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Timber Corrosion Test Methodology

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Abstract

BRANZ has developed a two-stage accelerated test methodology to determine the aggressivity of timber treated with different preservation chemicals towards mild steel and hot dip galvanised nails. In comparison with conventional techniques using high temperature and high humidity to accelerate corrosion, this method can more reliably simulate natural exposure as it establishes a more realistic environment inside the timber. Results derived from this method also confirm that ACQ and/or CuAz treatments are more corrosive than CCA treatment and the corrosion acceleration factors determined were similar to those obtained from field exposure tests. However, over-estimation was still found and possible approaches for further improvement are discussed.

Keywords: corrosion; accelerated test; timber; preservation; CCA; CuAz; ACQ; nail; mild steel; zinc; hot dip galvanising

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1. INTRODUCTION

Metallic components are used in a wide variety of timber structures [Risbrudt 2005]. However, most metals are thermodynamically unstable and subject to corrosion in the presence of moisture and/or oxygen in the cellular structure of timber. Corrosion of metal and the resulting degradation of timber can lead to the premature failure of structures. Therefore, information concerning degradation behaviour, corrosion performance and service life of metals in timbers is critical to the design, construction, maintenance and retrofit of timber structures.

Corrosion of metal in timber is an extremely complicated process that is influenced mainly by moisture content and/or the presence of preservation chemicals. Any environmental factors that can change the timber moisture content and/or the state of preservative will change the micro-environment at the metal-timber interface, thus changing the deterioration behaviour of metal.

Currently, there is a lack of understanding of the mechanisms and processes causing metal corrosion in timber. However, there have been many attempts to create testing techniques to evaluate the performance of specific metals when in contact with timber. Results derived from these tests provide useful information for timber-metal design, construction and maintenance schemes.

2. REVIEW OF TEST OF METAL CORROSION IN TIMBER

2.1 Metal Corrosion in Timber

Under most service conditions, susceptible metals in contact with timbers will corrode through various chemical and/or electrochemical processes. Factors influencing corrosion that are specific to timber include timber moisture content, natural timber constituents and/or presence of preservation chemicals (both organic and inorganic).

Timber moisture content is perhaps the most crucial factor for initialisation and progress of metal corrosion in timber. Timber naturally contains some moisture as a result of its production from a living tree. In addition, timber is hygroscopic which means it has a tendency to absorb moisture from the surrounding environment. At some stage an equilibrium moisture content will be achieved [Wiedenhoeft and Miller 2005; Rowell 2005]. When the moisture content is high enough (>18-20%), acetyl radicals present in the timber will be hydrolysed to acetic acid. This results in most timbers being slightly acidic, with a pH of 3 to 6, and therefore corrosive to metals. A further impact is that when the moisture content is high it facilitates ionic transport, i.e. the electrical conductivity of the timber is increased.

Various preservation chemicals are added to timbers to improve resistance to biological, chemical, mechanical, photochemical and/or thermal attack [Ibach 1999]. These chemicals are considered to influence the corrosion of metals in such treated timber however the mechanisms have not been systematically researched as yet. Galvanic reaction between cupric ions (Cu^{2+}) in copper-bearing water-borne preservatives and metals (such as iron or zinc) has been proposed as one such mechanism although experimental results were inconclusive [Baker 1988].

Investigating corrosion processes in treated timbers is further complicated by the variations in the formulation of preservation chemicals. For example, the most widely used preservative, copper chrome arsenate (CCA), can be formulated from either a mixture of potassium dichromate, copper sulphate and arsenic acid or from a mixture of chromium trioxide, copper oxide and arsenic acid. The potassium dichromate formulation has a higher conductivity by virtue of the formation of potassium sulphate and is believed to be more corrosive [Hendrix 2006]. In addition to the formulating materials, the preservative species themselves may affect corrosion. For example, chromium (Cr^{6+}) may passivate steel [Murphy 1998], but copper (Cu^{2+}) is cathodic to iron and zinc and promotes galvanic corrosion. Chloride is another constituent of some preservatives and can be present at significant levels in some alkaline copper quaternary (ACQ) preservative systems. Chloride is particularly influential on metal corrosion properties. In addition, timber can also absorb pollutants (chlorides, sulphides, etc.) from its surroundings, such as marine and industrial settings, making the micro-environment even more complex.

2.2 Methodologies for Test of Metal Corrosion in Timber

The performance of metallic components in timber is critical to the durability and stability of any timber-metal structures. Many accelerated and non-accelerated techniques have been developed to provide qualitative or quantitative results that could be used to determine the corrosion performance of a specific metal in contact with a timber. Typical testing methods and the results derived are briefly discussed in the following sections.

2.2.1 Outdoor exposure / Field testing

One of the most reliable (and simplest) methods to investigate the corrosion of metal in timber is to field test the timber-metal assembly by exposing it directly to the environment of interest [Baker 1992]. After a specified time, the metal can be retrieved from the timber and both the metal and the surrounding timber inspected visually or microscopically for any signs of corrosion. Corrosion products can also be removed completely so the cleaned samples can be weighed to measure their mass loss (corrosion) rates. Field testing has been widely employed to provide useful results for the development of preservatives, timber based construction materials and timber-metal structures.

However, field testing has some disadvantages from a scientific research perspective. Outdoor exposure has to be carried out in several locations and these will likely differ in temperature, rainfall, wind pattern, solar irradiation or atmospheric pollution. Such climatic elements can significantly influence the micro-environmental conditions at the timber-metal interface, thus affecting the corrosion processes and rates. Consequently, corrosion data gathered in one specific location cannot directly be applied to another. In addition, climatic conditions may change over time (climate change might be more pronounced in the long term), making repeat field tests problematic.

2.2.2 Simulated natural exposures

In consideration of the limitations imposed by the variation of outdoor climatic conditions, researchers have tried to design and conduct experiments under well-controlled conditions, particularly temperature and humidity.

Baker exposed specimens (metallic nails embedded into treated Southern pine) to a controlled humidity room to simulate conditions that “would have a corrosion rate higher than that expected in wood foundations” [Baker 1992]. The exposure at 27^{o}C and 98 ± 2% relative humidity (RH) lasted for 14 years. For comparison, Baker buried another set of samples in soil for 17 years at the Valley View Exposure Site in Madison, Wisconsin, USA.

Results showed that the constant high temperature and relative humidity environment accelerated the corrosion process of most nails compared to the nails buried in soil. For example, after 14 years of exposure at 27^{o}C and 98% RH, hot dip galvanised (HDG) steel nails in timbers treated with CCA-I and CCA-II (oxide formulation) lost 3% and 4% (weight) when buried in soil, compared to 16% and 18% in the exposed test environment. While this information is useful, a key challenge of such simulated natural exposure research is to achieve a better correlation between testing and in-service performance.

2.2.3 Accelerated exposures

In order to overcome the location variations of field tests and the long time required by natural exposure, researchers have developed approaches to accelerate the corrosion process by making the environment at the timber-metal interface more aggressive.

Three approaches widely employed to achieve this goal are:

1. Increasing the timber moisture content and the environmental temperature;
2. Placing the metal in direct contact with damp sawdust; and
3. Introducing additional chemicals that are aggressive towards metals.

2.2.3.1 Moisture content and temperature

In timbers with high moisture content, transportation of charged ions will be faster and electrical conductivity higher when compared with timbers having low moisture content. Consequently, metal corrosion reactions will tend to occur at a higher rate in high moisture content timbers. At high temperatures, the kinetics of some thermally activated corrosion reactions, e.g. diffusion and mass transportation, can also be increased. Based on this understanding, studies have attempted to accelerate metal corrosion in timber by increasing the environmental temperature or relative humidity, or both [Rammer *et al*. 2006].

Barnes *et al*. tested the corrosion performance of some typical metals (carbon steel, galvanised steel, aluminium, brass and copper plate coupons) sandwiched between two blocks of timber (treated with CCA, ACA (ammoniacal copper arsenate), pentachlorophenol, organic and organometallic treatments) at 38°C [Zelinka and Rammer 2005]. This methodology is very similar to the recommendations of American Wood-Preservers' Association (AWPA) E12 standard that addresses corrosion of metal in contact with treated timber [AWPA 2004]. In the methodology associated with the E12 standard, a metal coupon is sandwiched between two pieces of preservative treated timber. These timber-metal assemblies are then placed in a conditioning chamber of 49±1°C and 90±1% RH for an accelerated exposure of >240 hrs.

Based on the AWPA E12 testing procedure, Simpson Strong-Tie developed a modified procedure to investigate how fasteners performed in treated timbers [Simpson 2006]. Their results showed qualitatively that ACQ, CuAz and sodium borate with $NaSiO_2$ was more than twice as corrosive as CCA for the average of G90 and G185 hot dip galvanised steel samples.

Tests following the AWPA E12 standard have also been widely used by timber preservers and fastener manufactures. However, the testing conditions are not representative of the environments that the timber-metal structures will be exposed to and test results cannot be extrapolated to reliably predict corrosion rates under real service conditions. Furthermore, exposure in an environmental chamber cannot evaluate the potential influences of atmospheric pollutants in various environments, such as marine (chlorides), industrial (sulphur-containing species) and urban (nitrogen-containing species).

Another weakness of such testing is that the geometric configuration of the sample is different from that of real structures. Gaps are introduced within the sample sandwich structure affecting the ingress of water and oxygen and different sample tightnesses may impact on corrosion mechanisms.

2.2.3.2 Damp sawdust

Some researchers have put metal samples directly into water containing a sawdust suspension to accelerate corrosion processes [Bartel-Kornacka 1967]. The sawdust can be obtained from the cutting of treated timbers or clean sawdust can be treated to achieve a full and uniform penetration of the preservative used for normal timber treatment. To simulate the way fasteners are used in service, the fasteners were driven into the timber and removed before being placed into the experiments. Similar to the increased moisture and humidity tests, damp sawdust tests are only able to give relative and qualitative results on the corrosivity of treated timbers.

2.2.3.3 Salt spray

Salt spray (continuous or cyclic) can significantly increase the wetness of samples and also the aggressivity of the environment (through increased temperature and introduction of corrosive species). This use of salt spray to accelerate metal corrosion in timber is considered to be of some use in estimating the behaviour of materials in marine atmospheres or in contact with sea water.

Richolson of the U.S. Navy's materials laboratory, ran salt spray tests to determine the aggressivity of five types of timbers (white oak, teak, mahogany, bald cypress and Douglas fir) used in ship building. Various metallic fasteners (bright steel, galvanised steel, brass, chrome-plated brass and silicon bronze) were evaluated with these timbers following ASTM standard B-117 [Zelinka and Rammer 2005; ASTM 2003].

Product manufacturer SENCO[1] also tested the corrosion performance of fasteners made of stainless steel, hot dip galvanised and electro-galvanised steels in timbers treated with non-arsenate preservation formulations according to ASTM-B117 and ASTM-G85. Their tests indicated that white and red rust appeared at approximately 300 hours in the ASTM-B117 test on the hot dip galvanised and electro-galvanised fasteners. In comparison, stainless steel nails did not exhibit any signs of corrosion.

Ajith Peter and Edwin tested the corrosion performance of nails (copper, iron, painted and galvanised iron) in CCA treated timbers [Ajith Peter and Edwin 2008]. One set of panels were exposed in a salt spray chamber maintained at a temperature of 35^oC and 95% RH. This test was conducted for a period of 480 hours. The other set of panels were exposed adjacent to an estuary for 100 days. Experimental observations showed that the corrosion rate of the samples in the chamber was significantly higher than that of the samples exposed in the field. The authors believed the high salinity of the salt spray exposure resulted in the high corrosion rate observed. The higher temperature and lower pH of salt spray solution in the chamber may also have influenced the corrosion rate.

Exposures in fog chambers using distilled water were also discussed in an un-published commercial testing report authored by T. F. Shupe *et al.* in 2009. This report, entitled "Corrosion effects of ACQ treated wood on metal fasteners in an accelerated test" evaluated the corrosion resistance under exposure to fresh water and chemically treated timber of two coated fasteners [Shupe *et al*. 2009].

Salt spray using NaCl solutions can be valuable if the timber-metal structures will be exposed in coastal areas. However, introduction of chloride and other ionic species into timber through this approach is different from the deposition of airborne salt, and hence it is very hard to establish a reasonable salt concentration and gradient in timber.

Further, there is no universally accepted or standard specified correlation between the number of hours to the appearance of red rust (corrosion of iron-based substrate) in a salt spray chamber versus the number of hours of real-world performance. More importantly, due to the large differences in temperature, wetness and chemical state/concentration the corrosion mechanism might be quite different from that in treated timbers exposed naturally. Salt spray cannot precisely measure the real aggressivity of treated timbers towards metallic components and test results cannot be directly related to 'real world' corrosion performance.

2.2.4 Immersion techniques

2.2.4.1 Immersion of metal in preservation treating solution

This procedure is recommended by AWPA standard, E17 [AWPA 1999]. Test coupons are suspended in jars of treatment solution. The assembled test jars are agitated on a platform shaker. The treatment solution is changed at specified time intervals. When completed the corrosion products are removed and then the weight loss of the coupon determined to calculate corrosion rate.

Following this procedure, Kear *et al.* tested the corrosion performance of mild steel, hot dip galvanised steel and AISI 316 stainless steel coupons in the dilute solutions of CCA, CuAz and ACQ [Kear *et al*. 2008a]. Their results showed that hot dip galvanised steel samples exhibited the highest corrosion rate while the corrosion rates of the mild steel and stainless steel samples were exceptionally low compared to those measured within treated timbers. Further it was shown that CCA solution was more corrosive than CuAz and ACQ treatment solutions, opposite to the findings with real timber-metal structures exposed to the atmosphere.

Based on their experimental findings from this and other testing methods, Kear *et al.* believed that corrosion tests performed within aqueous solutions containing preservation chemicals cannot properly reflect the degradation behaviour of mild steel and hot dip galvanised steel embedded into timbers treated with the same preservatives due to the following reasons:

1. The pH values of the dilute preservative solutions were different from those in timbers treated with identical preservatives. For example, it was found the CuAz and ACQ treatment solutions were strongly alkaline, while the 'wash-water' of the timbers treated with these two chemicals were slightly acidic.
2. In timbers, Cu, Cr and/or As species would be fixed onto fibres through various reactions and processes. This fixation would significantly change the diffusion and migration of ions in timber, thus affecting metal corrosion or passivation. Obviously, in dilute preservative solutions, the fixation would be absent and hence the metal corrosion behaviour would be different from that in timber.

Measurements of this type may lead to a significant underestimation of the potential corrosion risk that will be encountered in timbers treated with CuAz and ACQ.

2.2.4.2 Immersion of metal in extract solution

A corrosion test using ACQ chemicals directly extracted from ACQ treated timber investigated corrosion performance of various fasteners over 30 days of exposure [Panasik 2009]. The fastener surfaces displayed a wide spectrum of results including no corrosion, small amounts of corrosion and extensive corrosion. In general, stainless steel of the 300 and 400 series performed well as did 410 series stainless steels with specific proprietary coatings. In some cases particular coatings seemed to allow more corrosion than others. Some types of carbon steel with proprietary coatings had very limited amounts of corrosion whereas hot dip galvanised materials were covered with corrosion.

2.2.5 Electrochemical methods

Electrochemical methods for corrosion measurement and assessment measure the current density at which corrosion takes place. The current density can then be converted to mass loss or depth of corrosion penetration. Electrochemical methods have become well established for the study of corrosion in aqueous solutions since corrosion rates can be derived rapidly with small scale samples. Their use in investigation of metal corrosion in timber is limited though they are believed to be promising for application in this area [Dennis *et al*. 1995; Simm and Button 1985; Jack and Smedley 1987].

2.2.5.1 Polarisation resistance

Linear polarisation resistance (LPR) is an electrochemical method that measures the direct current flowing through the metal/electrolyte interface when the electrode is polarised by a small electrical potential (~±30 mV or less offset). This current is related to the corrosion current (related to the Tafel slopes) and in turn is directly proportional to corrosion rate [Stern and Geary 1957; Evans and Koehler 1961]. This measurement of corrosion rates allows almost instant feedback to operators.

The polarisation resistance R_p is the reciprocal of the slope of the polarisation curve at the corrosion potential when plotted with current density on the ordinate and voltage on the abscissa. It is inversely proportional to the corrosion current density, which can be transformed to a corrosion rate for uniform corrosion through the following equation:

$$i_{corr} = \left(\frac{1}{2.303R_p}\right)\frac{b_a b_c}{(b_a + b_c)}$$

where b_a and b_c are the anodic and cathodic Tafel slopes, respectively.

