Trace element fingerprinting of ceramic building material from Carpow and York Roman fortresses manufactured by the VI Legion

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A B S T R A C T

Trace element compositions of raw clay, fired clay and Roman ceramic building material (CBM) from Carpow Roman fortress, Newburgh, Scotland and the city of York, England have been determined through the use of inductively coupled plasma-mass spectrometry (ICPMS). It is confirmed that the firing of clay does not disturb any of the sample's trace element composition and that trace element protolith identification is an applicable tool for fingerprinting the source of material used in CBM construction. It is also demonstrated through the particular proportions of LREE/HREE; Th/Co; La/Sc; La/Lu; Eu/Sm values on CBM from Carpow that these material were likely manufactured from clay used in the York Roman tile manufacturing industry, therefore suggesting importation. The transport of CBM from York to Carpow provides a useful addition to known examples of the long-distance shipping of CBM. It is postulated that similar analysis to that conducted here could be used to identify important trade networks within the Roman Empire.

1. Introduction

The use of trace element analysis to characterise geological samples is a common practice within geological science and one which has also been successfully utilised in archaeological studies to match particular clay sources to archaeological ceramic building material (CBM) such as brick and tile (e.g. Mallory-Greenough et al., 1998; Meloni et al., 2000; Gomez et al., 2002; Montana et al., 2003) as well as pottery (e.g. Beier and Mommsen, 1994; Montana et al., 2003; Vince, 2004, 2007; Vince and Steane, 2004). To date this technique has been limited to regional source identification of material, for example in Sicily where Montana et al. (2003) investigated thin-walled ware from an Hellenistic-Roman site at Segesta. However, the technique has only had limited use as a tool for localised identification of source material. The lack of reporting of results from geochemical analysis of individual samples was identified as an issue in early studies by Ives (1975) and the reporting of full trace element data or that of Rare Earth Element analysis has continued to be sparse. Specifically, although similarities in alluvium, soil and manufactured archaeological material have been noted, the geochemistry of the clay protolith from known extraction sites has not been compared to final manufactured material. To illustrate the use of trace elements as a tool to identify the raw materials used in the manufacture of CBM we have carried out analysis of known clays used for CBM manufacture by the Roman VI Legion garrisoned in York and at Carpow Roman fortress, Fife (Fig. 1).

Carpow Roman fortress lies on the south bank of the River Tay and underwent excavation throughout the 1960s and 1970s (Birley, 1965; Dore and Wilkes, 1999). The fortress was constructed by a vexillation (a detachment of a Roman legion usually formed for a specific purpose) of the VI Legion previously stationed in York (or the VI Legion minus a vexillation) during a single period of occupation between c. AD 180 and c. AD 220, which was followed by a single phase of methodological destruction (Dore and Wilkes, 1999). Stamped tiles at Carpow confirm the presence of the VI Legion at the site with examples stamped LEG VIC B P F being present; the titles victrix and pia fidelis were used by the Legion from its arrival in Britain and throughout the Roman period while the letter B represents the title Britannica which could relate either to Commodus who took the title in AD 184 or to Septimius Severus who took the title with his sons in AD 210 (Collingwood and Wright, 1992; Warry, 2010). Two stone buildings were recorded (the headquarters and legate's residence) and 224 fragments of CBM were examined from the Carpow site (Birley, 1965). It has been suggested that there is no reason that tiles could not have been...
made on site (Birley, 1965), however, fieldwork at Carpow (this study) revealed few local clay deposits. The similarity of CBM from Carpow and York has previously been remarked upon (Butler, 1897) but no further study was made. Given the lack of suitable clay at Carpow it seems possible that tile may have been imported to Carpow from the VI Legion base in York as both the River Ouse at York and River Tay at Carpow were navigable to the sites of the respective forts. The presence of the VI legion in both York and Carpow offers the potential to determine the level to which the site at Carpow was self-sufficient in terms of resources or whether supplies were transported from the legionary base in York.

Tile manufacture was dependant on the availability of suitable clays, temper, fuel, water and transport links (Swan, 1984). Clay would be dug during the autumn, be left to weather over the winter when wood would be collected (Warry, 2006). Temper (usually sand [Swan, 1984] and so lacking any major potential for geochemical contamination) would be added and tiles would be manufactured in summer and autumn (Collingwood and Wright, 1993). Following manufacture the tiles would be air dried and then fired at ~1000°C.

