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Direct current testing to measure corrosiveness of wood preservatives ☆

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Abstract

A qualitative test that mimics the corrosion behaviour of metals in contact with treated wood without using wood specimens would be of great value in rapidly evaluating the corrosiveness of new wood preservatives. The objective of this study was to determine whether the linear polarisation resistance of metals immersed in a solution of preservative chemicals is related to corrosion of metals in wood. This technique was used to measure the corrosion rate of four types of metals in three different aqueous solutions of wood preservatives. The four metals were UNS G10180 (SAE 1018 steel), UNS S30400 (AISI 304 stainless steel), UNS S43000 (AISI 430 stainless steel), and UNS Z15001 (zinc). The metals were subjected to various concentrations of alkaline copper quat (ACQ-D), ammoniacal copper citrate (CC), and chromated copper arsenate type C (CCA-C). It was found that the corrosion of metals in the solutions of the wood preservatives did not correlate well to what is known about the corrosion of metals in contact with wood.
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1. Introduction

Waterborne preservatives, such as chromated copper arsenate (CCA) and ammoniacal copper zinc arsenate (ACZA), have been used to treat bridges, wood foundations, and other exposed wood products to extend their service life. While some of the preservative bonds to the wood and becomes insoluble, a small percentage remains in soluble ionic form in the wood. These ionic components help to protect the wood; however, they also increase the corrosiveness of the wood environment, especially if the wood has not been given ample time to fully fixate before being put into service.

As a result of the voluntary withdrawal of CCA from residential use, many designers are now choosing to use alternative preservatives such as alkaline copper quat (ACQ) and copper azole (CA). Little has been published on the effect of these ammonia- and amine-based preservatives on the corrosion rate, although ACQ and other new preservatives are believed to be much more corrosive than CCA.

Electrochemical test methods that measure the corrosiveness of treated wood show great promise in their ability to rapidly evaluate the corrosion of metals in contact with wood [1,2]. Electrochemical tests have numerous advantages over metallic weight loss methods because they can be conducted at any temperature or wood moisture content. The linear polarisation resistance test method (LPR) has been used successfully to measure the corrosiveness of CCA treatments by embedding the electrodes in treated wood [3–5]. However, LPR tests run in treated wood are complicated by the inhomogeneities in the wood, such as gradients in moisture content or gradients in the level of preservative treatment. In addition to its non-uniform structure, wood has a high electrolyte resistance that adds additional complications to LPR testing [3]. Finally, the wood specimens used in LPR tests require intricate, time consuming machining for each test replicate. If a qualitative test could be developed that mimicked the corrosion behaviour of metals in contact with treated wood without using wood, it then could be used as a rapid means to evaluate the corrosiveness of new wood preservatives before they are brought to market. Such a methodology may also be useful in evaluating the corrosion resistance of new fastener materials or coatings to wood preservatives.

2. Objective

The objective of this study was to determine whether the linear polarisation resistance of metals immersed in a solution of preservative chemicals is related to corrosion of metals in wood. LPR tests that are run in solution could then be used early in the development stage of new wood preservatives. The solutions of preservatives were not expected to perfectly simulate the intricate treated wood environment; however, the experiment was conducted to examine whether this environment would correlate to what is already known about corrosion of metals in treated wood. The solutions of wood preservatives were chosen because the preservatives are already manufactured in large quantities, commercially available, and standardized by the American Wood Preservers' Association (AWPA). This experiment was a first attempt at applying the LPR method using a solution to assess the corrosion behaviour of metals in contact with a variety of preservative treatments. As such, many experimental variables were held constant to ensure the virility of the test method.

3. Experimental

Linear polarization resistance measurements were run on metals immersed in an aqueous solution of preservative chemicals. The aqueous solutions were made to mimic the preservative loading applied to wood used in timber bridges. Several wood preservatives were used and a small subset of structural metal alloys was tested in each solution.

