

# Recent Archaeometric Artefact Studies at the University of Helsinki

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## Abstract

In recent years, a multi-method research environment for materials science artefact studies has been established at the University of Helsinki. A variety of inorganic archaeological materials, including ceramics, plasters, glass, glazes, metals, and sediment samples, has been subjected to archaeometric examinations and geochemical analysis by employing different analytical techniques, such as scanning electron microscopy with energy dispersive spectrometry (SEM-EDS), particle induced X-ray emission (PIXE), and portable X-ray fluorescence spectrometry (pXRF). The analysed materials derive from archaeological contexts both in Finland and abroad, and the analytical work relates to various multi-disciplinary national and international collaborative projects. Foreign materials, especially from Sweden and Estonia, have been sampled as comparanda for Finnish archaeological finds to carry out interregional comparative analyses for artefact provenancing purposes. The nature of these geochemical studies varies from multi-site provenance-driven investigations to rapid qualitative tests of stray and metal detector finds. This article offers an overview of the archaeological results and the recent methodological developments in archaeometric artefact studies at the University of Helsinki.

## 1 Introduction

Archaeometric, i.e. materials science artefact studies approach archaeological, historical, and art historical research questions by employing geochemical research methods, which allow for the geochemical grouping and raw material identification of inorganic archaeological finds. In this field, typical research questions address raw material exploitation in artefact manufacture, geochemical sourcing (provenancing) of artefacts, and characterisation of ancient technologies, such as glass and ceramic production and metallurgy. In principle, geochemical sourcing aims to identify the raw material sources or production areas of the studied objects of unknown origin and thereby determine the geological origin, provenance, of the artefacts based on their geoche-

mical (or mineralogical) fingerprint. The idea of scientific provenance studies and the application of chemical analysis in artefact source determination derives from the so-called *provenance postulate* that was formulated in the 1970s: ‘there exist differences in chemical composition between different natural sources that exceed, in some recognizable way, the difference observed within a given source’ (Weigand et al. 1977: 24).

In addition to geochemical sourcing, scientific artefact studies are employed to investigate the nature of ancient technologies and reconstruct the production process (*chaîne opératoire*; see e.g. Schlanger 2005). Furthermore, innovations, adaptations, and transitions of technological traditions are studied in archaeological materials science. In terms of archaeological interpretation, the key questions also relate to

the underlying socio-cultural and economic catalysts (e.g. changes in resources, exchange patterns and contacts, diet or food preparation practices) and to the outcomes of the technological changes (e.g. changes in the organisation of production). From a broader perspective, cross-communal and cross-regional patterns of manufacture and exchange can also be investigated by examining aspects such as specialised or centralised production. For example, there are cases in which a certain workshop supplies products for a wider customer base outside the immediate community or a specific workshop specialises in the manufacture of a certain artefact type (possibly related to local raw material resources, specialist technological know-how, or well-established distribution networks).

## 2 Reviewing analytical options

Many geochemistry methods are available for techno-compositional artefact studies today. Perhaps the most commonly applied methods are *inductively coupled plasma mass spectrometry* (ICP-MS), *energy or wavelength dispersive X-ray fluorescence spectrometry* (ED/WD-XRF, portable ED-XRF/pXRF), *instrumental neutron activation analysis* (INAA), *scanning electron microscopy with energy dispersive spectrometry* (SEM-EDS), *particle induced X-ray emission* (PIXE), and *X-ray diffraction* (XRD). These methods vary in terms of their analytical precision and accuracy, and there are method-specific requirements for sample size and processing, as well as varying analytical costs. Importantly, the applicability of different methods to archaeometric research questions also varies – one method may be better suited to explore a given specific research theme or study material than another.

