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PREPARING HISTORIC WROUGHT IRON FOR PROTECTIVE COATINGS: QUANTITATIVE ASSESSMENT TO PRODUCE EVIDENCE-BASED PROTOCOLS

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Abstract

Painted historical wrought iron commonly occurs outdoors exposed to the prevailing climate. Maintaining protective paint layers is an interventive process that often involves removing existing paint layers and repainting. Whilst prior surface preparation greatly affects the longevity of any paint layer, its impact on the metal surface and paint performance has received limited research within heritage contexts, making their selection anecdotal or driven by manufacturers' guidelines. Historic Scotland-funded research at Cardiff University is quantitatively investigating the effect of surface preparation methods on wrought iron corrosion rates prior to paint application. The feasibility of using historic sample material in this research has been investigated and is reported. Testing historic wrought iron samples in quantitative studies of corrosion offers more direct linkage to heritage scenarios thus facilitating interpretation of results and extrapolation to real time heritage contexts. The use of an oxygen consumption technique to quantitatively determine the corrosion rate of five samples of historic wrought iron in controlled conditions of 90% relative humidity and 20 °C is reported. Results returned corrosion rates indicating a level of reproducibility that, with an error calculation, will allow corroded historic wrought iron to be used for production of test samples to be employed in experiments designed to determine the impact of surface cleaning techniques on the corrosion rate of corroded heritage iron.

Keywords

Historic wrought iron, standards, guidelines, corrosion rates, surface preparation, paint, oxygen consumption, climate chamber, sample standardisation.

Project aims

This project aims to:

- investigate whether a remote oxygen consumption technique is suitable for determining corrosion rates of historic wrought iron in real time testing within controlled environments;
- assess whether heritage iron, having undergone uncontrolled corrosion in a normal working context over centuries, can return sufficiently reproducible corrosion rates to justify its use for generating samples for experimental study aimed at producing quantified data;
- establish the feasibility of using historic wrought iron rather than generating analogues that act as proxy heritage samples, thus offering alternative experimental designs in heritage research.

Introduction

Existing guidance and the need for quantitative data

Historic wrought iron is ubiquitous in Western Europe where responsibility for planning or specifying its treatment falls to a range of individuals who may or may not have experience of heritage iron or a working knowledge of conservation principles. This includes home owners, museum professionals, house managers and property surveyors. Realpolitik often dictates that there is no budget for specialist consultation prior to specifying conservation work for historic wrought iron, yet it involves a large number of variables including choice of surface preparation methods, coating systems and maintenance regimes. Written guidance is available in the form of technical bulletins, leaflets, short articles, book chapters and occasional case studies in specialist journals and conference proceedings

(Ashurst and Ashurst 1988; Barker 2010; Blackney and Martin 1998; Blackney 2010; Cheltenham Borough Council (online); Davey 2007; Davey 2009; Meehan 2010; Mitchell 2005; Taylor and Suff 2010; Topp 2010; Schütz and Gehrke 2008; Watkinson 2005; Wilson et al. 2010) published by heritage bodies, government agencies, local councils and heritage ironworkers. However, an overall absence of evidence-based heritage standards for the treatment of historic wrought iron means decision making is reliant on un-scaled comparisons and, at worst, guesswork. Although detailed industrial and commercial standards and guidelines exist (ASTM 2008; British Standards Institute 2000, 2005/2006, 2006/2007, 2012; Corus 2004), their focus on modern materials, without any ethical constraints of the type found in conservation, limits their direct use in the historic contexts to specific situations.