Three wood preservatives were tested, each at three different solution concentrations. Four different metals were tested for each combination of preservative solution and concentration, for a total of 36 tests. Only one replicate was tested for each combination of preservative, concentration, and metal. In addition, ancillary tests were conducted to measure variability, measure the Tafel slopes, and partially determine the effect of pH.

3.1. Metals

The four metals tested were UNS G10180 (SAE 1018 steel), UNS S30400 (AISI 304 stainless steel), UNS S43000 (AISI 430 stainless steel) and UNS Z15001 (zinc). Galvanized steel is currently specified for use with preservative treated wood [6]. While each fastener manufacturer may have a different galvanizing process, zinc is the major component of these galvanized coatings. Therefore, zinc metal was chosen to estimate the corrosion performance of a galvanized fastener. Type 304 stainless steel was chosen because it is the least costly stainless steel alloy available for construction and would be the most likely replacement if galvanized steel were found to not withstand preservative treatment for wood. Type 430 stainless steel was chosen as a control because several standards for measuring the corrosiveness of solutions specify the use of this metal [7,8].

3.2. Preservatives

The wood preservatives tested were alkaline copper quat (ACQ-D), ammoniacal copper citrate (CC), and the oxide formulation of chromated copper arsenate (CCA-C) [9].

The aqueous solutions were chosen to correspond to three retention levels of preservative in wood. The concentration of the weakest solution was 4 g/kg, corresponding to the lowest retention level specified for wood not in contact with ground or water according to AWPA use category UC3-B [10]. The strongest solution was 40 g/kg, corresponding to the highest retention level for solid sawn wood in contact with saltwater, as specified by AWPA use category UC5-A,B,C [10]. The concentration of the third solution was between the two extremes: 12.8 g/kg. This solution does not correspond to any AWPA standard retention level. While the AWPA standard [10] specifies retention on a per-volume basis, the solutions were made on a per-weight basis in the lab. These two solutions should be nearly equivalent if the density of the solvent (water) is close to unity.

Because the amount of CuO in these wood preservatives is allowed by the manufacturing standard [9] to vary as much as 9%, the dilutions used in this experiment were made in large quantities and distributed equally among the test matrix. The solutions were used immediately; each solution was used for only one test to prevent cross-contamination between tests.

Small portions of the solutions were set aside for chemical analysis before corrosion testing. The results of chemical analysis are shown in Table 1. The large variance in concentration between the ACQ-D solution used for the variability experiments and the ACQ-D solution used for the main set of experiments was due to variation in the stock solution of ACQ-D.

Before running the polarisation test, oxygen was removed by bubbling nitrogen through the solution for an hour. Oxygen in solution can contribute additional cathodic reactions that would need to be accounted for. The removal of oxygen from the solution has been documented for electrochemical corrosion testing [7,8,11]. While oxygen is available for corrosion reactions in preservative treated wood, the concentration of oxygen in wood is not well documented and is believed to vary along the depth of the fastener [12,13]. To eliminate this unknown, it was decided to deoxygenate this solution for these tests.

The flow rate of nitrogen varied from 120 to 160 cc/min depending on the solution. A low flow rate (120 cc/min) was used for the ACQ-D solution because of its low surface tension; bubbles formed in the solution and created foam on the top of the beaker. In all cases, the nitrogen purge was continued throughout testing.

3.3. *Procedure*

Potentiodynamic polarisation resistance testing was chosen because it is repeatable [11,14], the data analysis is straightforward, and there is an established standard [8].