The starting point of an archaeometric artefact study should always be an archaeologically relevant research question – an issue that is potentially resolvable by scientific analysis (Tite 1999: 226). The proposed question defines our analytical strategy, that is, whether a qualitative approach (which elements are present in our samples?) is sufficient or wheth-

er a quantitative analysis (providing elemental concentrations) is required. In many cases, a qualitative, non-invasive analysis is adequate to answer the proposed research questions. For example, a portable XRF can rapidly and non-invasively define whether we are dealing with a bronze or copper object. However, if we wish to examine more complicated research themes, such as form high-resolution geochemical groups, or to trace the geological provenance of the studied artefacts, invasive sampling and laboratory-based analytical methods (ICP-MS, ED/WD-XRF, NAA, or PIXE) are typically required. The need for an invasive strategy applies particularly in the case of heterogeneous materials, such as coarse ceramics. In some cases, invasive sampling may be the only option in order to acquire the information that we seek; for example, high-precision quantitative chemical compositional data may be required for provenancing, or we may need to review a cross-section of our sample material to carry out microstructural and technological analysis. To quote Martín-Torres and Rehren on technical ceramics, ‘*subjecting them to analysis, particularly invasive analysis as is necessary to obtain cross sections and quantitative analytical data, is a hugely productive process, generating a plethora of information not otherwise obtainable. This is not a destructive analysis – it is a constructive analysis of the first order*’ (Martín-Torres & Rehren 2014: 130).

Another defining factor in planning an analytical strategy is formed by the characteristics of our selected sample material. For instance, to successfully group coarse-grained pottery geochemically, one should, ideally, produce a homogenised powder sample that is prepared as a pellet or glass bead to control the effects of tempering and mineralogy in the chemical grouping and attain a representative sample. In addition, it is recommended to prepare a cross-section for microstructural and mineralogical examination using petrography or a scanning electron microscope (SEM) for at least half of the chemically characterised samples to evaluate mineralogical effects in the bulk chemistry of the samples (Bishop et al. 1982: 279). This type of combined



Figure 1. A map of archaeological sites where Corded Ware pottery sherds were sampled for the provenance and technological study (Finnish sites nos. 1–13; Swedish sites nos. 14–17; Estonian sites nos. 18–24). 1 – Hauho Perkiö; 2 – Helsinki Malminkartano; 3 – Mynämäki Aisti; 4 – Virolahti; 5 – Espoo Näkinkylä; 6 – Tammela Myllykylä; 7 – Vantaa Jönsas; 8 – Kirkkonummi Tengö Nyåker; 9 – Porvoo Böle; 10 – Halikko Märy; 11 – Inkoo Ragnvalds; 12 – Raisio Kankare; 13 – Tammela Uusi-Markkula; 14 – Lilla Malma Hagtorp; 15 – Dunker Barrsjö; 16 – Kil Vallby; 17 – Fjälkestad Rötved; 18 – Köpu Ia; 19 – Vöhma I; 20 – Ruhnu Valgi; 21 – Veibri; 22 – Riigiküla XIV; 23 – Narva-Jõesuu I; 24 – Narva-Jõesuu Ila. Drawing: E. Holmqvist.

analytical approach, however, requires a fairly substantial invasive sampling (e.g. 6 grams of powdered material and a profile section sized 1–2 cm) of a ceramic sherd, which can be unattainable, considering the poor state of preservation and often limited size of archaeological ceramics that are typically recovered at Finnish

sites. Small ceramic assemblages can also affect the research design of multi-site comparative projects – in practice, it can be difficult to follow the principle that suggests that one should sample 15–20 sherds per site (Tite 1999: 197) because suitable artefacts are not always recovered in such quantities at Finnish sites.

