

Chapter 43 Materials Analysis of Ceramics

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The durability of fired clay—and the breakability of ceramic containers—has made ceramic materials ubiquitous on many archaeological sites from the Neolithic onwards. Yet, while historically, the ceramic technology has been closely linked with sedentism, agriculture, and animal domestication as a package of Neolithic technologies, it has become increasingly clear that these associations hold true primarily in Europe, a region which was a major focus of early archaeological research on Neolithic societies (Hommel 2014). Globally, the origins of ceramics—both in terms of fired clay as a material and vessels made from fired clay (pottery)—must be sought among hunter-gatherer societies of the late Pleistocene. Low-fired clay-silt figurines found at Dolni Vestonice in the Czech Republic dating to *c.*26 ka BP remain the earliest known fired objects, but materials analysis suggests that the transformation into durable material was incidental to their ritual function, which involved casting figurines into the fire—perhaps with the intention of having them shatter (Vandiver *et al.* 1989). In any event, there was no further innovation of ceramics as a medium for durable containers in temperate Europe for nearly 20 000 years. The earliest ceramic containers appear at hunter-gatherer sites in East Asia at about 20 ka BP. At present, sites in China have the earliest dates for pottery (Wu *et al.* 2012), but similarly early pottery traditions are documented at late Pleistocene hunter-gatherer sites in Japan and eastern Siberia. Africa hosted another centre of early hunter-gatherer ceramic innovation in the early Holocene, with dates around 7 000-9 000 BC (Hommel 2014, Jesse 2010, Jordan *et al.* 2016). In contrast, the innovation of fired clay containers in the Near East actually post-dates the beginnings of plant and animal domestication. Additionally, in the New World, there is a similar lack of correlation between early ceramics, agriculture, and social complexity (Hoopes 1994:42).

The versatility and geological ubiquity of clay materials has meant that humans have adapted it

to a wide range of different purposes, from building materials (both fired and unfired) to tools for storage, transport, cooking, and eating. Archaeological ceramics range from the mundane everyday wares to exquisite pieces of craftsmanship available only to the highest elites. While acknowledging that the archaeological ubiquity of ceramics colours assessments of their importance by archaeologists (Vickers 2010:13), the enormous array of uses to which humans put clay materials means that ceramic analysis can be used to shed light on almost every facet of human society, from economies and political relationships to ritual practices and cultural beliefs.

Given the ubiquity of ceramic materials on archaeological sites, ceramic analysis has been and remains a core component of archaeological research. The literature on the analysis of archaeological ceramics is enormous, including a number of recent syntheses (Rye 1981, Orton and Hughes 2013, Quinn 2013, Santacreu 2014, Rice 2015, Roux and Courty 2019, Gliozzo 2020b (and associated special issue papers)). These reference works have varying emphases, ranging from macroscopic analyses to natural science laboratory perspectives falling into the category of “material analysis” or “archaeomaterials research”. While it is sometimes useful, in discussions of analytical methods, to distinguish between more traditional investigations conducted with the naked eye or perhaps a hand lens, and those relying on chemical, mineralogical, and other laboratory methods, this is an artificial division. A programme of chemical or mineralogical analysis that ignores macroscopic observations of vessel shape, function, and decorative style will generate results of limited utility. Ware types identified through traditional macroscopic methods or low power binocular microscopy often inform the selection of sherds for more intensive analysis, and the results of petrographic analysis are often interpreted in relation to vessel shapes and decorative styles. The apparent division no doubt derives from the variety of skillsets involved. Acquiring detailed knowledge of region-specific ceramic chronological sequences involves very different kinds of training than developing a robust understanding of sedimentary geology and the thermodynamic transformations of clays at high temperatures, necessitating close collaboration between specialists.

It would be futile to attempt to recapitulate the entire corpus of literature on ceramic analysis in a single book chapter. My main goal here is two-fold:

- (i) to summarize the primary ways in which the analysis of ceramics in the laboratory contributes to a deeper understanding of the manufacture and use of archaeological ceramics, and
- (ii) to sketch the contemporary research landscape of ceramic materials analysis, highlighting recurrent themes and newly developing areas of research. In this sense, I aim to bridge the “how” and the “why” of ceramic analysis in a way that provides the ceramic analysis neophyte a well-signposted introduction, and offers the veteran archaeological scientist a useful synthetic perspective on an enormous, wide-ranging field of research.

The discussion is structured in reverse order of the typical process of formulating ceramic materials analysis projects. Normally, a ceramic materials project begins with an archaeological question, which then uses one or more analytical techniques to investigate aspects of the ceramic life history or *chaîne opératoire*. For instance, one might ask whether the emergence of centralized political institutions in a particular region was correlated with ceramic standardization or increased long-distance exchange. Elemental analysis and thin section petrography could then be used to shed light on the sources of ceramics, provide additional perspectives on systematization in the production process to answer these broader questions. In this review, however, I work backwards, beginning with a review of the ceramic *chaîne opératoire*, followed by discussing of how different analytical techniques illuminate various aspects of the production process. These two sections build towards a discussion of key research themes in ceramic materials analysis, including trade and exchange, the organization of production, and technology and innovation.

PRODUCTION PROCESSES

As might be expected for a technology with global reach and more than 20 000 years of use, there is huge variation in the ways humans have manipulated and transformed ceramic materials. Many exhaustive discussions of ceramic production processes are available elsewhere (Rye 1981, Roux and Courty 2019)—in this section, I provide a general sketch of the main aspects. In places, I foreshadow the subsequent discussion of analytical techniques by discussing in a

general sense the kinds of information archaeologists use to reconstruct elements of the *chaîne opératoire*. While the overall focus of this chapter is on laboratory analytical methods, it is worth pointing out that reconstructions of ceramic technology are often dependent on data *not* derived from the laboratory—for instance excavations of production workshops, ethnographic reports, textual accounts, and more traditional methods of macroscopic investigation.

Any discussion of the ceramic *chaîne opératoire* must begin with the prospection, collection, and preparation of raw materials. Clays deposits form through weathering of silicate minerals and rocks. Residual, or primary, clays form *in situ* in direct association with the original parent rock, while secondary clay deposits are formed through processes of sedimentary transport, often in an alluvial or marine context. Clays are geologically common in many parts of the world, especially relative to other raw materials of archaeological interest like metal ores and obsidian. As we shall see, this feature of their geographical distribution poses challenges to attempts at sourcing, as comprehensive sampling of possible sources becomes more time intensive. At the same time, not all clay deposits are suitable for making pottery, and even those deposits that were exploited archaeologically may require processing to produce a workable ceramic paste.