Simm and Button used the LPR method to measure the corrosion performance of metals in contact with European redwood treated with CCA [Zelinka & Rammer 2005]. Corrosion rates of several typical metals, including aluminium alloy, mild steel, stainless steel and pure zinc, were measured. It was found that the corrosion rate depended strongly upon the timber moisture content, which is consistent with gravimetric corrosion tests.

However, electrochemical tests undertaken by placing an electrode directly into timber are complicated as the resistivity of timber can change significantly with the moisture content (a variation over six orders of magnitude was found in some measurements). The resistance needs to be corrected to give any meaningful results on corrosion rate. In addition, timber normally has inherent in-homogeneity of structure, density variation, moisture content gradients and/or non-uniform distribution of preservation chemicals. Although researchers have taken care in the selection of timber and machining of the corrosion cell, most reported tests still show relatively poor reproducibility.

Concurrently, when a direct current is applied to the corrosion cell, the ionic components of the non-fixed chemicals in salt-based preservatives will be driven through the timber and permanently polarised. Therefore, it will not be possible to derive any further useful information from the timber sample.

Different direct current electrochemical approaches have then been attempted. Zelinka *et al*. performed LPR tests with metals (carbon steel, stainless steel and zinc) directly immersed into dilute aqueous solutions of preservation chemicals (alkaline copper quaternary, ammoniacal copper citrate and chromated copper arsenate) [Zelinka *et al*. 2007a]. It was found:

1. The measured corrosion rates for steel were found to be much lower than expected; and
2. The corrosion rate of zinc could not be accurately measured due to plating of the copper during testing.

The researchers believed that the poor correlation observed was mainly due to the fact that solutions of preservatives cannot act like to those within a treated timber environment. Also, in some cases, the Tafel behaviour cannot be observed simultaneously for both anodic and cathodic portions of the polarisation curves. Thus b_a and b_c cannot be estimated accurately for use in the corrosion rate calculation.

Kear *et al*. also performed voltametric scans (<±20mV) in aqueous preservative solutions (CCA, CuAz and ACQ) to estimate the corrosion rates of mild steel, hot dip galvanised steel and stainless steel components in timbers [Kear *et al*. 2008b]. However, these researchers could not obtain linear polarisation curves close to the corrosion potential in the majority of the systems examined. Qualitative analyses also indicated that hot dip galvanised steel was the most active material and suffered the highest mass loss. These observations were similar to previous results obtained using AWPA E17 procedures [Kear *et al*. 2008a].

Direct exposure of metal to preservative containing solutions as a model for exposure in treated timbers is generally based on the following assumptions [Zelinka *et al*. 2007a]:

1. The preservative does not react with the timber, or this reaction does not affect the corrosiveness of the preservative; and
2. The chemical constituents of wood are not corrosive, or at least much less corrosive than the preservatives.

Corrosion rates measured in dilute solutions of preservative are poorly correlated with corrosion rates of metals in treated timbers, indicating that the first assumption is not right. The second assumption is also flawed because corrosion rates of metals in contact with untreated timbers are sometimes comparable to those in CCA treated timbers. This implies that the cellular structures and/or their derivatives do have some effects (negative or positive) on corrosion.

Practically, it was also found to be very unlikely that a linear polarisation resistance response would be realised in most corrosion systems involving metal and preservatives since [Kear *et al*. 2008b]:

1. Anodic and cathodic mechanisms and polarisation behaviours are different; and
2. Several reactions may occur simultaneously during either anodic or cathodic polarisation. For example, in a solution containing CCA, five cathodic reactions may be occurring simultaneously, including the reduction of cupric ions, chromium-based ions, arsenic-based ions, oxygen, protons and probably the reduction of surface films. While in CuAz and ACQ containing electrolytes, many organic materials may exert their influences on corrosion reactions.

Corrosion rates of metals are strongly dependent on timber moisture content. There is also a threshold moisture content, around 18-20%, below which metal corrosion is insignificant, a region where corrosion rate increases with increasing moisture content, and a plateau above which the corrosion rate is constant with moisture content. These three regions correspond with the stages of water adsorption in timber, which at low moisture contents gets bound to hydroxyl sites within the cell wall, and as these sites get filled, free (unbound) water exists within the cell walls, and eventually the lumens. Zelinka *et al*. thus believed that corrosion in timber is an aqueous process that occurs in the free water present in cell walls and in lumens at higher moisture contents [Zelinka *et al*. 2008b].

Zelinka *et al*. then changed the solutions and made them more representative of the corrosive environment in treated timbers [Zelinka *et al*. 2008 a-b]. They created an extract of the treated timber by placing the sawdust of the treated timber in contact with water and ran LPR tests in water extracts of ACQ treated timbers with actual fasteners. The results appeared to have a relatively good correlation with those derived from the exposure of carbon steel and galvanised fasteners in a constant environment (27^{o}C and 100% RH). However, for aluminium fasteners, the correlation between the corrosion rates measured in solid timber and those derived from the polarisation test in extract solutions was very poor.

Further tests were run in an extract made from untreated Southern pine. The results did not correlate well with exposure tests and this extract was even more corrosive than the extract of ACQ treated timber. This result is not consistent with observations from natural exposures or other accelerated tests. These researchers believed that the deviations were related to various known and unknown chemical reactions occurring in the timber.

Based on these experimental findings, it is believed that polarisation resistance testing in extracts from timbers may have the potential to rapidly evaluate the corrosion performance of metallic fasteners in timber when:

1. the detailed chemical composition of the extract is quantified, and
2. the effect of individual chemicals on corrosion can be systematically investigated.

2.2.5.2 Electrochemical impedance spectroscopy

Electrochemical impedance spectroscopy (EIS) is an electrochemical technique that principally involves applying a small amplitude signal (at a voltage usually ranging from 5 to 50 mV) to the specimen of interest over a wide frequency range, typically from 1 mHz to 1 MHz. Both the magnitude and phase of the current relative to the voltage are measured and the real (resistance) and imaginary (capacitance) components of this complex impedance response of the system is then calculated [Walter 1986; Scully 1989; Rammelt and Reinhard 1992; Murray 1997]. EIS allows modelling of corrosion reactions with an equivalent circuit. This mechanistic circuit model can be used to predict how changes in the environment or other parameters will affect the corrosion rate.

Kear *et al.* used EIS to study the corrosion properties of mild steel, hot dip galvanised steel and stainless steel in aqueous preservative solutions (CCA, CuAz and ACQ) [Kear *et al*. 2008c]. Relative corrosion rates were derived as a function of metallic material and preservative. The results clearly showed that hot dip galvanised steel had an active dissolution behaviour while mild steel and stainless steel exhibited effective passivation under the testing conditions. As a result, the corrosion rate of the hot dip galvanised steel could be orders of magnitude higher than that of the mild steel and stainless steel. This was very similar to the findings using LPR tests in aqueous solutions of preservatives, but was quite different from the results derived from exposed timber structures. Kear *et al.* hypothesised that the big difference between the pH values of the aqueous preservative solution and the actual timber treated with the same preservative might be responsible for this.

Other researchers performed EIS studies using corrosion cells directly built on timber blocks. Jack and Smedley used this technique to determine how the corrosion rate of iron and zinc varied with moisture content and exposure time in untreated and CCA treated Pinus radiata [Jack and Smedley 1987]. Cross used EIS to estimate the relative life of the metals (mild steel, hot dip galvanised steel, zinc electroplated steel, AISI 304 stainless steel and AA 6063 aluminium) in contact with CCA treated timbers in roof environments [Cross 1990]. Although experimental data was fitted with complicated models, detailed explanation of the physical meaning of the components were not given.

To calculate corrosion rates, experimental EIS data need to be fitted with an equivalent circuit composed of resistors and capacitors. A key to the establishment of a reasonable equivalent circuit model is that behaviours represented by these components in the model can be understood in terms of physical mechanisms.

Unfortunately, it appears that the exact physical meanings of the equivalent circuits derived from EIS currently are hard to explain.

For EIS models to be appropriate and representative, a thorough understanding of the electrical properties of timber and timber-metal interfaces is needed and an understanding of the corrosion process for a given environment and geometric parameters must be developed. Zelinka *et al*. collected EIS spectra from southern pine equilibrated to 12% and 20% moisture contents to develop a basic equivalent circuit model of the electrical properties of timber at different moisture contents [Zelinka and Rammer 2006; Zelinka *et al.* 2007b]. These results could be used as a starting point for modelling the corrosion reactions in timber and to clarify the role of the timber–metal interface in electrical measurements.

In summary, both the direct current polarisation and the EIS method have been shown to be an option for measuring the instantaneous corrosion rate of metal in treated timber, particularly in wet timber. However, more work is needed to further develop these methods. There needs to be a better understanding of ionic conduction and resistivity of timber, as well as the corrosion process, before a meaningful EIS model can be fully developed.

2.3 Summary

Many techniques have been developed to evaluate the corrosion performance of metallic components when embedded in timbers, both untreated and treated. Most accelerated exposure tests and electrochemical techniques can provide corrosion rate results in relatively short time periods. However, poor correlations have commonly been observed between results derived using these methods and natural exposures.

The primary reason is that the testing conditions cannot properly reflect the service conditions and the corrosion mechanism might be completely changed due to this significant change of conditions. As a result, the results derived cannot be directly used for service life prediction. Lack of a fundamental understanding of metal corrosion in timber presents a large barrier to establishing a corrosion cell that can accurately simulate the complicated corrosion reaction processes (both known and unknown) occurring on a metal in a timber exposed to the environment. At the same time, the unknown chemical interactions between preservative and timber present challenges for establishing rapid techniques that can provide qualitative and/or quantitative results on corrosion of metal in timber.

3. BRANZ TESTING METHODOLOGY

3.1 Objective

BRANZ believes that a suitable testing methodology must be able to develop a range of moisture contents, to initialise chemical/physical changes and to establish a micro-environment at the timber-metal interface that is similar to that experienced by timber exposed outdoors for appropriate initialisation and progress of corrosion. It is clear that exposure of timber to an atmosphere of constant temperature and humidity cannot deliver this. This research aims to develop a new accelerated test methodology that can simulate natural exposure conditions in a better way and establish a more realistic micro-environment inside the timber for measuring metal corrosion.

3.2 Methodology Development

Precipitation is of primary importance for corrosion of metal in timber. It determines the moisture content of the timber. Further, it changes the state of preservatives and affects their hydrolysis, migration and re-distribution in timber [van der Sleet *et al*. 1997]. Water may dissolve and then mobilise some fixed components. Meanwhile during drying periods (upon solar irradiation), some of these released species may migrate within the timber and possibly move from deep within the section to the exterior surface. This process will increase or decrease the supply of active species to the corrosion reaction front, thereby affecting the metal corrosion rate.

In addition, physical defects, such as cracks and checks formed on the timber during natural weathering, can increase both the surface area and provide more easy paths for the access of moisture to the interior of the timber. This in turn leads to higher moisture content and facilitates hydrolysis processes and/or ion transport in the timber. BRANZ believes that a reliable testing scheme should have the capability to develop a range of moisture contents and to initialise chemical/physical changes similar to those experienced by timber exposed outdoors.

Continuous exposure of timber-metal assemblies at constant temperature and humidity cannot produce the above-mentioned reactions and effects. Therefore, a new exposure scheme should be developed.

Recently, BRANZ noticed that interrupted exposure tests, such as interrupted salt spray, consisting of sub-cycles of salt spray and air drying, is emerging as a promising approach for evaluating corrosion of metals directly exposed to atmospheric conditions [Zhao *et al.* 2009; LeBozec and Thierry 2010]. BRANZ also conducted an accelerated test to evaluate the performance of several types of stainless steels in direct contact with preservation treated timbers using a procedure combining exposure at constant temperature/humidity and interrupted spray (distilled water and NaCl solution) [Li 2008]. It was observed that corrosive attack on these steel specimens occurred quickly during the interrupted spray stage. Meanwhile, the timbers developed a greater degree of checking / cracking on their surfaces in the wetting-drying stage. This effect was not observed with exposures following the AWPA E12 procedure, indicating that interrupted testing schemes may establish an environment for metal corrosion that is realistic but still accelerated.

Furthermore, in the development of any accelerated atmospheric corrosion tests, several factors have been identified as important in cycling the surface of test materials through both wet and dry conditions [Lawson 2005]:

1. Precipitation – A liquid film present, containing dissolved pollutants and salts;
2. Evaporation – A liquid film present, salts and pollutants concentrating;
3. Dry – Liquid film absent, solid salts on surface; and
4. Re-wetting – Liquid film present, salts diluting, or possibly removed under rain conditions

Evaporation is significant because the corrosive materials may concentrate on the surface when drying, providing a more corrosive condition should the material be wetted by condensation rather than being cleaned by the washing action of rain.

Therefore, the testing methodology developed in this study was designed to consist of:

1. Well-organised wetting cycles that closely simulated natural rainfall events and achieved a moisture content similar to that of timbers wetted by rain; and
2. Drying stages that allowed timber to be dried to a moisture content level that was found in timbers exposed in the field.

The development of physical defects, such as cracks and checks, formed on the timber surface is expected to be mirrored by the wetting-drying cycles of the testing method. This could induce significant expansion and contraction of the timber due to temperature and moisture content variations.

BRANZ has recently completed a multi-year field exposure test aimed at evaluating the corrosion properties of metallic fasteners in timbers. These timbers were treated with different water-borne and copper-bearing preservatives to different levels. Corrosion rates of mild steel and hot dip galvanised steel fasteners (nails and screws) have been obtained after one and three years of exposure at BRANZ's Judgeford campus through mass loss measurements [Li *et al*. 2010]. In addition, BRANZ has measured the corrosion rates of nails in timber using several accelerated and non-accelerated techniques [Kear *et al*. 2005]. Corrosion rates measured from the method developed in this study will be thoroughly compared to the results derived from those techniques for procedure optimisation.

3.3 Treatment of Test Timber

Rough-sawn kiln-dried Pinus radiata sapwood with nominal cross-section dimensions of 100×100 mm was custom-treated with three commercially-sourced water-based preservatives:

1. CCA (oxide, H3.2 and H4);
2. CuAz (CA-B containing tebuconazole, H3.2 and H4); and
3. ACQ (ACQ-B containing didecylthyl ammonium chloride (DDAC) for H3.2 and ACQ-C containing alkylbenzyldimethyl ammonium chloride (BAC) for H4).

Treatment was undertaken at retention levels appropriate for Hazard Classes H3.2 and H4 as dictated by NZ 3640 [SNZ 2003], but to compensate for wood variability the solution strengths indicated were increased by 10%. A 'Bethell' process was chosen to maximise uniformity of the preservative retention between individual boards within each treated lot. To accomplish this process, a vacuum is drawn on the timber for a predetermined period of time before the treatment chamber is flooded with preservative while maintaining the vacuum. Once the flooding is completed, the pressure in the cylinder is raised and held until the timber refuses to absorb further preservative. Prior to the treatment, the density of each individual timber board was determined based on its weight, moisture content and volume. In addition, each board was weighed after treatment to assess its preservative uptake and active species retention.

Post treatment, the timbers were stored in a constant climate lab at, 25^{o}C and 55% RH. These timbers were then cut to the required dimensions (~90×90×200 mm). Original surfaces with any potential precipitated preservation chemicals were removed. During cutting, the longest dimension of all these timber blocks was selected to run in parallel to the wood grain. To minimise the influence of fast moisture transportation, the open ends of the timber blocks were sealed with enamel.

3.4 Establishing Timber Moisture Content

Moisture content is critical to many processes occurring in timber, such as preservative leaching, hydrolysis of cellular components and release of acidic species, timber decay, metal corrosion, etc. Timbers exposed to the test conditions should have a moisture content that is close to that of timber exposed to the atmosphere.

As mentioned earlier, fog chamber spraying with either sodium chloride solution or distilled water has been used before to evaluate the corrosion performance of metallic components embedded into timbers. The spraying process was carried out either continuously or discontinuously. Detailed information regarding the testing conditions is not given in the literature. It also appeared that some critical testing conditions (e.g. quantity of water sprayed, time of spraying and drying and/or drying temperature) were not carefully correlated with actual environmental conditions. Continuous water spraying deviates significantly from the atmospheric conditions that most timber structures will experience during service. Tests with wetting and drying cycles, but without well-controlled temperature and water quantity will still have difficulty in establishing a micro-environment that is similar to timber exposed naturally.

