostat (Gamry Instruments, Warminster, Pennsylvania). Ten replicates were run for each metal.

The Tafel slopes and corrosion rates were calculated for each polarisation resistance curve using Mansfeld's method [17,18], which was implemented using a non-linear least squares routine. To confirm the Tafel slopes, large perturbation polarisation scans were run in the potentiodynamic mode at the same scan rate as for the polarisation resistance experiments, 0.166 mV/s. The open circuit potential was measured 60 min after immersion, after which, the potential was increased at a constant scan rate of 0.166 mV/s over a range of 500 mV starting 250 mV below the open circuit potential.

3. Results

The results of polarisation resistance testing in the extract of ACQ treated wood are summarized in Fig. 1, which contains the average corrosion rate for each metal, where the error bars represent the uncertainty in the mean (the standard error). Additionally, data from a previous study [3], a one year exposure test in ACQ treated wood at 27 °C, 100% relative humidity are included for comparison. The corrosion rate of carbon steel in untreated wood from Baechler's [19] 20 year exposure study is included as a baseline.

Representative large perturbation polarisation curves are shown in Fig. 2 for each fastener. Neither the aluminium nail nor the electroplated screw exhibited obvious Tafel behaviour in any of the replicates. On the other hand, both the hot-dip galvanized and carbon steel nails exhibited Tafel behaviour on the anodic portion of the curve. While the cathodic portion of these curves also exhibits linear behaviour, the magnitudes of these slopes are very high, which suggests that the reaction is exhibiting concentration polarisation at these large perturbations and not true Tafel behaviour [20]. The average Tafel slopes, calculated by Mansfeld's method [17,18], along with those calculated from the polarisation curves, are presented in Table 2 along with the coefficient of variation (the ratio of the standard deviation to the mean). These two methods give similar results for the hot-dip galvanized fasteners, but different results for the carbon steel nail.

Another noticeable feature of Fig. 2 is the aluminium fastener appears more noble than the carbon steel fastener, which is in contrast to what we would anticipate for the galvanic series based on seawater [21]. Additionally, for all aluminium fasteners tested, the asymptotic limit of the large perturbation polarisation curve was more noble than the open circuit potential measured 60 min after the start of the test (Fig. 3a). We believe these features of the aluminium data are due to a passive layer and a reduction of cupric ions, respectively, and will return to the subject in the Discussion.

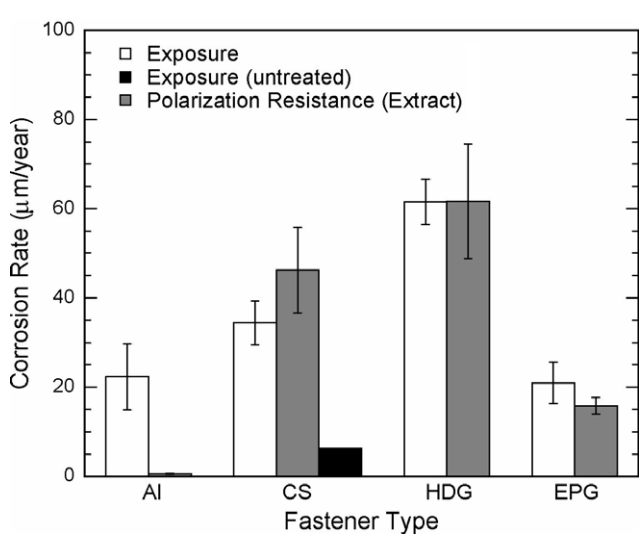

Fig. 1. Corrosion rates calculated from polarisation resistance measurements in an extract of ACQ treated wood (grey) for an aluminium nail (Al), carbon steel nail (CS), hot-dip galvanized nail (HDG) and an electroplated galvanized screw (EPG). For reference, Corrosion rates from exposure tests run in ACQ at 27 °C, 100% relative humidity environment (white) [3] and exposure tests run in untreated wood at 27 °C, 100% relative humidity [1] are shown for reference. The error bars represent the uncertainty in the mean.

