Journal of Archaeological Science: Reports 29 (2020) 102136

Contents lists available at ScienceDirect

Journal of Archaeological Science: Reports

journal homepage: www.elsevier.com/locate/jasrep

Characterization of medieval glazed ceramics of the East Plain Cilicia survey

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ARTICLE INFO

Keywords:
Medieval glazed ceramics
Materials characterization
SEM/EDX

ABSTRACT

Macroscopic analysis identifies the presence of Zeuxippus ware and Port Saint Symeon ware among the bulk of medieval glazed ceramic sherds collected during the archaeological survey in East Plain Cilicia. These well-known medieval glazed ceramics have a wide distribution all over the Mediterranean basin. The initial goal of this study is to detect the characteristics of the glaze, such as composition and application methods, of the ceramic sherds. For this purpose, further examinations of the glazed sherds were carried out by optical microscope, scanning electron microscope/energy dispersive spectrometer to identify the microstructural and compositional characteristics of three basic components i.e. ceramic body, slip and glaze in sherds. For the experimental studies, cross-sections of the samples were prepared by metallographic methods and the materials were investigated by optical and scanning electron microscopes and quantitative analyses were carried out in order to reveal the microstructural features. Results were compared to understand the similarities and differences between Zeuxippus and Port Saint Symeon wares.

1. Introduction

Medieval glazed ceramics were collected in bulk during the archaeological survey of the Osmaniye Province in East Plain Cilicia, where glazed sgraffito ceramics constituted the majority (Tülek, 2007a,b, 2009, 2010a, 2012a). Plain Cilicia is located along the Eastern Mediterranean coast. During the medieval period, the early Byzantine towns and castles in the region were taken over by armies of Islam, followed by constant battles among the Crusaders, Armenians, Byzantines, and Arabs. The Mamluks ended the turmoil and ruled the region for more than two centuries, until the Ottomans finally took over. Archaeological researches conducted at the ancient towns and settlements in Plain Cilicia yield a rich cultural heritage (Tülek, 2010b; Tülek, 2012b). The bulk of Osmaniye ceramics were collected from unstratified contexts that have been destroyed during tilling, thus the collected ceramic sherds belong to various ceramic traditions of diverse cultures, including Islamic, Byzantine, and Crusaders, that occupied Plain Cilicia from the 8th to the 15th centuries CE (Tülek, 2018). Among the rich wide variety of glazed sgraffito ceramic samples collected, this study focuses on the Zeuxippus Ware (ZW) and Port Saint Symeon Ware (PSSW), which are argued to have been produced simultaneously by people of the Christian religion but of different cultures and at different production centers in distant geographical regions of Anatolia.

Zeuxippus Ware was named after the Baths of Zeuxippus by the Hippodrome in Constantinople, where kilns and wasters were uncovered. The kilns and associated finds were dated to the 11th to the 15th centuries CE, based on coin findings (Megaw, 1968). Zeuxippus Ware has a fine red ceramic body, dense and hard, which was fired at high temperature and covered with a glossy transparent or green colored glaze. Chemical analysis of the ceramic body showed that the clays used to make the ZW contain several major oxides (Al, Ca, Mg, Fe, Ti, Na, Mn, Si, K, P) and minor oxides (V, Cr, Ni, Zn, Rb, Zr, Sr, Ba, La, Ce) (Megaw and Jones, 1983; Waksman and François, 2004). Megaw had placed ZW in two major groups – ZW I and ZW II – based on the firing technique and color. ZW I was fired erected in the kiln and had no color addition, whereas ZW II was systematical, fired inverted on a tripod stilt with color addition (Megaw, 1968). Both ZW types were covered with a white slip and decorated with incised concentric circles. The bowls were glazed on both the interior and exterior surfaces, while the plates were mostly slipped on their interior surface, and in some cases along the rim on their exterior surface with dripping glaze (Megaw, 1968). Megaw divided ZW I into three subgroups, A to C, however, no grouping was done for ZW II. Waksman and François examined Megaw's samples and distinguished eleven subgroups, A to K for ZW II (Waksman and François, 2004). In Megaw's classification, ZW IA has a colorless or a pale coloured glaze, and the forms are mostly bowls of medium size in a hemispherical shape (Megaw, 1968). ZW IB