The heritage sector guidance on surface preparation listed above is well-intentioned but limited in scope, frequently conflicting and reliant on an evidence base that is rarely quantified or offers data and methodologies that are difficult to translate into practical contexts. It is entirely understandable that application of this guidance becomes anecdotal or experience-based on many occasions. Identifying how to generate the necessary quantitative evidence-based data that will transform practice will rely on well designed, and preferably co-ordinated, research. Unfortunately, conservation of large heritage wrought iron assemblages is dominated by contractor-based private sector activity; understandably, research is not a priority here and gaining funding to support it is extremely difficult. Similarly, research within commercial coating companies and national standards bodies into developing evidence-based dedicated procedures and products for historic material is hampered by the lack of potential profit in the heritage sector. This leaves the beacon of research to be borne by academia and the heritage institutions themselves, where it must exist amongst many other priorities and

be subject to underfunding. A natural outcome of this situation is that surface preparation techniques developed for industrial contexts are employed in the heritage sector, despite a dearth of study into their suitability for historic wrought iron. The upshot is that the surface preparation of heritage iron to receive paint is unregulated, ad hoc and unable to support predictive conservation procedures. Exceptions occur when industrial standards can be adopted and adhered to; the wrought iron hull of Brunel's SS Great Britain was cleaned to Swedish Standard Sa 2.5 (ASTM 2008) prior to painting, as befitted the ethical constructs in place for a corroded hull that had been cleaned to the metal and painted periodically during its lifetime (Watkinson et al. 2005).

The impact of developing heritage standards for surface preparation

The experience of contractors and specialist conservation companies is of great value in identifying questions and procedures that can be addressed by research designed to produce quantitative and qualitative data to develop evidence-based best practice guidelines. This is essential for effective heritage management that will optimise use of limited resources through cost benefit calculations. To manage and plan successfully it is essential to know how effective conservation procedures will be; at present this information cannot be delivered. Quantitative testing and research can measure aspects such as loss of metal and longevity of paint layers as functions of intrinsic and extrinsic factors such as metal properties, time and environment; this will feed into cost benefit analyses. Experimental design, measurement techniques, analysis methods and their relevance to heritage contexts are important considerations, as is a preference for real time testing and the use of sample material that is, or closely represents, the heritage metal and its condition.

An initial step in delivering quantitative data is to understand the impact of pre-painting surface preparation techniques on wrought iron and paint layers applied subsequently. This should be followed by quantitatively determining the performance of protective coatings applied to them to offer a full evidence base suitable for devising a conservation and management plan.

To develop a full understanding of surface preparation techniques it is necessary to determine their:

- influence on corrosion rate of the metal they are applied to;
- impact on original surface and mill scale;
- action on iron oxides (removal, surface finish and adherence) and the possible corrosion protection offered by retention of oxides such as mill scale;
- effect on wettability, adherence and covering power of coatings on prepared surfaces;
- impact on chloride driven corrosion mechanisms;
- influence on coating performance;
- practicality for application in the field;
- impact on ethics and aesthetics.

The outcome will allow treatments to be compared and ranked in relation to specific requirements. This paper reports the preliminary work carried out to assess the feasibility of using historic wrought iron as sample material in experimental studies in place of analogues. It forms part of Historic Scotland's 'Traditional Ferrous Metals Research Project' examining conservation of cast and wrought iron to develop clear, widely available, evidence-based and reasoned guidance. Studies to be reported later will investigate the impact of surface preparation techniques on corrosion rates and performance of selected coatings on wrought iron.

Determining corrosion rates of cleaned wrought iron Sample material

The challenge of assessing heritage conservation methods experimentally lies in the nature of the sample material and the method of quantification adopted. The individual nature of heritage metals whose corrosion layers have developed over many years, often in unknown and variable environmental conditions, means that the production of analogues to represent them in experimental study can be challenging if results are expected to reflect the reality of treating heritage objects.

There are specific contexts in which attempts to deliver standardised analogues that represent heritage objects are useful for reasons of reproducibility. Degryny (2010) offers a clear methodology for generating standardised chloride-containing corrosion layers on copper alloys and iron on which to test the performance of protective coatings for corroded heritage metals. This worked effectively for producing comparative data between partners in the PROMET project (Argyropoulos et al. 2007). Nevertheless, there is inevitably a compromise in this approach between the imperfect representations of naturally generated corrosion layers and those grown in accelerated corrosion contexts, although this must be balanced against good reproducibility of analogue samples and the positive impact of this on data quality. Uniformity provided by analogues offers potential for ranking that can be fed back to real life scenarios by extrapolation using the context of 'expected performance'.

