

THE CHARACTERIZATION OF MEDIEVAL CERAMICS EXCAVATED FROM THE EĞİRDİR CARAVANSERAI (TURKEY)

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Thirteen potsherds from the Eğirdir Caravanserai (south-west of Turkey) were characterized by optical microscopy (OM), X-ray diffraction (XRD) and scanning electron microscopy (SEM). Mineralogical and petrographic studies of medieval potsherds in there show a ceramic body composed of a microcrystalline to amorphous matrix with various clasts and voids. The thin section, XRD and SEM analyses of samples showed that potsherds consist of K-feldspar (orthoclase), plagioclase (albite and anorthite), pyroxene (diopside), melilite (gehlenite), mullite, wollastonite, mica (biotite and muscovite), chlorite, leucite, amphibole, quartz, calcite, iron minerals (hematite and magnetite) and trace amounts of sphene. The obtained results showed that calcareous materials including illitic-kaolinitic clays were used for pottery production and the potsherds were fired in the temperatures from 800 to 1150°C.

INTRODUCTION

The interdisciplinary work of ceramists and archaeologists has lately attested to be of great help in understanding and often solving archaeological problems such as the provenance and characterization of ceramic materials and technologies used by potters [1]. Such work provides clues to interpret the social, political, economic and cultural contexts and evolution of ancient societies as well as their interactions within sites or regions. In the study of ancient pottery, it is important to combine geological, characterization and technological techniques to obtain more specific results. In addition, one technique is usually not enough to characterize and define the mineralogy and firing temperatures because sherds have been fired and exposed to weathering processes [2].

The close relationship between the firing of pottery and the natural processes leading to the petrogenesis of metamorphic rocks has already been pointed out [3], [4], [5]. Pottery can thus be considered as an artificial, hand-crafted rock produced by firing in a kiln "industrial metamorphism" and the investigation is analogous to the study of metamorphic rocks. In fact, in pottery, firing minerals commonly grow both replacing mineral phases present in the clay e.g., clay minerals, calcite and along grain boundaries of phases with contrasting chemistry

e.g., reactions between calcite or quartz with clay matrix [4]. These incomplete reactions allow deciphering some of the dynamic aspects of the process, because relic temper grains normally coexist with firing phases. The growth of mineral phases is related both to the firing temperatures and to the composition of the microsites in which they grow.

Aside from a few of works, ceramic characterization including the mineralogical and petrographic studies on ancient ceramics of Turkey is rare in literature. This paper presents the first study of the potsherds excavated from the Eğirdir caravanserai near the Eğirdir County, located north east of the city of Isparta (Figure 1). Eğirdir caravanserai was served on the caravan road located along with Konya (capital city of Seljuks) to Antalya at Mediterranean part during the Anatolian Seljuks period. Eğirdir khan or caravanserai or caravanserai was the fourth biggest caravanserai in over the hundred structures built during the Anatolian Seljuks period (1077–1307 AD) (Figure 2).

The excavations of caravanserai were completed at three seasons; 1993, 2006 and 2007. Before the excavation, caravanserai is a ruin and only the part of its wall could be noticeable. It was initially observed that this building having two main parts, courtyard and shelter, resembles to Seljuk's caravanserai. However, there was not any information regarding to past history

of this place. When the archaeological excavation was finished, architectural information such as construction plan, building materials and techniques used in the caravanserai have been mostly revealed. Moreover, the dating of caravanserai and the destruction reason were estimated roughly [6, 7, 8]

However, the archaeological evidence and information about the location of caravanserai are still limited to evaluate whether there was ceramic production in there or not during the medieval to Seljuk period. Hence, this study will not only be enlightened the ceramics of that age, but also present first and significant information about that region.