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https://doi.org/10.1016/j.jasrep.2019.102136
Received 27 May 2019; Received in revised form 11 November 2019; Accepted 27 November 2019
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group differs from the first group by having an orange–brown glaze and tripod traces. ZW IC is distinguished by its dark green glaze. The ceramics of ZW II group have specks of color in green, yellow–brown, and purple. Mostly the circular decoration at the center of the bowl was treated as a medallion containing incised floral motifs. As the studies on ZW finds from various sites continued, it became clear that ZW does not belong to a homogenous composition group nor represents the product of one center, thus new classifications have been proposed as the Zeuxippus Family, Zeuxippus Derivatives or Zeuxippus Type wares (Megaw et al., 2003) as well as Zeuxippus in the *strict sense- genuine,* and imitations (Waksman and François, 2004).

Even though initial find site of ZW was Constantinople, new production centers, such as Thessaloniki, Pergamon, Chersonesos, and Anaia-Kadıkalesi, have also been discovered as archaeological excavations continued. Thus, it has been understood that ZW has a wide distribution throughout the Mediterranean and the Black Sea (Megaw et al., 2003; Waksman, 2007; Kırmızı, 2013). A detailed map of ZW distribution is given by Waksman and François (Waksman and François, 2004). ZW ceramics found during the archaeological surveys and excavations conducted in the Cilicia region have been published in a corpus of medieval glazed ceramics of Anatolia (Böhlendorf-Arslan, 2004) which has not included the Osmaniye Provience finds such as the Kumkale excavations (Toydemir, 2001; Çambel et al., 2007). ZW sherds, identified as Zeuxippus Family, that were found in the excavations of Antiocheia (Waagé, 1948) and Port St. Symeon / al- Mina (Lane, 1938) were the only ones that were mentioned in publications. Even though ZW sherds were found during the Osmaniye archaeological survey, no production of ZW was identified in Plain Cilicia (Fig. 1).

The second glazed sgraffito ceramic group of the Osmaniye medieval ceramic repertoire is Port St. Symeon Ware, which was named after the recovery of the site with kiln wasters during the excavations of the Port St. Symeon / al- Mina (Wooley, 1937), situating at the mouth of River Orontes by the Mediterranean Sea in the Province of Antioch. PSSW is a fine glazed sgraffito ceramic, which has a red or reddish-brown clay covered with thick white slip on its interior surface and the areas below the rim of its exterior surface. Previous analysis revealed that the clay of PSSW is non-calcareous and rich in mica minerals. The micaceous clay is either ferruginous or purely argillaceous. The temper consists of several sand-sized minerals and rock fragments, including limestone, quartz, pyroxenes, amphiboles, feldspar, and fragments of igneous and metamorphic rocks. This composition is most similar to the local geology of the Antioch area (Goren, 1997). The slipped surface was incised and covered with splashes of copper oxide, iron oxide or manganese, and then glazed. After firing, the underglazed slip developed a yellowish color (Lane, 1938; Vorderstrasse, 2005).

Lane described the pottery found at Port Saint Symeon / al-Mina was homogenous, all dating to the Crusader Period level, thus he did not classify it into groups (Lane, 1938). However, Day divided these fine sgraffito ceramic finds of the Crusader period of al-Mina into four groups based on decoration styles. The first group of ceramics was similar to those found in Atlit, characterised by incised triangular shield ornaments of which Lane identified them as the products of Port Saint Symeon. The second group of ceramics was decorated with large figures and ornament all over the surface but with an undecorated rim, which also had slip and glaze on the exterior surface. The third group of pottery had less elaborate decoration than the previous groups but had an ivy rinceau decoration with coloured leaves. The fourth group of ceramics was decorated boldly with large ornaments on a spacious background (Day, 1939).