Ceramic ethnoarchaeology has documented a wide range of approaches to clay processing, involving the removal and addition of constituents, as well as homogenization and transformation of raw materials through aging or “souring” of clays to improve their properties (Eramo 2020; Rye 1981:17-20). Removal of constituents can be done by crushing, sorting, sieving and/or winnowing dry clays, or with the use of water through a process of levigation or decantation. Both processes take advantage of the fact that heavier fractions settle out of a sediment-water mixture more quickly than the finer fraction, which includes most clay minerals. Materials can be added to the ceramic paste to improve its workability (e.g., its plasticity), reduce cracking during drying, increase survival rates during the firing process, or make it more capable of resisting thermal shock during its use-life. Additions can include both organic and inorganic temper, a term used to describe intentional additions to the clay paste, rather than the more neutral term “inclusion.” The latter refers to both naturally present aplastic constituents (e.g., mineral and rock fragments from the original clay deposit) and intentionally added constituents. Clays can also be mixed to produce the desired properties.

Identification of clay selection and processing is a challenging process, one that relies heavily on both compositional and microstructural analyses (Eramo 2020, Ho and Quinn 2021). Thin section petrography can identify poorly hydrated and/or homogenized clay pellets, a possible indicator of mixing, while careful examination of the mineral composition of different inclusion size fractions may be able to identify a disjuncture between the geological origin of inclusions in the base clay and that of the added temper, assuming this difference exists. For the identification of organic tempering, one might expect organic remains to occasionally appear in the ceramic matrix as natural inclusions or accidental incorporations during production. However, if a ceramic matrix contains a large volume fraction of voids from the burnout of organics, the intentional addition of organic temper is a probable explanation.

Clay is shaped into vessels and other objects using a variety of techniques, such as coil building, paddle-and-anvil, and the potter's wheel. Many approaches to making pottery will incorporate multiple different techniques, for instance, using a paddle and anvil or a wheel to expand and shape a vessel initially roughed out through coiling or pinching. Different forming practices leave characteristic traces in the microstructure and macrostructure of clay vessels. Some of these traces are apparent on visual examination with the naked eye or with low power binocular microscopy. For these reasons, investigations of vessel formation practices often begin with the careful macroscopic examination of a large assemblage with an eye to characteristic features such as ridges, striations, poorly smoothed coil seams, or characteristic breakage patterns (Rye 1981:66ff, Roux and Courty 2019:158ff). Microstructural and macrostructural techniques like thin section petrography and digital radiography (see below) can reveal characteristic inclusion orientation patterns and wall thickness variations in ways that complement and expand upon macroscopic observations (Greene *et al.* 2017, Thér 2020).

The use of the wheel in ceramic production is an important innovation, one which is often assessed in relation to changes in the scale and specialization in ceramic manufacture. While “wheel made ceramics” is a common short-hand designation, the term encompasses a range of different methods, not all of which result in major increases in the rate of manufacture (Roux 2008:91). For example, wheel finishing, the use of a wheel to smooth an already-formed vessel,

and wheel shaping, the use of a wheel to shape and expand a rough-out, differ significantly from wheel throwing, which uses a (fast) wheel to transform a ball of clay into the more or less finished shape. Only the last method represents a significant improvement in the rate of production (Thér *et al.* 2017:1259). To complicate matters, wheel throwing and wheel shaping of a coil built rough-out can produce similar macro traces (Roux and Courty 2019:178). Detailed quantitative analysis of inclusion orientations and their rotational standard deviations in tangential thin sections, however, has is able to differentiate between these different uses of the wheel techniques (Thér 2016, Thér *et al.* 2017), though the time-intensive nature of such analyses, even with some automated image processing, may limit the widespread adoption of this approach.

Research on the origins of the wheel in the ancient Near East have suggested that the initial adoption of wheel shaping in the late 5th/early 4th millennium BC was not the result of attempts to increase the rate of manufacture in an economic environment that prioritized increasing scales of production (Roux 2008; Roux and Baldi 2016). In China, there is evidence of the use of a slow wheel to shape or finish vessels possibly as early as the 5th/early 4th millennium BC in the later phases of Yangshao Culture, with convincing evidence of wheel throwing appearing later at about 3000 BC. Interestingly the potter's wheel was not an innovation that was rapidly and permanently adopted everywhere. In some areas of the Near East, the use of the wheel continues to disappear and reappear over millennia, and only achieves a degree of permanency and preeminence in the 2nd millennium BC (Roux 2008).

In order to minimize failure rates, ceramics must dry thoroughly prior to firing. Ethnographic research has shown that in terms of workshop space, areas for drying ceramics equal or even exceed the space requirements for firing and fuel storage (Hasaki 2011:22). These requirements often mean that ceramic production is subject to seasonal ebbs and flows, when exterior space is available and warmer drier weather ensures faster, more complete drying (e.g., Degoy 2008:205, Roux *et al.* 2017:322).

Surface treatments of ceramics may occur at any number of points following the shaping of the vessel, including after an initial firing stage. Burnishing, the process of inducing a characteristic

sheen to the surface through rubbing and alignment of clay particles, produces different effects when carried out at varying points during the clay drying process (Roux and Courty 2019:201-202). Paints, slips, and glazes are frequently analyzed to study the technologies of production and the sources of raw materials (Gomez and Doherty 2000, Santarelli *et al.* 2019). Certain pigments, for example the cobalt blues in Chinese porcelain, are derived from sources of relatively limited availability with distinctive chemical profiles (Wen *et al.* 2007, Giannini *et al.* 2017). As these materials are generally applied to the ceramic surfaces in thin layers, microanalytical techniques tend to be favoured for analysis, though sampling is more typical for isotopic analysis of glazes (Wolf *et al.* 2003, Santarelli *et al.* 2019; but see also recent work by Zong *et al.* 2018). Care must be taken to avoid sampling the ceramic body, as this may impact elemental analyses and some types of isotopic measurements (Eiselt *et al.* 2019).

Firing is the irreversible process that transforms clay pastes into durable ceramic through heat. Mineralogical and chemical transformations include the loss of bound water (hydroxyl groups) from the mineral structure of clays, the vitrification and fusing of clay platelets, and the formation of new crystalline phases such as diopside, haematite, and mullite (for a very thorough treatment, see Gliozzo 2020a).

Research on ceramic technologies has devoted considerable effort towards reconstructing firing practices, including the thermodynamic conditions of firing (both temperature and redox conditions) and the structures used (e.g., open firings or enclosed kilns) (Gosselain 1992a, Livingstone Smith 2001, Chatfield 2010). While the chemical and mineralogical transformations of clays at different temperatures are reasonably well documented, especially in a laboratory setting, the reconstruction of firing practices from ceramic sherds faces many challenges. Chemical transformations of clays are not instantaneous, and are a function of both temperature and time, meaning that a ceramic fired for longer at a lower temperature may (within limits) undergo the same chemical and microstructural transformations as a ceramic fired at a higher temperature for a shorter period of time. Different clay pastes will also behave differently during the firing process, with the major differences between carbonate-rich and carbonate-poor clay pastes (Gliozzo 2020a).