In order to design a reliable interrupted spray testing scheme, BRANZ believes the following factors must be well understood:

1. How wet is wet enough? and
2. How dry is dry enough?

In other words, the timber moisture contents in water spraying and air drying stages must have a reasonably good correlation with the values observed in a timber exposed to a service environment.

However, moisture content and its fluctuation are not easily measured with timbers exposed outdoors since these parameters are strongly related to many factors including timber dimensions, orientation, preservation treatment and atmospheric conditions (ambient temperature, rainfall, and wind patterns). Currently, there is no moisture content baseline for timbers exposed to typical New Zealand environments.

Several timber gate structures were exposed at BRANZ's Judgeford site in previous research [Li *et al*. 2010]. These gates were constructed using timbers with the same preservation treatment and cross-sectional dimensions as the timbers used in this study. Their moisture contents were then measured to establish a rough moisture baseline for exposed timbers for method development.

These measurements were performed by using a portable pin-type electrical-resistance moisture content meter. The measurement times were intentionally selected so that the moisture contents immediately after rain and long after rain could be obtained. The timber structures were vertical and had one side facing directly to the north. The moisture contents on the north and south sides of one timber block were different due to the differences in rain direction and heating by sunlight. Five measurements were made in an "X" configuration on each surface. The moisture content for the timber side surface was then averaged from the results measured from these two sides.

Electrical resistance based moisture measurements are believed to be relatively limited in accuracy between 8% and 25% (wt./wt.) [SA/SNZ 1997]. Measurement errors also increase with higher water concentrations and so they might also be unsuitable when the timber moisture content is above 40%. Conductivity based measurements will be further complicated by the presence of ionically conductive preservation chemicals, such as CCA, CuAz and ACQ. However, moisture meters had to be used in this study, as moisture content determination using oven-drying was not practically suitable for samples exposed in the field. It is also believed that electrical resistance measurements can be relied upon to compare the moisture contents of the timbers of the same preservation treatment that are exposed naturally and artificially.

3.5 Establishing Operational Conditions for the Fog Chamber

Basic information, including annual rainfall, rain days, timber moisture content variation, was then retrieved from available databases or measured by BRANZ. These results were fundamental to the design of testing scheme.

The statistic information concerning the total rain and rain days during the time period of 2000 – 2009 at BRANZ's Judgeford site was retrieved from the NIWA National Climate Database (National Institute of Water and Atmospheric Research, New Zealand). The results are given in Table 1. The average annual rainfall at the field exposure site was calculated to be around 1212 mm. The information on monthly rain days was incomplete so an average was calculated from the data available and used to derive the total rain days per year. This led to an average annual rain days of about 128 days for the period of 2000 - 2009.

Table 1. Rainfall and rain days at the BRANZ Judgeford site

Year	Statistics	January	February	March	April	May	June	July	August	September	October	November	December	Annual
2000	Rainfall	121.8	21.7	41.5	100.5	101.2	130.8	47.1	71.3	140.2	114.3	47.5	69	1006.9
	Rain Day	11	7	9	13	-	9	8	-	15	16	8	-	-
2001	Rainfall	10.2	25.4	31	17.8	61.6	104.3	140	130	12	164.5	158.9	199.9	1055.6
	Rain Day	6	5	7	3	18	-	11	15	6	-	12	17	-
2002	Rainfall	81.8	70.3	74.4	59.5	74.3	163.5	99	151.8	111.3	48.5	119.5	113.5	1167.4
	Rain Day	10	9	9	-	9	-	-	-	17	-	-	13	-
2003	Rainfall	36.6	23.5	28.9	48.5	52.6	203.9	97.5	34.7	200.3	171.3	62.2	105.8	1065.8
	Rain Day	10	6	7	10	9	12	9	13	22	-	12	14	-
2004	Rainfall	108	413.5	61.1	51.3	67.1	129.2	140.9	260.7	149.3	83.6	111.2	162.9	1738.8
	Rain Day	12	17	12	8	12	15	-	19	12	12	10	15	-
2005	Rainfall	143.5	-	85.4	68.7	153.5	68.1	87.9	31.3	44	95.2	12.3	100.7	-
	Rain Day	7	-	-	9	16	-	17	10	9	10	7	-	-
2006	Rainfall	41.3	97.5	81.5	78.7	152.5	150.8	213.1	202.2	40.9	228.4	180.7	90.4	1558
	Rain Day	5	8	13	13	15	12	13	15	11	16	15	-	-
2007	Rainfall	116.3	13.8	42.1	52.4	51.7	65.8	132.4	69.2	48.2	165.8	45.8	55.5	859
	Rain Day	10	4	8	-	6	10	-	14	10	18	-	7	-
2008	Rainfall	68.5	29	96	132.8	33.4	180.3	265.6	158	76.9	117.2	58	122.6	1338.3
	Rain Day	5	-	7	12	-	-	-	-	-	-	9	13	-
2009	Rainfall	38.3	129.9	28.2	113.9	160.9	72.5	90.9	129	63.7	152.9	75.6	62.7	1118.5
	Rain Day	-	9	6	8	-	-	-	11	-	-	-	11	-

The weather conditions of New Zealand can be roughly divided into two seasons, wet and dry. The wet season has a relatively low ambient temperature and higher rainfall, therefore a higher moisture content develops in timbers directly exposed to the atmosphere. This can be clearly seen from Figures 1 and 2. For example, in April, the moisture content of the timber after a relatively long dry period could be as low as 15-20%, while in June and July, this value could be as high as 20-40%. The timber moisture contents after rain were similar, typically ranging from 30% to 70%.

However, it should be noted that NIWA's climate database indicates that the highest mean temperatures normally occur between December and March. Therefore, timber moisture contents in summer might be lower than those measured in this study which commenced in April (as noted in Figure 1).

The surface temperatures of the timbers exposed at Judgeford were also measured and showed that sunny day temperatures could rise to around 18-23^{o}C in July and 30-36^{o}C in April.

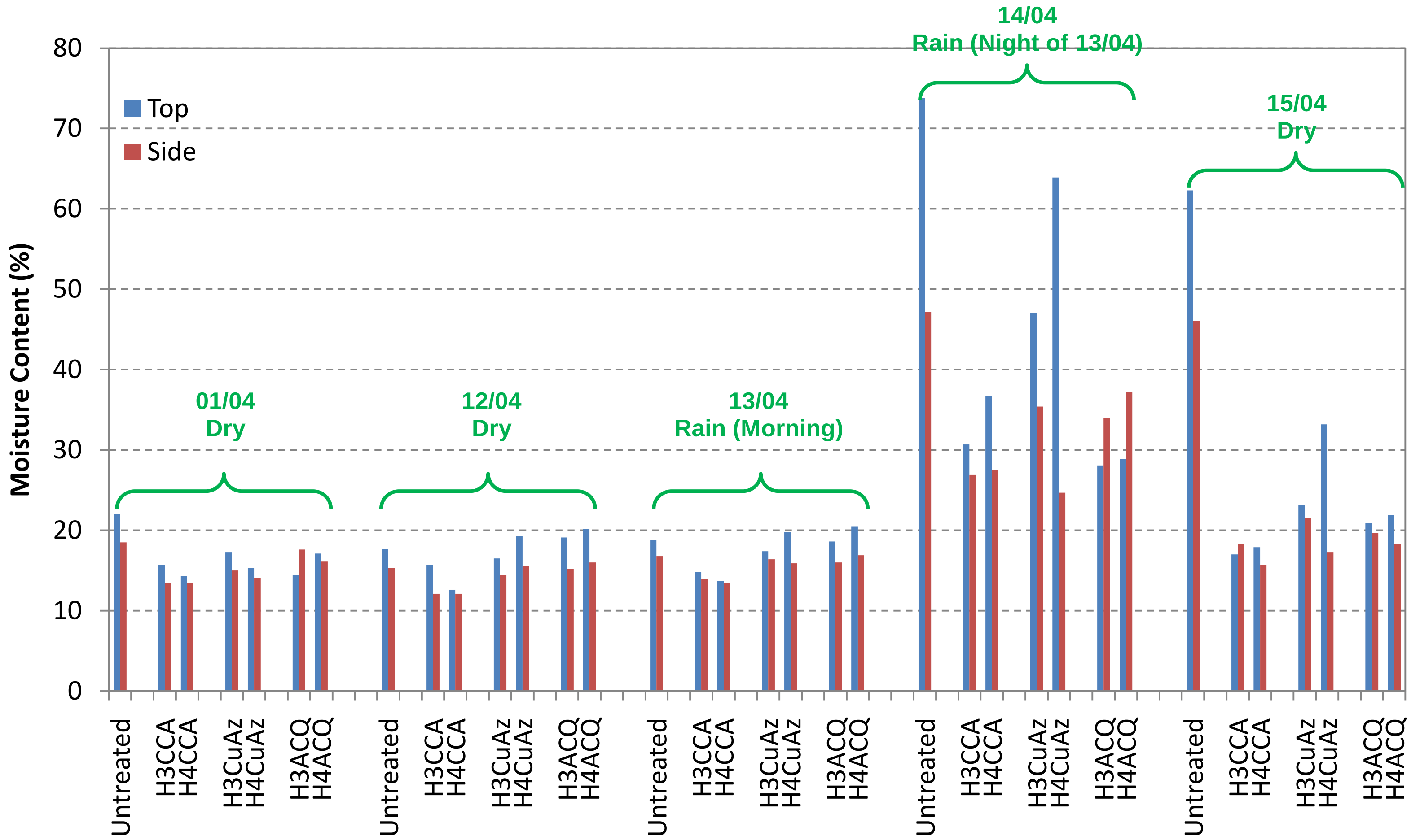


Figure 1. Variation of the moisture content of timbers exposed to the atmosphere at BRANZ's Judgeford campus from 01 April to 15 April 2010.

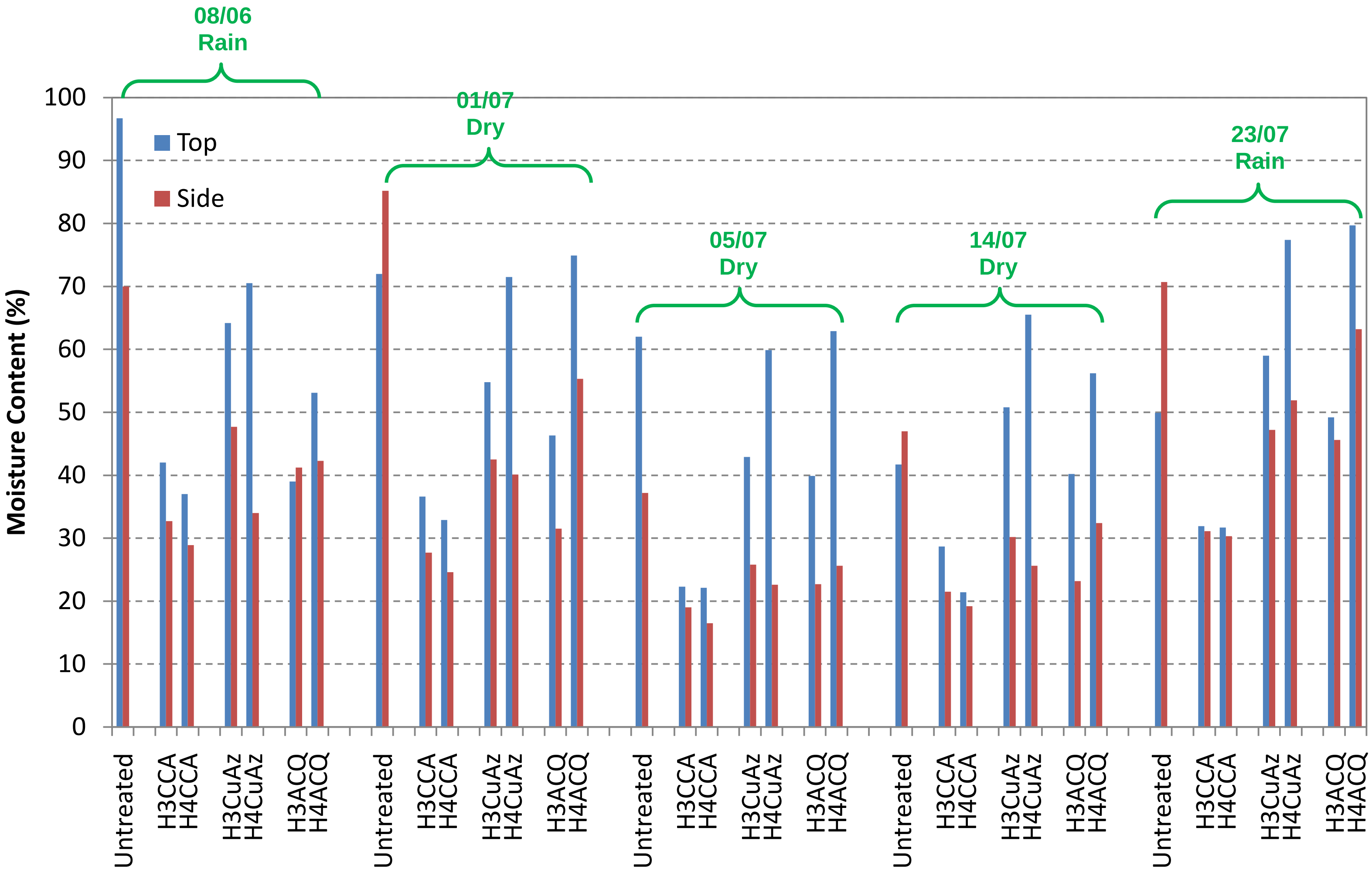


Figure 2. Variation of the moisture content of timbers exposed to the atmosphere at BRANZ's Judgeford campus from 08 June to 23 July 2010.

Based on the statistical information, the operational parameters for the fog chamber were then set up:

Compressed air pressure: 7.5 psi and

Water flow rate: 0.5-0.6 L/hr.

Deionised water (conductivity ~2 µS/cm) was used as the medium simulating natural precipitation without significant pollutants released from industry and/or agriculture. This minimised the influences of other ions contained in tap water on the corrosion processes in the timbers, particularly those treated with copper-bearing chemicals. Before these studies commenced the chamber had been run with distilled water for over 1000 hours. Therefore, the pipes for solution delivery, spray nozzles and chamber walls were thoroughly cleaned and free of NaCl contamination from any previous tests.

The wetting stage used in this study was carried out at one fixed temperature, 25°C (the lowest temperature that can be controlled accurately within the BRANZ Q-fog chamber). The drying stage was performed at two different temperatures, 25°C and 35°C. The former temperature was used to achieve a higher moisture content that simulates the rainy season and the latter was used to achieve a lower moisture content that simulates the dry season.

A series of preliminary tests were carried out using the above parameters. The volume of water sprayed was collected by funnels evenly distributed in the chamber to calculate hourly water spray. The moisture contents of the timber blocks installed in the fog chamber were measured during the artificial wetting-drying cycles. Comparisons between the timber moisture contents in natural and artificial wetting-drying cycles are given in Figures 3-5.

