Geochemical survey and metalworking: a case study from Exmoor, southwest Britain

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ABSTRACT: A series of geochemical surveys were undertaken on Exmoor as part of a multifaceted investigation into the rich ironworking history of this landscape. Exmoor was created by semi-metamorphosed sedimentary lithologies, which contain a range of iron ore sources. The Romano-British iron smelting site at Sherracombe Ford was one of several production complexes to exploit these ores and excavation demonstrated evidence of iron smelting and primary iron smithing. Site investigation strategies utilised a range of techniques, including geochemical survey, gradiometer survey, and excavation. This paper describes one geochemical survey applied at an intra-site level to investigate a large compacted smithing floor and a multiple-use furnace location. The discussion is focused on how geochemical surveys can be conducted on sites with archaeometallurgical remains and how such data can be analysed and interpreted. Significantly, geochemical and gradiometer surveys are compared and the datasets integrated, the resulting plot indicating a significant potential for the application of these technologies on ironworking sites.

Keywords: Geochemical survey, gradiometry, Exmoor, ironworking.
Figure 1: The location of the study area and Sherracombe Ford, relative to other significant Romano-British ironworking sites on Exmoor and the solid geology.
the slag heap to examine stratigraphy and site chronology, and quantitative sampling to characterise technologies and processes. Gradiometry survey was used as an exploratory technique and both gradiometry and geochemical surveys were used in the early stages of site investigation. This paper describes the evolution of the geochemical survey techniques applied at the site and describes the value of geochemical survey when used in combination with more conventional archaeological methods of gradiometer survey and excavation.

Introduction: geochemical survey

Geochemical survey has yet to develop as a standard archaeological technique. It has been used in an ad-hoc manner at a variety of different levels, ranging from the landscape (Aston, et al., 1998) to the site-specific (Linderholm and Lundberg, 1994). At one end of the spectrum it has been used in a univariate form to measure phosphate concentration (Proudfoot, 1976), whilst at the other end of the spectrum it has been applied as a multivariate analytical tool (Entwhistle, et al., 2000). Ambitious claims have been made about what geochemical survey can achieve, from site detection on a landscape scale (Jackson, 2001) through to analysis of intra-site activity (Cook, et al., 2003). However, there has been little consensus amongst practitioners regarding methodological approaches and achievable objectives, which has produced little critical development in application.

The basic premise of archaeological geochemical survey is that the activities of human beings can load geochemical deposition into their local, regional and global environments (Heron, 2001). Through detailed analysis of cations within the soil samples, it is possible to detect geochemical deposition resulting from human activity as part of the archaeological record. Traditionally this has been achieved through collecting soil samples and subsequent laboratory analysis. Recent advances, such as PXRF (portable X-ray fluorescence) allows the taking of measurements of metallic elements within the field (e.g. Craig, et al., 2007), offering considerable savings in both time and money, making the process more analogous to conventional methods of archaeological geoprospection.

Archaeological geochemical researchers have yet to fully and systemically investigate the relationship between geochemical deposition and human activities, and mechanisms of deposition and retention. However, Middleton and Price (1995) successfully demonstrated different activity zones within houses based on geochemical deposition, whilst Cook et al. (2003) defined Romano-British metalworking within buildings, both examples being local level spatial surveys. However, it is worth noting that archaeological geochemical deposition can occur on both regional and global scales, as shown by Hong et al. (1994, 1996) analysing Romano-British lead (Pb) and copper (Cu) pollution in Greenland ice cores transported through air borne pollution, and by Thorndycraft et al. (1999) analysing regional tin (Sn) pollution in alluvial sediments within valley floors.