Ethnographic research has shown relatively little correlation between maximum firing temperatures and the structures used for firing (Gosselain 1992a, Livingstone Smith 2001). Kiln-fired vs. non-kiln fired pottery is one common means of categorization, but there is a wide diversity in these firing structures, and a degree of ambiguity in what constitutes a kiln. Kilns are enclosed or mostly enclosed firing structures, but other attributes vary. Some definitions of kilns imply a degree of separation between the fuel and ceramic load (Rice 2015:166-167), but enclosed structures with little apparent fuel-ceramic separation are often described as (single-chamber) kilns (Gliozzo 2020a:260, Goldstein and Shimada 2007).

The diversity of firing structures means that many presumed advantages of kiln firing do not hold universally. More enclosed structures (either in kilns or in heavily insulated pit firings) theoretically allow a greater degree of consistency and control over the thermodynamic parameters of the firing (temperature, heating rates, redox conditions), and reduces failure rates due to sudden fluctuations in temperature. Interestingly, kiln-firing is not categorically more fuel efficient than open firing (Pool 2000:72). Separation of fuel from firing load also reduces scorch marks and ash defects on glazed surfaces (Rice 2015:180-181). Some methods of firing, such as days-long firing of stoneware and porcelain, often reaching temperatures in excess of 1200°C, are also not possible without a kiln (see, for example, Kerr and Wood 2004:365-375).

Firing times, heating rates, and maximum temperatures of kilns and open firings overlap considerably (Livingstone Smith 2001:998, Roux and Courty 2019:120), though there is a tendency to view kilns as more typical of slower heating rates and longer firing times (Rice 2015:171). Thus, in the absence of kiln structures, clear kiln waste debris, written accounts, visual representations, or ethnohistoric parallels, it is often difficult to reconstruct the details of firing technologies from sherds alone.

Despite its ambiguous relationship to actual firing methods, ceramic materials researchers are often interested in inferring firing temperatures, or more specifically, an “equivalent firing temperature” (Tite 1969:132n), corresponding to the level of transformation of ceramic body. Attempts to estimate firing temperature ranges generally fall into several categories. Broad estimates, sufficient to class ceramics into low-fired vs. high fired wares, can be done by

examining the degree of vitrification using microscopy.

A somewhat more constrained estimate of firing temperatures can be achieved through re-firing experiments. These experiments involve reheating ceramics, ideally in similar redox conditions to the original firing, to different temperatures for a period of time, and then comparing the microstructure with the original sherd—if the degree of vitrification remains the same, this would suggest that the sherd was originally fired to at least that temperature for at least that length of time (Chatfield 2010, VanValkenburgh *et al.* 2017). Measurements of magnetic susceptibility have also been used in refiring experiments to assess the firing temperatures of ceramics (Rasmussen *et al.* 2012).

Other methods of estimating firing temperatures involve the identification of ceramic neophases, which depend on both the composition of the paste and the temperatures at which they are fired (Gliozzo 2020a). Often this is done via X-ray diffraction (Bimson 1969), but for some ceramics, these newly-formed crystalline phases may be developed enough so that they can be investigated as discrete phases, for example through microanalytical techniques (see below) (Freestone and Middleton 1987:24).

One element of uncertainty in many firing temperature estimations is the length of time spent at that temperature (called “soak time” or “residence time”), as these affect how far microstructural and mineralogical transformations activated within a certain temperature range may progress. In other words, ceramics fired for longer times at lower temperatures can look like ceramics fired for shorter times at higher temperatures, though of course there are limitations to this range of equifinality. Soak times may vary considerably based on firing strategies and fuel type (Gliozzo 2020a, Livingstone Smith 2001). A focus on identifying firing temperatures in materials analysis, even when they are expressed as a range, tends to elide the wide range of time-temperature profiles and firing strategies documented in historical and ethnographic research. Even terms like “equivalent firing temperature” which partially alludes to this disconnect, do not fully resolve these ambiguities.

While not the focus of this chapter, it is worth saying a few words about ceramic use. From the

perspective of a life history approach, the use of ceramics is not fundamentally separate from the manufacturing process, but rather a subsequent stage in their ongoing material transformations. As with production processes, use, re-use, and repair of ceramics leave characteristic traces, in the form of mechanical wear and degradation, mineral and organic residues, and soot marks from burning. Organic residue analysis (Evershed 2008; [see chapter x](#)) is not typically grouped with other methods of ceramic materials analysis, in part because the methods (involving various forms of chromatography) are quite different from those used to investigate inorganic ceramic materials, and in part because of a conceptual segregation between production and use. Nevertheless, by providing information about organic sealants used for waterproofing or organic pigments used for decoration, such analyses often illuminate production techniques as well (Evershed 2008:904). Conversely, inorganic materials analysis is *not* solely useful for understanding the “production” side of ceramic life histories; it can also reveal how ceramics were used. For instance, analysis of copper-derived discoloration on Athenian white-ground lekythoi helped build a robust case for their use in cremation rituals (Walton *et al.* 2010).

ANALYTICAL TECHNIQUES

Most laboratory analysis of ceramic materials can be grouped into three broad categories: compositional analyses, microstructural analyses, and macrostructural analyses (Table 1). Compositional analyses include various techniques to identify the chemical, isotopic or mineralogical composition of ceramic materials, and shed light on the *chaîne opératoire*. Microstructural analyses of ceramics examine the arrangement and ordering of different components of the ceramics on a microscopic level. These techniques are often highly informative about various aspects of clay processing, paste preparation, and to some extent, vessel forming techniques. Macrostructural techniques include visual inspection and low power microscopy, but also include various laboratory techniques for non-destructively investigating the interior macrostructure of ceramic objects. Such approaches are perhaps most useful for examining vessel formation practices.

Ceramic composition, microstructure, and macrostructure are highly interdependent: the chemical and mineralogical compositions of clays, slips, paints, and glazes affect ceramic

colours, and clay compositions and microstructure affect what kinds of vessel shapes are possible to create without failure during drying, firing, or use. Individual laboratory techniques may be capable of multiple kinds of analyses; for instance a scanning electron microscope fitted with an energy dispersive X-ray detector (SEM-EDS) system provides both compositional and microstructural data. Likewise, thin-section petrography provides data on the mineral composition of ceramics, but also provides microstructural and microtextural data crucial for interpretations about raw material sources and the production process.