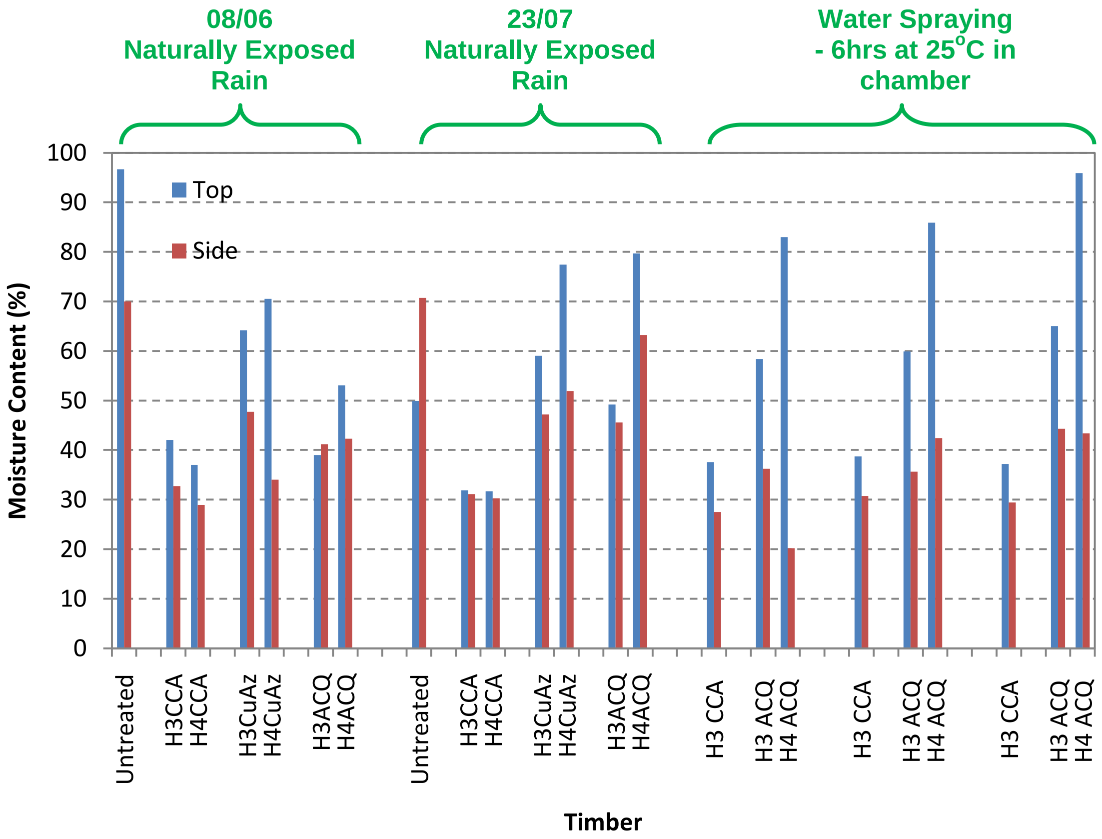


Figure 3. Comparison between the moisture contents of timbers exposed to natural and artificial environments (natural precipitation *vs.* artificial water spray at 25°C).

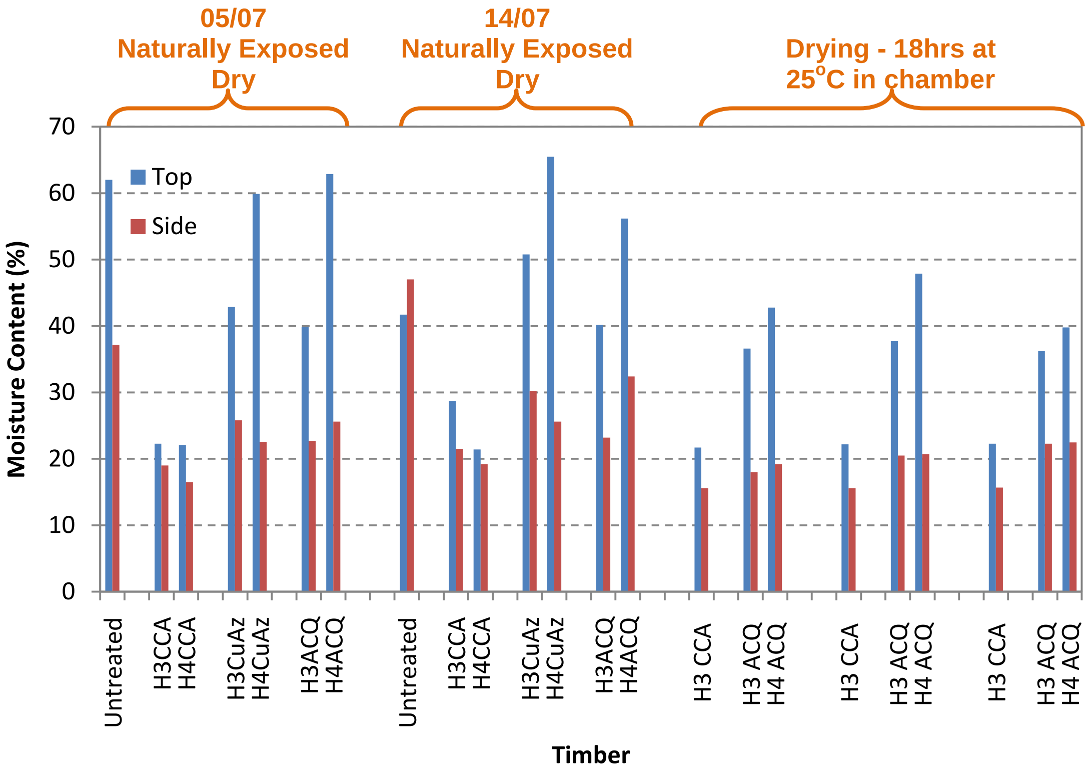


Figure 4. Comparison between the moisture contents of timbers exposed to natural and artificial environments (natural drying *vs.* artificial drying at 25°C).

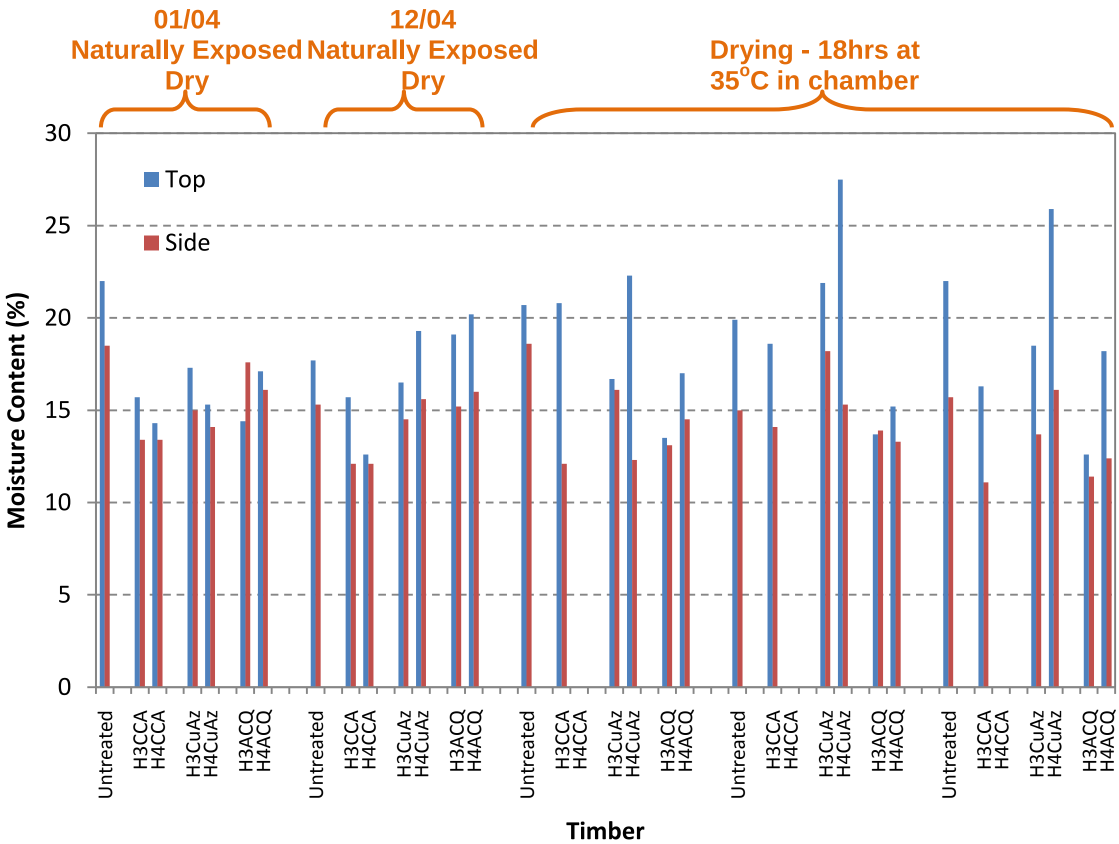


Figure 5. Comparison between the moisture contents of timbers exposed to natural and artificial environments (natural drying *vs.* artificial drying at a high temperature of 35°C).

From the results shown above, it can be seen that the artificial cycles containing water spraying and air heating could reasonably simulate the natural wetting-drying behaviour (the moisture content level and variation) of the timbers exposed to the environment at the Judgeford site.

In addition, it was found that after several wet-dry cycles, small cracks initialised on the timber blocks and it was observed that their depth and length developed gradually with repeated cycles.

4. TESTS AND RESULTS

4.1 Fasteners – Embedment and Cleaning

Two types of nails were used in the testing: mild steel nails of nominal dimensions 50×2.10 mm; and hot dip galvanised nails of nominal dimensions 30×3.15 mm. The thickness of the hot dip galvanising zinc coating applied onto the nails was measured according to the recommendation of ASTM A 90/A 90M – 01. The coating thickness, averaged from ten replicates, was determined to be 53.4±9.9 µm.

The precise dimensions of every nail were measured and recorded for surface area calculations. After cleaning with a mixed solution of ethanol and acetone, the original masses of these nails were measured with an electronic balance (0.1 mg accuracy). The nails were then manually inserted into the pre-cut timber blocks through pre-drilled holes (~60% of the nail diameter). Two locations were used: one was on the top; and the other was on the side. Three replicates were produced for each sample.

The timber-nail assemblies were then installed into the fog chamber. Their positions in the chamber were randomly changes every 10 days, this was to minimise the potential influences of uneven distribution of fog on the corrosion processes.

After exposure, all nails were retrieved by carefully splitting the timber. Their surface morphology was characterised visually and optically. Corrosion products formed on these nails were then completely removed by immersion into chemical solutions specified by ASTM G1:

1. Mild steel: 0.5L/L HCl + 3.5g/L hexamethylene tetramine (HMT), 20-25^oC, ~10 minutes; and
2. HDG: 100g/L ammonium chloride (NH_4Cl), 70^oC, ~5 minutes.

Cleaned nails were then rinsed with distilled water and acetone, followed by drying with warm air. Their mass was measured again to obtain mass loss for determination of corrosion rate (µm/year). Densities of mild steel and zinc used in calculations were 7.86g/cm^3 and 7.14g/cm^3, respectively.

4.2 First Trial – Single Stage Test

4.2.1 Sample configuration and chamber operational condition

In this trial, the nails were inserted into the timber top surface in a single line as shown in Figures 6 and 7.

The first test undertaken was a single-stage test. One complete cycle consisted of two sub-cycles: water spraying at 25^oC for 6 hours and air heating at 35^oC for 18 hours. A total of 30 cycles were completed (i.e. a test duration of 720 hours).

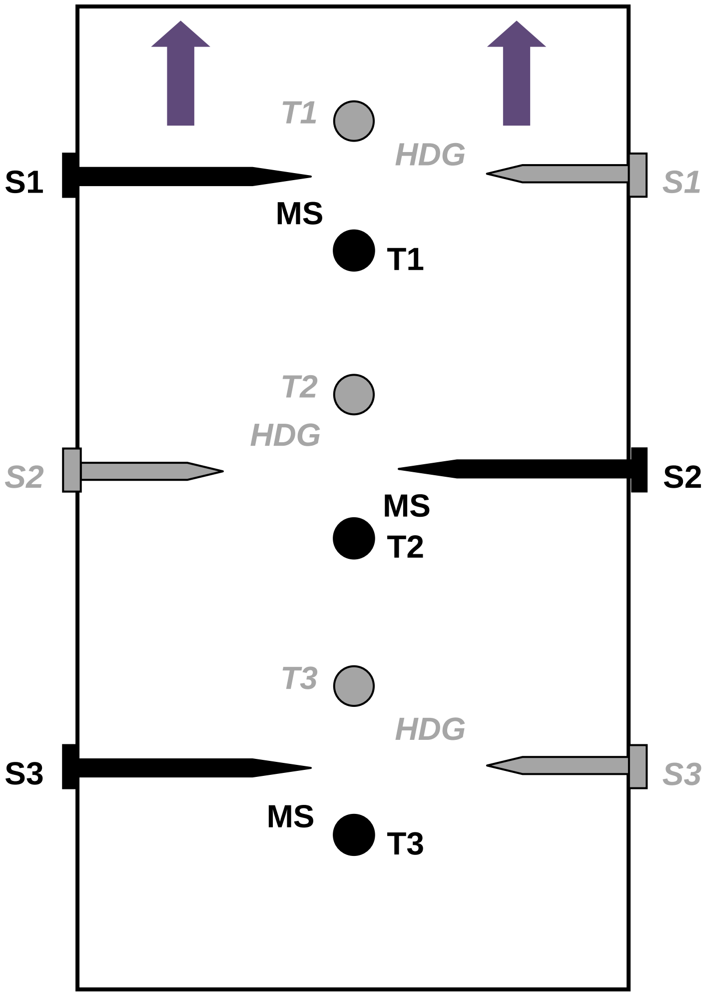


Figure 6. A schematic showing the sample configuration used in the first trial.

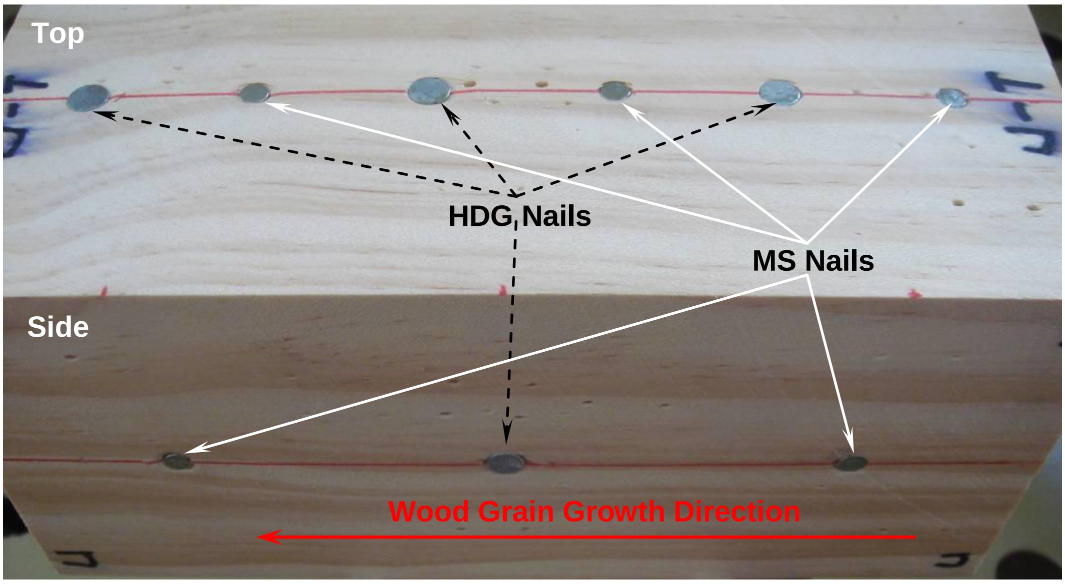


Figure 7. Nails driven into an untreated timber block.

4.2.2 Corrosion rate

The corrosion rates of the mild steel and hot dip galvanised steel nails, measured by weight loss, are presented in Figures 8 and 9.

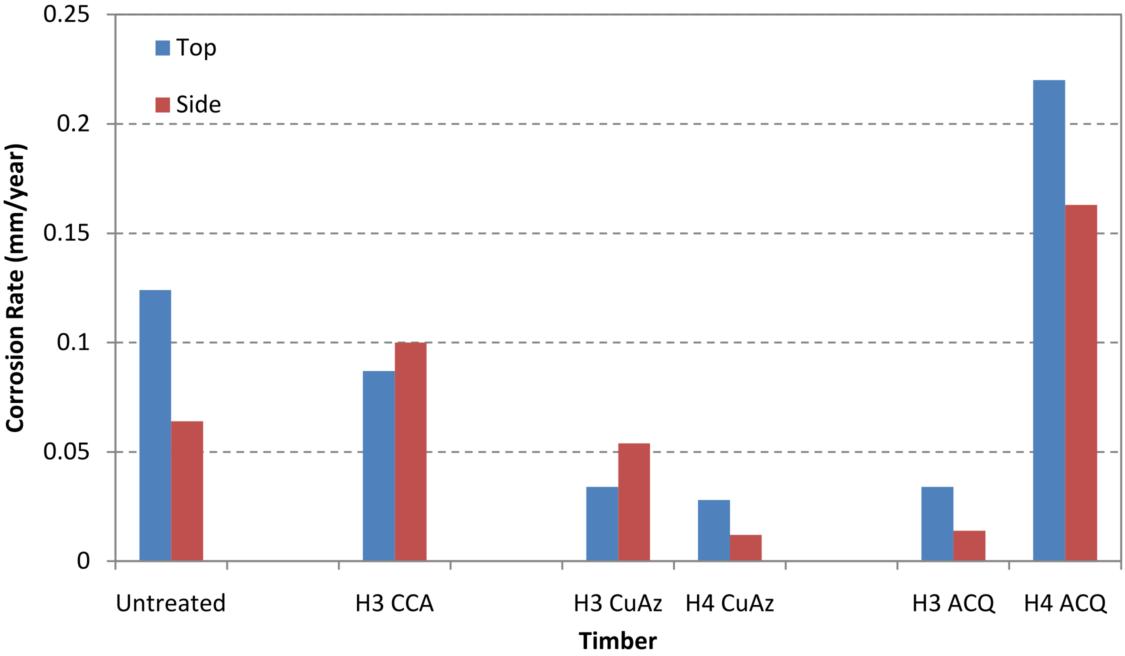


Figure 8. Corrosion rate of mild steel nails (Trial 1).