In contrast to heterogeneous sample matrices, a successful geochemical grouping of fine-grained and materially homogeneous materials, such as obsidian, can be effectively attained non-invasively, for instance, by pXRF or PIXE (Craig et al. 2010). Recently, non-invasive pXRF analysis has become the most commonly applied method for the chemical characterisation of archaeologically related obsidian, replacing, for example, the use of NAA – this glassy material can be successfully geochemically grouped and sourced without the need for sample preparation or invasive analysis. Non-invasive analytical strategies may also be applied in circumstances where there is already an expectancy of the chemical group structure or geochemical characteristics (particularly trace elemental patterns) of the sampled material (Speakman et al. 2011). Overall, considering sampling permission restrictions, artefact preservation and integrity, and advancing analytical techniques that allow the use of smaller sample sizes and non-invasive analysis, less invasive analytical techniques are becoming more popular in contemporary scientific artefact studies.

To conclude, the selected analytical approach – whether it is non-invasive or invasive – affects the data quality and, as a result, the attainable depth of the archaeological interpretation. It also defines our sampling strategy, the required sampling preparation procedures, the analytical costs, and the time required to carry out the analytical process, because non-invasive techniques allow vastly larger sample series to be analysed with a fraction of the resources needed for an invasive analysis. Below, this article discusses selected analytical methods and their application in recent scientific artefact studies at the University of Helsinki.

### 3 Recent archaeometric research themes at the University of Helsinki

In recent years, techno-compositional characterisation and provenance analysis of various

inorganic archaeological materials have been carried out by employing different analytical techniques at the University of Helsinki. Ceramics have been the most common study material to date, but other material types, such as glass, plasters, pigments, and metals, have also been analysed. The author of this paper has been involved in developing a multi-method research environment at the University of Helsinki by employing the analytical facilities of the archaeological laboratory at the Department of Philosophy, History, Culture and Art Studies, as well as the laboratory equipment based at the departments of Inorganic Chemistry (SEM-EDS) and Material Physics (PIXE) at the University of Helsinki. Research collaboration has also been carried out with foreign institutions (e.g. the Atomintstitute, Vienna). Given that, until recently, archaeological materials science research in Helsinki has been rather sporadic (see e.g. Lavento & Hornytzkyj 1996) and has lacked a strong research tradition or laboratory facilities, a great deal of work has been carried out to map the analytical facilities that are available at other departments and are suitable for archaeological materials science research. This has also required methodological adjustments to create sample preparation and analytical protocols for archaeological materials.

Currently, the most extensive techno-compositional investigation carried out in Finland is the project *Untangling Corded Ware: Provenancing Neolithic Battle Axe Culture Pottery of Southern Finland* (2012–2015), funded by the Academy of Finland and the Emil Aaltonen Foundation. This is a multi-site, provenance-driven project in which over 160 Corded Ware Culture (2900–2000 BCE) pottery sherds from a total of 24 sites located in southern Finland, Sweden, and Estonia were examined (Fig. 1). The selected pottery samples were subjected to techno-compositional characterisation and geochemical grouping by scanning electron microscopy with energy dispersive spectrometry (SEM-EDS) and particle induced X-ray emission (PIXE). This project is an interdisciplinary and international collaborative effort (the author as a principal investigator





Figure 2. A stereomicroscope image of the ceramic fabric of a Corded Ware pottery sherd recovered in Porvoo, Böle (KM 22004:6006), showing an abundant grog temper sized over 2 mm. Photo: E. Holmqvist.



Figure 3. The FESEM-EDS (Hitachi S-4800 with an Oxford Instruments 350 INCA energy-dispersive X-ray microanalysis system) instrument, based at the Department of Inorganic Chemistry at the University of Helsinki, employed in the archaeometric studies discussed in this paper. Photo: E. Holmqvist.

and the SEM-EDS scientist), and its main archaeological collaborators are Dr Åsa Larsson (Societas Archaeologica Uppsaliensis) and Prof Aivar Kriiska (University of Tartu), who are in charge of sampling Swedish and Estonian materials, respectively. Prof Jyrki Räisänen and Dr Vesa Palonen, together with their team at the PIXE laboratory (University of Helsinki), carried out the PIXE analytical work.