The experimental parameters of the test are closely related to ASTM G-59 [6]. The metal to be tested was placed into the degassed solution, and the open circuit potential (corrosion potential) was measured after 55 min. The potential was then held at 30 mV

Table 1
Results of chemical analysis

Preservative and concentration[a]	Metal oxide concentration (wt%)			Solution pH
	As_2O_5	CrO_3	CuO	
CCA-C				
Low	0.148	0.200	0.080	2
Medium	0.467	0.641	0.247	1
High	1.481	2.075	0.786	0.5
ACQ-D				
Low	–	–	0.230	9.5
Medium	–	–	0.716	9.5
High	–	–	2.888	9.5
CC				
Low	–	–	0.167	9.5
Medium	–	–	0.526	9.5
High	–	–	1.372	9.5
ACQ-D				
Medium[b]	–	–	0.926	9.5

[a] Low = 4 g/L, medium = 12.8 g/L, and high = 40 g/L.

[b] Variability tests.

below the open circuit potential for 5 min. At the end of that period, the potential was ramped up at a constant scan rate of 0.166 mV/s until a potential 30 mV greater than the open circuit potential was achieved. 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) as specified in ASTM G-59 [8].

Because the preservative solutions were opaque, a spacer was created so that the reference electrode was placed a constant distance from the surface of the working electrode for all tests.

The working electrodes (SAE 1018, AISI 304, and AISI 430 steel) were made from purchased 9.5-mm (3/8-in.) rods that were tapped on the top and fitted against a polytetrafluoroethylene (PTFE) gasket, as outlined in ASTM G-5 [7]. The zinc electrodes were removed by a rotary cutter from a sheet of 99.0% purity zinc. They were suspended in the solution with a commercially available PTFE gasket system designed to prevent crevice corrosion around the edges of the specimen. It was assumed that these slight differences in geometry would not change the measured corrosion rate.

Before the working electrode was placed in the preservative solution, it was prepared according to ASTM G-59 [8]. The electrode was polished with 240-grit sandpaper, rinsed with 18 MΩ-cm purity distilled water, and polished with 600-grit sandpaper. The electrode was then washed again with 18 MΩ-cm purity distilled water, degreased with acetone, and rinsed once more with 18 MΩ-cm purity distilled water before immersion into the preservative solution. Special care was taken to insert the electrode into the solution as soon as possible after polishing and rinsing to minimize any possible environmental oxidation before testing.

In certain cases, the 1018 steel had deep scratches from shipping or machining. These electrodes were polished with 120-grit sandpaper to remove the scratches. The electrodes were then polished with 240-grit sandpaper and prepared as described in the previous paragraph.

While it may be argued that this method of sample preparation creates a more active surface than fasteners may see in service, the method was chosen because it was repeatable and already used as part of an existing standard [8]. When fasteners are driven into wood, large shear forces are exerted on the surface of a fastener. It has been argued that this violent process changes the surface of the fastener and increases the corrosion rate by making a more active surface [15,16].

3.4. Ancillary tests

To obtain a standard deviation for data analysis, 10 tests were run in a medium (12.8 g/kg) solution of ACQ-D for each steel electrode, for a total of 30 additional tests.

To measure the Tafel slopes, potentiodynamic polarisation scans were run. The scan rate for these tests were the same as for the polarisation resistance experiments, 0.166 mV/s, and the potential was scanned from 0.25 V below the open circuit potential to 0.25 V above the open circuit potential. It was found in preliminary experiments that the Tafel slopes did not depend significantly on concentration, and therefore the Tafel slopes were calculated from metals run in the 12.8 g/kg (medium) solutions.

The chemical analysis revealed a large difference in pH between the CCA solutions and the CC and ACQ-D solutions. Because iron and steel are normally passive in the pH range of CC and ACQ-D, and active in the pH range of CCA, it was decided to lower the pH of the ACQ-D solution to determine the effect of pH on corrosion rate. Only one test was run on the ACQ-D solution at the lower pH. The pH of the solution was lowered from 9.5 to 2 by adding sulphuric acid.

Full analysis of the effect of pH on the corrosiveness of ACQ-D was outside the scope of this paper, but it is currently being studied for a future publication by the authors.