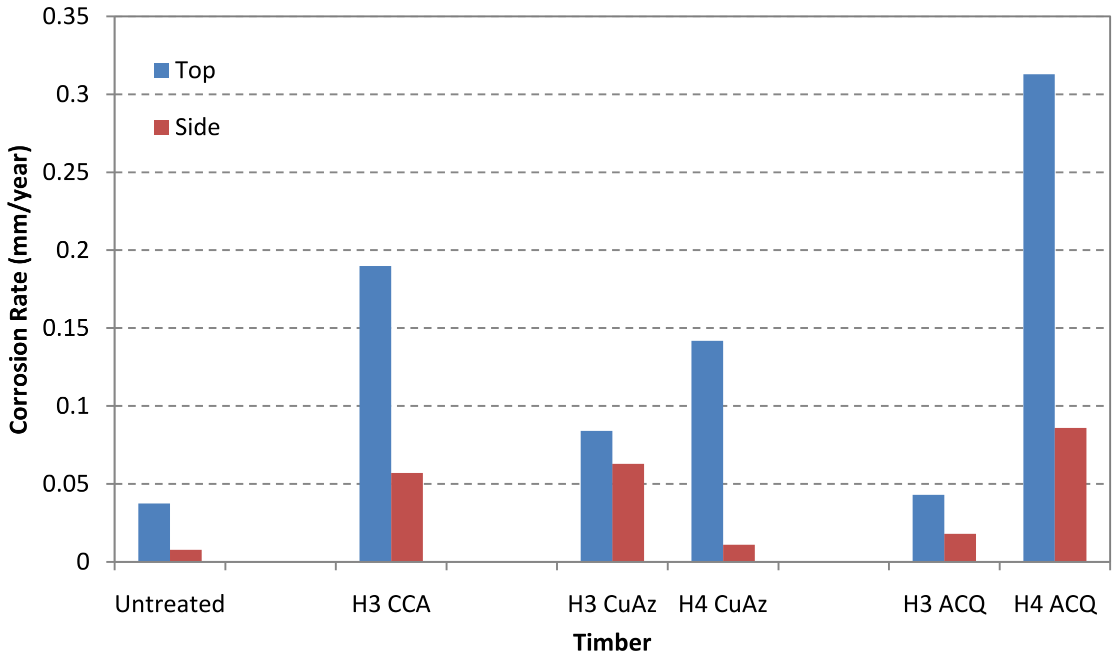


Figure 9. Corrosion rate of hot dip galvanised nails (Trial 1).

The most severe corrosion occurred on both mild steel and hot dip galvanised steel nails in timber treated with ACQ to an H4 level (see Figures 9-10). This was consistent with the findings of previous studies. However, all nails inserted into H3 ACQ treated timbers showed very low corrosion rates. The corrosion rates of mild steel and hot dip galvanised steel nails in H3 ACQ treated timbers were only ~15% of those in H4 ACQ treated timbers. This phenomenon has not been observed previously within accelerated and non-accelerated tests.

H3 ACQ / Top

H4 ACQ / Top

Figure 10. Surface morphologies of the nails retrieved from ACQ treated timbers (nails inserted into top surface of the timber block).

H3 CCA / Top **H3 CuAz / Top** **H4 CuAz / Top**

Figure 11. Surface morphologies of the nails retrieved from H3 CCA, H3 and H4 CuAz treated timbers (nails inserted into top surface of the timber block).

In general, timbers treated with CuAz were more corrosive than those treated with CCA under identical testing conditions. However, this behaviour was not reflected by the results of this first test (see Figure 11).

In most cases, the nails embedded into the side part of the timber block had a lower weight loss than those inserted from the top. This was fully supported by morphological observations (see Figure 12).

Top **Side**

Figure 12. Surface morphologies of the hot dip galvanised nails embedded into H4 CuAz treated timber.

From the corrosion rates measured using this single-stage testing method, it was difficult to determine the relative aggressivity of timbers treated with different preservation chemicals towards the most commonly used metallic fasteners (mild steel and zinc coated steel). Although this test confirmed that H4 ACQ treated timbers had the highest corrosivity when compared with untreated, CCA, and CuAz treated timbers, it did not reproduce the actual corrosivity of the CuAz and H3 ACQ treated timbers seen in field exposure. On the other hand, this test demonstrated that the corrosion behaviours of the nails inserted from the top and side surfaces were quite different. This agreed well with observations from field exposure tests.

4.3 Second Trial – Two Stage Test

4.3.1 Sample configuration and chamber operational condition

The configuration for the timber-nail assembly used in the second trial was exactly the same as that used in the first trial.

The major difference between this trial and the first trial was the operation of the fog chamber. In this test, two stages were introduced. One had a lower air drying temperature of 25°C while the other had a higher air drying temperature of 35°C. The aim was to create two different moisture content levels in the timbers to better simulate natural exposure cycles (dry and rainy seasons.

In the first stage, one complete cycle consisted of two sub-cycles: water spraying at 25°C for 6 hours followed by air drying at 25°C for 18 hours. A total of 30 cycles were completed (i.e., 720 hours). This stage was immediately followed by the second stage. In this stage each cycle comprised two sub-cycles: water spraying at 25°C for 6 hours and then air drying at 35°C for 18 hours. This stage also lasted for 720 hours. Consequently, this test lasted for a total of 1440 hours.

4.3.2 Corrosion rate

The corrosion rates of the mild steel and hot dip galvanised steel nails calculated from their weight losses after the two-stage exposure are presented in Figures 13 and 14.

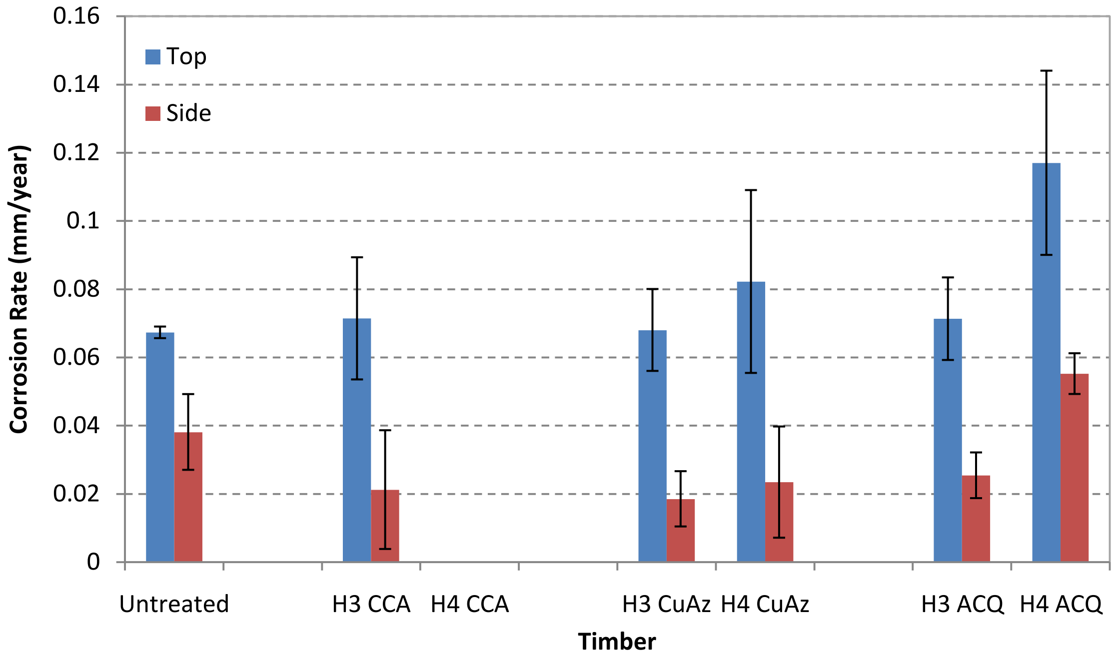


Figure 13. Corrosion rate of mild steel nails (Trial 2).

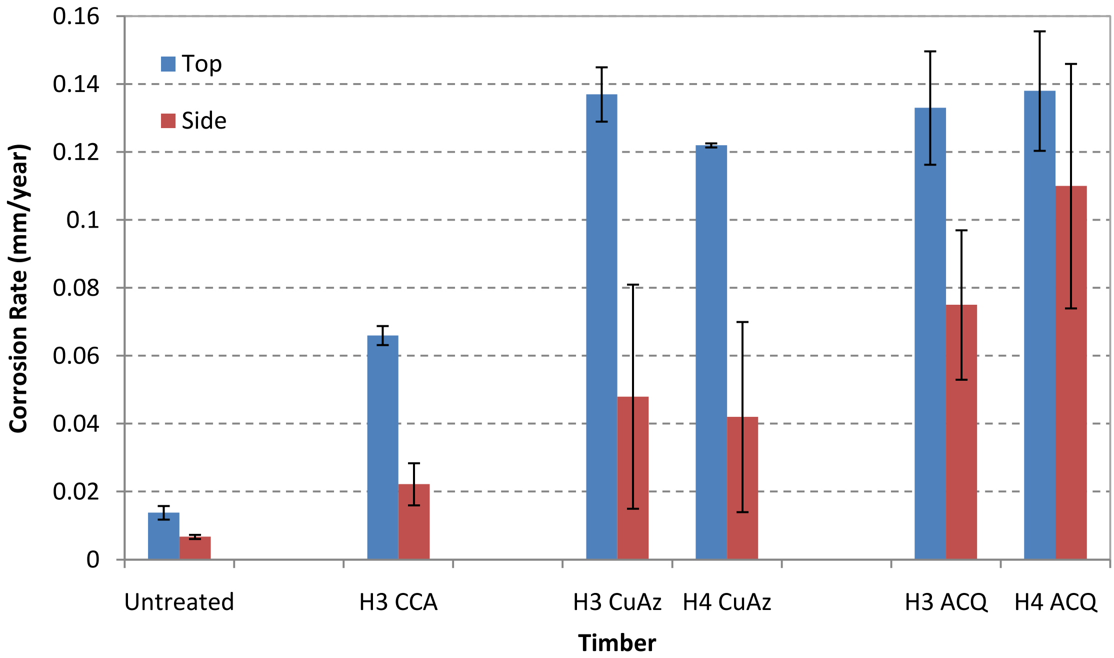


Figure 14. Corrosion rate of hot dip galvanised nails (Trial 2).

Based on the corrosion rates of mild steel nails, the test indicated that the aggressivity of timbers was related to their preservation treatment. H4 ACQ treated timber was the most corrosive, while timber treated with CuAz had a slightly lower corrosivity that was similar with timber treated with CCA and untreated timber. This is partly supported by morphological observations (see Figure 15). In addition, the trend of corrosivity with preservation treatment exhibited by the nails inserted from the top of the timber was very similar to that exhibited by the nails inserted into the timber from the side.

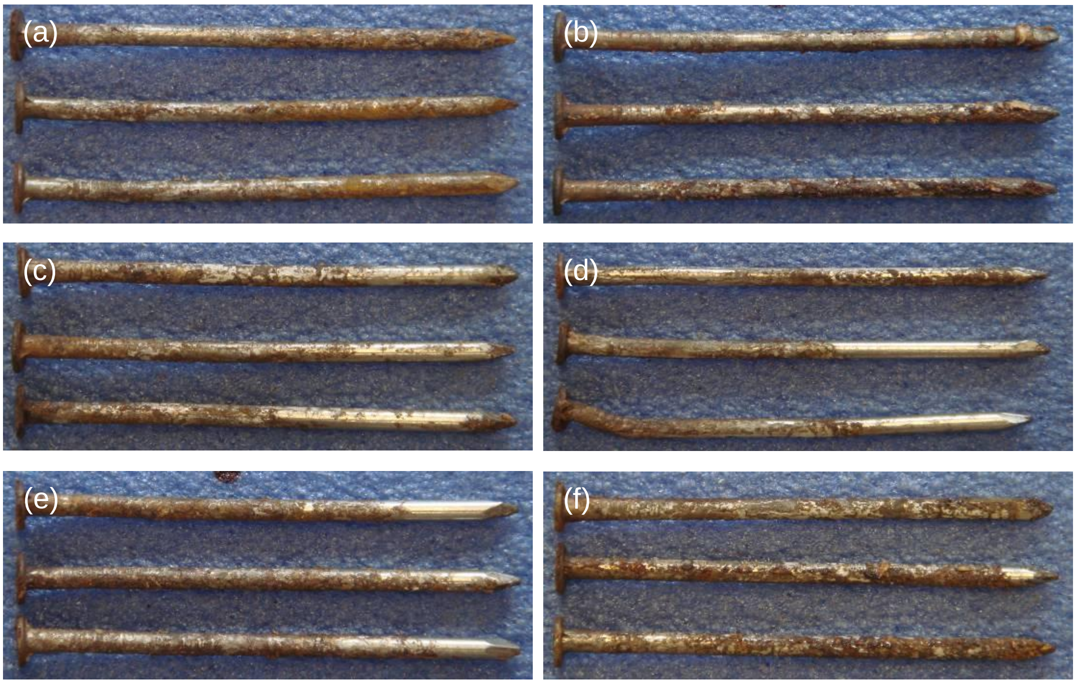
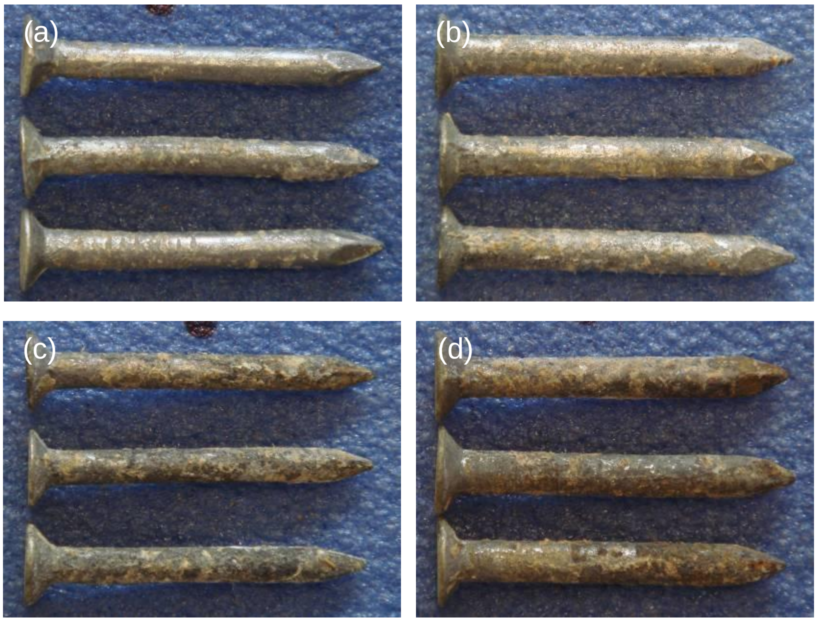


Figure 15. Surface morphologies of mild steel nails inserted into timber blocks from the top: (a) untreated, (b) H3 CCA, (c) H3 CuAz, (d) H4 CuAz, (e) H3 ACQ and (f) H4 ACQ.



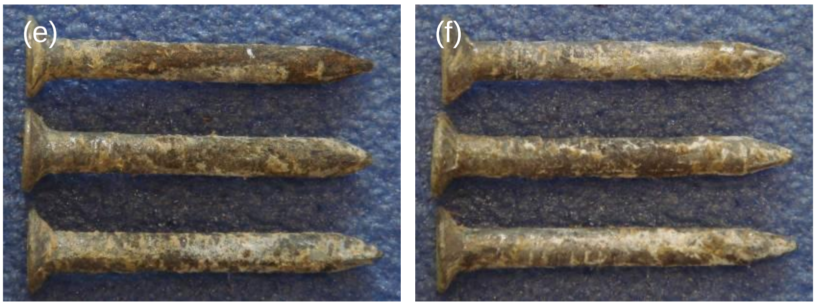


Figure 16. Surface morphologies of hot dip galvanised nails inserted into timber blocks from the top: (a) untreated, (b) H3 CCA, (c) H3 CuAz, (d) H4 CuAz, (e) H3 ACQ and (f) H4 ACQ.