The aim of this section is not to describe in full detail every analytical technique that has been used in ceramic analysis. Rather, it emphasises the different classes of analytical information produced in the laboratory investigation of ceramics, discusses how various techniques are used to illuminate various aspects of the ceramic *chaîne opératoire*, and highlights issues specific to the analysis of ceramic materials.

Compositional analyses

One major branch of compositional analyses deals with the elemental analysis of ceramic materials, covering major (roughly >1 wt%), minor (0.1-1 wt%) and trace (<0.1 wt%) elements, with the first two frequently reported as oxides in ceramic analysis. Major elements are generally less diagnostic for tracing the precise geological origins of ceramics, though they can be informative about the broad classification of the kinds of clays and other raw materials used. Minor and trace elements are far more commonly used in discussions of ceramic provenancing, and they often form the basis of multivariate statistical treatments aimed at identifying different sources of ceramics (Glascock *et al.* 2004).

Ceramics are heterogeneous materials, especially on the microstructural level, consisting of a mixture of clays and a range of organic or inorganic aplastic inclusions. As such it is worth differentiating between compositional techniques that analyse bulk composition, and microanalytical techniques that analyse the composition of a specific part of the ceramic fabric. Energy and wavelength dispersive spectrometers attached to scanning electron microscopes (SEM-EDS and SEM-WDS) and electron microprobes (EMPA) permit analyses of discrete

phases or small areas of the ceramic fabric with precise spatial control. However, comparably high limits of detection mean that it is most useful for assessing major and minor elemental compositions, with wavelength-dispersive spectrometers being somewhat better in this regard (Pollard *et al.* 2007:111). Nevertheless, the high degree of spatial control, combined with its imaging capabilities makes the SEM an extremely versatile and powerful tool. It is possible to analyse the chemical composition of very small inclusions via EDS, down to about 10 µm, depending on the material. These features, along with the relatively wide availability of SEM facilities, make the technique an extremely common feature of ceramic materials analysis projects (Froh 2004) (Figure 1). While in theory anything that can fit into the vacuum chamber of the SEM can be analyzed, for good quality quantitative chemical analyses, a polished, flat surface is required. As such, most ceramics analysed via SEM are sampled and mounted in polished resin blocks.

One fairly recent development in the use of SEM-EDS microanalysis is quantitative mineral compositional analyses by rastering thousands of EDS analyses over the surface of a polished sample and identifying minerals automatically by their spectra (Knappett *et al.* 2011). While this method has several advantages over other methods of determining ceramic mineralogy, its use in the field of archaeological ceramic analysis has been limited so far, as these analyses need specialist software and instrumentation that is not typically supplied with most SEMs available to archaeologists.

For lower detection limits X-ray fluorescence (XRF) is another commonly deployed methodology, which both has both portable and benchtop versions. Portable X-ray fluorescence (pXRF) analysis is increasingly common in ceramic analysis due to its versatility in the field (Frahm 2018), the speed of analysis, and the potential for non-destructive work, but pXRF analysis of ceramics faces significant challenges of calibration and issues of the representativeness of surface analysis (Hunt and Speakman 2015). Without a vacuum attachment or helium purge, pXRF analysis also struggles with the detection of light atomic weights elements, including some elements that are major constituents of ceramics (e.g., Na, Mg, Al, and Si). Ceramics may not corrode as readily as metal objects, but wet-dry cycles can lead to the formation of post-deposition mineral deposits on ceramic surfaces. With benchtop XRF units, it

is common to analyse ceramics as milled powders pressed into pellets or as fused discs/beads, which overcomes some of the limitations to quantification found in non-destructive surface analyses.

Various solution-based analyses, such as inductively coupled plasma mass spectrometry (ICP-MS), have become increasingly common for the analysis of ceramics, particularly at the trace level (Kennett *et al.* 2002, Little *et al.* 2004). These methods often have low detection limits, but they require chemically aggressive methods to dissolve the silicate minerals present in ceramics. Pairing an ICP instrument with a laser ablation unit (LA-ICP-MS) eliminates the need for solution-based analysis, offering an essentially non-destructive microanalytical technique with the high spatial control of an SEM with the very low detection limits of an ICP-MS. In ceramic analysis, LA-ICP-MS has seen its widest applications in the microanalysis of temper inclusions (Palumbi *et al.* 2014), glazes (Hill *et al.* 2004, Van Keuren *et al.* 2013), and paints (Eiselt *et al.* 2019). Being a microanalytical tool, it is not as effective as solution-based ICP-MS methods as a measurement of bulk composition of heterogeneous materials like ceramics.

Instrumental neutron activation analysis (INAA) has long been a popular method for bulk ceramic compositional analysis with an eye to provenance (Zuckerman *et al.* 2010), with the laboratory at the Missouri University Research Reactor (MURR) being a major centre for archaeological INAA in North America (Glascock *et al.* 2004, Hill *et al.* 2004). Neutron analysis is capable of detecting a range of trace and ultra-trace elements with relevance to ceramic provenance, though not all elements are analysable (Pollard *et al.* 2007:128). INAA facilities regularly analysing archaeological materials are relatively rare today (Pollard *et al.* 2007:123), with much of the data being produced by a relatively small number of laboratories.

In contrast to metals and glass, isotopic analysis of ceramics is considerably more rare (Carter *et al.* 2011, Henderson *et al.* 2020). Strontium and neodymium isotopes have been applied to ceramics, paralleling their increasing use in the study of glass over the last 15 years (Degryse and Schneider 2008, Degryse *et al.* 2010). Because the Sr isotope composition is related to the age of rocks, in theory it provides an independent means of distinguishing between clay sources derived from similar bedrock geologies of different geological age, something that trace element and

petrographic analysis might struggle to resolve (Carter *et al.* 2011). In practice, Sr isotope analysis has been useful for distinguishing between different lime sources used in high fired Chinese ceramics, analogous to its use in the provenancing of glass (Henderson *et al.* 2020). Lead isotope analysis has also, unsurprisingly, seen applications in the provenance analyses of the raw materials used in lead-glazed ceramics (Zong *et al.* 2018, Santarelli *et al.* 2019). The most likely reason for the lack of uptake (so far) in isotopic analysis of ceramics for provenance is that they do not offer revolutionary advantages over the combination of (trace) elemental analysis and thin section petrography, while at the same time being considerably more expensive (Henderson *et al.* 2020:201).