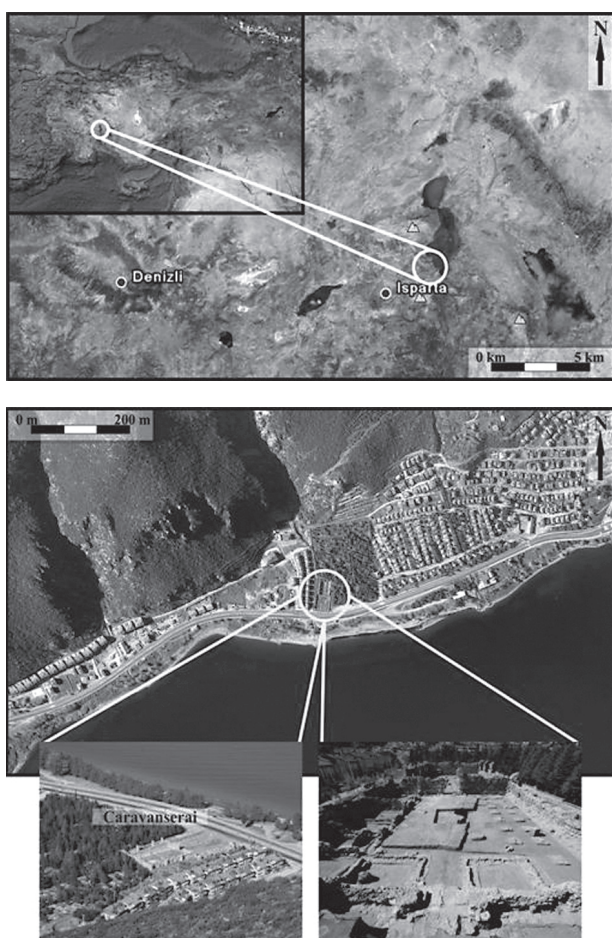


Figure 1. The map of Eğırdır caravanserai and general views of excavation area.

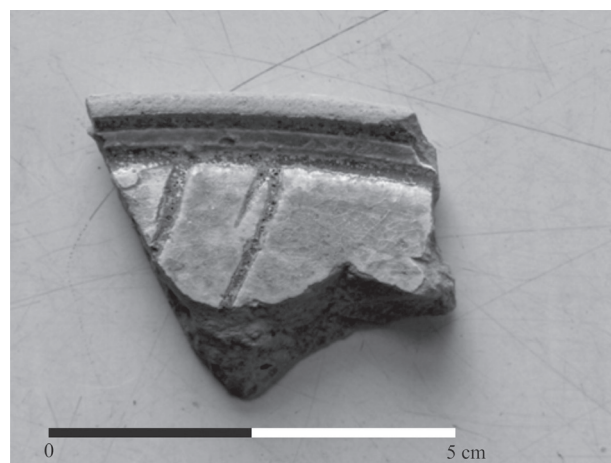
EXPERIMENTAL

Analytical strategy was developed at different scale levels: textural analysis (optical microscopy – OM and a scanning electron microscopy – SEM); mineralogical composition (X-ray diffraction – XRD).

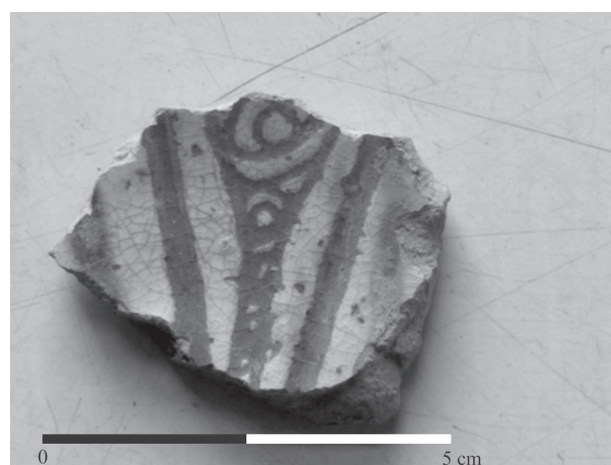
All ceramic pieces obtained from excavation were cleaned, made their drawings and grouped in terms of applied techniques and paste structures. Ceramic

fragments found in this place have various forms and size; some of them do not have any glaze, which could be classified as earthenware. Furthermore, mostly glazed earthenware fragments have single color glaze and decorated by sgraffito and champlévé techniques (Figure 2), which indicate that these ceramics belong to medieval period because other techniques such as stone paste or fritware were used after medieval. Indeed, single color glazed earthenware decorated with sgraffito technique was common at Anatolia region during the medieval period. A total of thirteen potsherd samples were studied.

Macroscopically, the surface (in typically 1-2 mm thickness) colours of potsherds were brown-pink-orange claret red, whereas the inner part of the samples, 2-3 mm in thickness, was gray-beige colours (Figure 3). In general, they comprised various minerals under the partially textured part in addition to rock fragments, which were bonded with body having moderate porosity. The binding materials-body was brownish-claret red colour, which contained amorphous materials and cryptocrystalline body including clay-iron oxide mixture.



a)



b)

Figure 2. Glazed potsherds have single color glaze and decorated by sgraffito (a) and champlévé (b) techniques.

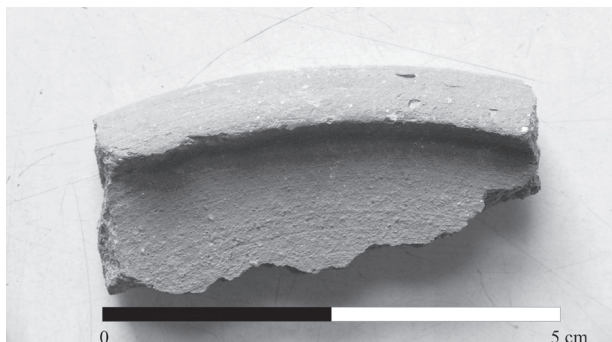


Figure 3. The surface colours of potsherds (sample no.5) are brown-pink-orange claret red.