Riverine clay deposits have been postulated as being a potential source for clay that could have been extracted for the manufacture of CBM at Carpow (Birley, 1965). The River Tay predominantly drains Grampian Highland (Ordovician – Devonian intrusive granites/granodiorites and meta-sediments) as well as some Midland Valley terrane sediments (carbonates, mudstones, sandstones and Mafic-untramafic shallow-extrusive volcanic units; Trewhin, 2002; Fig. 1). Clays utilised for tile manufacture in York are Devensian glacial deposits (Field observations, this study; Fig. 1A; Brenchley and Rowson, 2006). These glacial-lacustrine clays formed from local sediment inputs and weathered glacial material (e.g. Triassic mustones and sandstones; Whyman and Howard, 2005). Importantly these clays are lacking in any mafic, or igneous of that matter, input and so will be geochemically distinct from the River Tay clays at Carpow. For a full description of the relevant geologies see Brenchley and Rowson (2006) and Trewhin (2002). This distinction in geochemistry, combined with the known history and links between the two fortresses make this an ideal case to test whether trace element geochemistry can be used to fingerprint the source of CBM. We specifically use ratios of La, Th, Co and Sc as they reflect different mafic/felsic sources and are therefore strong indicators of provenance (Rollinson, 1993). In addition the ratios of Light Rare Earth Elements (LREE; La, Ce, Pr, Nd, Sm, Eu) and Heavy Rare Earth Elements (HREE; Tb, Dy, Ho, Er, Tm, …

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**Fig. 1.** A) Location map of Geological terranes, Roman fortresses sampled and relevant geography for this study. B) Drift geology map for the area surrounding Carpow Roman fortress. C) Drift Geology map for the area surrounding York Roman fortress, also showing Hungate clay sampling site. Both drift geology maps have been simplified from the BGS drift geology maps 1:50,000 sheet 48 East (Drift); 1:50,000 sheet 48 West (Drift) and 1:50,000 sheet 63 (Solid and Drift). D) Photo of Hungate archaeological site showing Roman ground level clay (diagonal shading) and a possible Roman clay extraction pit. See text for Discussion.
Yb, Lu] were examined as they are resistant to weathering disturbance (Rollinson, 1993).

We apply trace element analysis, including the full suite of Rare Earth Elements (REE), to identify whether CBM from Carpow was manufactured on site or imported from York. We do this through the analysis of clays known to have been exploited for CBM manufacture in York, CBM from York and CBM from Carpow, to identify the source of tile manufacture. We demonstrate, like Meloni et al. (2000), that the firing of clay does not disturb the utilised trace element composition of the clay and therefore CBM trace element composition is indicative of the source clay. Furthermore, we demonstrate that Carpow and York CBM samples are chemically similar and compare our data to published data (Mallory-Greenough et al., 1998; Meloni et al., 2000; Gomez et al., 2002; Montana et al., 2003) to show that CBM samples from different parts of the Roman Empire are geochemically distinct. We therefore conclude that the CBM from Carpow Roman fort was imported from York. In so doing we confirm the use of REE and trace elemental techniques to fingerprint CBM as well as suggesting new information about the logistical support and supply of the Roman army.

2. Materials and methods

2.1. Sample description and preparation

Three raw clay samples were collected from separate areas of exposed natural clay in the Hungate archaeological site, York. Given that the assumed site of the legionary tile kilns is located nearby, some 150 m to the north-west. The Hungate area may have been used as a clay source for Roman tile manufacture. The area also seems to have been used for clay extraction in the medieval period (Kendall pers. comm. Fig. 1). Seven CBM samples from York were provided for analysis by the York Archæological Trust and two unstratified CBM samples were collected from Carpow Roman Fort (Fig. 1). No raw clay was found in the vicinity (~100 m) of Carpow Roman fortress. The selection of clay from the Hungate site for the purposes of this study is significant; although the site of the VI Legion tilelery has not been found, the presence of waste and kiln debris associated with VI Legion tile stamps in both the Aldwark area and at Peasholm Green suggests that the Legionary kiln was located somewhere nearby (Brinklow et al., 1986; Betts, 1985; Swan and McBride, 2002). This places the probable location of the Legionary kiln to within 140–220 m of the Hungate site, making it highly probable that the Hungate area was used as a clay source for Legionary tile production.

