Leave no mudstone unturned: Geochemical proxies for provenancing mudstone temper sources in South-Western Cyprus

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ABSTRACT

A variety of ceramic fabrics bearing mudstone inclusions (either naturally existing in the clay or added as temper) are attested in several sites in South-Western (SW) Cyprus. Within the Mamonia terrane of SW Cyprus mudstone-bearing lithologies are divided into two main groups. Sediments of the Ayios Photios group (sandstones, siltstones, mudstones, calcarenites, occasional limestones and chert) were deposited in marine conditions and close to the continental slope. In contrast, the contemporaneous Dhiarizos group contains radiolarian mudstones and cherts deposited in deep-sea conditions. Mudstones and cherts from both formations share similar macroscopic characteristics (distinctive red colour, fine texture) and can be confused for one another, especially when examined only as small-scale ceramic inclusions. Being able to differentiate between the different inclusion types and to link them to one of the two formations leads to useful conclusions regarding the provenance of ceramic samples within the Mamonia terrane.

In this case study, geological samples from relevant mudstone sources were analysed as reference materials to describe the possible types of mudstone inclusions. Sr and Pb isotopic data were the primary means for distinguishing between the two major formations, as they closely relate to the differences in depositional setting. Based on the established isotopic classification, microtextural analysis and elemental mapping data were also compared to determine if members of the two formations are distinguishable at this level. Finally, as the isotopic approach proved to be the one offering better results, a theoretical outline for utilizing this type of information to determine the provenance of mudstones in ceramic sherds is presented.

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1. Introduction

Mudstones are fine-grained siliclastic sedimentary rocks composed predominantly of silt-sized (0.062–0.004 mm) and clay-sized (<0.004 mm) particles (Prothero and Schwab, 1996). When more than 50% of the constituents are silt-sized the term siltstone can be used, whereas if more than 50% of the particles are clay-sized the term claystone is more applicable. In both cases, individual grains are too small to be distinguished without a microscope. Few, if any, rock fragments are present, with quartz and feldspars being the predominant ones. Similarly, a sedimentary rock comprising more than 50% either silt- or clay-sized transported carbonate grains is referred to as a calcilutite.

Mudstones have been attested as major inclusions or intentional temper in various prehistoric contexts from the Eastern Mediterranean – pithoi from Messenia (Matson, 1972), Crete (Boileau and Withley, 2010; Day, 1988) and Cyprus (Jacobs and Borgers, 2009; Xenophontos et al., 2000), storage vessel fragments from Anti-Ithyera (Pentedeka et al. 2010) – and even later contexts – 7th–5th century B.C. Corinthian transport amphorae (Whitbread, 1995). A common parameter in all the above cases is the use of mudstone temper for large to medium, thick walled, hand-formed vessels. Thus, the use of mudstone (or shale) is associated with the need to reduce plasticity (improve workability) of the clays – allow shaping of the vessel and prevent collapse under the clay’s own weight (Matson, 1972; Whitbread, 1995). Additionally, the same studies, argue that mudstones play a role in reducing cracking during the drying process. The preferential use of mudstone over other apastics is considered to be a matter of its availability in the natural landscape and/or according to potting tradition (Matson, 1972).

In the context of Cyprus, mudstones are primarily found in ceramics attributed to sites within the Mamonia terrane, at the SW of the island. Mudstone outcrops are abundant in this region (Fig. 1b) meaning that mudstones will be both a natural constituent in the local clays but also readily available to potters as potential temper.

In the case of several Late Cypriot pithoi, studied by Xenophontos et al. (2000), the addition was intentional. Three models have been
proposed for Cypriot pithoi manufacture and distribution (Keswani, 2008): ‘centralized’ production, localized manufacture or itinerant potters. Within the Mamonia terrane, it is possible to define regions where mudstones of different origins are more prominent. Therefore, if pithoi were manufactured at one cluster of workshops the mudstone inclusions would be of one single origin. In the case of localized production and itinerant potters, pithoi from each site might exhibit a preferential use of the mudstone type that is in closest proximity. Based on the above arguments, mudstone inclusions could be used as an indicator of ceramic provenance within the Mamonia terrane if a strong proxy is defined for linking mudstone temper to a specific geological formation.