Figure 2: Earthwork plan of the Sherracombe Ford Romano-British ironworking complex, with key aspects of site investigation defined (drawn by Rob Wilson-North).
In this study, geochemical survey has been applied at an intra-site level and is used to investigate areas/features within a site. Sampling intervals are small (analogous to geophysical survey intervals), with the aim of detecting large geochemical signatures caused by metallurgical activity. The small sample interval creates a more representative sample population. For example, a 1m sample interval allows one sample to represent 1m$^2$ of archaeology. A 10m sample interval (as routinely employed on landscape scale survey, e.g. Jackson, 2001) is used to represent an area of 100m$^2$. Such large sample intervals are clearly unsuitable for intra-site analysis and in general raise issues about the representative nature of a geochemical survey plot. In using geochemical survey to detect past metallurgical activity, the following assumptions are made:

1. All human activities deposit chemical elements and compounds into their immediate environment.
2. Much of this geochemical deposition is ephemeral and undetectable to the archaeological geochemist.
3. Metalworking will deposit chemical elements into the ground in the vicinity of the activity.
4. Some of this deposition from metalworking will be persistent within the soil profile.
5. This persistent deposition can be recognised against the background variance within a survey area/activity area. The deposition of the metallic elements from metalworking is greatest in the immediate vicinity of the location where the metallurgical activity took place and can be deposited in the form of macro-artefacts (e.g. slag) and also micro-artefacts (e.g. microscopic particulates and gaseous deposition).

Moving away from the activity focus, the level of geochemical deposition decreases. In addition, some features found in the archaeological record are impermeable to geochemical deposition, e.g. a stone wall. Therefore, as well as identifying activity areas, geochemical survey has the potential to identify associated infrastructure, such as walls, through abrupt changes in geochemical concentrations.

This geochemical survey utilised a multivariable approach in both data capture and analysis. From individual soil samples, multiple elements were measured and intervariable correlation via Principal Components Analysis (PCA) was used to treat the data and identify correlations which were used to infer metalworking areas. Comparison of the original variables to the PCA allowed the identification of important elements, specifically defining impurities associated with the ore sources exploited.

Materials and methods

Field method: Gradiometer survey

The gradiometer survey carried out across platform A2 used a Geoscan FM36 gradiometer, using a 0.5m sample interval, walking 0.5m traverses. Following initial interpretation, several important geophysical anomalies thought to represent in situ remains from iron smelting related activities were further refined using a 0.25m sampling interval on 0.25m traverses and a 0.1m sampling interval on 0.1m traverses. All data was processed in Geoplot and exported into ArcGIS (v. 8.3) via Surfer (v. 8.0). All gradiometer surveys were undertaken by Substrata (Ross Dean).

Field method: Excavation and geochemical survey

The excavation strategies adopted were guided by the results of the gradiometer surveys. A large open-area trench of 12x6m positioned across the platform was the focus of excavation. Geochemical survey was undertaken during the excavation at a sample depth of c. 50cm within the excavation trench. Initial excavation exposed a blanket of colluvial ‘hillwash’ covering the archaeological remains. This was excavated to reveal the upper archaeological contexts and it was at this depth the geochemical samples were taken, c. 0.5m BGL (Below Ground Level). The excavation trench was cleaned by trowel, to give a coherent surface for geochemical sampling. Samples were taken using a trowel cleaned in distilled water and sample sizes were c. 10g. When collecting soil samples using a trowel, the sample will generally be context specific, i.e. the soil comes from a shallow scrape at one defined depth, which in most cases will lie within a single context. It is important to note that the extensive compacted smithing floor and furnace location, two of the central features of the excavations on the platform, were not fully exposed at the level at which the geochemical sampling took place.

Laboratory method: Geochemical survey

All samples were freeze dried after collection. After drying, each sample was homogenised by sieving through a 2mm stainless steel sieve. The element analyses required an acid digestion of the sediment sample, allowing some matrix decomposition and cation exchange. All samples were subjected to a pseudo-total acid digestion, a nitric acid (HNO$_3$) and hydrochloric acid (HCl) procedure which displaces the loosely bound cations within the samples but does not break down the silicate structure of the sample. This extraction procedure is less influenced by the underlying geology and soil structure, as the silicate lattice of the soil structure is not destroyed (Van Loon, 1985).