Previous petrographic analysis has been undertaken on 7721 kg of Roman tile from 213 archaeological excavations undertaken in York since 1973 and 584 kg of tile from excavations at Haslington East undertaken by Department of Archaeology of the University of York giving a total of 8.11 metric tons of tile. The fabrics of every individual sherd (c. 36,000 of them) were identified with a ×10 hand lens producing the sequence of 19 fabrics (McComish, 2011). Representative samples of these fabrics have since been analysed using a petrological microscope and a dredogram was produced using Ward Linkage and Euclidean Distance. This concluded that there were five fabric groups (Finlay, 2011), of which those that constitute >75% of York Roman CBM have been analysed.

Three areas of raw clay from Hungate were sampled. The raw clay was sampled by removing ~10–20 cm of surface clay (to reduce potential contamination or mobilisation of elements from weathering), then digging out a sample with a trowel. The removed surface clay was then returned to the sample site. The raw clay samples then had ~5 mm of surface clay removed to waste to avoid any surface contamination. The clean clay was then air dried over 48 h, after which an aliquot sub sample was removed, this was then fired in an oven at 800 °C for ~8 h. The CBM samples had all surfaces removed with a rock saw to remove any weathered material, cleaned with a diamond polishing wheel to remove any potential contamination from the rock saw, and then thoroughly washed in MilliQ ultra pure water to remove any surface contamination. The air dried clay, fired clay and CBM samples (> ~50 g) were then powdered in a zirconium mill to prevent any further metal contamination.

2.2. Analytical methods

Powdered samples were prepared for trace element analysis following the standard Durham University Earth Science Department protocols established by Ottley et al. (2003). Briefly, this involved digesting 0.100 ± 0.001 g of powder in a 4 ml 40% HF – 1 ml 69% HNO₃ solution for 48 h before evaporating to dryness and redissolving in HNO₃ acid, the final solution being 3.5% HNO₃. This solution then had a Re and Rh internal standard added to compensate for possible calibration drift, matrix suppression and dilution errors. The samples were then analysed for Sc, Ti, V, Cr, Mn, Co, Ni, Cu, Ga, Pb, Sr, Y, Zr, Nb, Cs, Ba, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Hf, Ta, Pb, Th and U on a Perkin Elmer Sciex Elan 6000 ICPMS. Calibration was achieved via the use of in-house standards and international reference materials (W-2, BHVO-1 and AGV-1 standards) as well as a blank and standard sample being run every 10 samples to ascertain instrument calibration stability. This produces detection limits of less than 5ppb for the REEs except Ce and less than 10 ppb for high field strength and large-ion lithophile elements. Reproducibility was monitored through replicate runs of sample AF05-10 and was consistently less than <8 RSD and typically <3% RSD (see Table 1). Furthermore solutions of international standards BE-N and BIR-1 (not used for instrument collaboration) were repeatedly analysed and are consistently similar to accepted values (Potts et al., 1992).

3. Results

Full elemental abundances are presented in Table 1. In this study particular attention is paid to Th/Co, La/Sr and LREE/HREE ratios as they reflect differing geological inputs (see Discussion). Th/Co ratios range from 0.50 to 0.89 in the raw clays, 0.68 to 0.78 in the fired clays, 0.56 to 0.79 in the York CBM and 0.85 to 0.89 in the Carpow CBM. La/Sr ratios range from 2.8 to 3.1 in the raw clays, 2.8 to 3.4 in the fired clays, 2.6 to 3.3 in the York CBM and 2.7 to 2.8 in the Carpow CBM (Fig. 2). LREE/HREE ratios range from 14.6 to 17.5 in the raw clays, 15.0 to 17.9 in the fired clays, 15.5 to 18.8 in the York CBM and 15.4 to 15.8 in the Carpow CBM (Fig. 3). All of the Th/Co, La/Sr, Lu/Eu and LREE/HREE ratios overlap between the raw clay, fired clay, York CBM and Carpow CBM.