Most provenance studies of mudstones rely on chemical methods because the separation of individual minerals is difficult (Potter et al., 2005). Major element analysis has been a staple in mudstone provenance studies and is best employed to determine the extent of weathering of the source terrain. It constitutes a convenient approach as it can be conducted rapidly and inexpensively with methods such as X-ray fluorescence (XRF) or energy dispersive–scanning electron microscopy (SEM–EDX). Some trace elements may be above detection limit for these techniques, but many of those of greatest interest require techniques such as instrumental neutron activation analysis (INAA) and inductively coupled plasma-mass spectrometry (ICP-MS). Another indicator of provenance is the type of primary detrital material that was incorporated into the mudstones, and whether it originated from an igneous, sedimentary or crustal source. To this end, isotopic analysis provides a useful proxy, as isotopic ratios strongly differentiate between these categories of materials (Potter et al., 2005 and references therein).

The current case study focuses on effectively differentiating between the two major mudstone-bearing formations of the Mamonia terrane, the Ay. Photos group and the Dhiarizos group. Even though mudstones from both formations share macroscopic characteristics (colour, texture) they differ in relation to the depositional environment in which they were formed (Fig. 1a). Reference samples representing different mudstone types were collected and assigned to either of the two formations based on their Sr and Pb isotopic composition, as these two systems closely relate to the differences in depositional setting and source rock material. Once the samples were provenanced, additional data (microscopic appearance, grain-size, phase analysis using elemental maps—all collected using SEM–EDX) were compared to determine if they are distinctive enough to discriminate the two types of mudstone. The isotopic approach proved to be the one offering the best resolution between the formations. Therefore, a methodology to utilize this data to determine the provenance of mudstone inclusions from bulk isotopic measurements on ceramic samples is also presented. Within the limits of this work, this is just a theoretical demonstration. However, it shows sufficient potential and will be tested on relevant archaeological material within the context of future research.

2. Background to the present study

2.1. Geological context

The Mamonia terrane records the break-up of a preexisting continental margin (Fig. 1a) during a period of convergence with the Troodos microplate (Edwards et al., 2010). Mamonia lithologies are divided into
two main groups: those deposited relatively close to the continent (Ayios Photios group) and those formed in deep oceanic conditions (Dhiarizos group). The former consists dominantly of Triassic–Upper Cretaceous (252–90 Mya) clastic sedimentary rocks (sandstones, siltstones, mudstones, calcarenites and occasional limestones) and chert. Terrigenous material from the mature continental landmass and occasionally platform carbonates were transported to greater depths and intermixed at the foot of the continental slope by turbidity currents. In contrast, the Dhiarizos group is made up mostly of Jurassic–Upper Cretaceous (200–90 Mya) radiolarian cherts and mudstones deposited on the sea floor, as well as some Triassic (250–200 Mya) volcanic rocks and reeval limestones, remnants of existing oceanic islands. The Kannaviou formation is a bentonitic clay matrix with which both Mamonia and Troodos fragments were mixed during the collision of the two terranes, creating a lithological mélangé (Kathikas formation). Finally, during the same tectonic event, some Dhiarizos lithologies were buried, metamorphosed and later exhumed, creating the Ayia Varvara formation.