X-ray diffraction (XRD) has proved useful for identifying the mineral composition of ceramics of ceramics, especially of clays and other minerals not identifiable through thin section petrography (Quinn and Benzonelli 2018). It is important to keep in mind, however, that the firing process alters the chemical structure of clay minerals, so XRD spectra of ceramics made from a particular type of clay may differ considerably from unfired samples of the same clay (Gliozzo 2020a). Indeed, XRD can be a useful as a way of identifying the transformation of clays at high temperatures and the formation of new phases such as mullite, an aluminosilicate neo-phase that forms in high-fired ceramics. As X-ray powder diffraction involves the homogenization of a sample of ceramics, the XRD spectra will be dominated by minerals that are present in large quantities, meaning that such spectra are often dominated by clay minerals and very common (and therefore less diagnostic for provenance) minerals such as quartz. Identification of minerals which comprise more minor but still interpretively significant components of the ceramic paste, are often better identified through other methods, though there are more recent developments in X-ray diffraction instrumentation that permit microanalysis (Quinn and Benzonelli 2018).

Fourier-transform infrared spectroscopy (FTIR) has also been used to estimate firing temperatures, as the shape and position of key peaks in the spectrum changes as the ceramic is fired. These changes are the result of mineralogical changes (or more precisely, changes in the chemical bonds within those minerals) taking place as the ceramic transforms at high temperatures. Recently Yan *et al.* (2021) developed an absorptivity ratio method to track changes

in the intensity of absorption peaks during firing, allowing them to estimate the firing temperatures of ceramic moulds used in bronze casting.

Microstructural analyses

By far the most common microstructural analyses used in ceramic analysis are thin section petrography and scanning electron microscopy. Thin section petrography is a widely used technique used to investigate a range of different aspects of ceramic technology and provenance. It relies on preparing a thin (normally 30 µm thick) slice of the ceramic and viewing it with a transmitted light optical microscope with various polarizing filters. Microstructural analysis using thin sections can identify clay mixing (Ho and Quinn 2021) and identify organic temper through the morphology of voids left after burnout, and identify vessel forming practices (Thér 2016). Thin section analysis can also help to distinguish between natural mineral inclusions and intentionally added temper (Quinn *et al.* 2017, Eramo 2020). Bimodal inclusion size distributions are highly suggestive, though perhaps not absolute, evidence of tempering, which can be challenging to detect when the clays and the tempers come from similar geological environments.

Microstructural features of ceramic thin sections can provide some basic information about firing temperatures. The presence of optical activity (birefringence) in the clay groundmass, when viewed in thin section under cross-polars, suggests a firing process that preserved the crystalline structure of clay minerals and did not result in extensive vitrification. However, this optical activity may be obscured in low-fired ceramics by incomplete burnout of organics.

The optical properties of minerals in thin section also make it possible to identify the mineral composition of ceramics. Micro-contextual associations of different minerals, for example in polymineralic rock fragments (e.g., Figure 1) can provide rich and potentially diagnostic information about the clays and/or tempers used to make ceramics. This geological information complements and extends the data produced through bulk trace element analysis, so there is considerable benefit to using these techniques in tandem. In practice, however, it is difficult to identify small mineral inclusions (<50 µm in diameter) in thin section, so other methods are

often required to identify very small mineral inclusions (typically via SEM-EDS), and the clay minerals themselves (typically via XRD). It is partly for this reason that fine earthenwares and high-fired porcelain tend to be investigated more via SEM-EDS than via thin section petrography. While standard thin section preparation involves layering the ceramic section between a glass slide and a coverslip, it is also possible to prepare a thin section without a coverslip, leaving a polished sample surface exposed. This method, while increasing sample preparation time (and cost, if prepared commercially), permits analyses via SEM-EDS or electron microprobe on the thin section itself (as seen in figure 1).

Thin section analysis relies on transmitted light polarizing optical microscopy. Reflected light polarizing optical microscopy—far more common in the analysis of metals and metallurgical slags—can be used with polished sections of ceramics to examine minerals that remain opaque in thin section, but such approaches are rare, as opaque inclusions commonly found in ceramics are often small and difficult to identify conclusively via reflected light methods alone. These inclusions are best identified through microanalysis of inclusion composition.

The compositional dimensions of the SEM-EDS instrument have already been discussed in the previous section, but it is worth discussing its imaging capabilities and utility for microstructural analyses here. SEMs permit higher magnifications than thin section analysis, and produce two kinds of imagery. Backscatter images, in which the brightness depends on the atomic weight of the material, are useful for distinguishing between inclusions which may have similar optical properties when viewed in thin section (quartz and certain feldspars, for instance). It can also be useful for imaging slips, glazes, and pigment layers, which may contain higher atomic weight elements as colorants (e.g. copper, iron, lead, or manganese), which contrast with the aluminum and silicon-rich clay minerals of the ceramic body. Microstructural features of crystalline phases can also help to distinguish minimally altered inclusions that formed the original components of the ceramic fabric or surface decoration, and new phases formed during firing (e.g. melilite) or post-depositional alteration (e.g. in-filling carbonates in pores). Secondary electron images are useful for imaging sample topography, and are often used to assess the degree of vitrification and estimate firing temperatures (e.g., VanValkenburgh *et al.* 2017). While most SEM investigations of ceramics, especially where chemical analysis is involved, are conducted on flat polished

sections mounted in resin, assessments of microstructural effects of vitrification are often done by looking at (ideally fresh) fracture surfaces, for which secondary electron images are best suited.

Macrostructural analyses

Among the macroscopic laboratory science techniques, macroimaging techniques like digital radiography and its predecessor, xeroradiography, have seen wide application to the study of ceramics (Greene *et al.* 2017). Aside from imaging the contents of sealed ceramic vessels without having to open them, the most frequent uses of X-ray macroimaging techniques is the study of vessel formation practices—distinguishing, for instance, between coil-building, paddle-and-anvil construction and wheel throwing. Variations in inclusion densities, orientations of inclusions, and smoothed-over coil joins are often visible using these techniques. In many cases, these features are more apparent in digital radiographic images than they are in macroscopic examination. Traces of vessel forming techniques may also be visible on a microstructural level when viewed in thin section, but the ability to work non-destructively and to examine inclusion orientation and distribution on a whole-object level is an advantage of these imaging techniques. The image processing power of digital radiography represents a significant advantage over traditional X-ray systems reliant on photographic plates.

Laboratory analyses of physical properties of ceramics might also be grouped under the category of macrostructural techniques, as they both deal with the macroscopic behaviour of ceramic materials. Such measurements include testing of thermal shock and fracture resistance. Often, such techniques are used to test whether technological choices made in the manufacturing process altered the macroscopic performance characteristics of the finished product (Bronitsky and Hamer 1986, Schiffer *et al.* 1994).

RESEARCH THEMES

To this point, discussion has primarily focused on the details of analytical techniques as applied to understanding various aspects of the ceramic *chaîne opératoire*, from the selection and

processing of raw materials to the shaping and transformation of those materials into finished products. The ultimate aim of ceramic materials analysis is to understand past human behaviour. The final section of this chapter ends with a brief review of some of the major themes and questions that structure and give meaning to the data produced by the analytical techniques. While these themes are discussed in separate sections, they are deeply interwoven in much ceramic materials research.