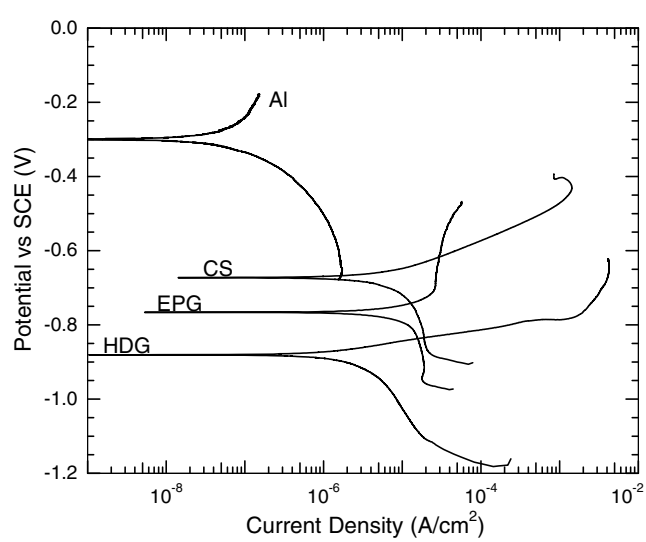

Fig. 2. Representative large perturbation polarisation curves, measured with respect to the saturated calomel electrode (SCE) for the fasteners tested: aluminium nail (Al), carbon steel nail (CS), hot-dip galvanized nail (HDG) and an electroplated galvanized screw (EPG).

Table 2
Tafel slopes (in mV per decade) calculated from polarisation resistance and large perturbation polarisation

	Polarisation resistance		Large perturbation	
	β_a	β_c	β_a	β_c
Aluminium	42 (4)	50 (5)	–	–
Carbon steel	47 (1)	67 (5)	83	–
Hot-dip galvanized fasteners	37 (5)	86 (25)	34	–
Electroplated galvanized fasteners	24 (3)	36 (3)	–	–

The standard error is shown in parenthesis.

For the carbon steel fasteners, an attempt was made to quantify the changes in the solution during testing by measuring the pH as well as the concentration of iron and copper. We found that within experimental error, for the polarisation resistance tests, there was no change in copper, iron or pH. For the large perturbation tests, on average there was a detectable increase (0.3 mg/L) in iron after polarisation. There was no measurable change in copper concentration or pH in these tests.

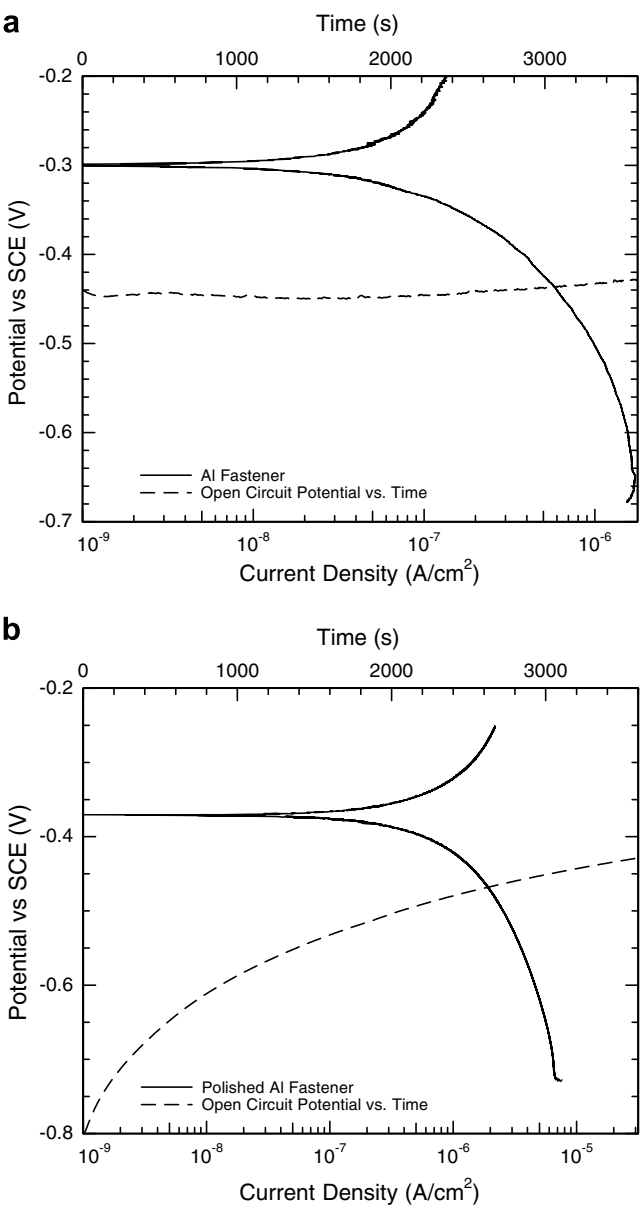

Fig. 3. Large perturbation polarisation curve for aluminium with the open circuit potential as a function of time overlaid.