PSSW has a wide distribution throughout the central and eastern Mediterranean. Multiple production centers of PSSW were also identified in Plain Cilicia along the East Mediterranean Coast. Archaeological works in Plain Cilicia uncovered kilns and/or wasters of PSSW at Tarsus, Misis, Toprakkale, Kinet, and Epiphaneia (Fig. 1) (Day, 1939; Blackman and Redford, 2005; Hild and Hellenkemper, 1990; von Wartburg, 2001)[1]. Day (1939) identified PSSW in Tarsus-Gözlükule mound excavations, and suggested that the production continued well after the termination of the Crusader period in 1268 to the beginning of the 14th century. Blackman and Redford approved Day's assertion stating that in Kinet Mound PSS ceramic production continued well into the 14th century after the Crusades (Blackman and Redford, 2005). Particularly, the kilns recently uncovered at Misis yielded a wide range of glazed ceramics, of which macroscopic and microscopic analyses continue and have yet to be unpublished, thus testifying the long term production of ceramics during the medieval period.

Scientific analysis of medieval ceramics found at the archaeological sites in Cilicia has always been limited to macroscopic examination, mostly based on naked-eye observations of decoration, shape, clay and glaze colors, as well as clay ingredients visible to an extent. There is almost no microscopic analysis carried on the medieval ceramic finds from Cilicia regarding any ceramic tradition, except two PSSW sherds that were examined by SEM/EDX and WDX together with other Byzantine ceramics (Armstrong et al., 1997). Kinet Mound excavation team carried out chemical analysis on medieval ceramics and wasters of the kilns and identified two groups of PSSW (Blackman and Redford, 2005). As for the scientific analysis on medieval ceramic finds of Akko, petrographic analyses were carried out, which provided some information about the microstructure of PSSW as an imported ceramic (Goren 1997; Shapiro 2012). On the other hand, there are plenty of publications concerning the microscopic analysis of the ZW ceramics found at the initial excavation spots, as well as throughout the western Anatolia (Megaw and Jones, 1983; Berti and Gelichi, 1997; Armstrong and Hatcher 1997; Waksman, et al., 2008; Kırmızı, 2013).

In the bulk of Osmaniye ceramic repertoire, ZW and PSSW are represented in a variety of glazed sgraffito ceramics (Fig. 1). The ceramic fragments, which best represent their ceramic tradition and bear their respective diagnostic features, are chosen as samples for compositional characterization and microstructural examinations. Although the sample set is too small to reach any conclusions, our work can be taken as a preliminary case study that contributes to a better understanding of the composition and technology of the medieval ceramics collected from East Plain Cilicia, as previous analyses as such are limited. During the evaluation of the present data, published values are also included for comparison (Armstrong et al., 1997; Waksman and François, 2004; Kırmızı, 2013). Initial goals of the present analyses are to detect the distinctions in glaze components and manufacturing techniques of these ceramic sherds. The results were also compared to understand the similarities and differences between ZW and PSSW.

2. Material and methods

2.1. Material

Medieval glazed ceramics were collected in bulk during the archaeological survey of the Osmaniye Province in East Plain Cilicia (Tülek, 2010). The potsherds selected for this study were collected from the surface mixed together with sherds belonging to different ceramic traditions and production periods. Thus, the macroscopic analysis served to place the samples into stylistic groups according to their decoration, glaze, and form. In this study, nine sherds were chosen as

[1] Adana Museum archaeologists, recently, excavated kilns in Misis uncovering plethora of wasters of glazed ceramics testifying production activity from the early Medieval into the Ottoman Periods, of which the scientific examinations are on going. Examination of the medieval glazed PSSW ceramics found in Toprakkale Castle is on going, as well, which contains finds of trivet and unglazed half manufactured pieces (the unpublished report of the Project no BAP 2012/73, on the multidisiplinary examination of the Toprakkale Castle in Osmaniye Province by the corresponding author). The Crusade Castle Kumkale yielded a couple of thousand medieval glazed sherds of PSSW but no wasters or trivets to testify a production (Toydemir, 2001; Çambel et al., 2007).