From each potsherd, two slices were cut, in order to prepare thin sections for the mineralogical and petrographic studies in polarized light. Visual examination took place under both plane polarized and cross-polarized light using a Nikon Eclipse E200 Pol research grade petrographic microscope. Under plane-polarized light, the overall matrix color of the potsherd and other textural and structural features within the sherd were examined and described. The aplastic components were evaluated based upon grain size, shape, frequency, and homogeneity of distribution. Proportions of components were determined by visual examination and point counting techniques [9].

Using a 2.5 \times objective (25 \times total magnification at the eyepiece), the grain size differences of the aplastic components were measured using a calibrated micrometer that is part of the petrographic optic system. This involved a quick strip-grid count to evaluate whether the grain size distribution was uniform, bimodal or trimodal. Non-plastic components greater than 0.1 mm (overall dimension) were defined as temper and documented for the crystal shape, color, pleochroism and presence of alteration. Opaque minerals were also evaluated using some of these afore mentioned criteria. Cross-polarized light was used to define the identity of the aplastic components, as well as separating mineral grains from rock fragments. In addition, it was also used to access the isotropic behavior of the paste, the variation in firing (oxidized versus reduced zones), and to determine the presence of original void spaces. In the thin-sectioning process it is possible to “pluck” out mineral (or rock fragment) grains and produce a void that mimics original void space. Often this difference can be identified by observation under cross-polarized light.

The samples of ceramic fragments for mineralogical analysis were prepared by milling in an agate mortar. XRD analysis was performed at room temperature with a Rigaku Miniflex diffractometer operating at 30 kV and 20 mA, at a scan speed of 2 $^{\circ}$ /min, from 2 to 70 $^{\circ}$ 2 θ , using CuK $_{\alpha 1}$ radiation with λ : 1.54051Å. The diffraction patterns were interpreted with the use of integrated X-ray powder diffraction software (PDXL: Rigaku Diffraction Software) and PDF-4 Mineral Databases.

The chemical analyses of potsherds were determined by a Spectro X-Lab 2000 X-ray fluorescence (XRF) spectrometer using clay standards. Potsherds, ground to fine powder, were mixed with lithium tetraborate for chemical analysis.

A Jeol 6340 SEM with an energy dispersive system (EDS) was used to analyze chips on fresh fractures. Samples for morphological studies were mounted on Al stumps using a conductive glue. A thin coating of Au and Pd was applied to the surface of the stumps.

RESULTS AND DISCUSSION

Mineralogical and Petrographical analysis

The examination for the geological background of region where the excavation took place is crucial to understand raw materials used for the ceramic pieces. Eğirdir region exhibits basement of calcareous rocks occurred from mainly ophiolitic rocks and the stack of marine sediment dated Mesozoic age at the south part of the district. The Quaternary alluvium is founded on these basement rocks.

It was not encountered any clay deposit around the Eğirdir in consequence of field study. However, the clay levels were determined near to the vicinity of field (Isparta region) at Neogene basin. Clay levels alternated usually with carbonated sedimentary such as marl, clayey limestone and travertine, which contain more or less carbonated compounds. The clay occurrences have been classified within the lacustrine clays and fluvial clays by Deniz et al. [10]. The fluvial clays at the region were accumulated mainly at the platform in the middle of mountain, valleys and polje. These clays, typically yellowish with light brown colours as well as indefinite layer and weak hardened, has the average thickness varying between 2 and 3 m. General paragenesis including quartz + mica + chlorite + smectite + kaolinite + feldspar was stated for the fluvial formation. Diagenetic mineral paragenesis at lacustrine formation includes usually quartz + clay minerals (smectite, illite and kaolinite) + carbonate minerals (calcite, aragonite and dolomite) + feldspar [10, 11]. In addition, the presence of carbonate minerals such as calcite, aragonite, and dolomite indicate rapid change of physical and chemical conditions at diagenetic stage.

The modal mineralogical and petrographical data of the potsherds were obtained from OM and XRD. Mineral contents were estimated from OM studies. In thin sections, all pottery pieces were composed of matrix (40-75 %), clasts (25-60 %) and voids (generally 2-4 % and infrequently 10 %). The matrix was a clayish mass transformed upon firing. Depending of the intensity of the thermal changes, the matrix was either microcrystalline or amorphous. Petrography served to distinguish only one fabrics from the Eğirdir site. Generally speaking, there is limited number of minerals identified in thin-section: quartz (3-5 %), pyroxene (1-12 %), micas (biotite 4-5 %

and muscovite 1-2 %), opaque mineral (mostly hematite, 3-5 %), feldspar (primarily detrital grains of plagioclase and orthoclase, 12-40 %), mafic minerals (amphibole, 1-3 %), alteration mineral (chlorite, 2-3 %) and trace amounts of sphene (Figure 4a). Rock fragments (4-12 %) are generally pieces of igneous rocks (pumice) (Figure 4b), likely metamorphic and negligible amounts of sedimentary rocks (limestone) (Figure 4c).