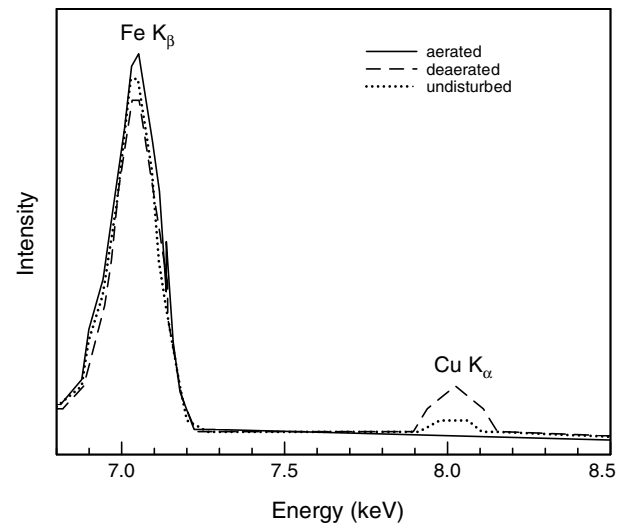

Fig. 4. EDX spectra confirming the presence of copper on the deaerated and undisturbed carbon steel nails.

The data from the additional tests, in which the solution was bubbled with gas, contained much more noise than the test with no bubbling, which we attribute to a high voltage piece of equipment with a xenon lamp being moved into the lab near the potentiostat. An accurate calculation of Tafel slopes from these data was not possible due to noise, although the polarisation resistance could still be calculated by measuring a tangent at the origin. The mean polarisation resistance (standard error), in units of kΩ cm^2, was 2.0 (0.4) for the aerated specimens, 3.2 (0.2) for the deaerated specimens, and 3.1 (0.3) for specimens that were neither aerated or deaerated (i.e. the main tests).

The nails polarized in deaerated solutions all had reddish brown colouring, which suggested they may have been plated with copper from the wood preservative. To test this, representative fasteners were examined with EDX (Fig. 4). The EDX detected copper on the fastener polarized in the deaerated solution, whereas no copper was detected on the fastener polarized in the aerated solution. Copper was also detected for the fastener polarized in the undisturbed solution, even though it was less obvious with the naked eye.

4. Discussion

The purpose of the experiment was to determine whether or not a relationship exists between polarisation resistance scans of fasteners in an extract of treated wood and exposure tests of fasteners embedded in the wood.

The extract likely contains many types of ionic species, most of which are unknown leachates from the wood. Because these ionic species could lead to different anodic and cathodic reactions occurring in solution at the same time, they might interfere with the corrosion rate measurement. However, the polarisation resistance method should work if the corrosion reaction is dominant [22] and if, at least in the local region around i_{corr}, the reaction can be described by the Stern-Geary equation [23].

There is good correlation, Fig. 1, between polarisation resistance tests and exposure tests for the carbon steel nail, hot-dip galvanized nail, and the electroplated screw, but poor correlation for the aluminium nail. For the steel and galvanized metals, it seems plausible that the corrosion reaction dominates, so that the polarisation resistance method gives the correct result.

In contrast, the large perturbation polarisation tests show that away from i_{corr} the data do not exhibit clear Tafel behaviour in the majority of cases. The cathodic data exhibit concentration polarisation due to the lack of stirring or gas purge. For the two anodic reactions that exhibit linearity, only the hot-dip galvanized nail had a Tafel slope that matches the one calculated from the polarisation resistance data. The most likely reason that the apparent Tafel slope for the carbon steel differs from that calculated in the vicinity of i_{corr} is that the linear region is the sum of two reactions, in which case if we knew the total composition of the extract it may be possible to deconvolute these two to give the true Tafel slope [20].

One noticeable feature of Fig. 1 is that the error bars are large, especially for the hot-dip galvanized and carbon steel fasteners. For comparison, in the previous study [10], where all metals were polished to a standard finish, and run in deaerated solutions, the coefficient of variation (the ratio of the standard deviation to the mean) on ten replicates of UNS G10180 (SAE 1018 steel) was found to be about 30%. The coefficients of variation in the current study ranged from 33% for the electroplated galvanized screw to 62% for the carbon steel nail. It is likely that a portion of this increased variance is due to variations in surface finish among fasteners because the coefficient of variation is highest for those fasteners which appeared to the naked eye to have the largest variability in surface finishes.