Atomic absorption spectrophotometry (AAS) was used to measure element concentrations via a Unicam 939 atomic absorption spectrometer with the Department of Geography sediment laboratories at the University of Exeter. AAS provides a relatively quick and simple method of univariable analysis. The detection limits are good (between 1 and 100ppm, depending on the element measured) and the analytical precision is high. Soil organic content was measured in each sample using Loss on Ignition at 450°C.

Data analysis and display

Principal components analysis (PCA) was used to analyse and transform the multi-element geochemical datasets, due to its ability to factorise the data matrix into (soil) sample related and (elemental) variable specific information, thereby visualising...
relationships between soil samples and elements through factor scores and loading plots. PCA takes a group of variables measured over a sample or population of observations, and looks at the interrelationships between them (Johnston, 1978), attempting to summarise data from a large number of variables in a smaller dimensional space, allowing data display in graphical or numerical forms. The PCA will produce a new principal component that is positioned through correlation of the original variables. Where metalworking is present and a survey is conducted on a close interval basis, it can be expected that a strong level of inter-variable correlation will exist. Therefore, when metalworking is present, a principal component can define the geochemical signature caused by deposition of metallic residues. By examining the factor scores it is possible to identify which of the samples had the most significant influence on the positioning of the component. Such samples will have high geochemical loadings and a high degree of inter-variable correlation, and are the sediments likely to have been contaminated by metalworking residues.

A GIS system was used to visualise and examine the geochemical data (MAPINFO, v. 6.5). Within a GIS environment, interpolation functions are generally used to produce a surface model from a dataset of z values. By using an interpolation function it is possible to create a continuous sub-surface from the original geochemical sample points that represents the concentrations of an element across a survey area or transformed data values, such as factor scores from a PCA. An Inverse Distance Weighted (IDW) function was used, which calculates the value of grid cells over the mapping area, with each data point in the calculation weighted by its distance from the centre of the cell. Further data modelling was undertaken within ArcGIS (v. 8.3) to allow synthesis of the geochemical data with excavation and gradiometer data sets, and data modelling and visualisation in a pseudo 3D environment.

Results

The excavation of platform A2 revealed a range of well-preserved and significant ironworking features, including a large furnace setting and smithing floor. The furnace setting survived to a height of 1.5m and preserved evidence of at least 10 episodes of reconstruction and use, set within a stone-revetted hollow and connected by means of a heavily trampled ramp to an extensive smithing floor (Fig. 3). The smithing
floor is exceptional, reaching in places 0.4m in depth of solid compacted residues formed during the primary smithing of hot iron blooms extracted from the adjoining furnace. On the platform, the remains of several other furnaces that had been truncated by successive re-cutting of the platform to reconfigure the working area were found.

The unwanted slag and waste materials from the processes operating on the platform were dumped downslope on the accreting slag heap. Excavation through the heap revealed the cyclic nature of deposition. The contexts which formed the slagheap could be categorised into three generic types:
1. Re-deposited ‘natural’ soil, often stony with little other material.
2. Technological debris layers, containing abundant slag, furnace lining, and charcoal. Many of these contexts were very loosely packed with large cavities but little infilling soil matrix.
3. Mixed layers with varying quantities of the 1 and 2 type of layers in them. The technological debris in these layers is generally smaller in size and quantity than in 2.

Returning to the geoprospection surveys over the platform, gradiometry identified the smithing floor in the 0.5m interval survey as anomalies GA2 and GA3 (Fig. 4). However, the exact shape of the smithing floor was not defined, and the pre-excavation interpretation of these two anomalies was as possible furnaces. It is interesting that the smithing floor produced two apparently distinct magnetic dipoles. These have subsequently been interpreted as two areas of heavily concreted smithing slag within the floor. The furnace location was clearly identifiable as the large dipole GA1.