The samples showed a highly birefringent, micro-crystalline matrix, typical for illitic-kaolinitic clays. Thin layers of an amorphous isotropic phase surround the clay particles and reflected the sintering process. Some samples had an isotropic matrix composed of glass mixed with an amorphous phase i.e. clay minerals with collapsed structures.

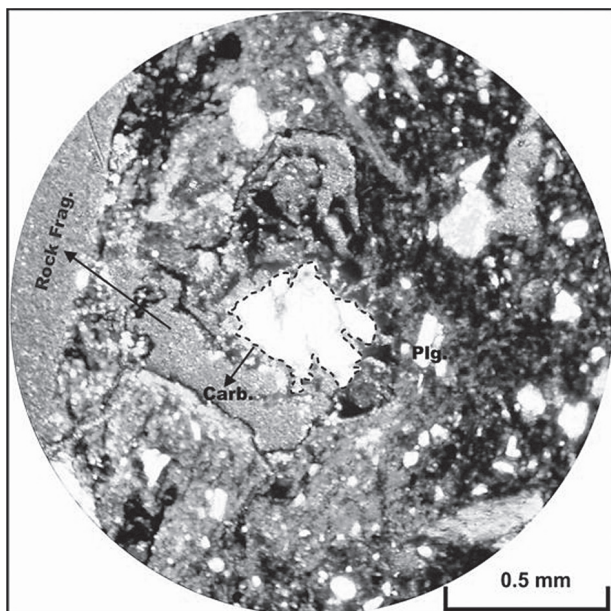


Figure 4a. Sedimentary rock (limestone) fragment in the thin section image of potsherd sample No.9. 2.5× (magnification), cross polarized light (XP): Scale bar = 0.5 mm; Rock Frag. - Rock fragment; Carb - carbonate mineral (calcite); Plg. - plagioclase.

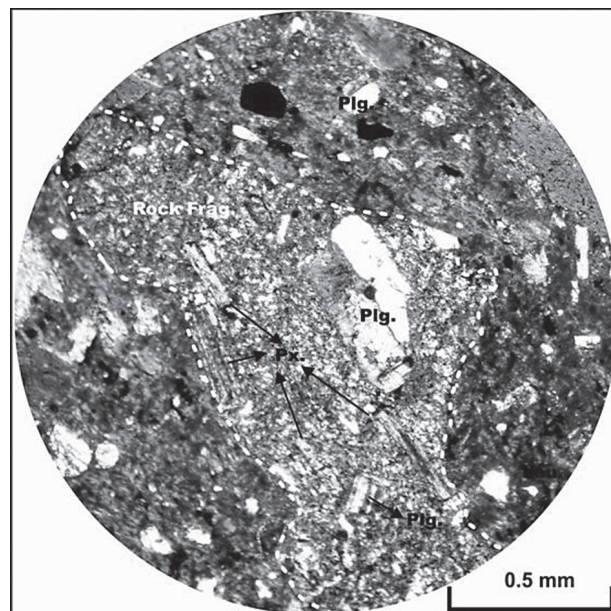


Figure 4b. Igneous rock fragment (pumice) in the thin section image of potsherd sample No.12. 2.5× (magnification), cross polarized light (XP): Scale bar = 0.5 mm; Rock Frag. - Rock fragment; Carb - carbonate mineral (calcite); Plg. - plagioclase.

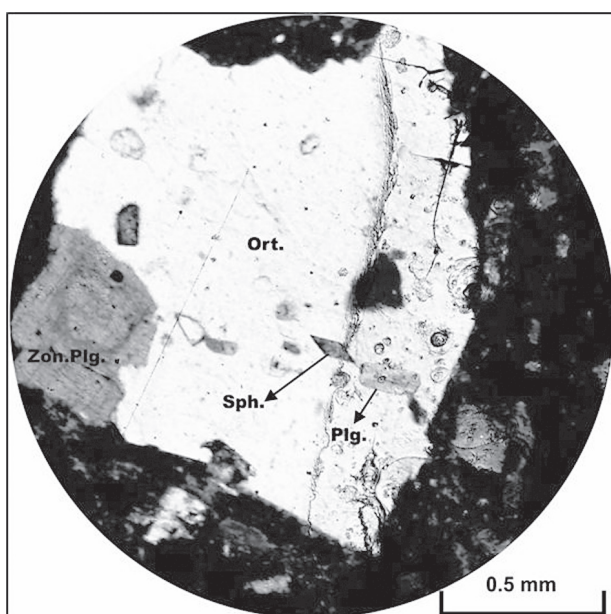


Figure 4c. Thin section image of potsherd sample No.8. 2.5× (magnification), cross polarized light (XP): Scale bar = 0.5 mm; Zon.Plg. - zoning plagioclase; Plg. - plagioclase; Ort. - orthoclase; Sph. - sphene.