There are several incongruities with the aluminium fastener. Not only is the measured corrosion rate of the aluminium fastener lower in the extract than in the treated wood, but the open circuit potential is more noble than expected and changes after cathodic polarisation. To examine the effect of a possible passive layer, an aluminium fastener was polished to 600 grit immediately prior to polarisation (Fig. 3b). The OCP of the polished fastener was much lower than the unpolished fasteners, but increased with time, eventually approaching the OCP of the unpolished fastener, which suggests that the aluminium fasteners did contain a passive layer. However, like the unpolished fasteners, the asymptotic limit of the polished polarisation curve was higher than OCP measured prior to polarisation.

We believe that the asymptotic limit of the polarisation curve is higher than the OCP due to the cupric ions in the extract. Once the cathodic polarisation began, cupric ions would be reduced and increase the open circuit potential of the fastener. In fact, the open circuit potential measured from the asymptotic limit of the polarisation curve is about −0.3 V with respect to the SCE, which agrees with the open circuit potential measured for copper in seawater [21]. For the aluminium fastener, it is plausible that we were essentially measuring the corrosion rate of copper, which explains why the measured corrosion rates in the extract did not match the exposure data.

From the additional experiments, it is clear that dissolved oxygen affects the cathodic reaction in wood extracts. In the deaerated solution, the predominant cathodic reaction appears to be the reduction of the cupric ions in solution, as evidenced by the deposition of copper on the fasteners. This deposition was not observed on fasteners polarized in the aerated solution, which suggests that there was a different cathodic reaction, possibly the reduction of dissolved oxygen in the solution.

Both the polarisation resistance and EDX data suggest that the fasteners polarized in undisturbed solution exhibit similar characteristics to the fasteners polarized in fully deaerated solutions. Even though the bulk solution contained oxygen, it acted as a deaerated environment because the oxygen near the fastener was consumed very quickly, and the reduction of cupric ions occurred faster than the diffusion of new oxygen to the fastener surface. It is likely that the rapid consumption of available oxygen occurs in solid wood where the diffusion of oxygen to the fastener surface is affected by the cellular structure of wood. To confirm this mechanism, the surfaces of fasteners in future exposure tests with ACQ treated wood should be examined for the presence of copper.

The additional tests were run approximately six months after the main tests were run. Along with the fasteners polarized in aerated or deaerated solutions, baseline fasteners in undisturbed solutions were also run. These baseline fasteners were run in exactly the same manner as the fasteners whose data appear in Fig. 1. The mean polarisation resistance (standard error), in units of $k\Omega\ cm^2$, was 2.0 (0.4) for the specimens run with the additional tests, whereas it was 4.2 (0.9) for the tests run six months earlier. Since the tests were identical, except that the extract had aged for six months in the case of the additional tests, we believe that the extract does change over large periods of time even when stored at 1 °C.

5. Conclusions

This paper presents a method for running polarisation resistance tests in water extracts of ACQ treated wood with actual fasteners. Copper plating on the carbon steel fasteners suggests that the major cathodic reaction in the extract

was the reduction of cupric ions from the wood preservative.

The method is shown to have good correlation with a constant environment (26.7 °C, 100%RH) exposure test for carbon steel and two types of galvanized fasteners. Because of this correlation, it may be possible to rapidly evaluate new steel and galvanized steel fasteners using electrochemical methods in wood extract instead of relying on long term exposure tests to qualify new products for use in construction. To be used as a screening test, the methodology should be evaluated with different wood preservatives and metals. Since many fasteners have non-metallic coatings, it may be necessary to use impedance spectroscopy to evaluate their performance.

In addition to being used as a screening test, this methodology could be used to better understand the corrosion of metals in contact with treated wood. Once the chemicals in the extract are quantified, the corrosion effects of individual chemicals could be systematically altered to investigate possible synergies and better understand the corrosion reactions between wood preservatives and metallic fasteners.

At this point in time, only ACQ treated wood has been tested using polarisation resistance on wood extracts. The next step in understanding the relationship between corrosion in wood extracts and corrosion in solid wood is to use these methods to examine untreated wood as well as wood treated with different chemicals. For example, the new micronized copper quaternary (MCQ) is chemically similar to ACQ but the copper in MCQ is insoluble. Therefore it is likely that fasteners in MCQ extracts would have a different corrosion mechanism than fasteners in ACQ extracts since we believe that the reduction of cupric ions contributed to the cathodic reaction in ACQ extracts. While the extract test method appears promising, it should not be used to evaluate different wood preservatives until the extract method is proven to work for preservatives with a different corrosion mechanism.

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