4. Discussion

For it to be possible to identify the source clay involved in CBM manufacture to be effective we need to confirm that:

1) Firing of clay does not disturb the trace element abundance and composition.
2) CBM samples need to be chemically similar to that of the source clay (e.g. Glascock and Neff, 2003 and references therein).
3) There has to be a difference in trace elemental ratios of CBM from different sources (e.g. Glascock and Neff, 2003 and references therein).

This study tests the effect of firing by comparing the elemental analysis (n = 35) of both fired and unfired aliquots of York clay
samples. Importantly there was no evidence of any geological heterogeneity within the clay. To identify the source of the CBM this study concentrates on the REE compositions of the samples as weathering has been shown to have little effect on REE composition in clastic sediments (McLennan, 1989) making them the ideal tool for provenance studies. REE have been used for provenance studies by a wide variety of authors (e.g. Mallory-Greenough et al., 1998; Meloni et al., 2000; Gomez et al., 2002; Montana et al., 2003) however very few have analysed for the full suite of REE (e.g. Mallory-Greenough et al., 1998). Therefore data from previous studies must be treated with caution. Also utilised are Th/Co, La/Sc, Eu/Sm and La/Sm ratios as these are good indicators of provenance (Rollinson, 1993). For example, La and Th are found in greater quantities in volcanic rocks than in sediments (Ballhaus et al., 1991). As such, the use of Th/Co is a useful indicator of sedimentary vs. volcanic input into the clay. La/Sc and Eu/Sm ratios are also useful as they are not significantly affected by weathering or metamorphism (Mallory-Greenough et al., 1998). However, when the elemental abundances for the fired and unfired samples are compared the highly linear regression (AF01-10 v AF01-10F, R² = 0.998) indicates that Th and Co are immobile during firing, supporting previous studies (Ehlers et al., 2002; Gomez et al., 2002). Therefore the use of Th/Co, La/Sc, Eu/Sm and La/Sm ratios as good indicators of provenance is supported by previous studies.
rock type (for example high LREE/HREE is characterised by rocks enriched in garnet and zircon; Rollinson, 1993). Furthermore, as the REE composition of sediments is chiefly governed by protolith these changes will be reflected in separate clay sources.

As already noted, most previous studies only publish averaged data of groups assigned by the authors and there are few published studies of CBM in which the full suite of REEs have been analysed. Therefore it is only possible to compare the new data presented in this study to averaged data of groups assigned by previous authors, a situation that is less than ideal. For example, Meloni et al. (2000), analysing CBM from Roman Pavia, provide ratios of HREE/LREE despite not analysing Pr and Er. Furthermore samples from archaeological pottery local to York (Vince, 2004, 2007; Vince and Steane, 2004) as well as Bronze Age and Roman Cyprus (Gomez et al., 2002) and Roman Sicily (Montana et al., 2003) have not included full REE analysis, making it difficult to find meaningful comparisons of Roman CBM for our data. Therefore the new data from this study is compared to that reported for Nile pottery (Mallory-Greenough et al., 1998) and distinct from that of Italy (Meloni et al., 2000), however, as previously noted this data is incomplete and so must be treated with caution. Since the York and Carpow CBM REE values are indistinguishable to those of the source clay from York, but are distinct from other global CBM samples, it is likely that both the York and Carpow CBM was manufactured in York.

The Th/Co and La/Sc (Fig. 2a) ratios from York and Carpow are similar and the La/Lu and Eu/Sm (Fig. 2b) from York and Carpow are indistinguishable supporting the hypothesis drawn from the LREE/HREE data that they were constructed from the same clay protolith.

The LREE/HREE values of samples of raw clay, fired clay and CBM from York are similar (Fig. 3), demonstrating that any possible additions to the clay during manufacture do not significantly alter the REE composition of the samples. Furthermore the LREE/HREE values of the samples from York are indistinguishable to those from Carpow. Unfortunately, no local clay samples were available from Carpow to allow a comparison with the CBM.

The spread in data from York and Carpow is distinct from that reported for Nile pottery (Mallory-Greenough et al., 1998) and distinct from that of Italy (Meloni et al., 2000), however, as previously noted this data is incomplete and so must be treated with caution. Since the York and Carpow CBM REE values are indistinguishable to those of the source clay from York, but are distinct from other global CBM samples, it is likely that both the York and Carpow CBM was manufactured in York.