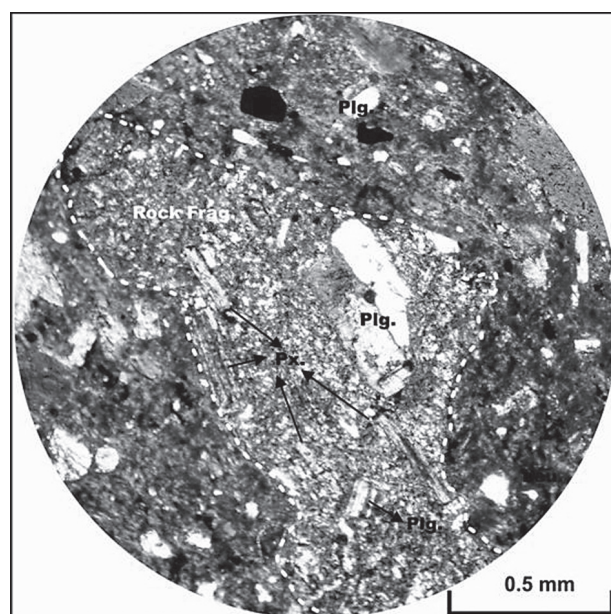


Figure 4d. Thin section image of potsherd sample No.11. 2.5× (magnification), cross polarized light (XP): Scale bar = 0.5 mm; Bio - Biotite.

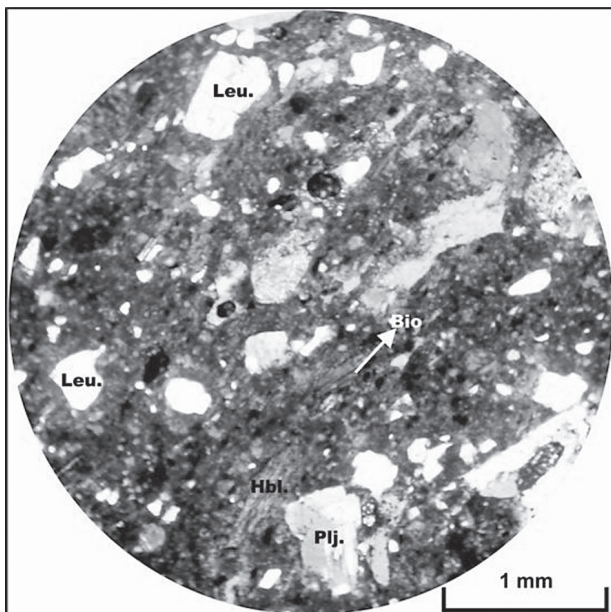


Figure 4e. Thin section image of potsherd sample No.7. 5× (magnification), cross polarized light (XP): Scale bar = 1 mm; Leu - Leucite; Bio - Biotite. Plg. - plagioclase; Hbl - Hornblende

When viewed in plane polarized light, all the samples showed a brownish-red translucent to opaque matrix, due to hematite grains. Hematite grains have a common occurrence, being opaque in transmitted light. Also zones which were full of amorphous vitreous materials made up of iron oxides with a reddish colour. Feldspar grains retain their original morphology and optical characteristics. Grain boundaries are sharp and well defined, many are twinned crystals. Mica (biotite and muscovite) lamellae did not preserved their original characters, such as shape and optical characteristics and they became opaque. Biotite displayed decreasing of pleochroism (Figure 4d) and muscovite became yellowish. Quartz grains did not revealed any noticeable morphological transformation.

The results of the chemical analysis of ceramic bodies, since all analysed fragments are characterised by a high percentage of SiO₂ (values ranged between 55.8-65.3 %), Al₂O₃ (21.8-22.7 %) and Fe₂O₃ (9.7-10.4 %) in the ceramic body, they can be identified as iron-rich siliceous body ceramics. Moreover, considering the moderate Na₂O (1.1-3.8 %) and K₂O (3.0-8.1 %) contents, the addition, in the ceramic paste of a high amounts of alkaline feldspars, plagioclase and leucite (Figure 4e) can be assumed.

Petrographic examination suggests that part of the temper could have been artificially added. Thin-section petrography revealed an approximately bimodal temper grading consisting mainly of sand-grade quartz and pumice fragments. The posherds were made with a silica-based, predominantly calcareous, raw clay with a high percentage of non-plastic material. The petrographic and mineralogical features of the potsherds, i.e. clasts and

matrix, compare well with the neighbouring geological formations such as illitic-kaolinitic clays thus suggesting a local production source.