From a provenancing perspective, the Corded Ware pottery, which is known for its grog temper characteristic, is a complex study material. Its complexity derives from the possibility of internal compositional contamination caused by the grog temper. Problems arise from the possibility of grog deriving from imported pots used in local pottery manufacture. The grog, often sized over 2 mm in diameter and applied in abundant quantities (Fig. 2), would, if originating from an imported pot, alter the bulk chemical composition of the ceramic fabric by adding a ‘foreign’ compositional factor. For this reason, grog-tempered pottery cannot be approached by employing a typical analytical strategy for a ceramic provenance study, namely the preparation of a homogenised powder sample that represents the sample bulk composition. Instead, the ceramic matrices – the chemical composition of the ceramic matrix is related to the composition of the raw clay exploited in pottery manufacture (see Buxeda i Garrigós et al. 2003: 14–15) – of both the studied pots themselves and the grog temper present in their fabrics must be analysed separately following similar analytical procedures. By following this analytical strategy, the pot and grog matrix can be used as comparative samples. As a result, the compositional patterns of both the examined pottery and the previous generation of pots present as grog temper can be examined.

In this project, a FESEM-EDS (Hitachi S-4800 with an Oxford Instruments 350 INCA energy-dispersive X-ray microanalysis system) instrument (Fig. 3) based at the laboratory of Inorganic Chemistry at the University of Helsinki was used to examine ceramic cross-sections prepared as polished blocks. The

same samples were used for the PIXE analysis, which was carried out at the PIXE laboratory at the Physics Department, University of Helsinki. The results of this project (final report in preparation) highlighted a wide-ranging ceramic transport phenomenon across the Baltic Sea during the Corded Ware Culture period; these exchange patterns became apparent in the form of the imported sherds recovered, particularly in the grog data, which represented the remaining essence of the imported pots. It appears that the majority of the imported pots were recycled as grog temper at their destination. The study also found significant technological variation between different regional traditions of Corded Ware manufacture, such as different clay processing practices.

Cross-Baltic-Sea exchange patterns were also encountered in another pottery study that compared Finnish and Estonian material, medieval Red Ware pottery, by SEM-EDS. This study found pottery imported from Tallinn and probably continental Europe to coastal Finland, as well as evidence for early glazed ware pottery manufacture in Finland (Holmqvist-Saukkonen et al. 2013; Holmqvist et al. 2014). We also examined technological aspects of the pottery and found that, compared with the imported glazed vessels, the pots that appeared to represent the early stages of glazed pottery technology in Finland also displayed technological faults, such as uneven glazes (Fig. 4), thus reflecting the potters’ challenges in learning and adapting this new technology in their manufacture. This Red Ware pottery study was part of the *Medieval history of Vantaa* project, funded by the EU Central Baltic Interreg IV A Programme, Vantaa City Museum, and Svenska Kulturfonden. The project is currently being continued by the analysis of additional samples from Tallinn, Vantaa, and Turku, the last of which is a known Finnish manufacturing site of medieval Red Ware pottery.

The studied materials are not restricted to Finland and its neighbouring areas. To date, the most exotic samples analysed in Helsinki are pre-Columbian pottery sherds recovered in Brazil, which were analysed as part of the project