Nevertheless, it would be preferable to use heritage material as samples and subject these to real time testing. In keeping with a focus on real life contexts, corroded historic wrought iron samples are used in this study rather than analogues. The sample material, sourced by Historic Scotland, is an un-provenanced wrought iron railing from an Edinburgh scrap yard. Vestiges of failed coating systems remain on approximately 40-50% of the surface, adhering closely in some areas and loosely in others. Corrosion products also cover the entirety of the railing in the form of closely adhering, coherent oxide layers as well as laminating and powdery corrosion products and pitting (Fig. 1).

Oxygen consumption and corrosion rates

Equally important is the corrosion measurement method. It should preferably represent the reality of corrosion, which means recording corrosion rate data in environments to which heritage iron objects will be exposed. This should

be in real time, rather than by accelerated ageing. Thus, while coating performance can be ranked by techniques such as EIS (Cano et al. 2007; 2010) or corrosion rate by Ecorr (Hollner et al. 2007), this necessitates immersion of samples in solutions whereas, in reality, heritage iron will be subject to vastly varying conditions with wet and dry cycles and a range of humidities in a temperate climate. Measuring oxygen consumption in real time within controlled relative humidity and temperature environments, relating it to oxidation of iron and converting this data to corrosion rates offers a clear record of corrosion for specified 'atmospheric' conditions.

Sample characterisation

Wrought iron is by nature an inhomogeneous material with local compositional differences possible within the same piece of metal (O'Sullivan and Swailes 2009, 260-261). To minimise the chances or extent of compositional variation between the samples they were cut from a continuous length of flat bar iron (Fig. 1). However, the skill of wrought ironworkers is such that the bar may have been produced by welding shorter lengths of the metal together without leaving any macroscopically visible indication (Chris Topp heritage blacksmith pers. comm.).

Samples were cut from the wrought iron bar using a hand hacksaw with white spirit as a lubricant to avoid heat from machine sawing. Sample size was 400 mm x 300 mm x 100 mm as dictated by the dimensions of the bar and the diameter of the aperture of the reaction vessel used for the oxygen consumption tests. Each sample was weighed and minor discrepancies in overall dimensions, and hence surface area, were recorded.

Paint layers were examined in profile and elementally analysed using a CamScan Maxim 2040 scanning electron microscope (SEM) equipped with Oxford Instruments energy and wavelength dispersive X-ray spectrometers. Polished sections of the railing were also analysed using the SEM to determine the composition of the wrought iron. Samples of corrosion products were removed from exposed areas and beneath exfoliating paint and analysed using a PANalytical X'Pert Pro (Cu) X-ray powder diffraction.

Following this, five samples of the iron were tested to determine their corrosion rate. The samples were uncleaned, contained residues of paint and areas of corroded iron on their major faces and had two fresh cut edges (Fig. 2). Each sample was sealed within an individual reaction vessel containing silica gel conditioned to 90% relative humidity (RH). Corrosion rates for the samples were determined using remote recording of oxygen concentration within the reaction vessel. This used an Oxymini Micro fibre optic meter to record the quenching of fluorescence in an oxygen sensitive dye contained within a sensor spot adhered to the inside of the reaction vessel. The airtightness of the 250 cm³ reaction vessels was tested for oxygen ingress by adding nitrogen to a jar and determining oxygen concentration over 257 days. Control jars were set up containing only conditioned silica gel. Each jar contained a MadgeTech humidity and temperature sensor accurate to ± 3% RH and 0.5 °C. The reaction vessels were kept in a

Binder KBF series climatic chamber to control temperature to 20 °C ± 0.5 °C which maintained the RH within the reaction vessels and facilitated reproducibility in oxygen measurements carried out in the chamber.

