Matching Display Relative Humidity to Corrosion Rate: Quantitative Evidence for Marine Cast Iron Cannon Balls

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Abstract

Cast iron cannon balls excavated from the wreck of King Henry VIII flagship, the *Mary Rose*, have been actively corroding on mixed material display at 55% relative humidity (RH). A Cardiff University study has examined corrosion rates of cannon balls treated by hydrogen reduction, alkaline sulfite and Hostacor IT. Oxygen consumption of six cannon balls has been measured at five relative humidities between 20% and 60% RH as proxy corrosion rate. Results show a noticeable increase in corrosion rate at 50% to 60% RH and also returned differences in corrosion rates of cannon balls based on previous treatment. Display RH of 55% for showcases containing organic materials and cast iron is found to be unsuitable for the cannon balls and their display in these conditions is not recommended. This has implications for contextual display at the Mary Rose Trust and elsewhere. Further study with greater sample numbers is providing statistical evidence of the effect of treatment regime on corrosion rate to inform decision making for future treatment, display and storage.

Keywords

cast iron, cannon balls, display, relative humidity, corrosion rates

Introduction

Context

The Mary Rose Trust houses the fleet flagship of King Henry VIII which capsized at the Battle of the Solent in 1545 and was raised in 1982. Excavation of the wreck uncovered a time capsule of Tudor life with many thousands of everyday and military objects. Recent redisplay of this important collection in an imaginative, purpose-built museum presents objects to the public in mixed material showcases designed to contextualise artefacts and offer insight into everyday life on board ship in Tudor times. This avoids the unnatural concept of sub-division by material type which represents best practice for long term preservation.

A display of cannon, cast iron shot and associated organics, including rope and polyethylene glycol treated wooden gun carriages, vividly recreates the gun deck of the ship (Figure 1). Relative humidity (RH) was controlled to 55% (monthly averages 54.5 – 57.7% RH between March 2013 and February 2014) to serve the needs of the organic materials but resulted in extensive cracking and physical break up of cast iron cannon balls previously treated by various methods including: hydrogen reduction; alkaline sulfite; and immersion in Hostacor IT.

Figure 1. Gun deck showcase at the Mary Rose Trust Museum conditioned to 55% relative humidity

Cardiff University quantitatively determined the corrosion rates of selected cast iron cannon balls as a function of ambient RH. Their corrosion rates were measured at RH values around and below the 55% target of the display area. This provided an evidence base for predictive management that related the needs of the cannon balls to the nature of the display and its current target RH, including any decision to withdraw the cannon balls from display.

The hydrogen reduced cannon balls did not break up in the 55% RH controlled environment of the gun deck display. Ethical restrictions related to metallographic modification at the high operating temperatures (North et al. 1976; Barker et al. 1982) and operating costs elevated by the risk of oxy-hydrogen explosion mean this is effectively a discontinued treatment, yet in theory chloride ions (Cl^-) can be completely removed by this method (North and Pearson 1977). The dialogue for and against the use of hydrogen reduction could be assessed in a rationalised manner in light of the data produced here and ongoing testing at Cardiff.

Cast iron: the corrosion problem

The Mary Rose Trust cannon balls comprise grey cast iron which contains 2-5% silicon and carbon, mostly as graphite flakes embedded in a ferrite-pearlite matrix (Cottrell 1975). Corrosion effectively scours the ferrite out of the graphite matrix leaving a graphitised structure that retains the shape of the cannon balls (North 1976; 1982), which appear visually unchanged but are lightweight and highly corroded with much reduced densities. The corrosion front lies at the metal/graphitised structure interface where Cl^- ions concentrate to counter the charge of ferrous (Fe^{2+}) ions produced by corrosion (North 1976; 1982). Akaganeite (β-FeOOH) is a commonly occurring corrosion product in marine iron (Argo 1981; North 1982; Kergourlay et al. 2010) as it forms at low pH in high concentrations of Cl^- and incorporates Cl^- in its crystal structure and adsorbed on its surface (Remazeilles and Refait 2007).