The correlation between timber corrosivity and preservation treatment given by the corrosion rates of the hot dip galvanised nails also agreed well with that of the mild steel nails. The only difference was the untreated and CCA treated timbers had a much lower aggressivity when compared with the CuAz and ACQ treated timbers (see Figure 14). The nails inserted into the timber from the top and the side also showed an almost identical trend between corrosion rate and timber treatment.

In comparison with the corrosion rate measurement results obtained from the single stage trial, it was clear that:

1. H4 ACQ treated timbers exhibited the highest aggressivity towards mild steel and hot dip galvanised steel nails; and
2. The nails inserted into the timber blocks from the side always had a lower corrosion rate than those inserted into the same timber block but from the top (compare Figure 15 (c-d) with Figure 17).

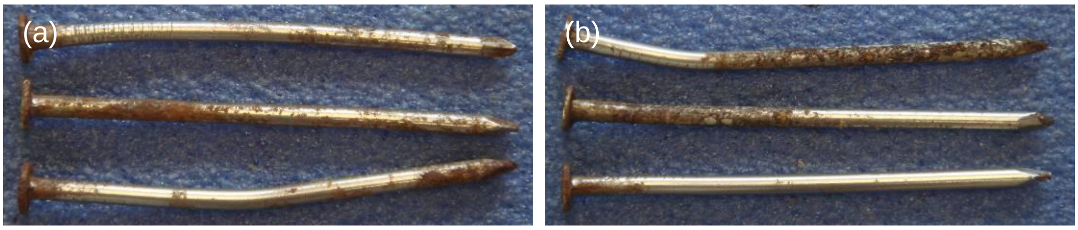


Figure 17. Surface morphologies of mild steel nails embedded into the H3 (a) and H4 (b) CuAz treated timbers from the side.

The corrosion rates derived from the two-stage trial were lower than those derived from the single-stage trial and also showed a clearer trend between timber aggressivity and timber treatment.

4.4 Third Trial – Two Stage Test

4.4.1 Sample configuration and chamber operational condition

In the third trial, the mild steel nails and hot dip galvanised nails were inserted into the timber top surface in a different pattern. It was thought that the original configuration might have had some negative influences on the corrosion properties of hot dip galvanised steel nails, introducing errors in the corrosion rate calculations. During the water spraying stage, it had been observed that a thin and continuous water film was developed on the timber top surface. It is possible that iron ions released from the corrosion of the mild steel nails might have diffused within this film. Some of the ions might have arrived at the surface of the hot dip galvanised nails and contributed to slightly enhanced zinc corrosion through the galvanic effect.

Mild steel and hot dip galvanised steel nails were then grouped separately and driven into different timber sections. A shallow groove was cut between these two sections to avoid the possibility of a continuous water film on the top surface (shown in Figure 18).

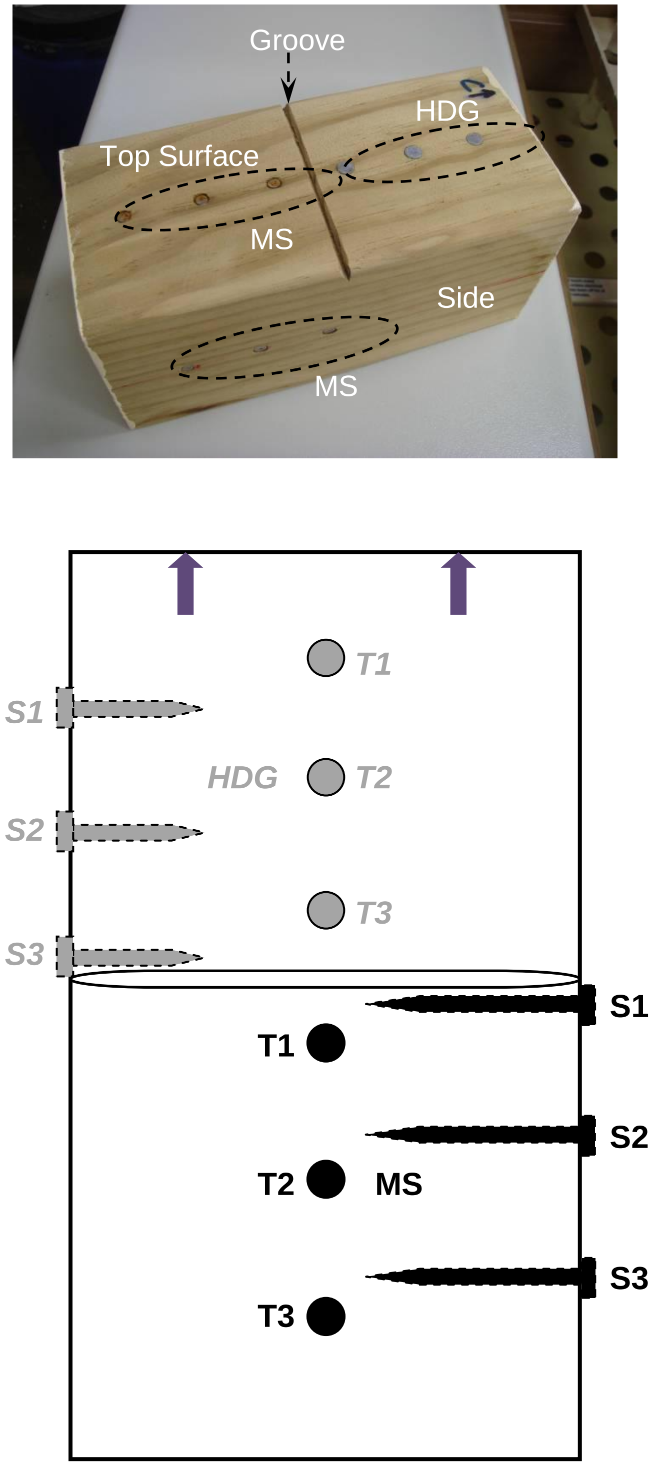


Figure 18. Sample configuration for the third trial.

It is well known that corrosion rates can be significantly affected by the time at which an atmospheric exposure starts [Wypych 2008]. For example, if the exposure was started during the winter there is little UV in the solar irradiation. If the exposure was started in summer, the sample may be subjected to more intense UV irradiation. This may cause more degradation not only in that first summer but also in the following winter.

The ambient temperatures in rainy and dry seasons are also quite different. Consequently, timber moisture contents in these two seasons will be somewhat different. Moisture content is critical to the corrosion of metal embedded into timber; any change in the initial stage of exposure may result in observable changes in metal corrosion behaviour. To investigate this, the arrangement of the drying stage was slightly modified in the third trial.

This third trial also had two stages. In the first stage, one complete cycle consisted of two sub-cycles: water spraying at 25°C for 6 hours and air drying at 35°C for 18 hours. A total of 30 cycles were completed (i.e. 720 hours). This stage was immediately followed by the second stage in which one cycle included water spraying at 25°C for 6 hours and then air drying at 25°C for 18 hours. This stage also lasted for 720 hours. Consequently, this test lasted for 1440 hours in total. Hence, this trial was different from the second trial in the arrangement of the drying stage. Its first stage had a higher drying temperature while the first stage of the second trial had a lower drying temperature.

It should be mentioned that the field exposure test at Judgeford site was started at September. The timber blocks might have a high initial moisture content.

4.4.2 Corrosion rate

The corrosion rates of the mild steel nails derived from this modified two-stage exposure are presented in Figure 19.

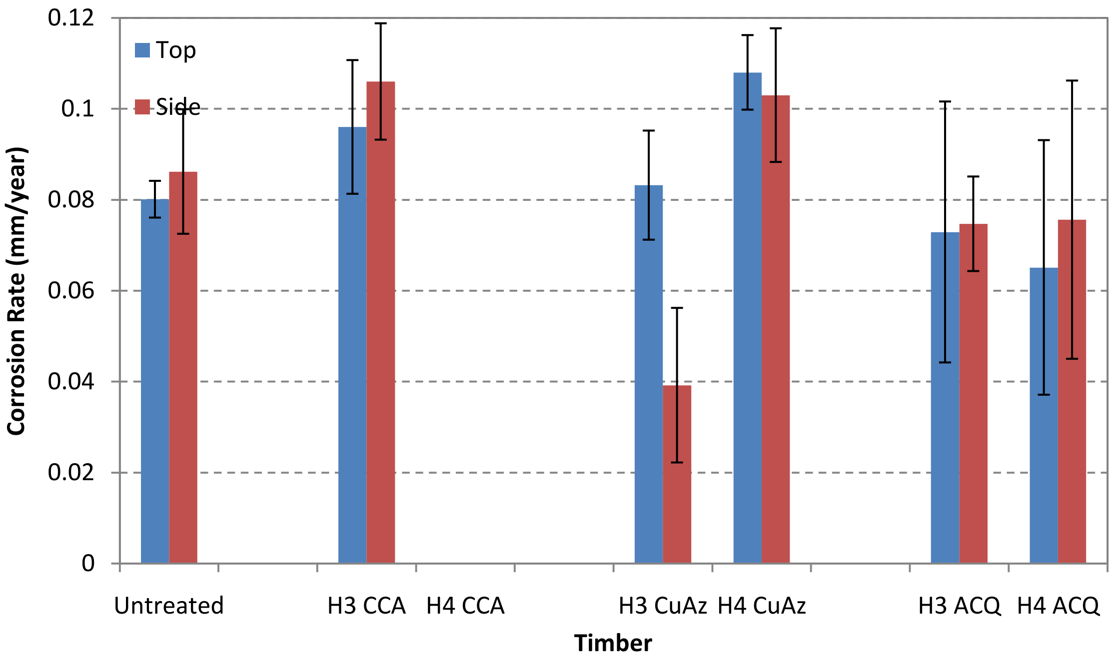


Figure 19. Corrosion rate of mild steel nails (Trial 3).

It was found that the timbers treated with CCA and CuAz had a very similar aggressivity towards mild steel nails. Also, their aggressivity was only slightly higher than the untreated timber. This correlation between timber aggressivity and preservation treatment is very similar to that observed in the second trial. However, Figure 19 implies that the timbers treated with H3 and H4 ACQ were less corrosive when compared with timbers treated with other preservatives (see Figure 20 for a morphological comparison). This result does not agree with the results obtained in the second trial or in the field exposure tests.

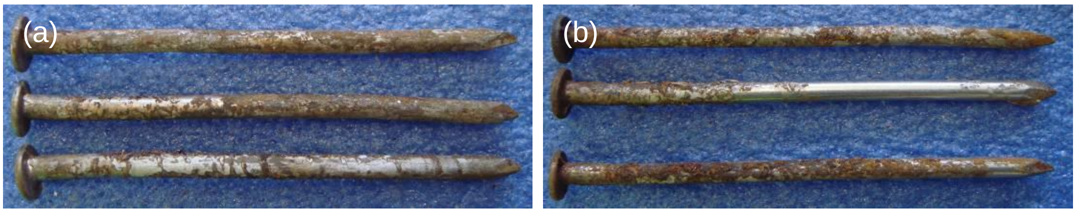


Figure 20. Surface morphologies of mild steel nails embedded into the H4 CuAz (a) and H4 ACQ (b) treated timber top surfaces.

Figure 19 also demonstrates that the nails inserted into the side surface of the timber block corroded at a slightly higher rate than those inserted into the top surface (see Figure 21 for a surface morphological comparison), the exception was for those in CuAz treated timbers (see Figure 22). This phenomenon is unusual and differs from the results from the second trial and from the field exposure.

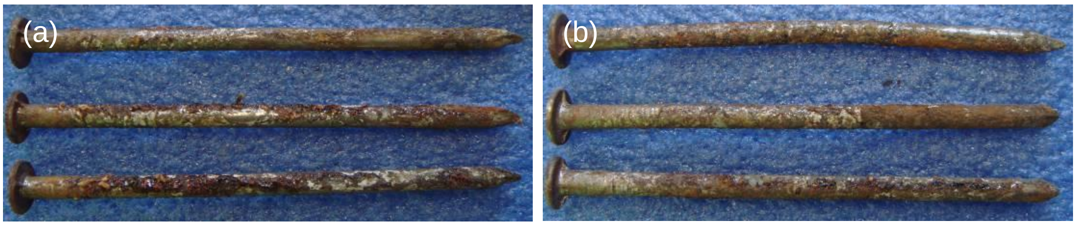


Figure 21. Surface morphologies of mild steel nails embedded into the H3 CCA treated timber from the top (a) and side (b) surfaces. Corrosion products were very similar on these nails.

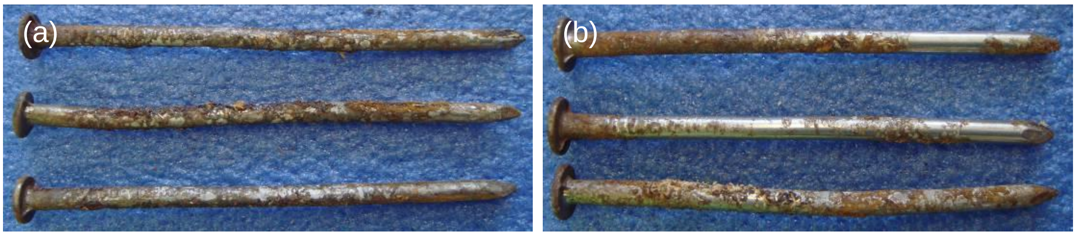


Figure 22. Surface morphologies of mild steel nails embedded into the H3 CuAz treated timber from the top (a) and side (b) surfaces.

Corrosion of hot dip galvanised steel nails showed a different trend with respect to timber preservation treatment when compared with mild steel nails (see Figure 23). The untreated and H3 CCA treated timbers exhibited a similar aggressivity towards HDG nails. The timbers treated with CuAz and ACQ attacked the HDG nails at very similar rates and these rates were much higher than those observed in the timbers treated with CCA. These observations are similar with those obtained in the second trial and are supported by surface morphological characterisations (see Figure 24). In field exposure tests, timbers treated with H4 ACQ generally had an aggressivity that was 1.5 to 2 times higher than those treated with H3 ACQ under identical testing conditions. This effect was not reproduced during the third trial.

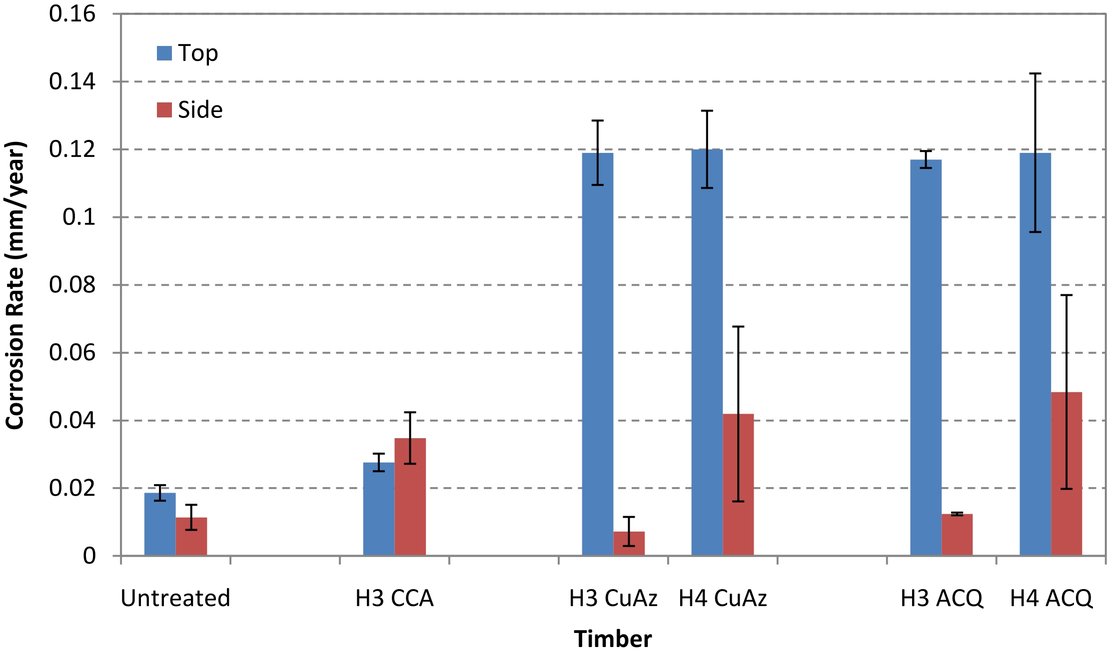


Figure 23. Corrosion rate of hot dip galvanised nails (Trial 3).

