Journal of Testing and Evaluation, Vol. 35, No. 1
Paper ID JTE100040
Available online at: www.astm.org

TECHNICAL NOTE

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Uncertainties in Corrosion Rate Measurements of Fasteners Exposed to Treated Wood at 100 % Relative Humidity

ABSTRACT: This paper evaluates the effect that uncertainties in measurements of time, weight, and surface area have on the determination of the corrosion rate of metal fasteners in contact with wood. Three different types of nails were driven into alkaline copper quaternary (ACQ)-treated wood and exposed to 26.7°C (80°C) at 100 % relative humidity environment for up to 1 year. It was observed that uncertainties in measurement of corrosion rate of less than 5 % could be obtained in 6 months of testing. This paper gives a description of the methods used as well as recommendations for future corrosion exposure tests in wood.

KEYWORDS: corrosion, exposure test, wood, preservative, ACQ, alkaline copper quaternary

Introduction

Because of the voluntary withdrawal of chromated copper arsenate (CCA) for residential use, many designers are now choosing to use alternatives to CCA such as alkaline copper quaternary (ACQ) and alkaline copper azole (CuAz). Limited research has been published on the effect of these alkaline-based preservatives about the corrosion rate, although researchers believe that ACQ and other new preservatives are more corrosive that CCA [1].

Currently, there is no method to correlate between in-service performance and controlled corrosion experiments in wood [1]. One step in developing this correlation is understanding how uncertainties in measurements affect the calculated corrosion rate in a controlled environment. The corrosion rate in any exposure test is commonly calculated from Eq 1 where m_i and m_f are the initial and final masses (g), t_i and t_f are the initial and final times (h), respectively, A is the surface area (cm^2), ρ is the density (g/cm^3), and K is a constant (87 600 mm cm^{-1} h $year^{-1}$). The term m_c (g) was added by the author to represent the additional loss of base metal that results from removing the corrosion products:

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

Freeman and Silverman [3] have analyzed how uncertainties in the values on the right side of Eq 1 statistically propagate through to the calculated corrosion rate for test runs in accordance with ASTM G31 Standard Practice for Laboratory Immersion Corrosion Testing of Metals [2]. Assuming independence and additivity of measurement errors, uncertainties were approximated by Eq 2, where V_{unc} is the variance of the uncertainty, σ_m represents the standard deviation in the measurement of mass, σ_t represents the standard deviation in the measurement of time, and σ_A represents the standard deviation in the measurement of area. The subscripts "*i*" and "*f*" and "*c*" stand for initial, final, and cleaning, respectively.

$$V_{unc}(R) \approx \left[\frac{\partial R}{\partial m_f}\right]^2 \sigma_{mf}^2 + \left[\frac{\partial R}{\partial m_c}\right]^2 \sigma_{mc}^2 + \left[\frac{\partial R}{\partial m_i}\right]^2 \sigma_{mi}^2 + \left[\frac{\partial R}{\partial t_f}\right]^2 \sigma_{tf}^2 + \left[\frac{\partial R}{\partial t_i}\right]^2 \sigma_{ti}^2 + \left[\frac{\partial R}{\partial A}\right]^2 \sigma_A^2 \tag{2}$$

The work of Freeman and Silverman [3] gives excellent guidelines on uncertainties of measurements from ASTM G31 standard tests, where the surface area of a simple sample geometry (coupon) is well defined and consistent for each test. However, corrosion tests in wood usually involve real fasteners, whose surface area is difficult to measure and which depend on the manufacturer and type of fastener. Additionally, most fasteners have a lower starting mass and surface area than corrosion coupons, so that uncertainties in these measurements are magnified.

This paper builds on the work of Freeman and Silverman [3] to determine the point at which uncertainties in the measurement of time, mass, and surface area become sufficiently small that they will cease to have an appreciable effect on corrosion rate measurements in wood. This paper calculates uncertainties caused by the measurement of time, mass, and surface area in reference to an experiment run by the author at 26.7°C (80°F), 100 % relative humidity environment. This environment was chosen to correspond to previous work on fasteners exposed to CCA-treated wood [4,5]. Baker [4] has shown that the mass loss of fasteners in this environment increased linearly with time over the length of his 14-year experiment, meaning that the corrosion rate is constant with time for these conditions. The assumption of a constant corrosion rate is used throughout the paper.

Manuscript received April 11, 2005; accepted for publication May 26, 2006; published online July 2006.