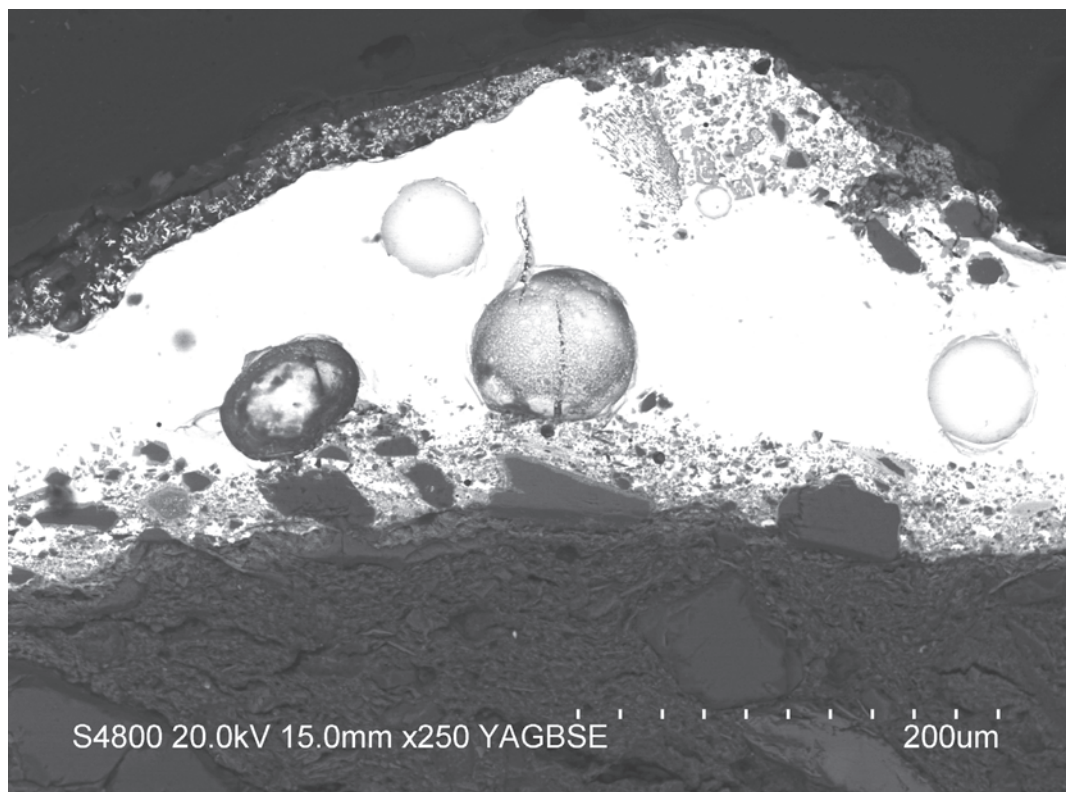


Figure 4. A SEM-BSE micrograph of a cross-section of a medieval Red Ware pottery sherd recovered from Mankby, Espoo, showing an even lead glaze (the bright layer). This pot represents the early glazed ware manufacture in Finland (see Holmqvist et al. 2014). Photo: E. Holmqvist.

*United in Diversity: Monumental Landscapes, Regionality and Cultural Dynamism in Pre-Columbian Western Amazonia*, funded by the Academy of Finland and directed by Prof Martti Pärssinen (University of Helsinki). Like the Corded Ware pottery samples, many of these pots are grog-tempered. Therefore this work requires a combined analytical strategy employing both microstructural and microchemical analysis by SEM-EDS and trace elemental analysis by neutron activation analysis (NAA; in collaboration with the Atominstutute in Vienna). The report on this project is currently under preparation.

Furthermore, the compositional patterns of Iron Age pottery samples from the Rapola cairns (Finland) were studied by SEM-EDS (funded by the Eino Jutikkala Foundation, directed by Doc Kristiina Mannerman), and

metal and ceramic finds recovered from the Levänluhta bog burial/sacrificial site were analysed by pXRF as part of the research project directed by Dr Anna Wessman and funded by the Emil Aaltonen Foundation. SEM-EDS was also used to examine the chemical concentrations and ceramic microstructures of the Neolithic Early Asbestos Ware, Typical Comb Ware, and Late Neolithic Asbestos Ware pottery sherds from the Lake Saimaa region in relation to the multidisciplinary *Argeopop* project (Oinonen et al. 2014). The project examined cultural continuation in the area at the time of the Vuoksi breakthrough. Together with other data, the ceramic results highlighted interruptions in the ceramic manufacturing traditions that were linked to cultural transitions – changes in the exploited raw materials, namely the use of different raw clay sources and the



abandonment of asbestos-tempering practices, in addition to the dissimilar styling of the pots, which may be linked to interruptions in habitation patterns (Oinonen et al. 2014).