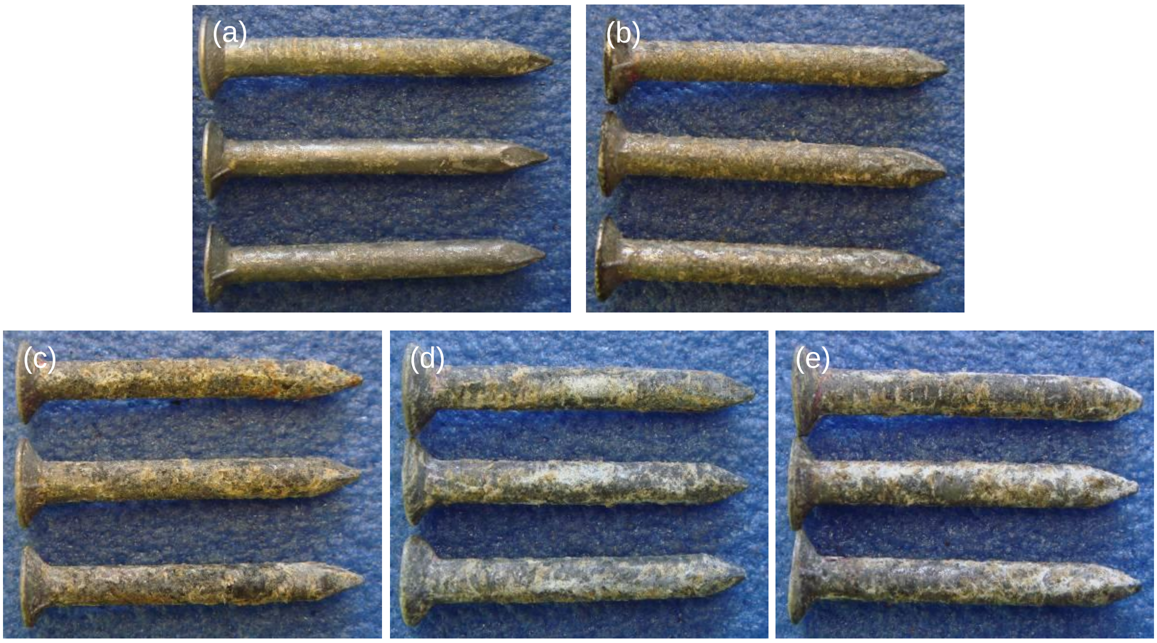


Figure 24. Surface morphologies of hot dip galvanised nails inserted into timber block from the top: (a) untreated, (b) H3 CCA, (c) H3 CuAz, (d) H3 ACQ and (e) H4 ACQ.

The surface morphological results, shown in Figure 25, clearly demonstrated that nails inserted into the timber block from the side surface were corroding at a lower rate than those inserted into the block from the top surface. This finding agrees well with the results obtained from the BRANZ field tests.

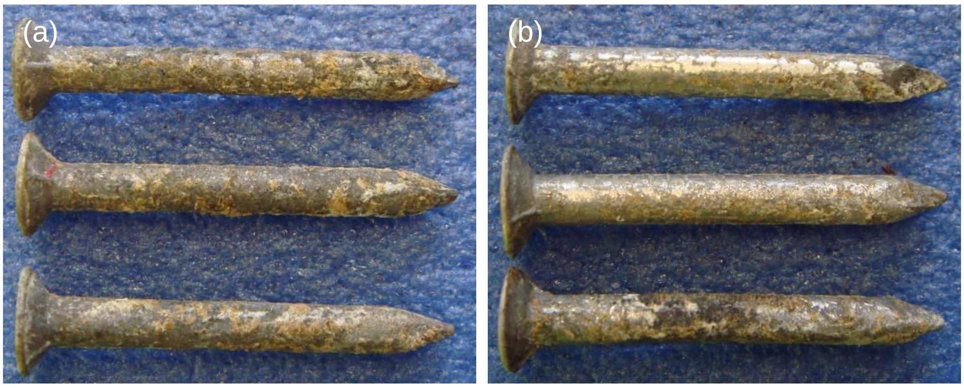


Figure 25. Surface morphologies of hot dip galvanised nails inserted into H4 CuAz timber block from the top (a) and side (b) surfaces.

5. DISCUSSION

5.1 Single Stage Test

Results obtained from the first trial showed that the corrosion rates measured with this single stage test were lower than those measured by the AWPA E12 recommended procedure and the BEFT method, and then more close to those obtained from the field exposure tests. This could be partially attributed to the lower temperatures used in this trial (25^{o}C during water spraying and 35^{o}C during air drying). Both AWPA and BETF tests employ a much higher temperature of 49^{o}C. This implies that a high testing temperature could accelerate corrosion processes of metal in timber, but can lead to serious over-estimation of corrosion rate.

It was shown that this single stage test had difficulty in determining the relative aggressivity of timber towards metal. The results indicated that timbers treated with H4 ACQ had the highest corrosivity which could be two times higher than that of the timbers treated with CCA. However, these results indicated that timbers treated with CuAz had a lower aggressivity towards mild steel and hot dip galvanised nails than those treated with CCA. This is not supported by findings from the field exposure tests, but is somewhat similar to the trends observed under the AWPA test. This might explained by the following:

1. The environmental conditions established by the single state test might only correspond to one specific season of a year which has several seasons. Corrosion of metal, a process that is strongly influenced by timber moisture content, was not be fully simulated. The seasonal variation of moisture content of timber exposed to atmosphere could not be reflected.
2. During this test it was observed that the sealing of the ends of the timber blocks partly failed, leading to the formation of cracks. These physical defects could provide fast routes for inward diffusion of moisture. This modified moisture transportation might change moisture-driven processes in the timber, therefore affecting metal corrosion.

In consideration of these observations, a single stage test might not be appropriate for corrosion tests and multi-stage tests would be better.

5.2 Comparison of Corrosion Rates Obtained in the Second and Third Trials

Both the second and third trials were two-stage tests with the difference being the sequential arrangement of the wetting and drying stages. The second trial had a lower drying temperature, 25°C, in the first stage, and a higher temperature of 35°C in the second stage. The arrangement in the third trial was reversed. The main purpose of this was to determine the potential influence of starting conditions on the corrosion performance of nails in treated timbers. A comparison of the corrosion rates derived from the second and third trials is given in Figures 26 and 27.

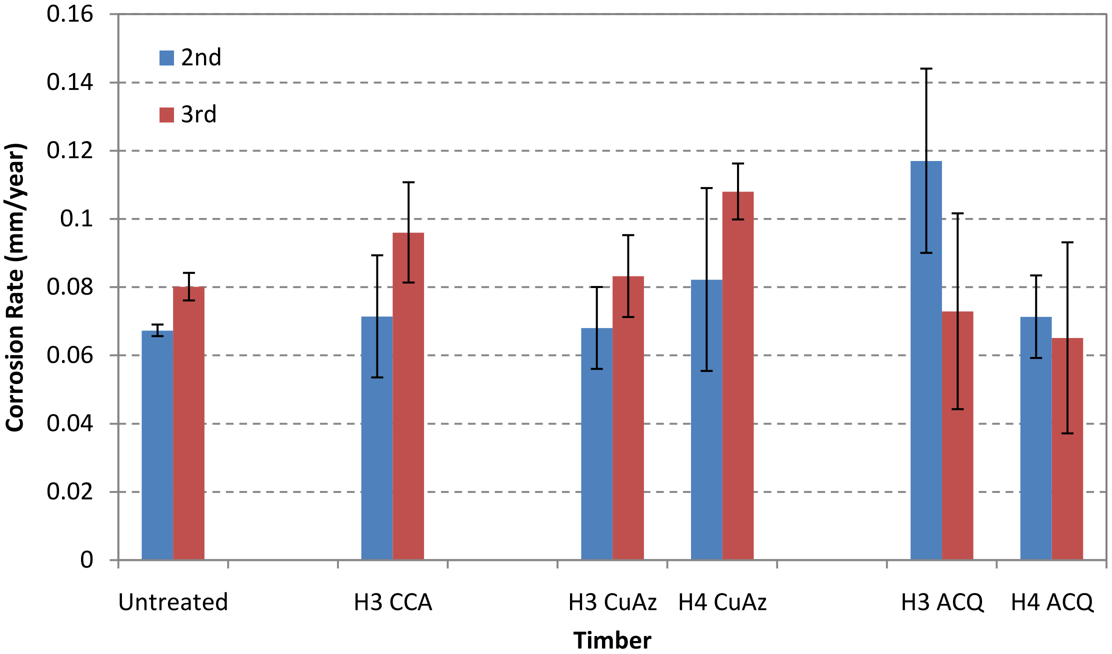


Figure 26. Comparison of corrosion rates of mild steel nails (Trials 2 and 3) (nails inserted into top surface of the timber block).

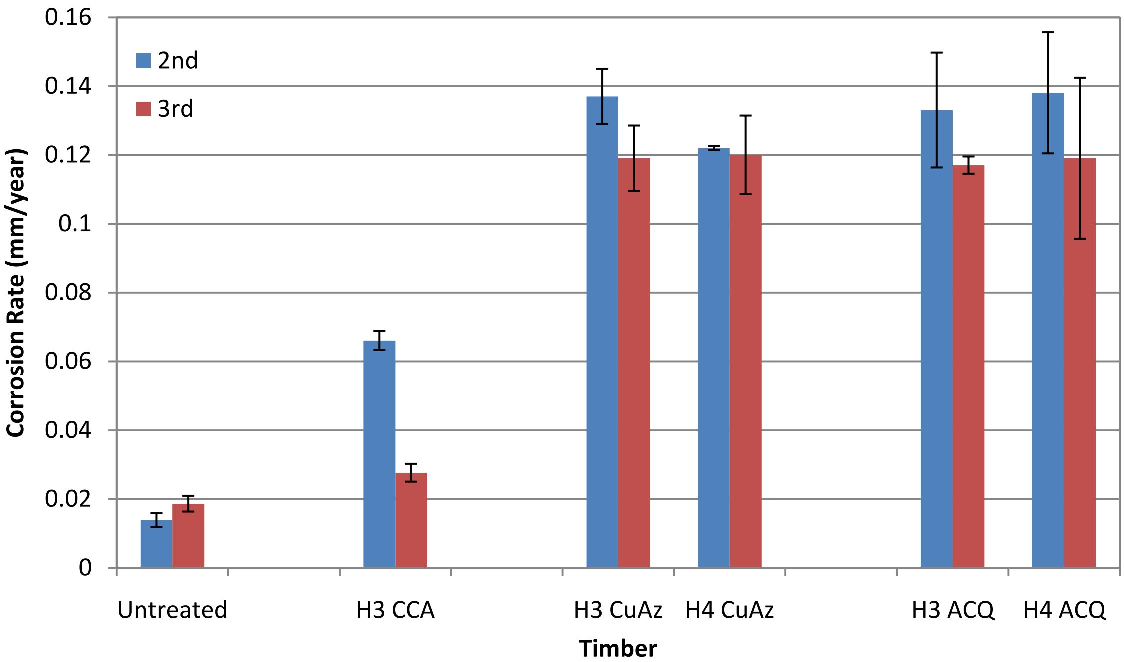


Figure 27. Comparison of corrosion rates of hot dip galvanised nails (Trials 2 and 3) (nails inserted into top surface of the timber block).

From the results shown in Figures 26 and 27, it can be seen that the exact influence of starting conditions on the corrosion of mild steel nails is not very clear. In untreated timber and timbers treated with CCA and CuAz, it appeared that a higher drying temperature in the first stage led to higher corrosion rates. However for mild steel nails embedded into ACQ treated timbers and hot dip galvanised nails in most timbers (except the untreated timber), the situation was different. For these a higher drying temperature in the first stage corresponded to a lower corrosion rate. These observations may support the hypothesis that the starting condition does have an influence on the corrosion processes of the nails inserted into timber, but this influence might be controlled by many factors, such as type of material, corrosion mechanism and characteristics of corrosion products.

Moisture content is fundamental to the corrosion of metals in timbers. In general, a higher moisture content is expected to contribute to a higher corrosion rate if other conditions are not significantly different. In the third trial in this study, a higher drying temperature, 35^{o}C, was used in the first stage. This resulted in a lower moisture content in the timber when compared with the first stage in the second trial. A lower moisture content in the first stage would probably lead to slower corrosion processes. For hot dip galvanised nails, a lower corrosion rate in the first stage may indicate less damage to the zinc coating (i.e. less reduction of coating thickness). Retaining the zinc coating provides protection to the underlying substrate when exposed to the second more aggressive stage.

However this mechanism might not occur for nails made of mild steel. Corrosion of mild steel in timbers, particularly in timbers treated with waterborne copper-bearing preservatives, is severe. Corrosion products formed on mild steel have high concentrations of chemical and/or physical defects and provide very limited protection to the steel substrate. Additionally, surface morphological characterisations found that most of the corrosion products developed on the mild steel nails did not fully cover the surfaces (this is as expected given the relatively short testing duration). Under these conditions, the corrosion process occurring on the mild steel might not be significantly affected by the test starting conditions.

In addition, a higher drying temperature may contribute to a higher corrosion rate in the first stage due to the enhanced mass transportation. It may also increase the rate of reaction.

The moisture content gradient in the timber block may also affect the corrosion processes occurring on the nails. The timber blocks used in this study had typical cross-sectional dimensions ~90×90 mm. Wetting by water spray was mainly from the top surface. The section close to the top surface would have a higher moisture content than the core. During the drying stage, the moisture in the near-surface section would be expelled more easily, resulting in a larger moisture content variation during the test.

The hot dip galvanised nails used in this study had a shorter length (~30 mm) than the mild steel nails (~50 mm). Corrosion on the HDG nails would be expected to be more readily affected by the experimental conditions. Any changes in the timber moisture content would be reflected by the measured corrosion rates since the whole surface would be affected. On the longer mild steel nails, a moisture content gradient may exist and wetting and drying may only influence the corrosion on part of their surface. This moisture content gradient may also induce an oxygen gradient that will affect anodic and cathodic processes on different surface sections, introducing more uncertainties.

5.3 Comparison of Corrosion Rates Obtained with Different Methods

Corrosion performance of mild steel and hot dip galvanised steel nails has previously been assessed by BRANZ using different testing methodologies, including AWPA E12, BEFT (BRANZ Embedded Fastener Test) and field exposure.

5.3.1 Corrosion rates of mild steel nails obtained with AWPA, BEFT and field exposure

From Figure 28, it can be seen that after one year exposure at BRANZ's Judgeford site, the comparative corrosivity of timber treatment follows a clear trend:

H3 CCA ≈ H4 CCA < H3 CuAz < H4 CuAz < H3 ACQ < H4 ACQ.

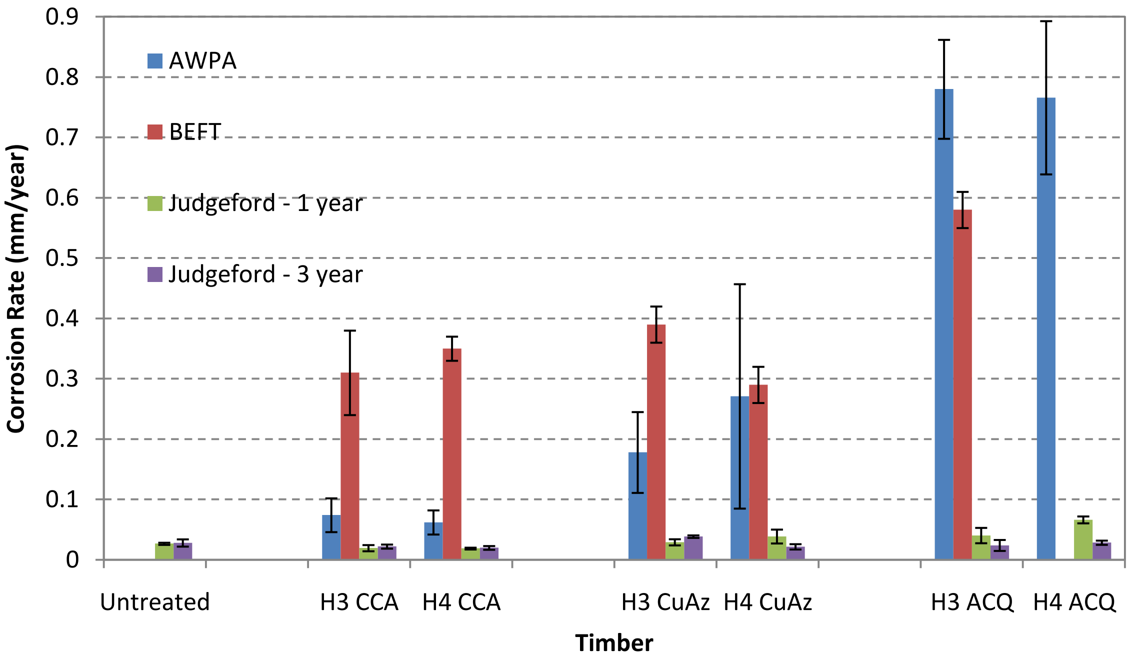


Figure 28. Corrosion rates of mild steel nails measured by accelerated and field exposure tests.