Cast iron from marine contexts is notoriously unstable post-excavation and its storage immersed in water within sealed containers limits oxygen availability and thus corrosion and oxidation of corrosion products. In the atmosphere, oxygen and moisture facilitate oxidation of Fe^{2+} at the metal surface to oxyhydroxide FeOOH (Equation 1). Hydrolysis of Fe^{2+} produces a low pH at the corrosion interface (Equation 2) and the presence of Cl^- favours β-FeOOH formation as a voluminous corrosion product that pressures and cracks the overlying graphitised layer (Figure 2) (North 1982; Turgoose 1982). The corrosion products β-FeOOH and ferrous chloride ($FeCl_2.4H_2O$) can form as the cannon balls dry and are capable of corroding iron at 20% RH (Turgoose 1982; Watkinson and Lewis 2005). Additionally, oxidation reactions involving sulphide corrosion products will generate volume changes that damage the graphitised layer (North 1982).

Figure 2. Cast iron cannon ball showing break up at metal/corrosion layer interface (diameter 65mm)

$$Fe^{2+} + H_2O + ½\ O_2 \rightarrow FeOOH + H^+ \quad (1)$$

$$Fe^{2+} + H_2O \rightarrow FeOH^+ + H^+ \quad (2)$$

Treatment of the *Mary Rose* cannon balls was designed to remove Cl^-: hydrogen reduction volatilises Cl^- bearing corrosion products (Barker et al. 1982); aqueous alkaline sulfite aims to wash out soluble Cl^- including that surface adsorbed on β-FeOOH (North and Pearson 1977; Gilberg and Seeley 1982); water solvates soluble chloride and the anodic corrosion inhibitor Hostacor IT (triethanolamine salt of acylamido carboxylic acid) aims to inhibit further corrosion at the metal surface (Argyropoulos et al. 1999).

Aim

- To determine the impact of display relative humidity on the corrosion rate of cast iron cannon balls recovered from a marine archaeological context.

Objectives

- Measure the corrosion rate of selected cast iron cannon balls of known provenance using oxygen consumption of individual cannon balls measured over a fixed time period at a fixed relative humidity.
- Use oxygen consumption to advise the Mary Rose Trust on the impact of display relative humidity on the corrosion of cast iron cannon balls.

Method

Sample

The Mary Rose Trust supplied what would have been upon manufacture standard 9 lb cannon balls (4082 g × 100 mm diameter) (Table 1). Two had been treated by hydrogen reduction for 100 hours at 850°C then impregnated with a thermosetting resin; three by washing in changes of 0.5M alkaline sulfite solution at 60°C and coating with 15% (w/v) Paraloid B48 in acetone; and one by washing in changes of 2% (v/v) Hostacor IT in tap water and application of microcrystalline wax with graphite powder.

Table 1. Details of sample cannon balls and their condition prior to corrosion rate testing

Sample	Treatment	Mass (g)	Condition prior to corrosion rate testing
HRed 1	Hydrogen reduction	1514	Surface complete; cracking on one area developing a flake
HRed 2	Hydrogen reduction	1268	Surface complete; no cracking
Host 1	Hostacor IT	2367	Surface lost one small flake to depth of 2mm; no cracking
AlkSul 1	Alkaline sulfite	1686	Surface lost one large flake to depth of 2mm exposing blow holes from casting faults; no cracking
AlkSul 2	Alkaline sulfite	1173	Surface complete; no cracking
AlkSul 3	Alkaline sulfite	1426	Surface lost one large flake; very significant cracking post-treatment

The experimental study employed these archaeological objects as test samples since reproducible standardised analogues offering similar properties could not replicate the properties of real objects. This introduces limitations in experimental reproducibility due to the wide range of variables whose values are unknown.

Method

The methodology followed the procedure used for oxygen consumption rate measurement employed previously for corrosion testing at Cardiff University (Emmerson and Watkinson 2014; Watkinson and Rimmer 2014; Emmerson and Watkinson 2016). The oxygen consumed by each cannon ball was determined at 20%, 30%, 40%, 50% and finally 60% RH as a function of time in a closed vessel. Each cannon ball was cradled in Netlon for easy movement and placed in its individual reaction vessel with silica gel conditioned to the appropriate RH for the test and a MadgeTech RHTemp101A datalogger to record internal RH (±3%) and temperature (±0.5°C).

