CALCAREOUS MICROFOSSILS AND PROVENANCE STUDIES OF EARLY SASANIAN PERIOD CERAMICS FROM THE ARCHAEOLOGICAL SITE OF QIZLAR QAL'EH (NORTH-EASTERN IRAN)

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Introduction

Microfossils can provide detailed information concerning the provenance of ceramic matrices (Riley 1981; Quinn and Day 2007a; Tschegg *et al.* 2009; Wallis *et al.* 2011; Kramar *et al.* 2012, 42; Wallis *et al.* 2014), building materials (Wilkinson *et al.* 2010; Tasker *et al.* 2013), sediment blocks (Mentzer and Quade 2013, 87), mosaics (Wilkinson *et al.* 2008), and the geomorphological context of archaeological sites (Boomer *et al.* 2007; Sblendorio-Levy and Howe 1998).

Microfossils can occur naturally in clay as non-plastic inclusions. A comparison of the microfossils in a clay matrix with those in clay sediments in the field can help to identify whether or not they are naturally present or intentionally added as part of a sand temper to the clay during its preparation. In addition, the detailed study of microfossils enables us to reconstruct the palaeoenvironmental conditions of the original deposit. Since microfossils recur with other mineral inclusions, comparing these minerals with those in clay samples from the area surrounding the site, helps to identify the secondary deposits in which microfossils were found.

This paper discusses petrographic analyses of 13 coarse ware samples (Figure 1) and three local clay samples from Qizlar Qal'eh. The site is located just east of Komishan, on a surrounding plain, *c.* 18 m above sea level, and 32 km west of the Caspian Sea, next to the Gorgan River. Qizlar Qal'eh has spectacular ruins of a fortress from the early to the late Sasanian period, and it was designed as a military fort. It is connected to the Gorgan Wall, dated to late Sasanian period (Figure 2).

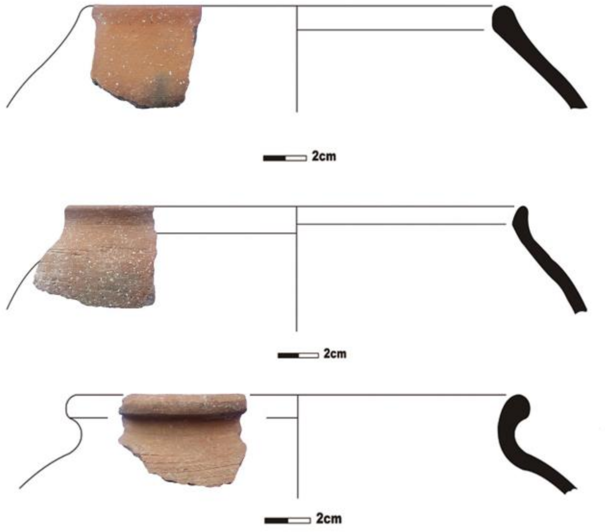

Figure 1. Coarse wares from Qizlar Qal'eh containing calcareous microfossils; the lower sherd (no. 3) was TL dated .

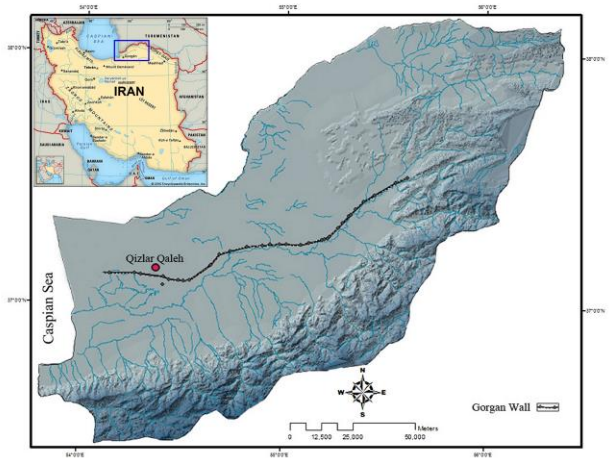

Figure 2. Map of the Gorgan Wall and location of the archaeological site of Qizlar Qal'eh in the western part of the Great Wall.