Trade and exchange

The themes of trade and exchange pervade many materials investigations of ceramics. Sourcing ceramic raw materials, from clays and tempers to glaze pigments and paints, is a major focus of ceramic materials research. Provenance studies¹ can trace objects to distant geological origins, or demonstrate that a vessel with foreign stylistic decoration was in fact a local imitation. This kind of information has major significance for understanding past economies, as well as the movement of people, materials, and ideas.

The central condition of any sourcing successful study, often referred to as the provenance postulate, is that variation between sources must exceed variation within sources (Hein and Kilikoglou 2020; see also **chapter x**). Stated another way, underlying geological variability in raw materials determines the spatial precision with which one can provenance archaeological objects. The provenancing of ceramic materials involves several specific considerations. Ceramic raw materials are generally less transformed chemically and mineralogically by the production process than the raw materials used to make metals and glass. As a result, ceramics typically preserve, to a greater degree than these other pyrotechnologies, the mineralogical and textural character of the original raw materials used to make them. High temperature ceramics, which undergo significant transformations, are an exception (Kerr and Wood 2004:711). Thus, while finished ceramics do differ from their raw materials in important ways, these differences do not pose the same challenges they do, for instance, in the provenance of metal (see Pernicka 2014).

¹ In different fields, the terms provenance and less commonly-used term provenience sometimes have different meanings. For the purposes of this chapter, the term provenance is used in the sense of the geological source of the raw material used to make an object.

On the other hand, the raw materials used to make ceramics are, with some exceptions, geologically abundant. Ubiquity makes the sampling of all possible sources more challenging. While raw material sampling is commonly used to establish a local baseline in studies of ceramic exchange (e.g., Lindsay *et al.* 2008), geographical scope of most clay sampling studies is necessarily smaller than it is for geologically rarer raw materials like metal ores and obsidian deposits.

It is important to remember that, despite significant scholarly interest in long-distance trade, much (if not most) of the ceramic inventory at any given archaeological site did not travel very long distances. Of course, the proportion of imported ceramics varies across vessel type (domestic cookwares vs. transport amphorae vs. high-status tablewares) economic and/or political contexts, and geographical areas. For example, the Pax Romana combined with well-developed marine transport routes and the huge markets for olive oil in Rome result in an enormous mound of oil amphorae of Spanish origins being deposited at Monte Testaccio (Blázquez 1992). At the same time, ceramics are heavy and prone to breakage, while clays are widely distributed, so people on the move may find it easier to apply their ceramic skills to local raw materials than they are to haul large quantities of ceramics hundreds or thousands of kilometres. For these reasons, and because of the geological constraints discussed above, it is important to undertake provenance research on assemblages with anthropologically useful (and otherwise unanswered) questions about ceramic sourcing, *in areas where the geological conditions are conducive to success*. Otherwise, there is a danger that considerable time and expense will be spent producing a result that is essentially self-evident (e.g., most cooking wares were locally produced) or worse, inconclusive. When deployed effectively in suitable geological contexts (e.g., Harrad 2004), provenance research can prove highly informative about the movement of ceramic materials, even if that movement is largely within a region. Provenance research can also inform discussions about community formation, interaction, and social relationships.

In light of these considerations, it is perhaps not surprising that some of the most successful ceramic sourcing studies focusing on the long-distance exchange involve maritime trade, often in island contexts (Zuckerman *et al.* 2010, Knappett *et al.* 2011, Dickinson *et al.* 2013). Bulk

transport of ceramics over long distances is facilitated by water transport, and island regions are often characterized by spatially restricted, geologically distinct clay sources, which contrast with the large—and largely geologically homogeneous—sedimentary provinces produced by major alluvial systems.

Another area of research where ceramic provenancing research has proved useful is in analysing cases of import substitution: instances where local imitations of foreign imports or “horizon” styles emerge (Zuckerman *et al.* 2010, Erb-Satullo *et al.* 2011). In these cases, especially if the imitations are effective, macroscopic analysis may prove inconclusive. The results of these studies have important implications for patterns of long-distance exchange and the nature of local negotiation of inter-regional economic and political relationships.

In other instances, ceramic provenance discussions have intervened in debates about the directionality and structure of exchange networks (Blomster *et al.* 2005, Stoltman *et al.* 2005, Lindsay *et al.* 2008). Though ceramics are of course not the sole items of exchange, and they were often among the least important (Vickers 2010:13), they are among the most archaeologically visible, making them a useful if not infallible proxy for broader networks of contact and exchange.

The organization of production

As one of the most archaeologically visible crafts, the study of ceramic production, its spatial distribution, and modes of organization, has contributed in a very substantial way to the literature on the economies of early complex societies (Rice 1981, Costin 1991, Stein and Blackman 1993). Archaeologists have devoted considerable effort to exploring how aspects like specialization, scale, and standardization in the ceramic industry correlate with broader social transformations (Blackman *et al.* 1993, Roux 2015). For instance, the significant archaeological attention to the innovation of the potter’s wheel derives from the fact wheel throwing *can* facilitate mass production of pottery, even if early use of the wheel as a means to shape rough-outs (termed wheel fashioning or wheel shaping) did not have a major impact on the rate of production (Roux 2008:91, Thér *et al.* 2017:1259).

Laboratory analysis of ceramic materials contributes to the investigation of the organization of ceramic production in several ways. Consistency in various elements of ceramic manufacturing has often been taken as evidence of a more rigidly organized production process. Standardization of bulk chemical composition, consistent methods of paste preparation, and metric standardization (i.e., vessel dimensions) have all been discussed in this regard (Blackman *et al.* 1993, Quinn *et al.* 2017). At the same time, it is vital to consider whether geological conditions in the study area favour production of ceramics with homogeneous material compositions irrespective of organizational factors. Large alluvial plains, for instance, will produce more geologically and mineralogically homogeneous clay sources than a mountainous environment with residual clays forming from distinct geological formations. Comparative measures of relative standardization (e.g., between different vessel classes or between periods) may help to control for these cases (Arnold and Nieves 1992). Even in cases where geological conditions do not favour homogeneity, it is important to assess standardization in ceramic manufacturing practices and/or products within their broader social context. Standardization need not occur only in the context of a closely administered, top-down mode of organization. Emergent bottom-up processes may also engender uniformity. There are also instances where some dimensions of standardization were not a primary concern, even among highly specialized potters producing large quantities of ceramics (Hein *et al.* 2020). In sum, regularization, standardization, and uniformity of practice must be assessed systematically with respect to its relative degree, its manifestation (e.g., in chemical/mineralogical composition, in vessel dimensions, in paste preparation), and its broader social and geological context.