Reaction vessels were 2 litre glass vessels with mild steel screw top lids (Clas Ohlson article no. 44-1319). These were sealed by casting 25 g Mould Life Plastisil Gel 10 silicone rubber into the base of each lid, tightening the lid onto the vessel and sealing the lid around the bottom of the threads with the same silicone rubber to create an airtight seal. The vessels were stored and their oxygen partial pressure measured in a climatic chamber maintained at 20°C to ensure that RH within the vessels was constant. Oxygen partial pressure within the vessels was determined via a sensor spot fixed to the interior of the vessel with Radio Spares Silicon Rubber Flowable Compound adhesive. Light from a fibre optic cable attached to an OxyMini Micro meter was fired at the spot through the glass to detect its fluorescence, which was directly proportional to oxygen partial pressure (±2 mbar). Readings were taken at intervals over the test period (between 20 and 60 days depending on RH) and oxygen consumption within the reaction vessels determined by plotting oxygen partial pressure against time.

Nitrogen filled control vessels were used to determine the effectiveness of the seal to the atmosphere. Additionally, oxygen consumption within a control vessel with silica gel and datalogger but no cannon ball was recorded for each humidity. This identified whether the differing RH inside the vessels impacted upon oxygen readings and the regular breaking and renewal of the seals tested whether seal quality was reproducible. Tests on cannon balls and controls were terminated when sufficient data points had been gathered to identify corrosion rates at the varying RH values.

Results

Leakage of 8 control vessels is recorded in Figure 3 and Table 2.

Table 2. Oxygen ingress for 8 nitrogen filled control vessels giving equation for linear regression trendline and average oxygen ingress (mbar.day^{-1}) for each vessel

Vessel	Gradient	Average oxygen ingress rate (mbar.day^{-1})
1	y = 0.1733x – 0.0812	0.17
2	y = 0.1702x – 0.0965	0.17
3	y = 0.1663x – 0.0321	0.16
4	y = 0.1513x – 0.0061	0.14
5	y = 0.1662x – 0.0179	0.17
6	y = 0.1633x + 0.0255	0.16
7	y = 0.1587x - 0.1221	0.17
8	y = 0.2122x + 0.1112	0.21

Oxygen consumed as a function of time (proxy corrosion rate) for individual cannon balls at the five RH values tested are shown in Figures 4-8. The oxygen consumption recorded is not adjusted for the oxygen ingress detected in the control vessels. This is considered when interpreting the data. Control vessels with conditioned silica gel and no sample showed no overall oxygen consumption. Fluctuations of partial pressure were within the error of the meter.

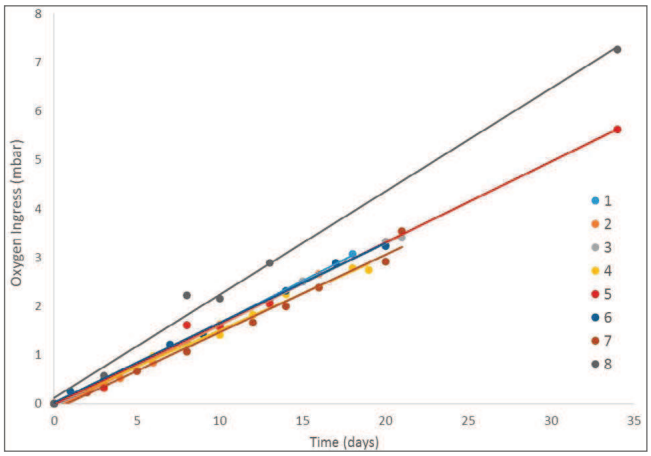

Figure 3. Change of oxygen partial pressure within 8 control test vessels containing nitrogen over time. Leakage rates are minimal given atmospheric oxygen partial pressure of 210mbar