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Again, it is unfortunate that analytical work on archaeological material close to York (Vince, 2004, 2007; Vince and Steane, 2004) does not include all of these elements making comparison impossible. Furthermore the Th/Co and La/Sc data from Italy (Meloni et al., 2000) and Sicily (Montana et al., 2003) plot in close to the British samples, yet the exact likeness of the data is not known since the raw data was not published. However the La/Lu and Eu/Sm (Fig. 2b) data from Italy (Meloni et al., 2000) and Sicily (Montana et al., 2003) plot separately from the British samples. This suggests that the similarity in the Carpow and York data is due to the CBM at the two sites being manufactured from the same protolith rather than being due to natural overlap.

To be certain of our results further work is needed to constrain the natural variability in clay sources throughout Roman Britain, and work is underway to carry this out. However, the data presented in this study demonstrates that the process of firing for CBM does not perturb the trace elemental composition of the material compared to that of the protolith clay. Furthermore, the comparison of York CBM with the assumed source clay shows that they are geochemically indistinguishable and likely confirms that trace element analysis, specifically for REEs can be used to identify the protolith of the CBM. Finally the similarity of the REE and trace elemental data from York and Carpow, and distinction from CBM from other areas of the Roman Empire (Sicily, Cyprus and Italy), makes it likely that the Carpow and York samples were constructed from the same protolith.

4.3. Implications for the logistical support of the VI Legion in Roman Britain

It is proposed that tiles from York were used for building works at Carpow. This has previously been suggested by Butler (1897), although Birley (1965) thought likely is that the tile was manufactured at Carpow. It has been noted that the four VI Legion tile stamp-dies from Carpow are unique to the site and it has been suggested that this indicates that the tiles were not manufactured in York (Bets, 1985; Collingwood and Wright, 1992) otherwise the dies concerned would also be present in York. The geochemical data presented in this study suggests, however, that this was not the case, with the Carpow CBM originating in York. While the variation in stamp-dies between York and Carpow is difficult to explain it is postulated that the Carpow dies may have been used only on specific batches of CBM manufactured in York that were destined for transportation to Carpow.

The use of tiles from elsewhere at Carpow may be a reflection of the relatively short period of occupation at the site. Forts would initially be constructed of timber and roofed with thatch, being rebuilt in stone and tile at a later stage: it is possible, therefore, given the limited period of occupation at the site that only a few of the more prestigious buildings such as the headquarters or specialised buildings such as the baths at Carpow were ever built using CBM. If this was the case it may have been deemed more economical to ship a relatively small number of tiles into the site from elsewhere rather than to establish a tilery on site, especially given the lack of a local suitable clay source.

The distance between York and Carpow would suggest that any CBM was transported using a combination of river and marine transport along the east coast of the U.K. rather than being transported overland by road. Other examples of long-distance shipping of CBM are known from Britain, for example, a tile group recognised by distinctive calcareous clay inclusions are known from various sites across the south of England from Exeter to London which was likely transported around the south coast by ship; these tiles are present in sufficient quantity in London to suggest that they were imported deliberately rather than occurring as ballast (Bets and Foot, 1994). Long-distance shipping of tile has also been recorded elsewhere in the Roman Empire, for example by the first century AD tiles from Imperial brick factories in Dalmatia were being shipped across the Adriatic and along the Dalmatian coast with only a small proportion travelling by road, and roughly a third of the brick stamps seen in Dalmatia appear to have originated in north-east Italy representing further evidence of transportation by sea (Wilkes, 1979). The transportation of CBM from York to Carpow suggested by the present study therefore represents a useful addition to known examples of the long-distance shipping of CBM.

5. Conclusions

Through the utilisation of trace element analysis it has been demonstrated that:

1) Aliquots of raw clay and fired clay from the same clay sample are geochemically indistinguishable and thus indicate that no alteration of material results from the firing process.
2) CBM samples from York are geochemically indistinguishable to the known, local York source clay, therefore reflecting the source of manufacture
3) CBM samples from York and Carpow are geochemically similar, yet, distinct from other published CBM samples from the Roman Empire.

Therefore, this study hypothesises that CBM samples from Carpow Roman fortress were likely manufactured from clay from York, and then transported to Carpow, probably by boat. This further demonstrates the use of trace element analysis in identifying where CBM was manufactured and therefore, with further work, the technique could be used to provide significant information on trade networks within the Roman Empire.

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