Ethnoarchaeological perspectives on ceramics have infused many of the key research themes in ceramic materials analysis, but it has been particularly influential in research into the organization of production. In spatial terms, ceramic ethnoarchaeology has provided fundamental data about the relationships between production sites and resources. Archaeologically documented production contexts and ceramic resource extraction sites are rare relative to the quantities of ceramics discovered. Thus, ethnographic research fills an important gap, helping to constrain and shape archaeometric investigations (Arnold 2005). Among the most frequently cited results of this research is the concept of threshold distances, the distances potters will

typically travel to obtain fuel and raw materials for ceramic production (Arnold 1985:32ff). Ethnographic data has also informed discussions about standardization (Roux 2015), the organization of workshop space (Hasaki 2011), and community-level patterns of production and transmission (Lathrap 1983).

Ethnographic and archaeological research on the spatial dimensions of ceramic production and (to an extent) exchange has fostered a loosely grouped area of research called ceramic ecology or ceramic landscape research (Matson 1965, Arnold 1993, Doherty 2020). The more archaeomaterials-oriented approaches within this area of research are fundamentally interested in the ways in which potters interact with and adapt to the geography and geology of clay raw materials. This research tends to be well-rooted in a detailed understanding of the clay geology and natural resource landscapes, often involving field research and raw materials sampling alongside laboratory analyses of archaeological ceramics. Tracking the movement of ceramics may be an incidental outcome of this kind of research, but ceramic provenance and raw materials sourcing is decidedly not the primary goal. Rather, the aim is to reconstruct the choices made by potters in relation to their resource landscapes (Michelaki *et al.* 2015). For instance, a ceramic ecology approach might aim to identify and explain pottery preferences for a sub-optimal but proximate clay source requiring substantial processing over a better but more distant source.

Technology and innovation

Discussion of potter choices in relation to resource landscapes leads directly to another major area of focus in ceramic materials research: the study of technology and innovation. Research on the anthropology of technology has shown that technical choices carry important cultural meaning, and that not all choices can be explained from a purely material properties or resource availability perspective (Lechtman 1977, Lemonnier 1993). These concepts have particular resonance in ceramic analysis, given the huge array of ceramic production techniques documented archaeologically and ethnographically (Gosselain 1992b). Ceramic materials analysis excels at reconstructing the choices made during the production process and allows archaeologists to assess how these choices relate to geological conditions and the constraints of material properties.

Experimental archaeology has played a key role in defining how technical choices made during the ceramic production process affect the material properties and performance characteristics of ceramic vessels (Bronitsky and Hamer 1986, Schiffer *et al.* 1994, Gardner *et al.* 2020). Often these studies involve mechanical and thermal shock testing to directly assess these properties in a controlled laboratory setting. In these cases, clarity on the social and cultural conditions aspects of production emerge when technological choices cannot be attributed to a distinct improvement in ceramic material properties. Ethnoarchaeological work has provided insight into the ways in which ceramic technical choices interact with cultural and linguistic boundaries (Kalentzidou 2000, Degoy 2008, Roux *et al.* 2017). At the same time, materials analysis of ceramics is not simply a way to categorize each element of the *chaîne opératoire* as either culturally or environmentally determined, as culture and environment are not easily separated (Anschuetz *et al.* 2001). A clay source may be selected not just because of its proximity or ease of use, but for its relationship to culturally significant features of the landscape. Rather, analytical approaches to ceramic materials bring into focus how material properties, and resource distributions intertwine with human decision-making in the production process.

Ceramic materials research has also proven informative about the spread of innovation (Erb-Satullo 2020). Materials research has informed discussion around the world's earliest clay materials, including pottery, figurines, and hearths (Vandiver *et al.* 1989, Karkanas *et al.* 2004, Iizuka *et al.* 2020). Indeed, materials analysis, including compositional, microstructural, fracture pattern analysis, was central not just to the identification of the earliest fired ceramic technologies, but also to the argument that thermal fracture was an intentional outcome of early ceramic figurine making in the Upper Paleolithic (Vandiver *et al.* 1989). Materials analysis has played a key role in tracking and explaining innovation in a wide range of ceramic technologies, from the development of early refractory ceramics (Freestone 1989, Thornton and Rehren 2009), to the spread of the potter's wheel (Thér *et al.* 2017). Ceramic materials research has also proven instrumental in deconstructing patterns of technological continuity and change in periods of culture contact (Capone 2004, VanValkenburgh *et al.* 2017).

Glazing technologies, especially in the study of Chinese porcelain and Islamic glazes wares, is a

particular focus of research on ceramic innovation, and one where materials analysis has played a large role. Interestingly, while glazed clay objects (distinguished from glazed faience) appear with increasing regularity in the Egypt and the Near East in the Late Bronze Age (c.1600-1200 BCE), about the same time that glass innovation takes off, there is comparatively little research on glazed clay objects of this period. Some correlation between glass and innovation in ceramic glazes is seems reasonable given that they are both vitreous materials that appear around the same time in Mesopotamia. The site of Nuzi, for instance, is well known for its early glass materials (Shortland *et al.* 2018), but also yielded a range of glazed clay objects including wall nails and vessels (Erb-Satullo *et al.* 2011). At the same time, producing a glaze that will adhere to a ceramic substrate poses difference challenges to that of producing glass beads or vessels, and there are interesting asymmetries in the geographies of innovation. Egypt, a centre of early glass innovation, did not adopt glazed ceramics until the late first millennium BCE (Tite *et al.* 2002).

Analysis of Medieval and Early Modern glazed ceramics across Eurasia has charted a fascinating history of innovation, imitation, and exchange that capture the emergence of an increasingly globalized economy in the last 1000 years (Kerr and Wood 2004:709ff, Wood *et al.* 2007, Wen and Pollard 2016). It is difficult characterize the full sweep of this enormous research field in a few sentences, but archaeomaterials research has played a central role in bringing greater nuance to the impact of Chinese porcelain on the Islamic and European ceramic industries, shifting the model away from unidirectional imitation and charting the influences of local traditions and back influences that impacted the Chinese industries as well (Giannini *et al.* 2017, Matin *et al.* 2018).

CONCLUSIONS

In assessing the contributions of ceramic materials analysis to the broader field of archaeology, several key observations are worth highlighting. First, while ceramic provenance is perhaps the most widely known application of ceramic materials research among non-specialists, ceramic sourcing is in fact one small component of a much richer and more diverse research landscape. Successful raw materials sourcing research requires both suitable geological conditions, as well as a well-defined research question to which the provenance data can usefully contribute.