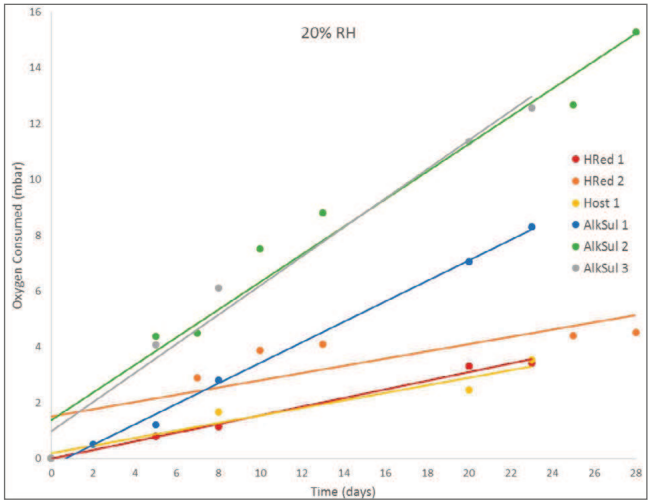

Figure 4. Oxygen consumption over time of cannon balls at 20% relative humidity with linear regression trendlines fitted

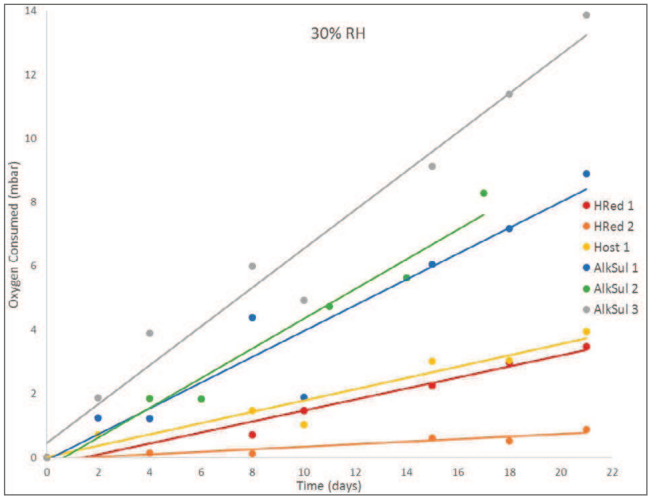

Figure 5. Oxygen consumption over time of cannon balls at 30% relative humidity with linear regression trendlines fitted

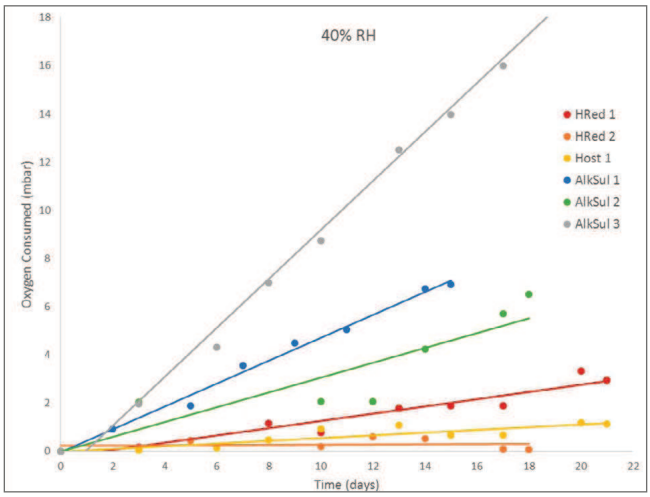

Figure 6. Oxygen consumption over time of cannon balls at 40% relative humidity with linear regression trendlines fitted