4. Results

The polarisation resistance (R_p) values for the metals and preservative treatments are shown in Table 2. While the LPR technique was attempted on zinc in the ACQ and CC solutions, copper from the solution plated and completely covered the zinc electrodes. When the zinc became completely covered by copper, its corrosion rate was no longer activation-controlled, and the linear polarisation resistance technique was no longer valid for measuring the corrosion rate in ACQ or CC solutions. Therefore, this data was not

Table 2
Polarisation resistance (R_p) values for different metals exposed to aqueous preservative treatments

Preservative	Metal	Loading[a]	R_p (Ω cm^2)
CCA-C	430 Stainless	Low	2.59×10^5
		Medium	2.69×10^5
		High	5.96×10^5
	304 Stainless	Low	6.47×10^5
		Medium	9.50×10^5
		High	3.36×10^5
	1018 Steel	Low	1.66×10^5
		Medium	1.57×10^5
		High	4.76×10^5
	Zinc	Low	2.37×10^4
		Medium	1.46×10^3
		High	9.76×10^2
ACQ-D	430 Stainless	Low	1.26×10^5
		Medium	1.09×10^5
		High	1.18×10^5
	304 Stainless	Low	1.25×10^5
		Medium	1.34×10^5
		High	1.26×10^5
	1018 Steel	Low	9.18×10^4
		Medium	1.03×10^5
		High	7.97×10^4
CC	430 Stainless	Low	1.32×10^5
		Medium	9.34×10^4
		High	9.04×10^4
	304 Stainless	Low	1.58×10^5
		Medium	1.33×10^5
		High	1.18×10^5
	1018 Steel	Low	1.23×10^5
		Medium	1.01×10^5
		High	9.28×10^4

[a] Low = 4 g/kg; medium = 12.8 g/kg; high = 40 g/kg.

included in Table 2. The R_p values are related to the corrosion current density, i_{corr}, which is directly proportional to the corrosion rate, through the Stern–Geary equation:

$$R_p = \left[\frac{\beta_a \beta_c}{2.303(\beta_a + \beta_c)}\right]\left[\frac{1}{i_{corr}}\right] \qquad (1)$$

where β_a and β_c are the anodic and cathodic Tafel slopes, respectively [17].

The Tafel slopes were calculated by using commercial software that fit the polarization data to the Butler–Volmer equation with a non-linear least squares method. For each solution, the curves were of the same shape, regardless of the metal type. Representative polarization curves, for alloy UNS S34000, and the Tafel slopes are shown in Figs. 1–3 and the resulting Tafel slopes for all polarisation curves are given in Table 3. In every case, the anodic portion of the curve did not exhibit Tafel behaviour over sufficient region to allow extrapolation. Therefore, β_a was estimated as equal in magnitude to β_c for the Stern–Geary calculation [14].

The calculated corrosion rates in micrometres per year (μm/year) for the steel electrodes are shown in Fig. 4. Only one replicate was run for each metal in each solution. The standard deviation of each alloy of steel was measured in ancillary tests by running 10 additional replicates in the medium (12.8 g/kg) ACQ-D solution. The coefficient of variation or ratio of the standard deviation to the mean was then calculated. It was assumed that the coefficient of variation remained nearly constant across solutions for each metal alloy. The coefficient of variation was then used to calculate the standard deviation for the other solutions. The error bars in Fig. 4 represent one standard deviation using the measured variability (medium ACQ-D solution) and calculated variability (all other solutions).

The calculated corrosion rates of zinc in CCA were orders of magnitude higher than those of steel and therefore are not shown in Fig. 4. A possible explanation for this behaviour is given in the discussion.