SEM analysis

General textures of potsherds observed by SEM, let identify melting features in some sherds. Occasionally, a continuous glass phase typical of high temperature ceramics was visible under the SEM. Melting and welding had occurred locally, and the degree of particle interconnection was significant, suggesting an increase in firing temperature (Figure 5a). This figure has areas within the matrix with smooth surfaces and welded particles around coarse grains suggesting incipient melting. Figure 5b has areas with bubbly and glassy textures, attributed to partial melting at temperatures higher.

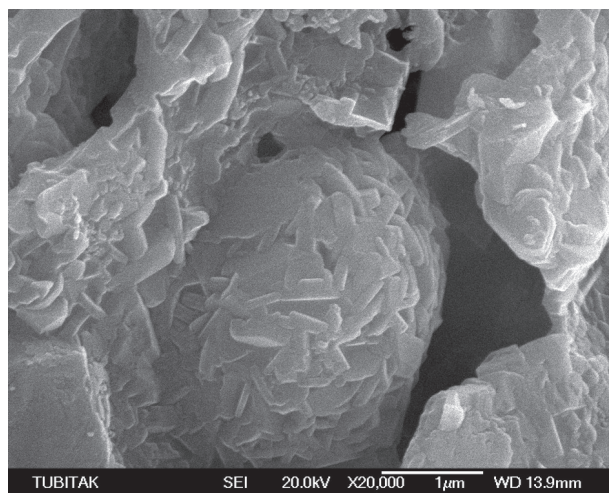


Figure 5a. SEM images of potsherds sample No.8 showing welded surfaces around grains in the potsherd. Elongated semi-tetragonal to irregular tetragonal shape of the mullite crystals.

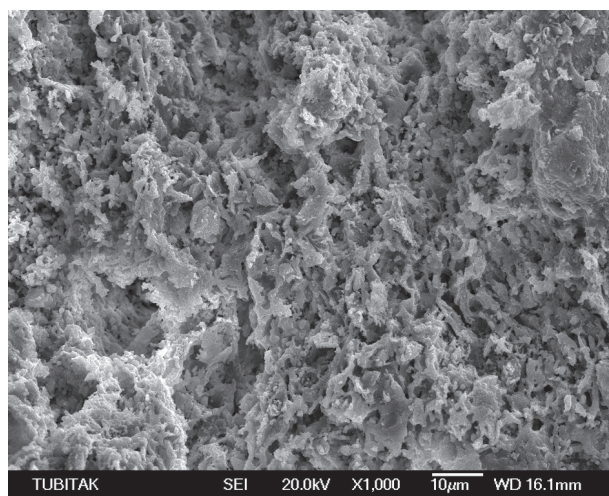


Figure 5b. SEM images of potsherds sample No.6 showing partial melting of clay matrix in the potsherd.

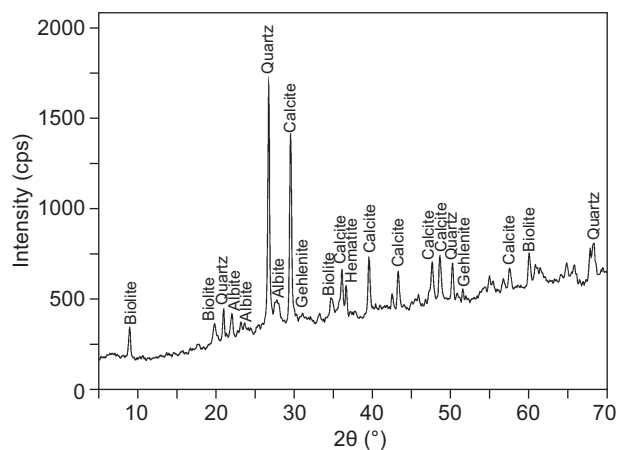
SEM images of the sample S8 (Figure 5a) revealed the particles of submicrometre size. Size ranges and morphology of the mullite crystals in sample S8 are explained below. Some crystals with elongated semi-tetragonal to irregular tetragonal shape were observed as well (Figure 3). Aggregates of semi-tetragonal particles predominated over coarser crystals. They consisted of stacks of a great number of thin tetragonal particles (0.25 µm wide by > 0.35 µm long and 0.01 µm thick).

Firing process

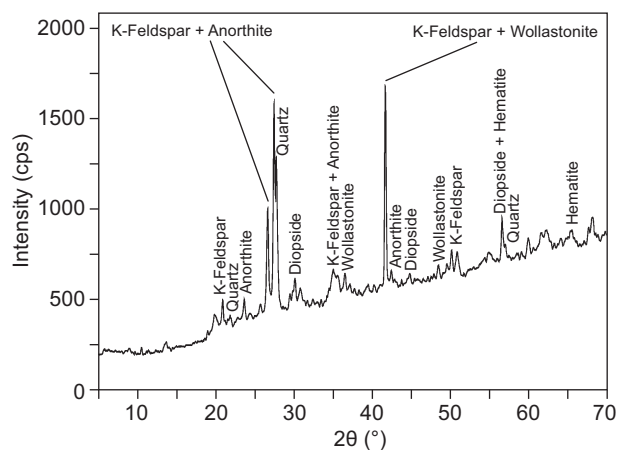
Firing process of potsherds was also investigated. Technological data have been inferred by comparing microtextures, mineral occurrence and their relative compositions. Firing temperatures are usually used in archeoceramic researches to determine the technological attributes of an historical sherd and to infer some of the aspects of the material of a given historical period [12].