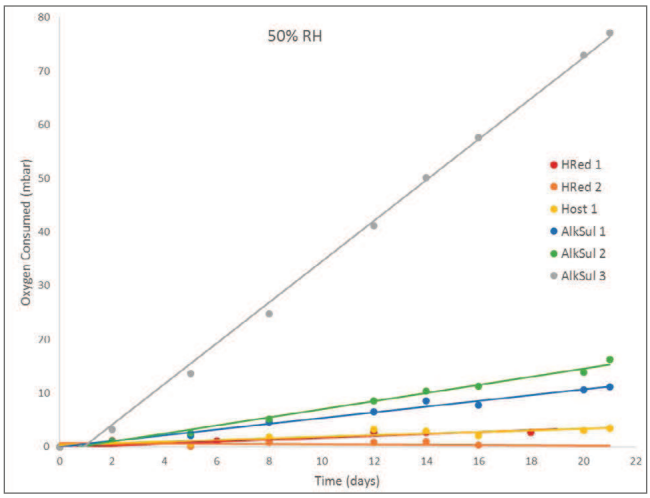

Figure 7. Oxygen consumption over time of cannon balls at 50% relative humidity with linear regression trendlines fitted

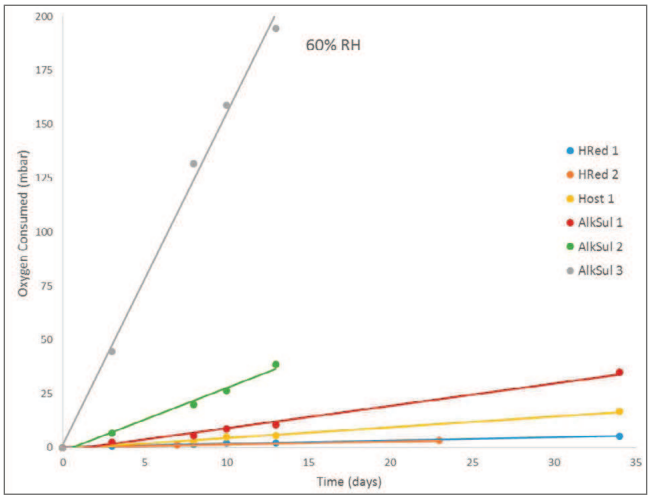

Figure 8. Oxygen consumption over time of cannon balls at 60% relative humidity with linear regression trendlines fitted

The relationship between oxygen consumption and time was linear for all cannon balls over the test period. Average oxygen consumption per day for samples at each RH are given in Table 3.

Table 3. Oxygen consumption rates (mbar.day^{-1}) of cannon balls at all test humidities

Cannonball	Oxygen consumption (mbar.day^{-1})				
	20% RH	30% RH	40% RH	50% RH	60% RH
HRed 1	0.15	0.17	0.17	0.15	0.15
HRed 2	0.16	0.04	0.05	0.07	0.14
Host 1	0.15	0.19	0.06	0.17	0.54
AlkSul 1	0.36	0.42	0.46	0.53	1.06
AlkSul 2	0.55	0.49	0.29	0.77	2.97
AlkSul 3	0.55	0.66	0.97	3.67	14.94

Cumulatively, there were no significant changes to the physical integrity of any of the cannon balls by the end of the five test periods (Figures 9 and 10).

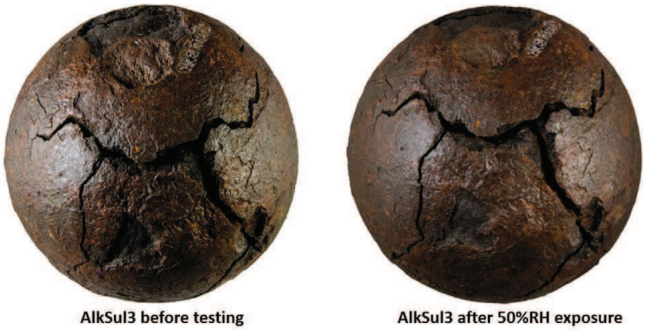

Figure 9. Physical condition of alkaline sulfite treated cannon ball before oxygen consumption testing (left) and after test runs through from 20% RH to 50% RH (right) (diameter 100mm)