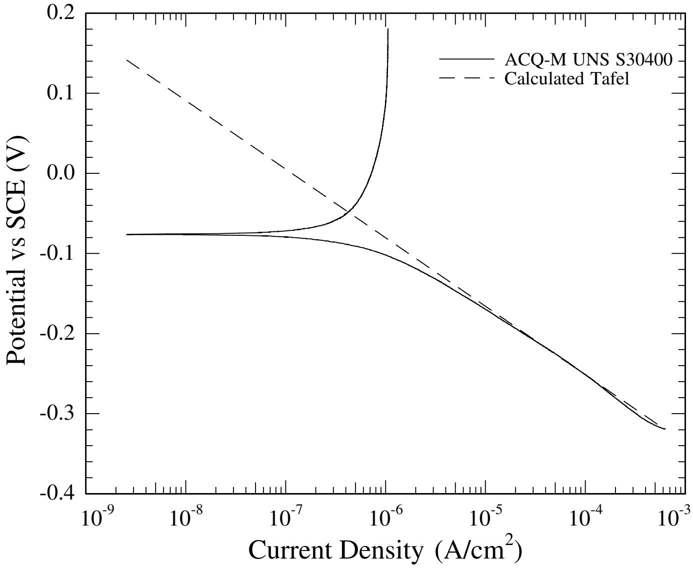

Fig. 1. Polarisation curve for alloy UNS S30400 in ACQ with cathodic Tafel slope indicated.

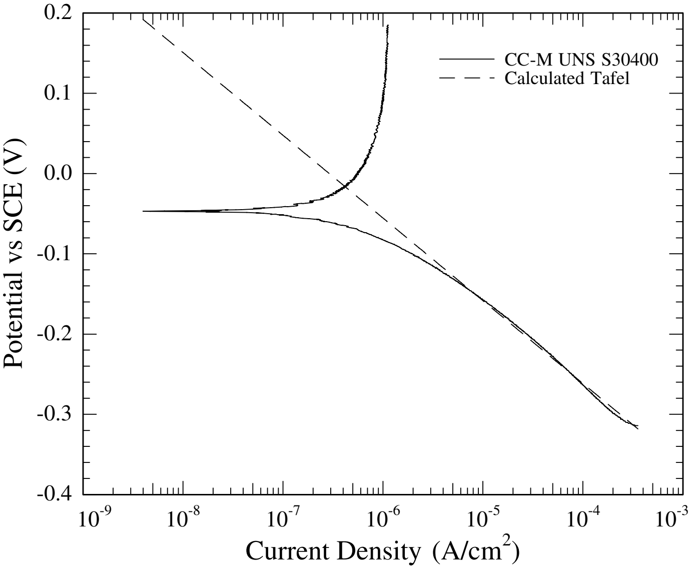

Fig. 2. Polarisation curve for alloy UNS S30400 in CC with cathodic Tafel slope indicated.

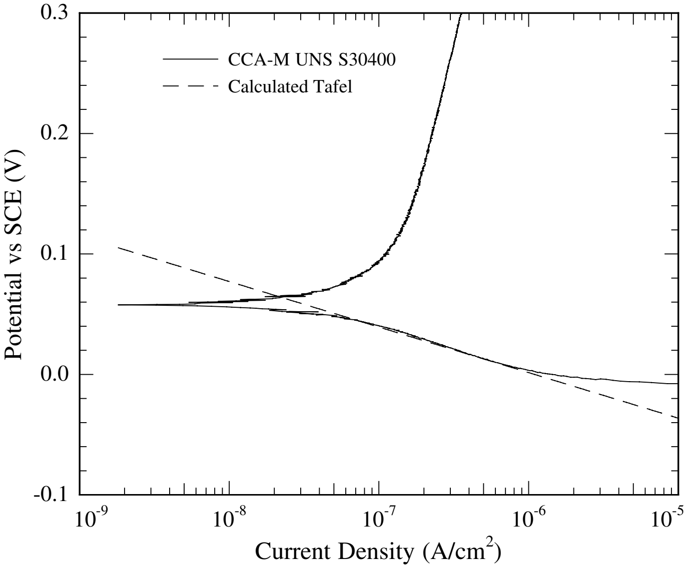

Fig. 3. Polarisation curve for alloy UNS S30400 in CCA with cathodic Tafel slope indicated.