Results of sample analysis

The sample dimensions and their weight range are shown in Table 1. The mean composition of the sample analysed was 99% iron, 0.37% silicon, 0.36% phosphorus and 0.27% manganese (Table 2) which is typical of a wrought iron. Slag content was moderate and relatively evenly distributed in stringers, although inclusions varied in size from <10 µm to >600 µm (Fig. 3). Corrosion was localised and pitting was present (Fig. 2). XRD identified goethite ($\alpha\text{FeO}(\text{OH})$ diffraction code 01-081-0462), magnetite (Fe_3O_4 diffraction code 01-085-1436) and lepidocrocite ($\gamma\text{FeO}(\text{OH})$ diffraction code 01-074-1877) to be present as corrosion products (Fig. 4). There were at least nine layers of paint on the samples and the compositions of these (Figs. 5 to 9) indicated the presence of lead based coatings, those with barium sulphate fillers, cobalt drying agents, copper based pigments and a suggestion of zinc. Overall the picture is of an ad hoc painting maintenance regime with a range of pigment and paint types such as might be expected in the protective coating of an outdoor railing.



Figure 1: Iron railing

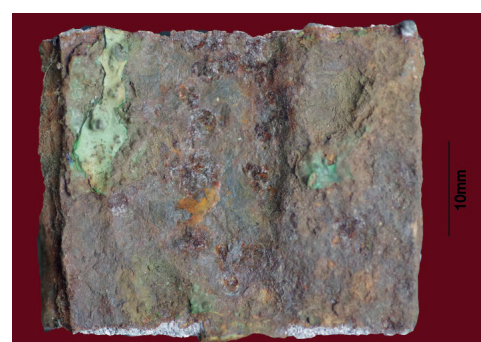


Figure 2: Sample of wrought iron railing used in control tests

Sample No.	Length (mm)	Breadth (mm)	Depth (mm)	Mass (g)	Consumption rate (mbar O ₂ /day)	Standard deviation of consumption rate from average	Consumption rate by mass (mbar O ₂ /day/g)
1	38.90	29.50	9.40	66.03	0.14	0.00	0.01
2	38.90	29.30	9.80	65.32	0.14	0.00	0.01
3	39.00	30.70	9.90	69.06	0.12	0.01	0.01
4	38.90	30.60	9.40	69.66	0.17	0.02	0.02
5	38.90	29.00	9.30	64.18	0.12	0.01	0.01
				Average	0.14		

Table 1: Dimensions and masses of the five un-cleaned samples with corrosion rates as oxygen consumption per day and oxygen consumption per day per gram of sample material over test period of 257 days.

	Weight %				
	Si	P	Mn	Fe	Total
Spectrum 1	0.4	0.3	0.28	99.02	100
Spectrum 2	0.36	0.41	0.33	98.91	100
Spectrum 3	0.36	0.36	0.21	99.06	100
Mean	0.37	0.36	0.27	99	100
Std. deviation	0.02	0.05	0.06	0.08	
Max.	0.4	0.41	0.33	99.06	
Min.	0.36	0.3	0.21	98.91	

Table 2: Results of SEM analysis of the wrought iron giving compositions of three areas analysed (spectra 1-3).

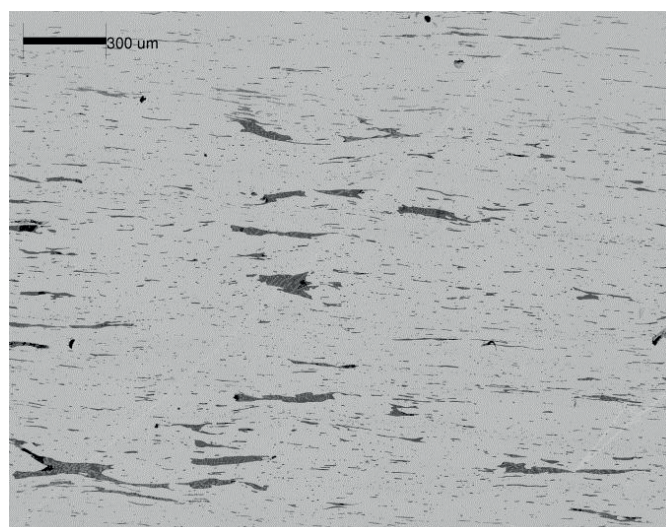


Figure 3. SEM backscattered electron image of a polished section of wrought iron railing showing slag distribution.