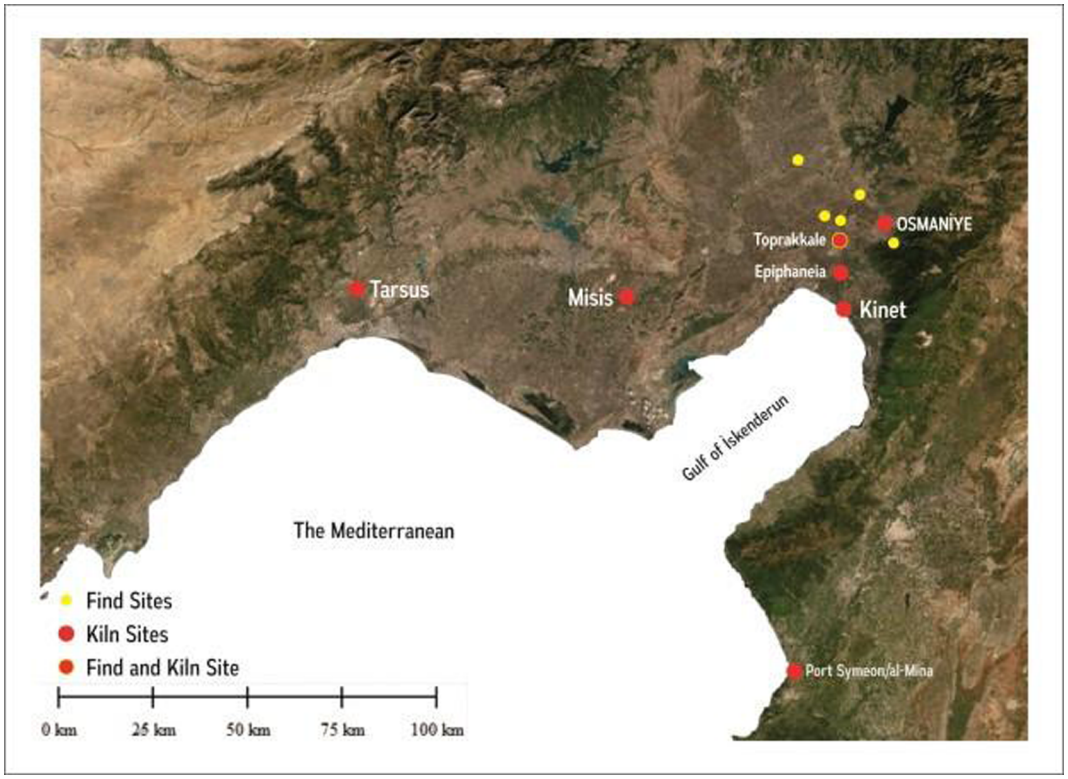


Fig. 1. Map showing the find sites of the PSSW and ZW sherds in Osmaniye Province included in this study and the production centers of PSSW in East Plain Cilicia.

Table 1
Images and descriptions of the ZW sherds included in this study.

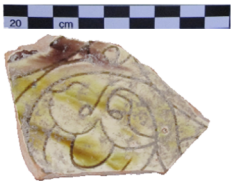
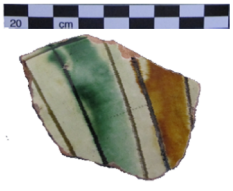
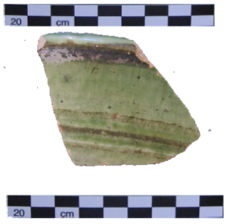
Code	Macro description	Macro image
OZW-1	Sgraffito, pale green glaze, white slip, very pale brown/buff clay (10 YR 8/4)	
OZW-2	Sgraffito, yellow glaze, white slip, pink clay (5 YR 8/4)	
OZW-3	Sgraffito, green glaze, white slip, reddish-yellow clay (5 YR 7/6)	
OZW-4	Sgraffito, green glaze, white slip, pink clay (5 YR 8/4)	

the best representatives of the ceramic traditions they belong to and are coded as OZW-1, OZW-2, OZW-3, OZW-4, OPW-1, OPW-2, OPW-3, OPW-4, OPW-5. Based on the macroscopic analysis, the first four samples were identified as ZW and the latter five samples were identified as PSSW. Tables 1 and 2 show the general description of the ZW and PSSW sherds under studied. According to the macroscopic analyses, all sherds have sgraffito type of decoration, green/yellow/transparent glaze, white slip, and red clay.