The addition of sulphuric acid to the ACQ-D solution caused several visible changes indicative of chemical reactions. The pH of the solution was lowered from 9.5 to 2. As the pH of the solution neared 4, a dark blue precipitate formed. Further addition of sulphuric acid caused the precipitate to re-dissolve in solution as it approached a pH of 2. The original ACQ-D solution was opaque and dark blue. The ACQ-D solution with a pH of 2

Table 3
Tafel slopes in V per decade measured by potentiodynamic polarization experiments

Solution	Loading[a]	Metal	β_a	β_c
CCA-C	Medium	430 Stainless	[b]	0.05
		304 Stainless	[b]	0.04
		1018 Steel	[b]	0.03
ACQ-D	Medium	430 Stainless	[b]	0.09
		304 Stainless	[b]	0.09
		1018 Steel	[b]	0.11
CC	Medium	430 Stainless	[b]	0.10
		304 Stainless	[b]	0.10
		1018 Steel	[b]	0.11

[a] Medium = 12.8 g/kg.

[b] Tafel behaviour not observed.

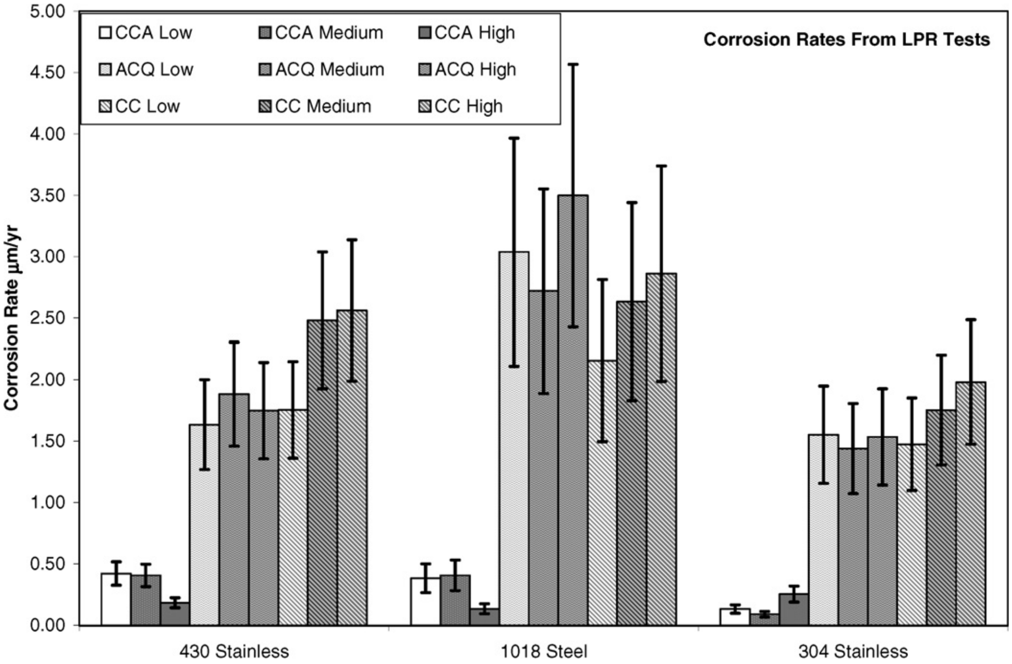

Fig. 4. Corrosion rate (micrometres per year) for steel in contact with different preservative treatments. Error bars represent one standard deviation.

was light green, translucent, and slightly cloudy. No chemical analysis was run on the altered solution or its precipitate.

The measured corrosion rate for the 430 stainless steel in the ACQ-D solution with a pH of 2 was 3.97 µm/year. This is roughly 1.9 times the corrosion rate of ACQ-D with a pH of 9.5 and 4.5 times the corrosion rate of 430 stainless steel in the medium CCA solution. It is critical to note, however, that at a pH of 2, not only is the corrosion of metals higher, but wood itself begins to dissolve and degrade. It would be unlikely that metals would be in contact with wood at this pH level in service.