Nevertheless, even in cases where the source of raw materials is either trivial or difficult to determine precisely, ceramic materials analysis contributes important data to debates about technological choices, the organization of production, and the processes of innovation.

Second, a strong understanding of raw materials, their formation, geographic distribution, and material properties, is fundamental to ceramic materials analysis. This foundation is not only key to provenance research but also to projects investigating technology and the organization of production. To be clear, it is not feasible to suggest that all ceramic analysis projects must involve field surveys and sampling of raw materials sources—in many instances field research is impossible or impractical given the constraints of time and funding. Even in these cases, however, ceramic materials studies are enriched by a thorough review of geological and environmental science literature relevant to the region. In my own work on Mesopotamian ceramics from present-day Iraq (Erb-Satullo *et al.* 2011), field sampling of clay sources was not possible. Instead, data from a sedimentological survey of heavy minerals in Mesopotamian alluvial sediments (Ali 1977) provided crucial support for the argument for a local production centre of the horizon style Nuzi Ware ceramics.

Finally, and perhaps most importantly, rather than separating ceramic materials research from the broader study of archaeological ceramics, the best analytical research is deeply intertwined with ethnographic, stylistic, and other field- or museum-based methods of macroanalysis. Chemical, mineralogical, structural, and physical analyses have enormous potential to enrich the study of ceramics, but only when they are well contextualized in relation to broader research questions.

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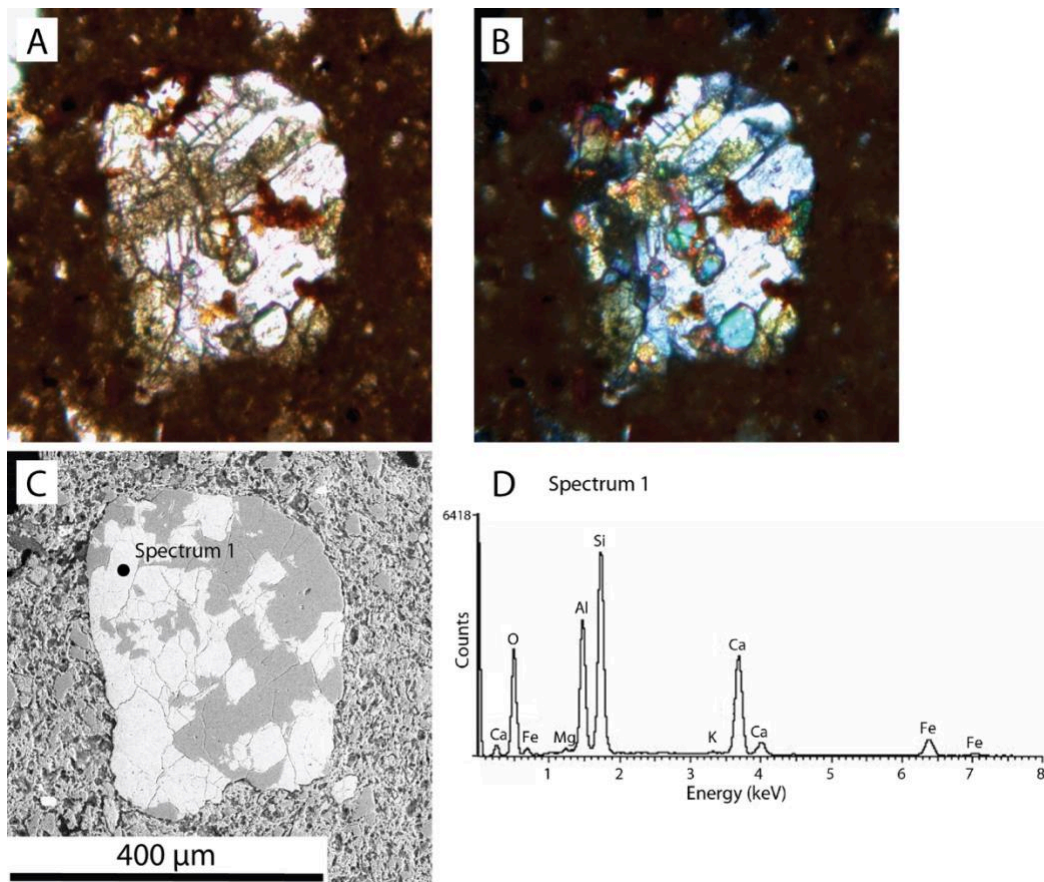
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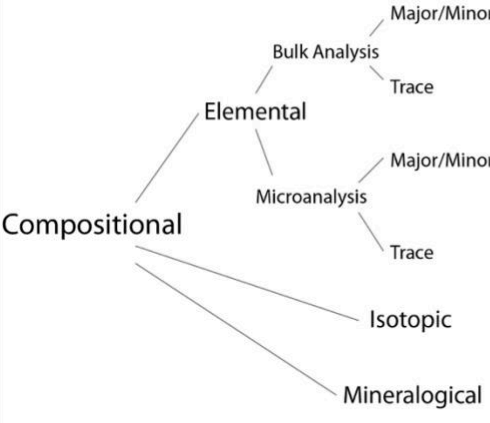
Modes of analysis	Example technique	Most informative about...
	<p>XRF</p> <p>INAA, ICP-MS (quadrupole), XRF</p> <p>SEM-EDS, EPMA</p> <p>LA-ICP-MS</p> <p>ICP-MS (multi-collector)</p> <p>XRD, Thin section petrography</p>	<p>Raw material identification, ceramic properties (e.g. refractoriness)</p> <p>Raw material sourcing</p> <p>Raw material identification, identification and analysis of surface treatments, firing process (identifying neophases)</p> <p>Sourcing ceramic inclusions and surface treatments (paints/slips/glazes)</p> <p>Identification of raw materials, potential for sourcing (not yet common)</p> <p>Raw material identification and sourcing, firing process (XRD)</p>
Microstructural	SEM, Thin section petrography	Paste preparation, vessel formation, firing process (degree of vitrification), identification of raw materials (organic burnout voids and partially reacted phase textures)
Macrostructural	Digital Radiography, Xeroradiography	Vessel formation techniques

Table and Figure Captions

Table 1 Different modes of materials analysis with corresponding examples of techniques commonly used in ceramics research and indications about what they reveal about the ceramic *chaîne opératoire*.

Figure 1 Rock fragment in the matrix of an archaeological ceramic prepared as a polished thin section, permitting both thin section petrography and SEM-EDS analysis. Plane polarized (A) and cross-polarized (B) optical microscope images, showing different relief and interference colors in the minerals. SEM backscatter image (C) showing compositional variation, accompanied by an EDS spectra (D) of a spot analysis of an epidote crystal, which is consistent with the optical properties shown via polarizing microscopy.



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Chapter 43: Materials analysis of ceramics

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