Corrosion in H4 ACQ treated timber was 3.5 times higher than that in H4 CCA treated timber. After three years of exposure, this trend slight changed to:

H3 CCA ≈ H4 CCA < H3 CuAz > H4 CuAz < H3 ACQ < H4 ACQ.

Therefore H3 CuAz treated timber exhibited the highest aggressivity towards mild steel. While timbers treated with CuAz and ACQ were still more corrosive than CCA treated timbers, the difference was smaller. For example, the corrosivity of H4 ACQ treated timber was only 1.9 times higher than that of the H4 CCA treated timber.

Tests following AWPA E12 showed a similar trend on the comparative timber corrosivity with that observed after one-year field exposure by BRANZ. One difference is that H3 ACQ was measured to have the highest aggressivity. In addition, the corrosivity difference between the timbers treated with different preservatives was also amplified. For example, H4 ACQ treated timber was measured to be 12.4 times more aggressive than H4 CCA treated timber.

BEFT testing derived another trend on the comparative aggressivity of timbers (no data was available for H4 ACQ treated timber):

H3 CCA < H4 CCA < H3 CuAz > H4 CuAz < H3 ACQ.

Therefore H3 ACQ exhibited the highest corrosivity while the H4 CuAz showed the lowest corrosivity. The H3 ACQ treatment was 1.9 times more corrosive than H3 CCA treatment. This value was lower than that derived from the one-year field exposure but very close to the three-year exposure result.

However, it must be noted that the corrosion rates derived from both AWPA E12 and BEFT tests were much higher than those measured from the field exposure. For example, the corrosion rate measured by the AWPA method for an H4 ACQ treatment could be 12 and 27 times higher than those measured after one and three years of field exposure, respectively. Meanwhile the corrosion rate measured by the BEFT method for an H3 CCA treatment could be 16 and 14 times higher than those measured after one and three years of field exposure, respectively.

5.3.2 Corrosion rates of hot dip galvanised nails obtained with AWPA, BEFT and field exposure

Figure 29 shows that AWPA E12 tests may have difficulty in determining the comparative corrosivity of treated timber. The corrosion rates of hot dip galvanised steel nails in CuAz treated timbers measured with this method were two to three times lower than those measured in timbers treated with CCA. Similar observations apply with mild steel nails, H4 ACQ treatment was showing a lower aggressivity towards HDG nails than H3 ACQ. The corrosion rate in H4 ACQ treatment was 69% of that in H3 ACQ treatment. This test method also tended to over-estimate the occurrence of corrosion processes. The corrosion rates in timbers treated with H3 ACQ were 19 and 15 times higher than those derived from one and three years field exposures, respectively.

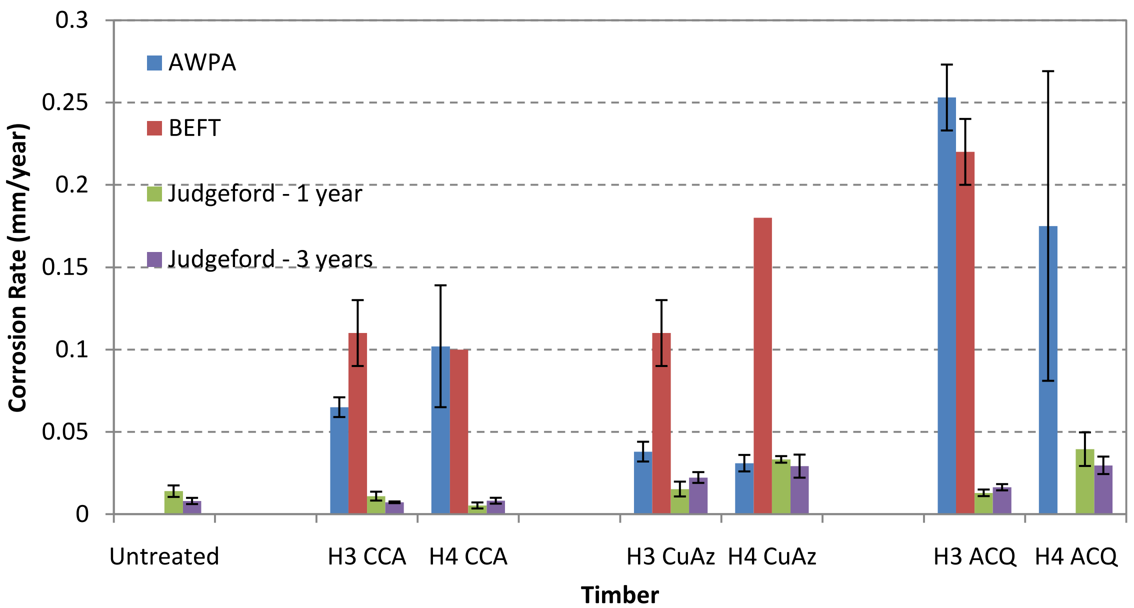


Figure 29. Corrosion rates of hot dip galvanised nails measured by accelerated and field exposure tests.

Comparatively, tests based on BEFT could reflect the comparative aggressivity of timber treatment towards HDG nails in a more reliable way. However, the corrosion rates derived could still be many times higher than those obtained from field exposure tests.

5.3.3 Corrosion rates obtained with the current method and field exposure

The corrosion rates derived from the accelerated two-stage tests used in this study indicate that ACQ and CuAz treated timbers could be more aggressive than CCA treated and untreated timbers. However, the corrosivity enhancement by these two preservation treatments over CCA treatment was not as significant as those observed with the AWPA or BEFT methodologies.

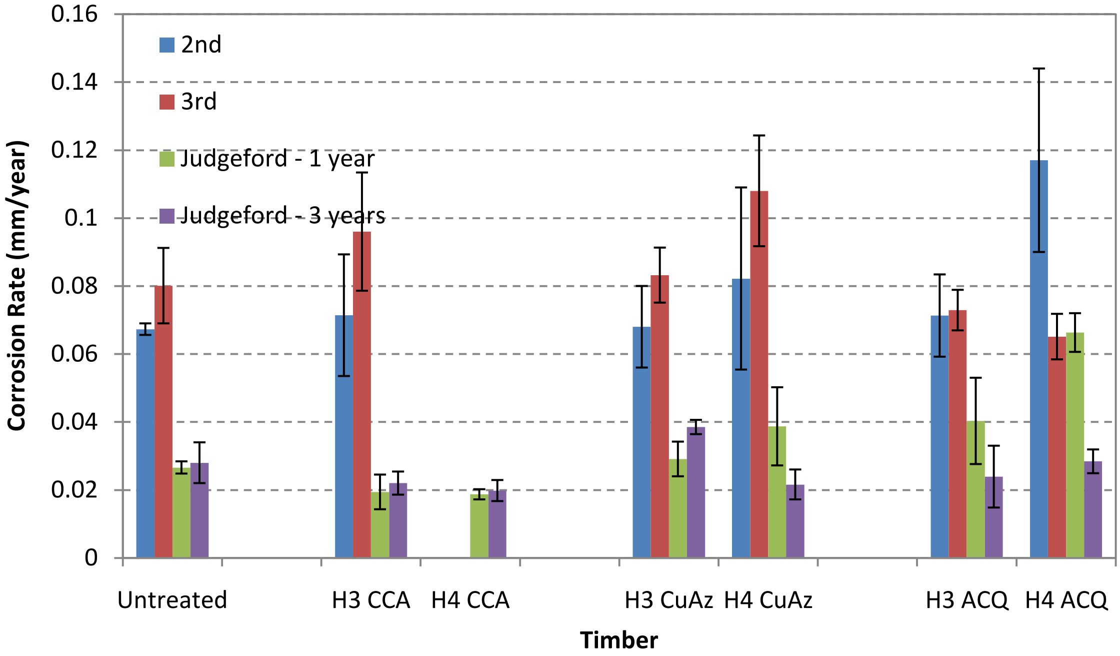


Figure 30. Corrosion rates of mild steel nails measured by accelerated two-stage and field exposure tests.

The third trial produced a different outcome with mild steel nails having a slightly lower corrosion rate in H3 ACQ treatment than in other treatments. This finding is somewhat similar to the observations obtained from the nails after three years of exposure at Judgeford. In the Judgeford field test, H4 ACQ and H4 CuAz treatments were only 1.4 and 1.1 times more corrosive than H4 CCA treatment. These enhancement levels are very similar with those calculated from the results of the second and third trials. However, it must be noted that corrosivity enhancement by ACQ and CuAz when compared with CCA treatment is more obvious based on the results derived from the one-year field exposure.

One encouraging observation is that the corrosion rates of mild steel nails derived from these two-stage accelerated tests were, in general, two to four times higher than those obtained from the field exposures. This indicates that the corrosion processes occurring under the present experimental conditions are more representative of those occurring on nails embedded into timbers exposed in the field.

Accelerated two-stage tests indicated that CuAz and ACQ treatments had a similar aggressivity towards hot dip galvanised nails. This behaviour aligns well with field tests, particularly with the results of the three-year exposure. Their aggressivity could be two to four times higher than CCA treatment, while the corrosivity enhancement of CuAz and/or ACQ over CCA was typically around two to seven times based on the field exposure tests. However, the two-stage test also over-estimated the aggressivity of the treated timbers. The corrosion rates derived from these tests could be three to ten times higher than those of field tests, although these enhancement values are lower than those (which can be as high as nineteen times) shown by AWPA and/or BEFT tests.

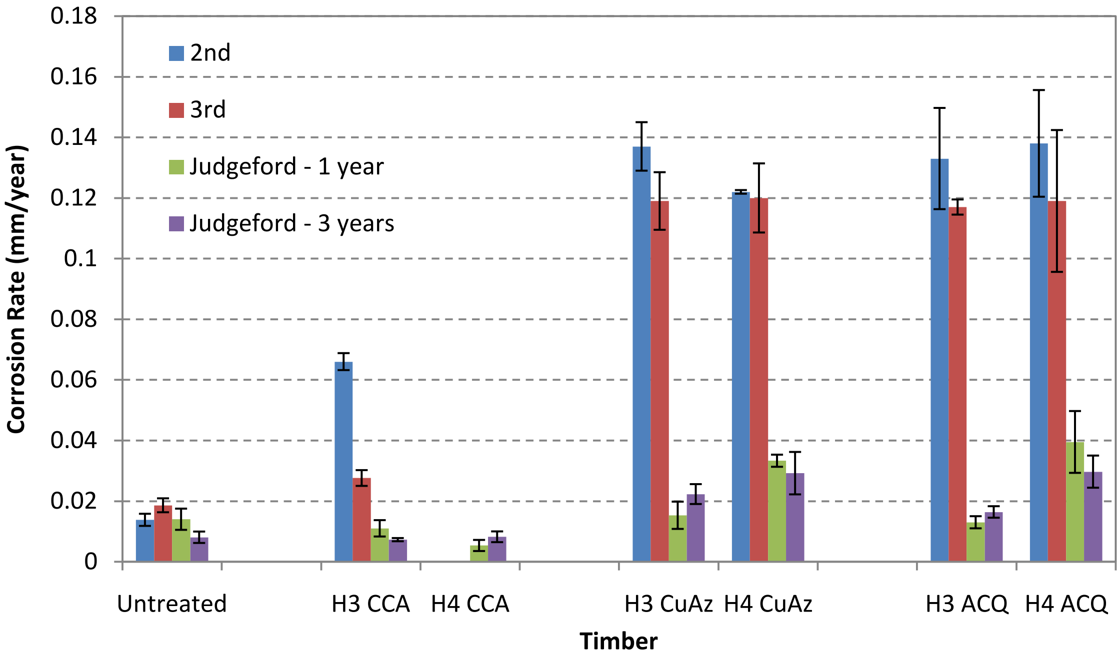


Figure 31. Corrosion rates of hot dip galvanised nails measured by accelerated two-stage and field exposure tests.

It is believed that the lowered corrosion rate over-estimation by these two-stage tests are related to:

1. lower operational temperatures (25°C and 35°C) that are more close to ambient temperature,
2. better simulation of timber moisture content and particularly its variation with season and of physical changes of timber, and
3. establishment of environmental conditions that could create a realistic micro-environment at the metal-timber interface for the initialisation and progress of corrosion.

In general, the correlation between the corrosivity and the type of timber treatment demonstrated by the corrosion rates measured using the nails inserted into the timber blocks from the side surface is somewhat similar with those obtained from the field exposure tests of two or three years duration. This is particularly true within the second trial reported here.

6. CONCLUSIONS

A two-stage testing methodology, consisting of sequential wetting and drying cycles, has been established and used to evaluate the comparative corrosivity of timbers treated with different preservatives by measuring the corrosion rates of mild steel and hot dip galvanised steel nails. The results showed that the increased timber aggressivity of CuAz and ACQ treatments can be reflected by this method. The corrosion enhancement level of CuAz and ACQ treatment over CCA treatment measured by this method is similar with that measured in field exposure tests. This method and other accelerated methods, e.g. AWPA E12 and BEFT, over-estimate the corrosion rate of nails, but it is clear that the corrosion rates derived from this two-stage method are closer to those obtained in the field tests. Further refining of the two-stage testing methodology could further enhance the reliability of the results.

7. LESSONS LEARNED

Corrosion of metal in timbers exposed to the atmosphere is an extremely complicated process since many environmental factors can exert an influence. The moisture content of the timber is always regarded as one of the most important factors and is itself a highly dynamic process. Both moisture content and, therefore, corrosion of metals are affected by dimensions, orientation, surface finish, preservation treatment, seasonal variations and local climatic conditions [Lebow and Lebow 2007]. A testing method that aims to evaluate the performance of metallic components embedded in timbers should be capable of simulating the wetting and drying behaviour of timber exposed to the service environment in an appropriate way. To achieve this, the target range of moisture contents must be established using a reliable base line. Moisture contents and their variations had previously been measured with the timber blocks exposed at BRANZ's Judgeford site. The results obtained were used in the design of this study. However, these measurements were not complete and cannot accurately reflect the seasonal variations of moisture content and/or timber temperature. More research is required to provide the data necessary to develop a better understanding of the changing behaviour of timber moisture content, a climate specific parameter.

Furthermore, the temperature of the exposed timbers should be monitored seasonally since corrosion kinetics are temperature sensitive. A better understanding of seasonal variation of timber temperature could lead to a more reliable set-up of the operation temperatures in the wetting and drying stages.

Wetting of test timber was achieved, in the present study, through water spray in a fog chamber. Water droplets generated by the spray nozzle were extremely fine and wetted the timber slowly. At the same time, the side surfaces of the timber blocks had a low probability of being wetted. This wetting behaviour does not mirror that of natural precipitation and therefore the value and gradient of moisture content in the test timber might be somewhat different from that of timbers used in field tests. A different wetting method might overcome this effect.

The timber blocks used in the present study have similar cross-sectional dimensions to those exposed at Judgeford. However the nails used in this study were shorter than those used in the field exposure tests. The nails used in the field tests had a length of ~60 mm, while the mild steel and galvanised nails used in this study were ~50 and ~30 mm long, respectively. In consideration of the moisture gradient along the depth of the timber block, the length difference may lead to differences in their corrosion performance.

In planning tests the embedding and grouping of nails should be considered together with the dimensions of the timber blocks to prevent interplay of corrosion on adjacent nails. Separation of different types of nails can be achieved through barriers to ion diffusion. The shallow groove used in the present study might not be the most appropriate as it damaged the timber surface integrity and potentially provided a path for fast inward penetration of water.

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