5. Discussion

It was hypothesized that linear polar resistance experiments in solutions of preservative treatment could be used to rapidly evaluate the differences between different metals and preservative treatments to rank the performance of the metals in preservative treated wood. Our results deviated from the hypothesized results in two major respects: first, the measured corrosion rates for steel were much lower than expected, and second, the corrosion rate of zinc could not be accurately measured in all environments due to plating of the copper during testing. Unlike the simple case of pure H_2SO_4 for which the ASTM G-59 [8] methodology was originally established, the solutions of preservative treatments have many different ions (Table 1) that work to alter the corrosion rate in different ways. The effect of the ions on the corrosion rate can be explained in terms of cathodic reactions, which balance the corrosion reactions.

For acidic solutions, specifically in this paper, the CCA solutions, the most probable cathodic reaction is the reduction of hydrogen

$$2H^+ + 2e^- \rightarrow H_2(g) \qquad (2)$$

For neutral or alkaline solutions, specifically in this paper, the ACQ and CC solutions, the reaction is usually the reduction of dissolved oxygen. Since care was taken to de-aerate the solution before testing, the cathodic reaction must be either the decomposition of water (Eq. (3)) or the reduction of the cupric ion (Eq. (4)):

$$2H_2O + 2e^- \rightarrow 2OH^- + H_2(g) \leftarrow \qquad (3)$$

$$Cu^{++} + 2e^- \rightarrow Cu(s) \leftarrow \qquad (4)$$

While it is conceivable to consider the reduction of chromates or arsenates in CCA as possible cathodic reactions, these ions are oxidizing inhibitors and do not affect cathodic reactions as much as do cupric ions [18]. The effect of oxidation inhibitors will be discussed later.

In the case of Eqs. (3) and (4), it is possible to determine which cathodic reaction is occurring. If the cathodic reaction were the reduction of the cupric ion, copper would plate onto the corroding metal, giving a visual sign of the reaction. Indeed, this plating of copper was observed for zinc in the ACQ and CC solutions.

Before a discussion of the performance of the individual metals in solution can take place, a discussion of the pertinent factors that affect the corrosion of metals in solutions of wood preservatives is needed.

5.1. Factors affecting corrosion rate of metals in treated wood

pH. In general, pH is one of the main factors that affect the thermodynamic stability of metals in aqueous solutions. Certain metals perform better (less corrosion) in alkaline solutions while others perform better in acidic solutions, depending on the stability of the oxides and hydroxides that form on the metals in solution. While the exact conditions of thermodynamic stability do not follow any general pattern across the periodic table, potential vs. pH diagrams that show the stability of metals in aqueous solutions were calculated by Pourbaix [18].

Cupric ions. In the electrochemical series, copper is noble to both iron and zinc. Therefore, there exists a thermodynamic driving force for copper ions to cause galvanic corrosion and plate out at the expense of the corroding metal. A much larger driving force for galvanic corrosion exists between zinc and copper than between iron and copper. The addition of chromium and nickel to stainless steel make this metal even more noble, and there is little, if any, driving force for galvanic corrosion.

Chromate and arsenate ions in CCA. Both chromate and arsenate ions protect iron in aqueous environments. The chromate ion protects metals in solutions with a pH greater than 2, while the arsenate ion is very effective at protecting iron in a solution with a pH of less than 2. The arsenate ion is not as effective in protecting other metals [18].

5.2. Steel in CCA solution

The pH of the CCA solutions ranged between 0.5 and 2. According to the Pourbaix diagrams for iron and chromium [14,18], both carbon and stainless steel are thermodynamically unstable in this region and should corrode rapidly. It was concluded that the experimentally measured rates of corrosion were low because chromate or arsenate ions inhibit oxidation. The fact that there were cupric ions in the CCA solution did not greatly increase the corrosion rate because chromate and arsenate ions inhibited corrosion.