Figure 4: The gradiometer survey results from the platform A2 survey.
Geochemical survey on platform A2 produced a PCI that accounted for 35% of the original variance in the dataset and a PCII that accounted for a further 19%. The location of PCI was heavily influenced by the elements Fe, Mn and Cu, with only Cr and Mg displaying a negative association with PCI, and Zn and K both having a weak positive association. The plot produced from the PCI factor scores clearly identifies the smithing floor (excavation contexts 49, 50, 69, 56 and 37; Fig. 5). These contexts all contain hammerscale, charcoal, and semi-molten slag that had been hammered out off the raw iron as it was forged and then trampled underfoot by the working blacksmiths to form a thick concreted surface. The area of higher PCI factor scores also extends over context 60, which had distinctive heavy black staining but did not contain large amounts of the compacted smithing slag.

The second major geochemical anomaly defines the location of the furnace setting. The location represents the boundary between two different sediment matrices and is defined by a clear geochemical boundary. The sediment units producing this geochemical change can be divided into two groups. The first is composed of contexts 8, 5 and 61, and represents the furnace location and the soil matrix that has infilled the furnace location. The second is the soil profile behind the furnace that represents the packing and the filling at the back of the furnace location. The furnace location is not defined through high PCI factor scores but through a geochemical difference between two areas, both of which have a lower geochemical loading than the smithing floor.

The geochemical definition of the smithing floor provides an interesting comparison with the 0.5 m gradiometer survey, redrawn as a simple polyline file (Fig. 5). The smithing floor gradiometer dipole (GA2) did not display a classic dipole form, and the magnetic response effectively identified an area of high Fe concentrations related to heating, which could be features such as hammerscale, slag deposition, or a furnace. The form of the archaeological feature is not apparent. In contrast, the geochemical image gives almost the exact definition of the smithing floor. However, the furnace setting is defined exceptionally well by the gradiometer dipole GA1 and correlates well with the geochemical change.

The examination of the individual elements adds further information. The Fe concentrations (Fig. 6) produce a good definition of the contexts that comprise the smithing floor (49, 50, 37 and 69) and also the furnace setting (8, 61 and 62). The Fe concentrations are still elevated in context 60 surrounding the smithing floor, although not as high as on the smithing floor. The Cu concentrations also produce a good definition of the smithing floor (contexts 49, 50, 37 and 69) and the furnace setting (Fig. 6), also with weaker concentrations over context 60. The Cu complexes and compounds in the smithing floor would have originated from the original ore material exploited for smelting. Further elements followed this same depositional pattern, including Mn (Fig. 7), which also displayed a high positive association with PCI.

Of further interest are the interrelationships between different metal elements and the magnetic responses recorded by the gradiometer survey (Fig. 7). Although already viewed as a simple line drawing, the interrelationships of these modelled surfaces can be viewed within a quasi 3D environment. Visually, the communality between data groups is obvious,
with the smithing floor a dominant feature in all of the plots, and with the furnace location most precisely defined by the gradiometer survey (nT) and the manganese concentrations.

In particular, examination of the Fe concentrations and the gradiometer nT scores demonstrates a close visual correlation, with the nT responses effectively representing a combination of the Fe concentration and its state of oxidation or reduction. The relationship of the Fe concentrations to the magnetic nT responses were subjected to a further PCA analysis using raw (unstandardised) data. The resulting PCI accounted for 61.4% of the original variance in the dataset, only removing c. 38% of uncorrelated variance. The resultant plot of the contribution of the factor scores succinctly defines both the smithing floor and the furnace setting, demonstrating a high level of correlation with the excavated remains (Fig. 8).

Figure 7: The visual inter-relationship of the gradiometer results, PCI factor scores and Fe, Cu and Mn concentrations.