Thermoluminescence dating

Thermoluminescence (TL) dating of one sherd from Qizlar Qal'eh (sherd 3, petrography no. 2281; Figure 1, lower) containing microfossils was conducted in the laboratory of nuclear physics of the Kashan University, Iran. The date obtained of 1817±84 BP suggests that the ware appeared in the early Sasanian period (Figure 3).

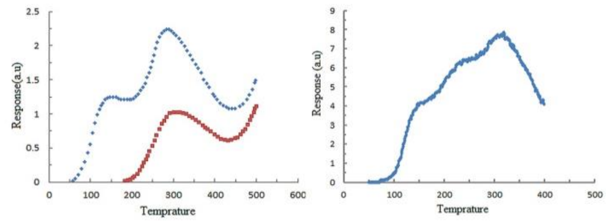

Figure 3. Thermoluminescence glow curves of a ceramic containing calcareous microfossils give a date of 1817±84 BP.

Petrographic analysis

The ceramics and clay samples from Qizlar Qal'eh were analysed in thin section. Microfossils, including different types of planktonic and benthic foraminifera, were identified in the thin sections at magnifications of 100-400X (Figures 4-7). The aims of the analyses were to identify the microfossils genus and species and assess the clay sources used in ceramic manufacture, as well as studying the technology, including firing temperatures.

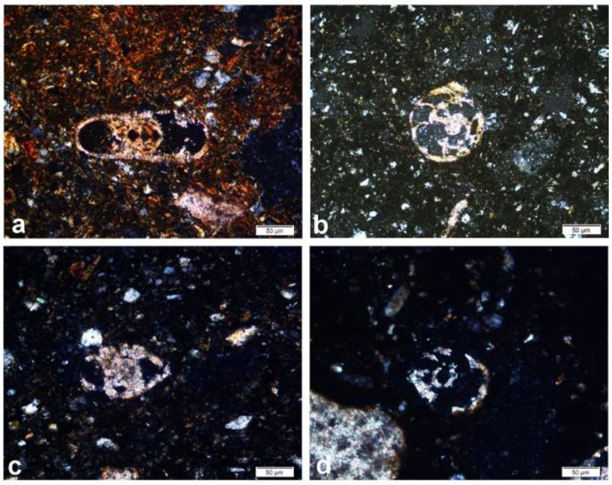

Figure 4. Qizlar Qal'eh: micrographs of benthic foraminifera of early Sasanian ceramics. (a) Cyclammina sp., planispiral, calcareous hyaline wall, seen in cross polarised light (XPL); (b) Rotaliidae sp., calcareous hyaline wall, equatorial section, seen in plane polarised light (PPL); (c) Rotaliidae sp., calcareous hyaline wall, axial section (PPL); (d) undetermined foraminifera, calcareous hyaline wall, equatorial section (XPL).

The microfossils were only found in the thin sections of the ceramics dated to the early Sasanian period. These ware types are generally manufactured by slow wheel-throwing and have a very coarse fabric. They are classified as storage vessels and appear for the first time in the IVA phase of the ceramic chrono-typological sequence (see Figure 1). The early Sasanian ceramics are concurrent with the construction of the Gorgan Wall, around the central fort; the poor quality of the ceramics and the shallowness of the archaeological layers suggest that the site was used as a temporary settlement for the builders during the Gorgan Wall construction.

The early Sasanian ceramics contain abundant planktonic and benthic foraminifera and mollusks, which are overgrown with secondary calcite. The recurrent inclusions are chert, quartz, sandstone, fossiliferous limestone, abundant calcite and occasional igneous rock fragments. The presence of chert, quartzarenite, limestone, metamorphic rocks and crushed shell fragments in the ceramic fabric and in the alluvial samples collected from the adjacent area to the Gorgan River, suggest a local source of alluvial sediments. The correlation between the sand grain size in the clay samples and the ceramics (Kramar *et al.* 2012, 42) indicates that the mineral inclusions in the pots are naturally occurring.