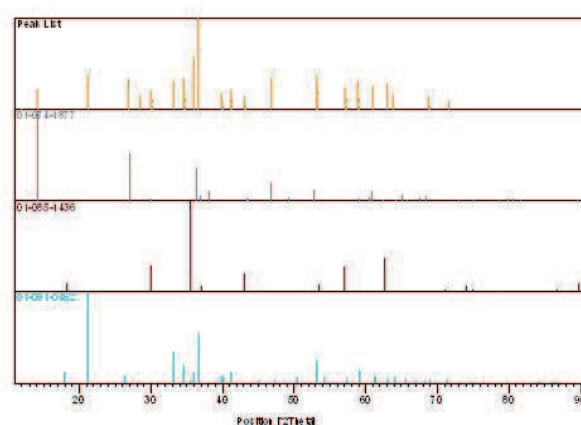


Figure 4: Diffraction patterns of corrosion sample and matching compounds (top to bottom): the corrosion product sample; lepidocrocite (01-074-1877); magnetite (01-085-1436); goethite (01-081-0462).

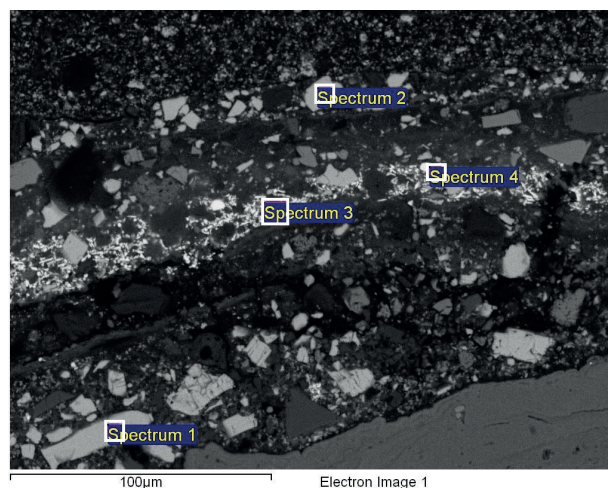


Figure 5: Backscattered electron image showing location of analyses of paint layers from the sample material (indicated by white squares). Spectra 1-4 are given (Figs. 6 to 9).

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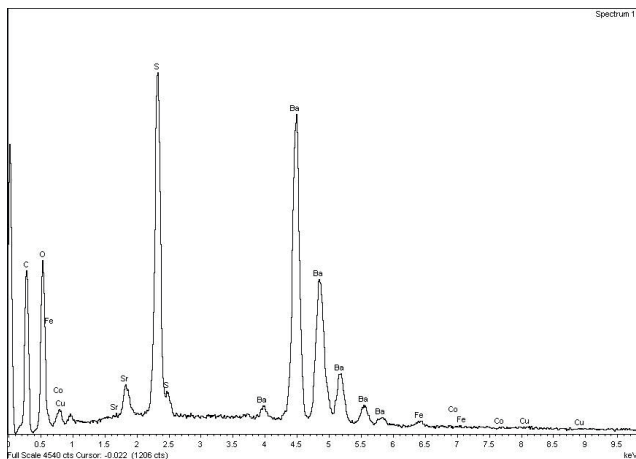


Figure 6: Spectrum 1 of SEM analysis of paint layers on sample material (location indicated in Fig. 5) showing major peaks for sulphur, and barium with minor peaks for copper, cobalt, strontium and iron.