Quartz (SiO₂), pyroxene (diopside [Mg Ca (Si₂O₆)]), plagioclase [albite (NaAlSi₃O₈) and anorthite (Ca₂Al₂SiO₈)], melilite [gehlenite (Ca₂Al₂SiO₇)], leucite (KAlSi₂O₆), mullite (Al_{4.64}Si_{1.36}O_{9.68}), wollastonite (CaSiO₃), iron minerals [(hematite (Fe₂O₃) and magnetite (Fe₃O₄)] and biotite [K(Mg,Fe)₃AlSi₃O₁₀(F,OH)₂] were detected by XRD for all samples as major mineral and phases (Table 1). Calcite (CaCO₃) was detected at only one sample (Sample 9) (Figure 6a).

The quartz content is high, the iron minerals (hematite and magnetite) occur in all samples. Peaks of clay minerals (particularly illite and kaolinite) are absent, according to the widespread textural modification of clay matrix occurred during the firing. Due to the partial collapse of the crystalline structure of clay minerals most likely illite, only the 4.5, 2.5 and 2.6Å reflections had been identified. Additionally, the intensity of the 4.5Å reflection (illite and mica) was diminished except Sample 4, 5 and 9.



a) sample 9



b) sample 13

Figure 6. X-ray diffraction patterns of: a) sample 9 and b) sample 13.

Table 1. XRD results and estimated firing temperatures of the potsherds.

Sample No	Main minerals/phases	Estimated firing temperature (°C)
1	Anorthite, diopside, leucite, K-feldspar, quartz, magnetite	900-1000
2	Anorthite, diopside, K-feldspar, quartz, magnetite, hornblende, muscovite (heated)	900-1000
3	Anorthite, diopside, leucite, K-feldspar, quartz, magnetite	900-1000
4	Anorthite, gehlenite, labradorite, quartz, biotite, albite (heat-treated), hematite	850-900
5	Anorthite, gehlenite, labradorite, quartz, biotite, albite (heat-treated), hematite	850-900
6	Mullite, anorthite, diopside, K-feldspar, quartz, magnetite	1050-1100
7	Anorthite, diopside, K-feldspar, quartz, magnetite, albite (heat-treated)	900-1000
8	Mullite, anorthite, diopside, K-feldspar (orthoclase), quartz, magnetite	1050-1150
9	Gehlenite, calcite, quartz, biotite, albite (heat-treated), hematite	800-900
10	Mullite, anorthite, diopside, K-feldspar, quartz, magnetite	1050-1150
11	Anorthite, leucite, K-feldspar, quartz, albite (heat-treated), hematite	1000-1100
12	Wollastonite, anorthite, diopside, K-feldspar, quartz, hematite	1000-1100
13	Wollastonite, anorthite, diopside, K-feldspar, quartz, hematite	1000-1100

The 7 Å reflection of kaolinite was missing in all samples. The continuous presence of hematite and magnetite as well as the red and orange colours of most potsherds indicate that the potsherds were fired in a permanently oxidising atmosphere, therefore suggesting a kiln operation. The type of firing atmosphere is related to the method of firing, and is partially responsible for certain characteristics of the fired ware, e.g. colour. Within the white portion firing minerals such as diopside-wollastonite is present (peaks in the range of 27–34° 2 θ) (Figure 6b). Mullite and gehlenite were also present. K-feldspars and plagioclase show anomalous asymmetric peaks. This could be related to a progressive lattice transformation and to the formation of coronitic reaction rims [13].

Dehydroxylation of most clays generally occurs between 450°C and 550°C [14]. The study of mineral transformations shows the persistence of clays (chlorite, illite, muscovite and kaolinite) crystalline structure up to 900°C during firing [14, 15, 16, 17, 18]. Nemezc [19] showed that certain diffraction lines of clay minerals such as illite and montmorillonite tend to diminish starting with 750–800°C. The lack of kaolinite lines in all samples did not indicate that this mineral was originally missing in the raw materials but suggests temperatures much above 600–650°C when the crystalline structure collapsed and metakaolinite formed. Decomposition of illite starts at about 850°C but traces of illite are still observed up to 1000°C [20]. Depending on the chemical composition of illite, a wide variety of resulting phases is possible in clay systems, e.g. hematite, corundum, feldspar, leucite, spinel etc. [21].