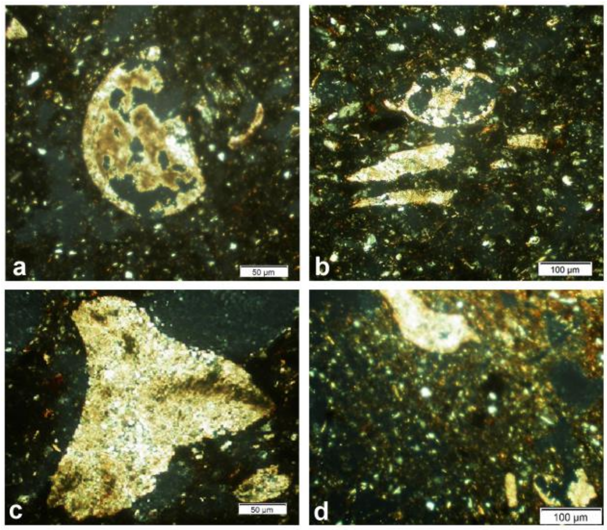

Figure 5a-d. Qizlar Qal'eh: micrographs of ceramic thin sections showing poorly preserved planktic Foraminifera, due to high firing temperatures.

On the basis of the microscopic examination of the inclusions in the samples analysed, three different petrogroups were distinguished: one containing microfossils, one group that was grit-tempered (with inclusions such as limestone and grog, or polymineralic temper), and also a group containing both microfossils and sand temper. Mineralogical analysis shows a single source for both coarse textured grit- and sand-tempered wares, from the alluvial clay from this region.

The compositional variability within the clay suggests mixing of clays from two or more local deposits (Papachristodoulou *et al.* 2006, 351). Because of the clay mixing from different local deposits (Bagnasco *et al.* 2001, 230; Quinn and Day 2007b, 164; Riley 1981, 336), and the distribution of homogeneous clay over a large area (Steponaitis *et al.* 1996; Tite 1999, 196), the mineralogical analysis cannot be adequate to identify the precise provenance of the raw materials used. Therefore, microfossil analysis was undertaken for comparison with the results of mineralogical studies.

Benthic foraminifera such as Rotaliidae sp., *Cyclammina* sp., (Figure 4), planktic foraminifera like *Dicarinella* sp. (Figures 5 and 6a, b) and ostracod (Figures 6c, d) were identified in the ceramic thin sections. Benthic and planktic foraminifera, which are naturally present in the ceramic matrix, came from the stratigraphic sediments of the Late Cretaceous, contemporary with the deposits of the Abderaz Formation, in the north-east of the Gorgan plain, and prove the primary origin of the microfossils that can be identified along the Gorgan River. Microfossils of post-Early Cretaceous are also combined with Quaternary microfossils such as recrystallised mollusks (Pelecypods), bivalves (large massive bioclast) and ostracods (Figures 6 and 7), which can be found along the Gorgan River.

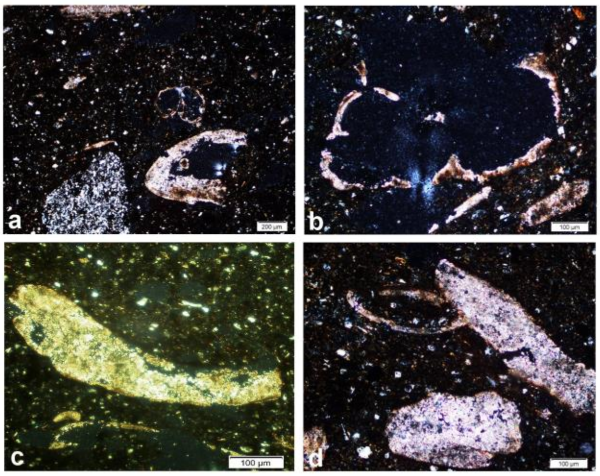

Figure 6. Qizlar Qal'eh: micrographs of thin section ceramic samples showing: (a) Dicarinella sp.? (b) Dicarinella sp.; (c) Ostracod and large massive bioclast; (d) Ostracod and undetermined bioclast (brachiopods?, echinoderms?).