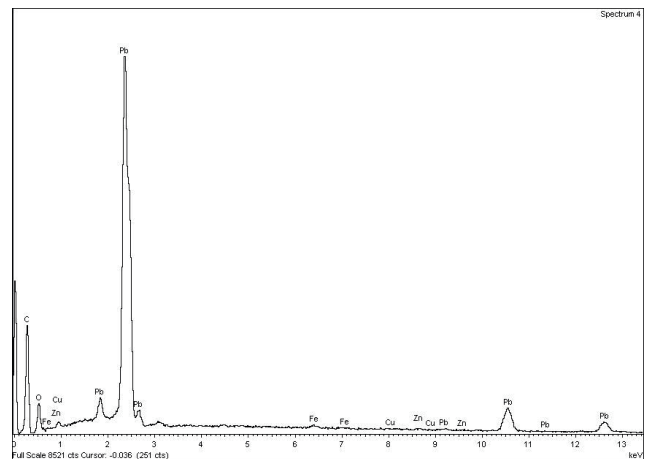


Figure 9: Spectrum 4 of SEM analysis of paint layers on sample material (location indicated in Fig. 5) showing major peaks for lead and minor peaks for copper, zinc and iron.

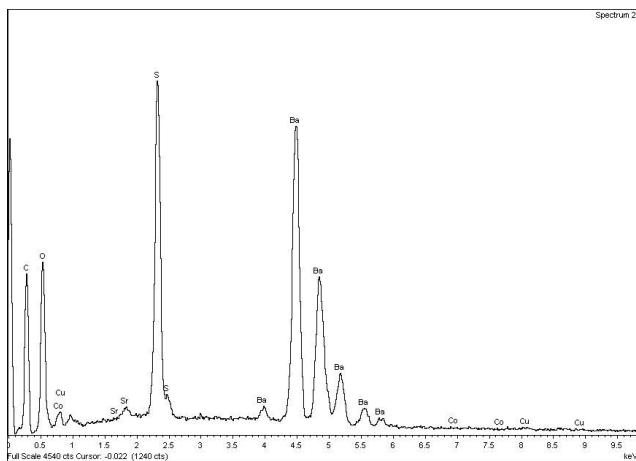


Figure 7: Spectrum 2 of SEM analysis of paint layers on sample material (location indicated in Fig. 5) showing major peaks for sulphur and barium and minor peaks for copper, cobalt and strontium.

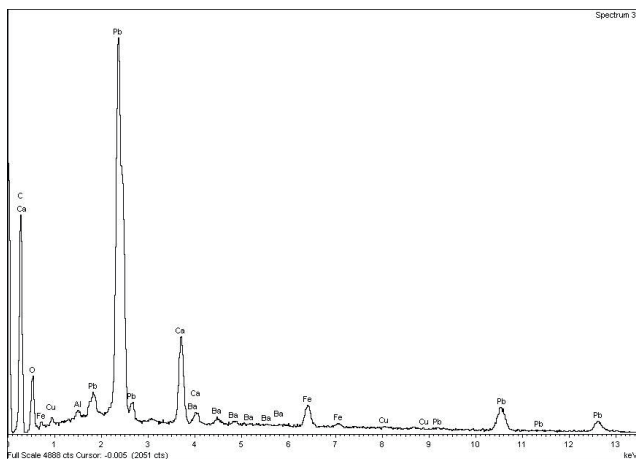


Figure 8: Spectrum 3 of SEM analysis of paint layers on sample material (location indicated in Fig. 5) showing major peaks for lead and calcium and minor peaks for aluminium, barium and copper.

The corrosion rate of each sample was recorded over a period of 257 days (Fig. 10). Representing the daily corrosion rate as oxygen consumed per sample reveals that the corrosion rate averaged 0.14 mbar/day and each sample was within 0.02 standard deviations of this value (Table 1).

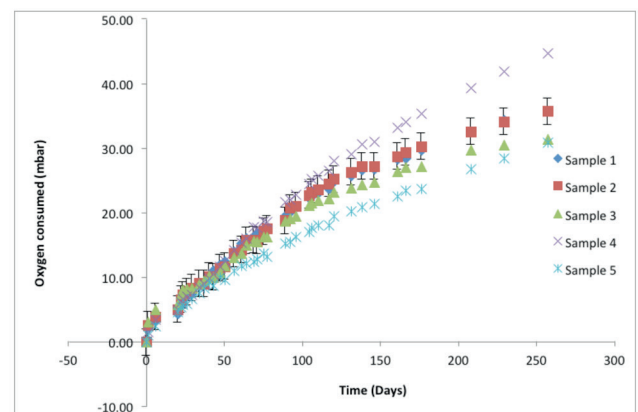


Figure 10: Graph showing oxygen consumption of five un-cleaned samples of wrought iron railing..