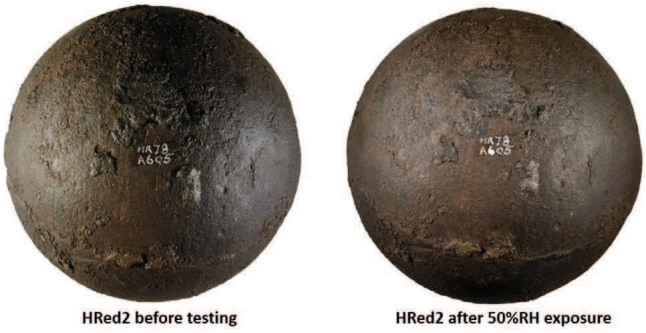

Figure 10. Physical condition of hydrogen reduction treated cannon ball before oxygen consumption testing (left) and after test runs through from 20% RH to 50% RH (right) (diameter 100mm)

Discussion

The leakage rate of the nitrogen filled control vessels is linear, with 7 of the 8 controls producing very similar leakage over the time frame of the experiments (Figure 3, Table 2). The average leakage rate of 8 vessels was 0.17 mbar.day^{-1} and of the 7 closest vessels 0.16 mbar.day^{-1}. Leakage is closely reproducible, indicating that renewing seals on the vessels was predictable. All the graphs for the cannon ball tests record oxygen partial pressure and have not been corrected for ingress of oxygen into the reaction vessel, which will contribute to replenishing oxygen used up in the oxidative corrosion processes occurring. Therefore, the slopes of all lines in Figures 4-8 would be slightly steeper if oxygen ingress was added to the total of oxygen consumed. All oxygen consumption and therefore corrosion is slightly greater than is recorded in Figures 4-8.

Oxygen consumption is an indicator of corrosion rate but it cannot be used in simple stoichiometric calculation of the amount of iron lost. The detailed mechanism of corrosion is not known and other oxidation reactions may occur including the oxidation of sulfides, although studies of cannon balls from the *Batavia* wreck have revealed low levels of sulfide compounds in its cannon balls (North 1976; 1982). Since the cannon balls are from the same deposit it might be expected that their generic corrosion patterns are similar. Oxygen consumption is linear for all cannon balls over the test periods (Figures 4-8). The overriding factor to guide storage and display RH at the Mary Rose Trust is that all six of the cannon balls tested here consumed oxygen over the 20% to 60% RH range of the study (Figures 4-8). Clearly all display environments within this RH range present a corrosion risk. Rates of corrosion differed considerably between cannon balls. At all RH values tested, the three alkaline sulfite treated cannon balls consumed the most oxygen (Figures 4-8). The Hostacor IT and hydrogen reduction treated cannon balls consistently consumed less oxygen than the alkaline sulfite treated samples at all RH values.

Accounting for the ±2mbar accuracy of the oxygen meter, AlkSul 1 and 2 virtually maintain their oxygen consumption rates at 20%, 30%, 40% and at 50% RH. AlkSul 3 remains the same at 20% and 30% RH but compared to its rate at 30% RH it is 1.5 times faster at 40% RH and 5.5 times faster at 50% RH (Figures 4-8). The rate of oxygen consumption increases significantly for all alkaline sulfite treated cannon balls moving from 50% to 60% RH (Figures 7 and 8): at 60% RH AlkSul 1 is 4 times faster; AlkSul 2 is 2 times faster; AlkSul 3 is 3.5 times faster. Translating this into oxygen consumption at 60% RH over 20 days: 20mbar (AlkSul 1); 70mbar

(AlkSul 2); 310mbar (AlkSul 3). For these three objects treated according to the same protocol to remove Cl^-, there is a lack of consistency in their corrosion rate despite their treatment to a similar end point.