2.2. Sr and Pb isotopic systems

Pb behaves as three radiogenic isotopes, $^{206}\text{Pb}$, $^{207}\text{Pb}$ and $^{208}\text{Pb}$, daughter products of $^{238}\text{U}$, $^{235}\text{U}$ and $^{232}\text{Th}$ decay respectively, and one non-radiogenic isotope $^{204}\text{Pb}$, used as the normalizing isotope ($^{206}\text{Pb}/^{204}\text{Pb}$, $^{207}\text{Pb}/^{204}\text{Pb}$, and $^{208}\text{Pb}/^{204}\text{Pb}$). Pb is mostly hosted in the silicate fraction, as $^{207}\text{Pb}$ replaces K in K-feldspar (Faure and Mensing, 2005). Sr has one radiogenic isotope, $^{87}\text{Sr}$, formed by the decay of $^{87}\text{Rb}$, and three non-radiogenic isotopes, of which $^{86}\text{Sr}$ is used as the normalizing isotope ($^{87}\text{Sr}/^{86}\text{Sr}$). Sr$^{2+}$ replaces Ca$^{2+}$ in Ca-bearing minerals like calcium carbonate, plagioclase and apatite, as well as K$^{+}$ in K-feldspar, although less favourably (Faure and Mensing, 2005). In both the Pb and Sr systems, such radiogenic/non-radiogenic isotopic ratios of a sediment or rock will be a function of the characteristics of its source lithology – initial isotopic composition, parent/daughter element ratios (U/Pb, Th/Pb, and Rb/Sr) – and its age (Faure and Mensing, 2005).

Rb, U and Th are preferentially incorporated into mantle melts that eventually lead to the production of continental crust. The enrichment of the continental crust in these parent elements coupled with its age implies that continental crust will have more radioactive daughter isotopes and consequently higher isotopic ratios than the bulk mantle (Faure and Mensing, 2005). The average composition of the continental crust is $^{87}\text{Sr}/^{86}\text{Sr} = 0.730$ (Veizer and Mackenzie, 2005), $^{206}\text{Pb}/^{204}\text{Pb} = 19.07$, $^{207}\text{Pb}/^{204}\text{Pb} = 15.74$, and $^{208}\text{Pb}/^{204}\text{Pb} = 39.35$ (Millot et al., 2004). Oceanic crust, which is depleted in these elements, will retain lower isotopic ratios closer to the original mantle composition. This difference will be reflected in the isotopic ratios of sediments deriving from either source. In the current case study, mudstones of the Ayios Photios group that contain material of continental origin are expected to have higher isotopic ratios than the Dhiarizos group sediments that are of deep-sea origin.

3. Materials and methods

3.1. Sampling

Sampling was conducted according to the following criteria: (i) to represent different types of mudstones and cherts from both the Dhiarizos and Ayios Photios groups (based on inclusion descriptions in Xenophontos et al., 2000), (ii) to take place in geologically well documented locales (based on Edwards et al., 2010), and (iii) to include sources in relative proximity to archaeological sites in order to be more relevant in the context of ceramic temper provenance. Therefore, sampling was conducted along the Dhiarizos and Chapotani rivers, relevant to the region of Palaeapaphos, and at the Mavrokolympos river, close to the site of Maa–Palaikastro. Furthermore, a Dhiarizos mudstone outcrop close to the Petra tou Romiou locality was identified and sampled.

Potential outcrops were identified based on the GPS coordinates and descriptions of locations in Edwards et al. (2010). Specimens were examined using a hand-lens and evaluated according to macroscopic criteria (colour, texture, fissility) to verify that they represented suitable references. The geological description of the outcrops and their coordinates provided an initial assumption regarding the formation to which each sample belonged. However, field relationships are complicated within the Mamonia terrane and thus the assignment of each sample to a geological formation had to be verified.

Additional material from the reference collection of the Vrije Universiteit Brussel (presented in Renson et al., 2013) was used for this study, in particular material from the Mamonia terrane as well as a selection of Circum Troodos sediments (CTS) (same selection as in Makarona et al., 2014), used as reference for local sedimentary clays.

3.2. SEM–EDX microtextural and elemental results

SEM–EDX was used to provide both a detailed morphological overview of the samples (backscattered electron images — BSE) as well as elemental composition data (EDX). The system used was a FE-SEM JEOL JSM-7000F (Research Group of Electrochemical and Surface Engineering, Vrije Universiteit Brussel). The EDX detector has an energy resolution of 129 eV. The data was collected at a voltage of 15 kV and at a working distance of 10 mm. The samples were coated with a thin layer of Pt/Pd, thus allowing C content to be determined as well. The BSE images combined with the elemental maps were used to investigate the mineral phases present in the samples. Measurements were conducted on individual grains of different phases and additionally the bulk composition of the samples was determined as that of the total mapped area (2 mm $\times$ 1.5 mm). Quantification of the results was performed using the ZAF method standard-less quantification offered by the proprietary software Analysis Station 3.8.0.31 (JEOL Engineering Co., Ltd).