Several authors [22, 23, 24, 25, 26] presented some mineralogical constraints for the firing mineral developed at different temperatures in Ca-rich and Ca-poor clays. Furthermore, experimental work of Riccardi, et al., [27] showed that the feldspar zoning develops at T: 950°C in Ca-poor clays. Calcareous clays or marl were discussed by Peters & Jenny [28] and Pollifrone & Ravaglioli [29]. Capel et al., [30] studied the formation of gehlenite and calcic plagioclase in calcareous clays. Gonzalez-Garcia et al., [31] verified the presence of gehlenite and anorthite phases when firing clays from the Guadalquivir valley which were originally composed of illite, kaolinite, quartz and calcite. The presence of periclase is usually observed (MgO) after firing ceramic bodies with magnesium carbonates. MgO has lower reactivity than CaO [16, 32].

Decomposition of calcite (CaCO₃), dolomite [CaMg(CO₃)₂], and magnesite (MgCO₃) occurs at temperatures between 800°C and 950°C [33]. Samples with poor content of CaCO₃ provide very simple mineralogical composition (quartz and hematite) [17]. Calcium carbonates have high reactivity, therefore they would easily react with quartz and other minerals from destroyed clays, producing crystalline phases such as gehlenite, anorthite and wollastonite [16, 18, 32].

Gehlenite is formed when a CaO nuclei reacts with clay's Si and Al. This reaction is observed above 800°C. This phase is metastable because it can react with quartz to give wollastonite and plagioclase (anorthite and albite) [18].

According to Carretero, et al., [34] in the calcareous body, the slow-fired samples present a larger amount of newly formed phases (plagioclase, clinopyroxene, melilite) if compared with the fast-fired ones, both at 1000 and 1100°C. At the higher temperature, a more abundant formation of plagioclase and pyroxene occurred, partially at expense of melilite and quartz. In contrast, only quartz, feldspars and hematite compose the calcium-poor body in similar amount after firing at 1000 and 1100°C.

By combining these results with various reference data [12, 13, 22, 25, 31, 34, 35, 36, 37, 38, 39, 40, 41, 42] the firing temperatures for the ceramics could be inferred. Samples showed sintering processes limited strictly to the contact between the particles and indicating at least 600°C. Some samples had an amorphous matrix, with more frequent melting and amorphisation processes, thus indicating temperatures over 750°C [39] but below 850°C.

Over 850–900°C the melting of matrix components was already common. Biotite started to decompose around 800°C and the partial decomposition, changes of birefringence, birefringence and cleavage planes as noticed in samples suggested firing around 850°C. The forming of gehlenite in samples pointed to temperature above 800–850°C [27] but the presence of not-transformed or only marginally decomposed inclusions, such as calcite grains or limestone clasts within the Sample 9, suggested firing temperatures below 850°C (calcite thermally decomposes in the interval 600–850°C in a heated, natural clay).

In Ca-rich clay, a simplified mass balance may be used to model the firing reaction. Illite and calcite represent the phases present in the natural clay, whereas K-feldspar and gehlenite are the firing phases. The breakdown of calcite reveals Ca, which is released CO₂ at 850°C as a component of the fluid phase in Ca-rich clays. At temperatures exceeding 850°C, albite changes its composition by reaction with the clay matrix. A rim of K-feldspar (KAlSi₃O₈) overgrows the albite crystals in the sample fired at 1050°C, according to the mass balance reaction. The zoning pattern is characterized by a steep decrease in the Na content with a concomitant increase of K [12].

The Ca-content of new phases formed within the clay matrix is enhanced with increasing temperature. These phases move towards the anorthite composition. At this point quartz starts to be involved in the reactions. At temperatures of 1050°C the presence of anorthite and diopside is evidenced by specific compositions and X-ray data; even if gehlenite is still present [27]. The neomineralization of mullite is observed from

temperatures of 1000°C upwards. The kaolinite layer-lattice structure undergoes a progressive reorganization through an intermediate cubic spinel-type phase and it finally reaches the chain structure of mullite-like [17, 33, 43, 44]. It was found that wollastonite and anorthite are stable in the potsherds for temperatures exceeding 950°C [3].

CONCLUSION

The characterization of ancient ceramics indicated that the medieval potsherds found in the Eğirdir caravanserai (Isparta, Turkey) were mainly produced from the clay deposits which are rich in calcareous materials including illitic-kaolinitic clays.

The potsherds displayed some of the most typical features of the hand-made range, uneven surfaces and moulding stage. On the basis of this information and the knowledge of the contemporary pottery-making process, it is probable that they were hand-made. The potsherds should have been exposed to different firing temperatures. The mineralogical/phase contents lead to conclude that the range of firing changes from 800 to 1150°C. It may be inferred from the estimated firing temperatures that either a uniform flame distribution could have not been set up during firing.

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