The two cannon balls treated by hydrogen reduction produced similar and very slow corrosion rates over the full 20% to 60% RH range, while the single Hostacor IT treated cannon ball shows similar slow corrosion rates from 20% to 50% RH and a slight increase at 60% RH (Figures 4-8). It is tempting to consider that both hydrogen reduction and Hostacor IT treatments are more successful than alkaline sulfite but this is impossible to determine using such a small sample of non-reproducible tests. Yet the differences in corrosion rate are very significant with AlkSul 3 being over 30 times faster than Hostacor IT and 60 times faster than the hydrogen reduction samples. Even at 60% RH, cannon balls treated by hydrogen reduction and Hostacor IT returned slow oxygen consumption rates. In contrast, the three alkaline sulfite treated cannon balls all consumed large amounts of oxygen at 60% RH and always corroded faster than the other cannon balls on test. This significant jump in oxygen consumption rate parallels the increase in the thickness of adsorbed water on metal surfaces at 60% RH to 3 monolayers (Cole 2010). This offers a thicker, more continuous layer of water to form electrolytes and compounds such as ferrous chloride can deliquesce at 55% to 60% RH to provide electrolyte ions (Watkinson and Lewis 2005).

Initial exploration of reasons why these differences occur may be that the hydrogen reduction treatment reached 850 °C where Cl^- volatilises as HCl gas (North and Pearson 1977). This might be expected to produce a Cl^- free object. Digestion of the hydrogen reduced samples would be required to confirm this, although their low rate of oxygen consumption at 60% RH tends to support this theory. Hydrogen reduction has been applied until the gaseous eluate is Cl^- free (North 1977) but quantitative testing of chloride residues within treated objects is limited. The thermosetting resin applied post-treatment may also contribute to their good corrosion resistance. The low oxygen consumption of the Hostacor IT treated cannon ball is less easy to explain. Diffusion controlled washing in an aqueous solution of Hostacor IT, unaided by alkali, is likely to be less efficient than alkaline sulfite at removing Cl^- and both treatment regimes are likely to leave residues of Cl^- (Watkinson and Al Zahrani 2008; Watkinson and Rimmer 2014). Inhibitors are often ineffective in the presence of Cl^- with Hostacor IT requiring Cl^- to be below critical minimum values (Argyropoulos et al. 1999). There has been no reproducible quantitative testing of the inhibitive effectiveness of Hostacor IT with Cl^- contaminated marine iron, but electrochemical testing evidenced that it either inhibits or slows corrosion of uncorroded wrought iron in polyethylene glycol solution (Argyropoulos et al. 1999). Microcrystalline wax coatings are not renowned corrosion barriers. Alkaline sulphite treatment has been shown quantitatively to reduce corrosion rate (Watkinson and Rimmer 2014) but variables such as treatment protocol will impact on its effectiveness and Paraloid B72 is not expected to offer good barrier properties on corroded porous cast iron surfaces.

These tests aimed to determine the stability of the cannon balls in display RH for the Mary Rose Trust but translating corrosion rates into longevity of cannon balls on display is impossible as the true measure is whether the cannon balls retain their physical integrity and serve their purpose as historic objects for public display. During the testing presented in this paper, no physical change was seen in any of the cannon balls although no measure was employed beyond simple visual observation (Figures 9 and 10). The short cumulative time period of the tests (approximately 100 days over the full range of five RH values) offers no guide to damage over normal display periods of years.

Conclusion

It is now possible to offer direct advice to the Mary Rose Trust that displaying their cannon balls may produce a slow corrosion rate at 40% RH with corrosion beginning to increase at 50% RH and increasing significantly at 60% RH. RH levels within showcases containing cannon balls should not exceed 40% and values of 30% or 20% RH are preferable. Mixed media displays of organic materials and cast iron offer conflicting RH requirements of the materials. Given the small number of cannon balls tested, it is not possible to identify which cannon balls will corrode at these humidities based on past treatment regimes. These guidelines must therefore be applied to all cannon balls in the collection until further data is available. These recommendations are applicable beyond the context of the Mary Rose Trust to other institutions housing collections of marine cast iron. The results here have encouraged the Mary Rose Trust to support a larger

experiment at Cardiff University with 45 treated cannon balls to investigate whether it is possible to determine the corrosion threat from laboratory records detailing treatment procedure. It will also identify whether hydrogen reduction is an effective treatment and may open up new evidence based dialogue on the potential future use of this largely discontinued treatment.

Acknowledgements

Thanks to Professor Mark Jones at the Mary Rose Trust for loaning the cannon balls for these experiments and to David Pearson and Dr Eleanor Schofield for the treatment records and environmental data.

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