Sample No.	Gradient
1	0.2246
2	0.2049
3	0.1866
4	0.2378
5	0.1702

Table 3: Gradients of the trendlines for the oxygen consumption rates of the un-cleaned samples during the first 75 days.

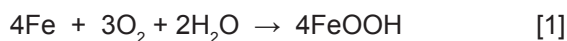
Discussion

The data revealed that it is possible to use the railing wrought iron to produce test samples for determining the effect of selected surface cleaning techniques on the corrosion rate of historical iron. The corrosion rate of the samples was sufficiently consistent to allow comparison between surface treatments that would not be influenced by the nature of the sample. The graph shows a good agreement between the oxygen consumption rates of the samples, demonstrated by the clustering of the points over the first 70 days. Rate agreement between samples 1 and 2 means their points on the graph overlie each other throughout the test period. The oxygen consumption rates do not form straight lines and to increase clarity no trendlines are shown. The trendline gradient for each sample over the first 75 days is given in Table 3. The slight falling off of the rate seen for each sample may be due to the limiting effect of diminishing oxygen concentration within the reaction vessels or a consequence of a build-up of newly formed corrosion products.

Errors need to be considered when interpreting these results; the first is inherent in the oxygen meter readings. Displaying the error bars relating to the meter error for each point renders the graph unreadable; inclusion for one sample (Sample 2) indicates the range of the error (1%) to be factored in for each measurement. A second error can be calculated to interpret results of surface cleaning tests, derived from the maximum difference between corrosion rates of the un-cleaned samples and is 0.05 mbar/day in this case (min. corrosion rate 0.12 mbar/day and max. 0.17 mbar/day).

It is surprising that iron retaining vestiges of paint in a random survival pattern should show good agreement of corrosion rate for samples of similar nominal surface area. Minor differences in sample mass appear to have no influence on corrosion (Table 1). Overall, the homogeneity of the slag distribution likely provides for even corrosion patterns over the metal surface more akin to general corrosion than extensive localised pitting. The actual surface area of the samples clearly differs from calculations related to sample dimensions as surfaces are uneven and pocked and it may be this, rather than the mass of the samples, that creates differences in corrosion rate. Digestion and analysis of samples following testing of surface cleaning techniques will determine chloride content and any variation will offer more insight into sample standardisation.

Additionally, the corrosion rate of the railing can be calculated as loss of metal using a simplified equation for corrosion [1] if conversion to a range of oxidation products is not considered. Analysis of corrosion products may reveal a more complex corrosion outcome but this simplified approach is based on the reasoned estimation that FeOOH predominates.



This can be related to total loss of the metal, assuming corrosion rate does not change with time, within a calculated number of years in a very humid mid-range temperature environment of 90% and 20 °C to provide some comparative indication of corrosion rate.

The calculation uses the change in pressure of oxygen in the reaction vessel over the test period (atmospheres), temperature (maintained at 20 °C), volume of gas within the reaction vessel (litres) and the gas constant ($R = 0.08205746$) to calculate the number of moles of oxygen consumed by the corrosion of the sample [2]. The ratio of oxygen moles to iron moles in the corrosion reaction is given in Equation [1] and is used to calculate the number of moles of iron converted to FeOOH during the test period by assuming that [1] is the only reaction occurring. Changes to oxidation state of iron in reactive corrosion product phases to balance dissolution of metallic iron can occur immediately after wetting and do not involve oxygen consumption (Stratmann and Hoffmann 1989). This would not be detected by the measurement technique used here but is likely to be insignificant as the samples are constantly at 90% RH. Any contribution from these reactions is not considered in this calculation. The mass of FeOOH per unit area can be calculated [3] and, using the density of iron (7.874 g/cm³) and, assuming uniform corrosion, the depth of metallic iron becoming FeOOH per unit time can be derived using [4] (Table 4).