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Experimental

Wood

Select structural grade Southern Pine boards nominally 50 mm by 100 mm (2 in. by 4 in.) treated with alkaline copper quaternary (ACQ) were purchased from a commercial supplier. The wood was selected so that the grain angle was approximately tangential to the 50-mm face. The ACQ-treated lumber was intended for use above ground according to American Wood Preservers' Association (AWPA) use category UC3-B and had a specified nominal retention of 4 kg/m^3 (0.25 lb/ft^3) [6]. The exact type and formulation of the ACQ was not specified on the commercially purchased lumber.

Environment and Time of Exposure

The 100 % relative humidity environment was created in sealed glass desiccators that were partially filled with water, allowing both water and water vapor to be at equilibrium at one atmosphere of pressure. The relative humidity was kept constant by placing the desiccators in a conditioned room with a constant temperature of 26.7 °C (80 °F). Assuming uniform corrosion, the measured corrosion rate should be the same, regardless of the exposure time. To see if shorter exposure times could accurately predict the corrosion rate, corrosion tests were run for times of 1, 2, 3, 6, 9, and 12 months.

Fasteners

Three different types of fasteners spanning several materials types were tested: an 8d common nail, an 8d hot-dipped galvanized nail, and a 4d aluminum alloy nail. Three replicates were run for each exposure time and fastener type for a total of 54 replicates in a split-plot experimental design.

Pre-exposure Procedure

Prior to insertion into the wood, the fasteners were cleaned, degreased, and weighed. The fasteners were cleaned in a three-step process. The fasteners were first placed in an ultrasonic cleaner with a soap solution for 5 min. The fasteners were then rinsed under flowing distilled water before being placed in a distilled water bath that was ultrasonically agitated for 5 min. The fasteners were degreased by rinsing with a 50:50 mixture of toluene and ethanol and again rinsed with distilled water. The fasteners were then weighed to the nearest 0.0001 g and driven into pre-drilled holes with a diameter of 2.26 mm to prevent wood splitting and to ensure uniform contact between the nail surface and the wood. The pre-drilled hole corresponds to approximately 90 % of the diameter of the smallest fastener.

Post-exposure Procedure

The fasteners were removed in such a way to minimize the damage to the fastener. Initially, two grooves were cut in the wood surrounding the fastener with a band saw. The wood was then placed in a vise. As pressure was applied, the wood split along the sawn grooves, and the fastener was removed without damaging the corrosion products.

Several high-resolution digital images were taken of each fastener to document the amount of visual corrosion products. Additional black and white photographs were taken under special lighting to give a silhouette of the fastener. These silhouette pictures were then fed into a computer program written by the author to compute the surface area of the fastener, which was used for the corrosion rate calculations. The computer program calculated the surface area (A) by measuring the diameter every five pixels, calculating the circumference of that disk, multiplying the circumference by the distance of five pixels, and summing over the length of the fastener. Ideally, the pre-test surface area should be used in corrosion rate calculations; however, no silhouette pictures prior to insertion were taken.

The fasteners were then cleaned with methods similar to those presented in ASTM Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens (G1-03) [7]. After cleaning, the fasteners were weighed and the mass (m_f) was measured. The weight loss from cleaning (m_c) was calculated by using the same cleaning process on uncorroded fasteners (Table 1).

TABLE 1—*Cleaning methods used to remove corrosion products.*

Material	Solution	Cleaning time,[a] min	m_c Weight change due to cleaning, mg	σ_{m_c} Standard deviation in cleaning, mg
Steel nail	50:50 mixture of distilled water and Evapo-Rust™	60	−1.2	0.9
Galvanized nail	Saturated ammonium acetate	60	−1.6	1.2
Aluminum nail	Concentrated nitric acid	5	+0.1	0.2

[a]The cleaning was performed with ultrasonic agitation.

[b]Evapo-Rust™ is a proprietary chelating agent manufactured by Harris International Labs Inc., Springdale, AR 72764.