5.3. Steel in ACQ and CC solutions

The rate of corrosion would be expected to be much higher in ACQ and CC solutions than in CCA because these solutions did not contain the chromate or arsenate ions. However, the ACQ and CC solutions had a pH of 9.5. At this pH, iron is naturally passive and does not corrode rapidly. The corrosion behaviour of steel in the ACQ and CC solutions was dominated by the effect of pH.

Note that the pH of the preservative changes when it is introduced to the wood environment [19]. Therefore, although steel is passive in the pH range of ACQ and CC solutions, it may not be passive in ACQ- or CC-treated wood. The pH of ACQ- and CCA-treated wood is estimated to be between 4.5 and 8 [20,21].

To test our hypothesis that the corrosion rate for steel in ACQ and CC was lower than it should be in comparison to its corrosion rate in CCA, a test was run with 430 stainless steel immersed in a 12.8-g/kg solution of ACQ-D following the above-described protocol in which sulphuric acid had been added to lower the pH to 2, a pH similar to that of the CCA treatment solutions. The measured corrosion rate was 3.97 μm/year, 4.5 times greater than the corrosion rate measured in the medium CCA solution. Again, note that the pH range of the CCA solutions (0.5–2) was much lower than the pH range in CCA-treated wood [19,22].

It is purely coincidence that the ACQ and CC solutions appeared slightly more corrosive than CCA in this experiment. For all three preservatives, the corrosion mechanism was different in solution than it would be in wood. In CCA-treated wood, the hexavalent chromium ions are reduced to a trivalent state and the pH rises as the preservative fixates [13,16]; in ACQ- and CC-treated wood, the ammonia and amine compounds also leave the wood with time, lowering the pH and raising the corrosion rate [22,23].

5.4. Zinc in CCA solutions

While zinc is less thermodynamically stable than steel in the presence of copper, it appears that the zinc is stable with respect to the cupric ion in CCA in these experiments.

The corrosion rate of zinc in the low concentration CCA solution was on the same order of magnitude as the steel. However, as the concentration of CCA was raised, and the pH was lowered, the corrosion rate jumped much higher. This is consistent with Pourbaix [18], who states that the chromate ion is effective at inhibiting corrosion at pH levels greater than or equal to 2. The fact that the corrosion rates of zinc in higher concentrations of CCA are much higher than the corrosion rate of steel in these environments may be associated with the fact that the arsenate ions are not a general oxidation inhibitor and only protects iron [18].

5.5. Zinc in ACQ and CC solutions

Like iron, zinc at a pH of 9.5 generally forms a hydroxide film that protects it from further corrosion [18]. However, it was observed that the zinc hydroxide layer does not protect zinc in the presence of the cupric ion. In these wood preservative solutions, the zinc metal was covered with a thick, green film, indicative of copper plating.

6. Recommendations and conclusions

A qualitative test that mimics the corrosion behaviour of metals in contact with treated wood without using wood could be used as a rapid means to evaluate new wood preservatives before they are brought to market. In this experiment, the linear polarisation resistance method was used to measure the corrosion rate of four different types of metals in three different aqueous solutions of wood preservatives to see if this test mimics what is known about the corrosion of metals in wood.

Much of the discussion was based on the thermodynamic stability of alloys in the presence of various ions over a range of pH values. The objective of this study was to determine whether running linear polarisation resistance measurements on metals immersed in a solution of preservative treatment could be used to make qualitative comparisons of the corrosion rate. To prove that the measured corrosion rates are due to differences in thermodynamic stability between alloys, additional tests would need to be run to determine the effect of pH, the chromate ion, and the arsenate ion.

Furthermore, the linear polarisation resistance method would need to be altered if it were to be considered as a qualitative method for estimating the corrosion rates of metals in contact with wood. The solutions of preservatives should be altered in some manner to make them act more similar to how the preservatives exist within a treated wood environment. Because these chemical interactions are not well understood, this presents challenges as well as opportunities for future research.

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