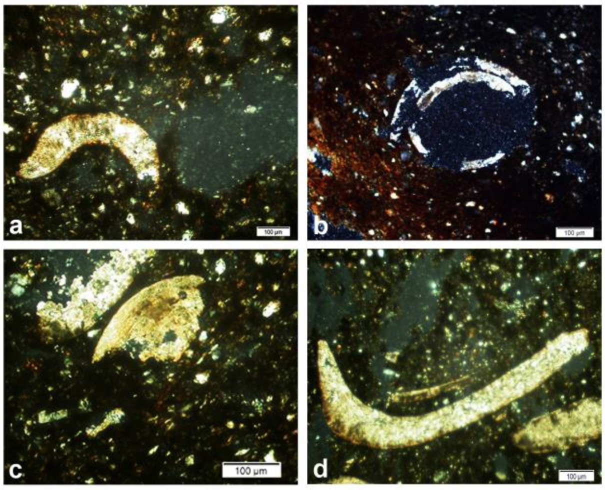

Figure 7. Qizlar Qal'eh: bivalves in thin sections of the early Sasanian period ceramics showing: (a) Pelecypod; (b) bivalve mould; (c) recrystallised mollusks (Pelecypods?); (d) brachiopod (all XPL).

The presence of the post-Early Cretaceous planktonic and benthic foraminifera in combination with the Quaternary microfossils and the shells can also be indicative of the reworked deposit. According to Quinn and Day (2007b, 164) "mixed microfossil assemblages in ceramics can also result from the exploitation of river clays containing reworked material".

Some microfossils in other samples are not well preserved because they were altered during the manufacturing and firing processes (Quinn and Day 2007a). This phenomenon prevents their identification to genus and species.

Discussion

As determined, the Quaternary facies that contain shell fragments, igneous rocks and debris grains indicate a marine Quaternary sediment that is reported from the Gorgan plain, a Kahrizak formation that is called the Apsheron Formation. Apsheron deposits are overlaid by Bakovian, which dates from Late Pliocene to Early Pleistocene, and contains a pelagic (deep-sea) fauna (Afshar 1989). Some microfossils present in low-fired ceramics are well preserved, others are not due to the shaping and firing processes.

A variety of benthic foraminifera such as *Rotaliidae* sp., *Cyclammina* sp., planktic foraminifera like *Dicarinella* sp., and other planispiral foraminifera were present in the early Sasanian ceramics. The foraminifera are characteristic of post-Early Cretaceous, derived from the sedimentary deposit of the Abderaz formation in the north-east of the Gorgan plain. Therefore, this area can be considered as a potential original basin (autochtonous) for raw materials used for the pottery production. The post-Early Cretaceous microfossils are mixed with Quaternary microfossil species such as mollusks (Pelecypods) and bivalves (large massive bioclasts) derived from alluvial sediment. Mollusks and bivalves were naturally present in the clay used in the production of early Sasanian ceramics. In addition, the presence of microfossils from two different geological ages that occurred in the ceramic matrices shows that they were reworked and transported from the original basin or deposit to a secondary one (allochthony) located in the surroundings of the site and adjacent to the Gorgan River.

Homogeneous distribution of crushed freshwater shell fragments throughout the ceramic paste and the raw clay samples analysed from the area around the site demonstrate that the coarse wares were produced from local raw materials. Crushed freshwater mussel shells, which are present along interior rivers and streams, can be utilised as a temper (Weinstein and Dumas 2008, 202).

The coarse wares mainly include chert, quartz, limestone, iron oxides, metamorphic and igneous rock fragments, minerals that are compatible with the local geology. Although such minerals occur in a wide area of the north and north-west of the Gorgan plain, the presence of shell fragments that naturally occurred in the soils surrounding the site and the ceramic thin sections show that they have the same petrographic characteristics. According to I˙ssi "these non-plastic materials may be a natural substance of the clays or they may be used as additional compounds" (I˙ssi *et al.* 2011, 2576). The presence of sand temper was identified on the basis of the particle size of minerals present in the ceramic matrices and those in the raw clay samples. Due to the mineral particles' angularity, it is also possible to hypothesise that the clay sediments used by the potters were collected not far from their original source.

The petrographic analysis of the sand- and grit-tempered ceramics, and the ceramic samples containing microfossils, dating back to the early Sasanian period, indicates that different local clay sources were exploited.