Results

The average corrosion rates, standard deviations, and coefficients of variation (standard deviation divided by the mean) taken over all exposure times are reported in Table 2. For the calculated uncertainty, both the initial and final times were measured to the nearest "day," where a "day" is an 8-h work shift, having an uncertainty of ±4 h. The resulting standard deviation in the measurement of time (σ_t), is therefore 2.3 h [3]. The standard deviation in mass due to the balance itself is at most 0.0001 g. This is the uncertainty for σ_{mi} and σ_{mf}. However, additional uncertainty is brought about by the cleaning procedure to remove the corrosion products. This uncertainty (σ_{mc}), is given in Table 1. Finally, the standard deviation in the surface area (σ_A), was measured by measuring the surface area of the same corroded fastener in the program ten times. It was

TABLE 2—*Average corrosion rates.*

	Corrosion Rate, mm/year	Standard Deviation, mm/year	COV, %
Steel nail	0.044	0.026	59
Galvanized nail	0.070	0.046	66
Aluminum nail	0.022	0.019	86

TABLE 3—*Uncertainties in the measurement of corrosion rate.*

Metal	Corrosion Rate, mm/year	Surface Area, mm^2	Time, months	Total Uncertainty,[a] %	Percent of Total Uncertainty Caused by		
					Mass	Area	Time
Steel	0.044	780	1	5.23	85.25	13.88	0.87
			2	3.07	61.81	37.60	0.59
			3	2.53	40.61	58.98	0.41
			6	2.10	14.64	85.21	0.15
			9	2.00	7.19	92.74	0.07
			12	1.98	4.12	95.83	0.04
Galvanized	0.070	650	1	5.16	96.21	2.89	0.89
			2	2.68	89.07	10.15	0.78
			3	1.91	78.25	21.02	0.72
			6	1.22	47.96	51.60	0.44
			9	1.04	29.39	70.34	0.27
			12	0.97	18.84	80.99	0.17
Aluminum	0.022	320	1	20.51	99.62	0.31	0.06
			2	10.30	98.77	1.17	0.06
			3	6.92	97.39	2.55	0.05
			6	3.58	90.64	9.31	0.05
			9	2.52	81.30	18.66	0.05
			12	2.02	71.06	28.90	0.04

[a]The "Total Uncertainty" was approximated by dividing the standard deviation of the total uncertainty in the corrosion rate by the corrosion rate.

found that the standard deviations in the surface area for aluminum, steel, and hot-dipped galvanized nails, were 3, 13, and 6 mm^2, respectively.

Discussion

It is imperative to define and differentiate between uncertainty in measurements and variance within a data set. Uncertainty in the measurement of the corrosion rate is limited by the precision and accuracy of the measurements used to calculated the corrosion rate and is the focus of this paper. Variance within a data set is a measure of the total "spread" of the data. It includes the uncertainty in the measurements and also includes all other sources of variation between specimens. Increasing the number of specimens allows the researcher to better understand the variance within the data set but does not improve the inherent uncertainty in the measurements. In the case of corrosion in treated wood, the variance includes variation in wood properties, variation in preservative retention levels with position on the board, variation in moisture content, as well as consideration that corrosion itself is a stochastic process. In this study, attempts were made to partition some of these variations by using a split-plot experimental design; a generalization of a randomized block design [8]. The coefficient of variation from three replicates ranged between 59 % and 86 % (Table 2).

Table 3 shows how uncertainties in the corrosion rates are expected to change with time for different fasteners. These approximations are based on the average corrosion rate over time and averages surface area for each fastener type. The percentage uncertainties in the rightmost columns were calculated by dividing the relevant terms in Eq 2 by the total uncertainty, V_{unc}. The uncertainty in the corrosion rate of aluminum alloys is higher because the aluminum fasteners were smaller than the other fasteners and have a lower density, which causes small variations in mass to have a large effect on the corrosion rate.

As can be seen from Table 3 for all fasteners tested, uncertainties drop below 5 % for test times 6 months in duration or longer. This is a significant finding because previous corrosion tests in wood with other wood preservatives [4,9] have run for longer periods of time: up to 20 years. From the analysis of this paper and previously published long-term data [4,9], it appears that corrosion tests run in a constant environment could be run for shorter periods of time, although the exact uncertainties should be calculated for each experiment on a case-by-case basis.

Conclusions and Recommendations

It was found from this study that the corrosion rates for different materials in ACQ treated wood could be measured in 6 months with a 5 % uncertainty in the measurement. It was also found that the data set had a high coefficient of variation, and more replicates would be needed to obtain a meaningful confidence interval of the corrosion rate. The largest uncertainties in the corrosion rate appear to be caused by the uncertainties in the measurement of mass and surface area.

Acknowledgments

The author thanks Douglas Rammer, P.E., and Dr. Robert Moon of the USDA Forest Products Laboratory, Madison, Wisconsin, for their fruitful discussions on this topic.

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