3.3. Sr and Pb isotopic analysis

Isotopic analysis was conducted on 200 mg of well-homogenized bulk sample. Sr isotopic ratios were determined for all samples, while Pb isotopic ratios were measured only for the samples gathered during this study. The procedure for dissolving the samples and performing the Pb separation was conducted as in Renson et al. (2011). As the resin used in the Pb separation does not cause fractionation of the Sr isotopes (the measured isotopic ratio would reflect the original value of the sample) the waste fraction of the Pb separation was collected and used to isolate the Sr content of the samples, following the procedure outlined in Makarona et al., 2014. This sequential separation eliminates the need for two dissolution procedures, reducing both sample preparation time and sample requirements. The isotopic ratios were determined using a Nu-plasma Multi Collector ICP-MS (MC-ICP-MS) system (Département des Sciences de la Terre et de l’Environnement de l’Université Libre de Bruxelles).

4. Results

4.1. Microtextural and elemental characterization

The SEM-BSE images and elemental maps are presented in Fig. 2 and the quantified values in Table 1. This information was used to classify the samples according to rock type, either as a mudstone/siltstone (with higher Na$_2$O + Al$_2$O$_3$ + K$_2$O values – representing the feldspar/clay fraction – and lower Si concentrations) or as a chert/silicified mudstone (high Si concentrations). Sample M33 was classified as a calci lutite, as it bears quartz and feldspar grains in a primarily carbonate matrix. The Dhiarizos group is characterized by fine-grained mudstones
with only very few detrital feldspar or quartz grains. The Ay. Photios formation includes coarser siltstones consisting of quartz, feldspar and carbonate grains with a grain-size between approximately 50–100 μm. The Ca rich inclusions (20–50 μm) that stand out in samples M23, M26 and M37 are also rich in P, and could be attributed to apatite grains. Moreover, in most samples minuscule (10–30 μm) Ti and Fe rich grains were observed, which could represent ilmenite inclusions.

The differences in grain-size between the two formations reflect their individual depositional environments. The Ayios Photios mudstones/siltstones are coarser due to the detrital nature of their source

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### Table 1

Bulk concentrations for major elements in the mudstone samples, measured using SEM–EDX, are presented as oxides (%, normalized values). The sum of Na₂O + Al₂O₃ + K₂O is calculated to represent the clay fraction of the samples. These constitute semi-quantitative results calculated by ZAF standard-less quantification/JEOL JSM 7000F.

<table>
<thead>
<tr>
<th>Formation</th>
<th>Ayios Photios group</th>
<th>Dhiarizos group</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mudstones/siltstones</td>
<td>Silicified mudstones/ chert</td>
</tr>
<tr>
<td>Sample</td>
<td>C (%)</td>
<td>Na₂O (%)</td>
</tr>
<tr>
<td>M32</td>
<td>11.06 ± 0.23</td>
<td>1.05 ± 0.13</td>
</tr>
<tr>
<td>M38</td>
<td>12.32 ± 0.22</td>
<td>1.19 ± 0.13</td>
</tr>
<tr>
<td>M34</td>
<td>6.04 ± 0.15</td>
<td>0.31 ± 0.15</td>
</tr>
<tr>
<td>M36</td>
<td>6.03 ± 0.15</td>
<td>0.28 ± 0.15</td>
</tr>
<tr>
<td>M23</td>
<td>22.53 ± 0.07</td>
<td>0.3 ± 0.15</td>
</tr>
<tr>
<td>M15</td>
<td>7.14 ± 0.29</td>
<td>0.43 ± 0.15</td>
</tr>
<tr>
<td>M26</td>
<td>7.51 ± 0.07</td>
<td>0.57 ± 0.15</td>
</tr>
<tr>
<td>M33</td>
<td>7.35 ± 0.10</td>
<td>0.34 ± 0.15</td>
</tr>
<tr>
<td>M22</td>
<td>12.89 ± 0.28</td>
<td>0.25 ± 0.15</td>
</tr>
<tr>
<td>M23</td>
<td>12.09 ± 0.28</td>
<td>0.25 ± 0.15</td>
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<tr>
<td>M37</td>
<td>12.09 ± 0.28</td>
<td>0.25 ± 0.15</td>
</tr>
</tbody>
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materials and their carbonate content can be attributed to the incorporation of small amounts of platform carbonates in the turbidity currents. On the contrary, the Dhiarizos mudstones are finer grained due to the calmer precipitation conditions during deep-sea. Their carbonate content is extremely low (below detection limit), as they were deposited below the calcite compensation depth (Prothero and Schwab, 1996).