Figure 8: A data plot showing the fusion of the Fe concentrations and the gradiometer nT responses, through a PCA analysis of these two variables and plotting of the PCI factor scores.
Discussion

The results from this study demonstrate the considerable potential of using close interval, intra-site, high-resolution geochemical survey to investigate metalworking remains in combination with gradiometer survey. The PCI factor scores defined a series of anomalies that correlated very closely with the excavated archaeometallurgical features. The smithing floor was a physically large feature within the survey area which had a strong geochemical signature. These two factors, the area it covered relative to the overall survey size and the level of its geochemical enhancement, made this feature clearly identifiable in the geochemical results. Undoubtedly, the high level of correlation between Fe, Mn and Cu, and the subsequent positioning of the PCI, is primarily defined by samples from the smithing floor. The furnace setting was identified as a ‘negative’ geochemical feature based on the PCI factor scores, with the position of PCI derived from the metallic residues of the smithing floor. This ‘negative’ feature was the junction between two types of sediment matrices.

The presence of high concentrations of Cu and Mn, strongly correlating with the high Fe concentrations, suggests that both these elements were impurities within the ores being smelted at Sherracombe Ford. Small amounts of chalcopyrite are commonly found within the iron ore deposits in the Devonian rocks on southern Exmoor, and Mn is frequently noted as ‘wads’ within the ore bodies (Edwards, 2000). Therefore, a mode of deposition is suggested where Cu was deposited onto the smithing floor by the removal of furnace slag trapped within the iron blooms through primary smithing. Certain elements, such as Mn, are valuable additions to the iron smelting process because they act as flux, replacing iron in the slag (Tylecote, 1986, p.127). It is possible that Mn bearing ores were deliberately sought at Sherracombe Ford for smelting, potentially delivering higher iron bloom yields. Alternatively, Mn could have been added separately to the furnace as a flux, although this is considered less likely.

The geochemical and gradiometer surveys produced a high level of correlation. The furnace setting stands out as an example of the combination of these two techniques. The metallic component of the furnace setting had been removed. The location is identifiable as a weak geochemical signature caused by the change in soil structure. The preservation of the furnace setting had left intact the clay surround that had witnessed multiple firings. This produced a strong dipole identified by the gradiometer survey. Likewise, the smithing floor was identifiable as a series of high nT responses, whilst the geochemical survey provided the plan of the structure, identified through strong geochemical enhancement.

The integration and synthesis of the iron concentrations with the gradiometer data undoubtedly refines the definition of anomalies on the platform, which can be reconciled with archaeological features. The combination of the two datasets defines the furnace setting and the smithing floor with a high level of accuracy. In fact, when compared to the excavation schematic, the combined geoprospection data is moving to a level of context mapping, within a pre-excavation environment. Of course, with multi-context environments in a 3D world which have vertically accreted overtime, the mapping of contexts will be limited to the depth to which the geoprospection data is gathered. Nonetheless, this survey highlights the potential for geochemical survey to be combined with gradiometer data to map contexts on a pre-excavation basis and also to aid in the interpretation of contexts in a post excavation phase.

Conclusion

The investigation of the ironworking remains at Sherracombe Ford is significant for two reasons. Firstly, new methods of site investigation were explored, specifically, the integration of geochemical data with gradiometer data and their comparison with excavation results. The methodological development of compressing multi-element data using PCA and expressing this as spatial data plots has made the technique analogous to more conventional methods of geophysical survey. This provides a more accessible tool for archaeologists to use and understand.

By virtue of the fact that this geochemical survey methodology has been applied on an archaeometallurgical site, there are high geochemical loadings in the immediate site environment caused by the metalworking processes. This demonstrates geochemical survey to be a suitable tool to investigate such residues contained within archaeological contexts. This survey represents the first step in using a robust geochemical survey methodology to investigate metalworking sites. There is a potential to identify features that are ambiguous upon excavation by virtue of their geochemical signature, e.g. small Bronze Age furnaces; to identify remains and features on a pre-excavation or in lieu of excavation basis, or to help inform excavation, as in this case, and also to provide an environmental perspective on the archaeology of pollution, e.g. lead residues.

From an archaeological viewpoint, the investigations at Sherracombe Ford have revealed extensive evidence for Romano-British iron smelting in the uplands of Dumnonia, an area of scant Romano-British archaeological remains. The discovery of these sites has challenged conventional models of landscape evolution in the southwest of Britain and modified the archaeological perspective of Romano-British control and settlement.

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