• Ideal gas law:

$$PV = nRT \quad \text{or} \quad n = \frac{PV}{RT} \quad [2]$$

P = pressure of gas (atm.)

R = ideal gas constant

V = volume of the gas (l)

T = temperature (K)

n = amount of substance (moles)

• Converting mass to moles:

$$\text{mass} = \text{moles} \times \text{molar mass} \quad [3]$$

• Calculating depth of iron converted to FeOOH:

$$\text{depth} = \text{mass loss} / \text{density of iron} \quad [4]$$

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Sample No.	Vol ume of gas in vessel (l)	Oxygen consumed during corrosion reaction over first 75 days ($\pm 1\%$) (mol)	Iron converted to FeOOH during test period (mol)	Iron converted to FeOOH during test period (g)	Sample surface area (cm ²)	Total iron conversion per unit area (g/cm ²)	Depth of iron converted per year (μm)	Time to loss of metal to depth 0.5mm on each surface (years)	
1	0.15	0.000105	0.00014	0.00782	35.8	0.000218	1.35	371	
2	0.15	0.000106	0.000141	0.00787	36.2	0.000218	1.34	372	
3	0.16	0.000107	0.000143	0.00798	37.7	0.000211	1.31	383	
4	0.15	0.000115	0.000154	0.00858	36.9	0.000233	1.44	348	
5	0.16	0.0000913	0.000122	0.00680	35.2	0.000193	1.19	419	
Average								1.33	378
Standard Deviation								0.09	26.0

Table 4: The results of calculations of mass of iron converted to FeOOH during corrosion at 90% RH over the first 75 days and extrapolation to loss of metal over time.

These results show an average rate of conversion of metallic iron to FeOOH of 1.33 μm per year and an expected average of 378 years to loss of metal to a depth of 0.5mm on each surface of a sample. Initially seeming unreasonably slow, these rates are likely to reflect protection afforded by existing corrosion products and a lack of chloride contamination. A depth of 0.5 mm represents conversion of approximately 20% of the metal which is no insignificant amount. Good agreement exists between calculated results for all five samples despite magnification of minor differences in rate over long time periods.

The reality of these calculations in practice is limited as the build-up of corrosion products may change corrosion rate which will also differ according to fluctuations in RH and temperature, rain events and continued loss of the remaining paint. The potential for localisation of corrosion in pits complicates calculations of expected lifetime, as does the realisation that railings would fail as functional items by becoming physically unviable well before all metal disappeared. Quantitative measures such as loss of metal cannot, alone, reflect the change in heritage value of an object. This is judged on criteria comprising tangible corrosion events and less tangible factors such as aesthetics and object context. Fit for purpose is also a factor as pitting corrosion could cause failure of a water tank; it is no longer fit for purpose despite perhaps 98% of the metal fabric remaining. This begs much broader questions of how the success of conservation is to be measured for historic objects if fit for purpose is part of the assessment of its heritage value.

Conclusion

The research reported here demonstrates that historic wrought iron can offer a degree of reproducibility that enables its use for producing test samples for quantitative experimental study using remote detection of oxygen consumption to assess small corrosion rates. Minor differences in sample material and levels of error can be factored into the results when data is interpreted. This offers encouragement to workers wishing to use historic sample material instead of analogues in research projects.

Future work

Samples of the railing with surfaces prepared by five preparation methods, whose selection was determined by prior testing by Historic Scotland (Wilson et al. 2008), are undergoing corrosion at 90% RH with regular measurement of oxygen consumption. The techniques are:

- Airbrasive blasting:
 - glass beads;
 - aluminium oxide;
 - crushed walnut shells.
- Wire brushing after:
 - immersion in sodium hydroxide solution;
 - flame cleaning.

This will identify the 'best performing' and most suitable method for surface preparation of historic wrought iron. Publication of these results is in progress. This will be followed by investigation of corrosion rates of samples prepared by the 'best performing' technique and coated with a range of paint systems commonly applied to heritage wrought iron to identify a 'best performing' coating system.

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