The effects of firing on the nature of calcareous microfossils and minerals

Although identifying the provenance of such samples is quite difficult, there is a potential to identify the ceramic firing technology (Quinn and Day 2007a, 776). Identifying thermal behaviors of the microfossils can be considered a useful method in understanding the ceramic firing technology. Through the study of the structural changes that occurred in microfossils and minerals, we can estimate the amount of heat received by the ceramics. The petrographic observation of the microfossils wall structure shows significant properties depending on the firing temperature. Microfossils which are well preserved, show that they were exposed to low temperatures, or a short firing period.

Petrographic examination of the thin section of one ceramic sample indicated structural changes taking place in the mollusk and bivalve wall structures with exposure to heat. The presence of benthic and planktic foraminifera, ostracods and mollusks with altered structures in the samples (Figure 5 and 7) confirm a firing temperature between 650 and 850 °C. Maritan *et al.* (2007, 535, 537) concluded that "shell fragments maintain their internal structure up to 650 °C. The shell fragments also decompose completely and all the internal structures are obliterated above 850 °C". According to Quinn (1999, 31; 1998, 87) "microfossils can be degraded by heating above 600 °C, and can be completely destroyed at temperatures of 800-900 °C". The presence of fossil moulds is highly indicative of biogenic calcite that has been lost in the ceramic matrix. On the other hand, there is no indication of lamellarity in the specimens. This means that all forms (including the ostracods) have been roasted beyond any structure except for overall morphology.

Extensive studies done on thermal behaviors of crushed shells show that the aragonite structure present in shell fragments irreversibly transforms into calcite after heating at 400 °C (Felicissimo *et al.* 2010, 2185; Feathers 2006, 92; Collins 2012, 3700). Bivalve shells were a mixture of aragonite and calcite (Adams and Mackenzie 1998, 33). The high proportion of calcite in the investigated samples confirms chemical decomposition due to firing which lead to the gradual transformation from aragonite to calcite after heating at 400 °C (Feathers 2006, 92).

The study of thermal behaviors of minerals in archeological ceramics (I˙ssi *et al.* 2011; Iordanidis *et al.* 2008) also enables us to estimate the firing temperature when the microfossils have been completely destroyed. The results indicate that the firing temperature, estimated by thermal behavior of minerals and microfossils, did not exceed 850 °C.

Besides the firing temperature, other factors such as length of the firing process and the firing atmosphere (I˙ssi *et al.* 2011, 2578) also influence the structure of the clay minerals in the matrix. For instance, calcite decomposition ends around 825 °C in kiln firing, but it extends to 875 °C in pit firing conditions (I˙ssi *et al.* 2011, 2578).

Conclusions

The preliminary results of mineralogical and microfossil analyses indicate that the two are complementary in terms of provenance and technology.

A detailed study of microfossils in the ceramic matrices shows foraminifera characteristic of the post-Early Cretaceous period which were derived from sedimentary deposits of the Abderaz formation in the north-eastern Gorgan plain. On this basis, the north-east of the Gorgan plain can be considered a potential source of raw materials used for the ceramic manufacture.

Since post-Early Cretaceous foraminifera were mixed with Quaternary microfossils, and also shell fragments and minerals which can be found around the site adjacent to the Gorgan River, it is thought that the raw materials used in the production of the early Sasanian coarse wares were derived from the alluvial sediments collected in the surroundings of the site north-west of the Gorgan plain. On the other hand, due to the lack of a stratigraphic sequence of the Cretaceous geological age in the north-west of the Gorgan plain, it is possible to hypothesise that post-Early Cretaceous foraminifera were transported elsewhere from their original deposit.

The occurrence of chert, quartzarenite, limestone, metamorphic rocks and crushed shell fragments in the archaeological ceramics and in the alluvial samples derived around the site adjacent to the Gorgan River, show that they are compatible with the local geology of the north-west of the Gorgan plain. Mineralogical analysis shows that a single source was exploited for both coarse textured microfossils and grit-tempered wares. Microscopic examination of microfossil structures suggests a firing temperature between 650 and 850 °C.

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BOOK REVIEW

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Isabelle C. Druc. *Portable Digital Microscope - Atlas of Ceramic pastes: Components, Texture and Technology*. Deep University Press, Blue Mounds, Wisconsin, 2015, 120 pages, 140 + colour photographs.