Even though the above assessment provides information about the samples that cannot be obtained by simple macroscopic or microscopic observation, it is not enough to clearly differentiate the two formations. Grain-size may appear to be a distinguishing factor, with mudstones initially assigned to the Dhiarizos group being finer grained than the siltstones of the Ay. Photos group. However, microtexture can vary even within the same formation and since the two formations do not differ significantly in terms of bulk elemental composition it would be difficult to definitively differentiate between them based on these criteria alone.

4.2. Isotopic provenancing of the reference mudstone samples

The isotopic composition of the samples (Table 2) provided the means for this classification. As seen clearly in Fig. 3 two broad groups of samples are identified: one with much higher Sr and Pb isotopic ratios, close to those reported for continental material, and one with a much less radiogenic isotopic signature. The first group, therefore, must correspond to material from the Ay. Photos group and the second to the Dhiarizos group. The additional Sr isotopic data helped to constrain the provenance of the samples from Renson et al., 2013, as their initial description was only that they belonged to Mammonia Middle Triassic–Upper Cretaceous lithologies.

The signature of the Dhiarizos group is much tighter in terms of Pb isotopes but the different rock types within it differentiate in terms of Sr signature. On the contrary, Ay. Photos samples exhibit a wider range of compositions. Siltstone and mélange samples from Renson et al., 2013 have similar Pb compositions but show variation in their Sr signatures. The two mudstone samples from this study and one of the formerly collected clay samples have higher Pb isotopic signatures than the previous ones but the same Sr composition. These differences might be attributed to differing degrees of sedimentary maturity. A targeted study of more samples with differing degrees of maturity and grain sizes is necessary to fully understand the variations within the Ay. Photos group. The Ay. Photos cherts (M24, M26) overlap with the Dhiarizos, in terms of their $^{207}$Pb/$^{204}$Pb and $^{87}$Sr/$^{86}$Sr ratios (Fig. 3c). According to the geological prospecting data (Edwards et al., 2010) only Ay. Photos cherts are present at the sampling location, so these samples are not falsely assigned. Moreover, since the two types of cherts differ in $^{206}$Pb/$^{204}$Pb ratios a differentiation between them can be achieved. The above assessment demonstrates that the combined use of Sr and Pb isotopic data is an effective approach to resolve mudstone types in Cyprus. The microtextural and elemental information is critical to interpret the isotopic data. The next step is to discuss how this result can be applied when examining mudstone inclusions in archaeological ceramics.

4.3. Application of isotopic mixing for mudstone temper provenance

The more straightforward approach would be to measure the isotopic composition of the mudstone inclusions individually and compare them to the values of the reference samples. The mudstones would need to be extracted from the clay matrix using a microdrill and enough material (at least 100 mg) needs to be collected for the isotopic analysis. In this case the ease of interpretation is outweighed by the actual procedure of isolating the mudstone inclusions, which can prove challenging. Laser-Ablation-MC-ICP-MS, which would allow the determination of both Sr and Pb isotopic ratios of individual inclusions would be an ideal technique for this type of in-situ measurements. However, such
instrumentation is less commonly available and requires a much more intensive data reduction procedure.