As discussed above, invasive sampling is not always possible due to sampling restrictions. In recent years, the use of non-invasive methods and portable energy dispersive X-Ray spectrometry (pXRF) in particular has become increasingly popular in archaeological materials science (see e.g. Davis et al. 2012; Forster et al. 2011; Grave et al. 2012; Heginbotham et al. 2011; Sheppard et al. 2011), although its application in geochemical characterisation and sourcing of heterogeneous materials, such as coarse-grained ceramics, can be difficult due to the lack of a vacuum chamber and data calibration issues (see Holmqvist forthcoming 2016 with references). However, non-invasive analysis is an effective way to attain qualitative or semi-quantitative compositional data.

A pXRF instrument was acquired for the archaeology laboratory at the Department of Philosophy, History, Culture and Art Studies in early 2013, and since then, the instrument has been actively used to examine the chemical composition of a variety of archaeological materials, including ceramics, metals, plasters, glass, pigments, and soil samples. At our laboratory, the instrument has been primarily used for qualitative characterisation and has proven to be a valuable tool in the material identification of excavation, stray, and metal detector finds, as well as museum objects. The greatest advantage of the pXRF instrument is that it allows completely non-invasive chemical analysis (thus, no sampling or analysis permissions are required). Moreover, because of its portability, the analysis can be carried out in museum contexts, thus eliminating the need to transport valuable and often fragile objects to the laboratory for the analysis. It is a rapid and cost-efficient technique, but as a surface analysis technique, it is also prone to sample surface irregularities, contamination, corrosion, and layering, which can affect the data quality. In principle, a pXRF can analyse ca. 30 elements (Mg–U), but their quantification is problemat-

ic. Light element detection is especially complicated in in-air analysis; for this reason, the highest precision and accuracy can be obtained for high Z elements ( $\geq 26$ , Fe) (Forster et al. 2011; Grave et al. 2012; Speakman et al. 2011; Holmqvist forthcoming 2016).

In Helsinki, pXRF has been used to chemically characterise the metal objects found in the Levänluhta bog burial/sacrificial site in Isokyrö, Finland (as part of the *Levänluhta* project). The method has been extremely useful in the identification of the metal compositions of these valuable objects, which are on display at the National Museum (Fig. 5). Without chemical analysis, it can be difficult to determine whether we are dealing with copper, bronze, or brass objects, as has been the case with these famous artefacts. The analysis was carried out at the exhibition hall of the National Museum when it was closed from the public; the analysis thus caused no interruptions to the exhibition. Similarly, pXRF analysis was applied to define the gold content of the medieval gold ring recovered as a metal detector find in Espoo. The ring was found to be 17.5 carat gold (with an Au concentration of 72.7%), in addition to silver (Ag 20.8%), copper (Cu 5.0%), tin (Sn 1.3%), and iron (Fe 0.2%) (see e.g. Helsingin Uutiset 28 January 2014). Other available methods of carat determination (e.g. the acid test) are invasive and are therefore not applicable to such invaluable artefacts.

#### 4 Final remarks

As summarised above, archaeological materials science has prospered at the University of Helsinki over the last few years. The current aim is to further develop the university's analytical facilities and multidisciplinary collaboration with different departments and research institutions and to broaden the diversity of the research themes, materials, and methods involved. Archaeometry-themed courses are also given at the University of Helsinki, establishing Helsinki as the only Finnish university where archaeological materials science is currently being taught. There are also a num-





Figure 5. A bronze bracelet from the Levänluhta bog burial being analysed by portable XRF at the National Museum. Photo: E. Holmqvist.

ber of master's theses related to archaeological materials science being prepared in Helsinki.

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