An alternative solution is to conduct bulk isotopic measurements on the ceramic samples, resulting in isotopic values representing both the mudstone inclusions (M) and the clay matrix (C). Each component contributes to the total signature according to its concentration of each element (SrC, SrM or PbC, PbM) and its percentage in the mix (fC, fM where fC + fM = 1). The mixed signature (T) relates to the primary isotopic signatures of the clay and the mudstones according to the following mass balance equations (Faure and Mensing, 2005):

$$\frac{^{87}\text{Sr}}{^{86}\text{Sr}} = f_C \cdot \frac{^{87}\text{Sr}}{^{86}\text{Sr}} + f_M \cdot \frac{^{87}\text{Sr}}{^{86}\text{Sr}}$$

$$\frac{^{206}\text{Pb}}{^{204}\text{Pb}} = f_C \cdot \frac{^{206}\text{Pb}}{^{204}\text{Pb}} + f_M \cdot \frac{^{206}\text{Pb}}{^{204}\text{Pb}}$$

The percentages of contribution to the mix can be easily derived from microscopic or petrographic examination of the sample, whereas Sr and Pb concentrations are routinely determined during the procedure for measuring isotopic ratios. Therefore, it is possible to calculate the expected isotopic signatures for different mixes between selected end-members.

The mixing lines between end-members in isotopic diagrams are generally hyperbolas (Langmuir et al., 1978). If the denominators of the isotopic ratios are the same (as in Pb–Pb isotopic diagrams) the hyperbola degenerates to a straight line. In a Pb–Sr diagram, the curvature of the mixing hyperbola for two end members, A and B, is determined by the factor:

$$r = \frac{Sr_A}{Sr_B} \cdot \frac{Pb_B}{Pb_A}$$

After the mixing line is drawn, the exact expected position of the mix is determined by the percentages of each end-member within the mix. By comparing these calculated signatures with the one measured for the sample the correct combination of end-members can be determined.

In Fig. 3 an example is provided to illustrate the above methodology. A ceramic sherd consisting of 70% clay matrix and 30% mudstone inclusions is the assumed unknown sample. The clay matrix will have a signature within the CTS field (assuming the sherd is locally made in Cyprus). Based on the microscopic and/or elemental characteristics of the mudstone inclusions an educated guess can be made regarding which formations should be checked as potential end members. In this example all possible mudstone types are considered. The final positions of the mixes along the lines are based on the 30% mudstone content of the sample. Observation of the isotopic diagrams reveals that the estimated positions are well resolved from one another. Therefore, the unknown sample can be matched to one specific calculated mix if and only if the two coincide in all isotopic coordinates. The fact that multiple conditions need to be satisfied increases the certainty with which the final identification is made.

Once the mudstone inclusions in the unknown sample are assigned to a geological formation and/or rock type, more interesting conclusions regarding the provenance of the ceramic itself can be extrapolated. For
example, by examining the geological context of the Dhiarizos valley in the proximity of the region of Palaepaphos, it is evident that it will be dominated by Dhiarizos group mudstones (Fig. 1b). Therefore, if a number of pithoi sherds are found to be tempered with Ay. Photios mudstones either (i) they were made locally with raw materials deriving from specific locations along the Dhiarizos river, where Ay. Photios outcrops are found, or from the outcrops along the Chapatami river or (ii) the pithoi were imported from a locale where Ay. Photios group mudstones are dominant, for example Maa–Palaikastro. The first hypothesis would give rise to questions as to why potters would consistently choose materials from these specific locations (proximity to the workshop or a tradition of itinerant potters). The second hypothesis on the other hand could be used to support a model of exchange between sites (Keswani, 2008). Naturally, to verify either hypothesis a sufficient amount of samples from each context should be investigated and the full petrographic information of the samples should be taken into account.

5. Conclusions and further work

The current case study demonstrates the challenges in provenancing fine-grained temper materials such as mudstones. Isotopic data proved to be in this case the most suitable technique for distinguishing the specific mudstone formations examined. The microtextural and elemental characteristics that were recorded contributed to the interpretation of the provenance of sherd but on their own would be insufficient to provenance the mudstones. The theoretical framework for applying these results in order to provenance mudstone temper from ceramic sherds is also presented and could prove useful in constraining the provenance of sherd to regions of the Mamonia terrane. A suitable case study for the application of this methodology on mudstone-tempered ceramics originating from Cyprus is being considered. Finally, additional mudstone samples that were collected during the course of the fieldwork for this study will be analysed to enhance the reference database.

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