2.2. Microstructural characterization

Samples of approximately 5 × 10 mm were cut off from the broken parts of body fragments. The characterization studies were carried out in two stages: (i) optical microscope (LM, Olympus BX41M-LED) examinations revealed the basic microstructural features such as the thickness of both slip and glaze layers of the sherd samples, (ii) scanning electron microscope/energy dispersive spectrometer (SEM, Jeol JSM 6060/ EDX, IXRF) revealed the microstructural features and elemental compositions of each component (ceramic body, slip, and glaze). Before LM and SEM investigations, the sherds were cold mounted, followed by grinding and polishing to minimize the highly topographical effect and reveal the borders of ceramic body, slip and glaze. The grinding was carried out using the 320, 600, and 1000 mesh size SiC abrasives, whereas the polishing was carried out using a 3 μm diamond solution. For the LM examinations, bright field contrast was used in order to highlight all components (ceramic body, slip and glaze) having a different refractive index. Before the SEM examinations, the mounting surfaces were sputtered using elemental Au/Pd that had a high purity to enhance the conductivity of surfaces during examinations. During the SEM characterization, both topographical and compositional contrasts were used in order to obtain the distinctive features. EDX was used to determine the elemental composition of each component. The accelerating voltage was 15 kV, the beam size was 60 μm, the collecting time was 150 s, and the dimensions of the analyzed area were 25 × 50 μm. An average of five measurements were

Table 2
Images and descriptions of the PSSW sherds included in this study.

Code	Macro description	Macro image
OPW-1	Sgraffito, colorless transparent glaze, white slip, reddish-brown clay (5 YR 5/4)	
OPW-2	Sgraffito, colorless transparent glaze, white slip, reddish-yellow clay (5 YR 6/6)	
OPW-3	Sgraffito, green glaze, white slip, reddish-yellow clay (5 YR 7/6)	
OPW-4	Sgraffito, colorless transparent glaze, white slip, reddish-yellow clay (5 YR 7/6)	
OPW-5	Sgraffito, green glaze, white slip, light grey clay (10 YR 7/2)	

taken by the SEM-EDX. The elements were determined as weight % (wt %) and the arithmetic averages were used as the final data. Under optimum conditions, the detection limit is minimum 0.2 wt% for EDX measurements. In order to define the crystals in the diffusion zone, spot analyses instead of the area were carried out.

3. Results and discussion

3.1. LM examinations

The cross-section images of sherds by LM are given in Fig. 2 and Fig. 3. The images clearly show that three basic components, namely ceramic body, slip, and glaze exist. These components are also seen as a layered structure from the margin to ceramic body. A layer of slip is applied to the ceramic body in order to execute the sgraffito decoration, which was later glazed over. The slip and glaze were mostly applied on the interior side of the samples, but OPW-5 sample is distinguished for having glaze on both sides.

Identification was made based on the microstructural features that are present in both slip and glaze layers (Armstrong et al., 1997; Tite et al., 1998; Molera et al., 2001). On the other hand, the thicknesses of both slip and glaze covered on the ceramic body may have an active role in macroscopic identification. Since, single and/or multi-layers have a strong effect on optical properties (transparency level, refractive index, absorption coefficient, etc.), which are decisive factors during the naked eye examinations. Not only the thickness of glaze but also that of the interface zone are affected by some variables like firing temperature, soaking time, the composition and viscosity of the suspension to be used as glaze material depending on glazing technology (dipping, spraying, burnishing, etc.) (Burleson, 2003). The differences in the thickness of glaze could be attributed to deficiencies caused by the used glaze application method as reported by Palamara et al., (2016). They studied on the technology issues of selected Byzantine glazed pottery and they reported that the thickness of the glazed layer was 60–100 μm, however, it could be wider and in some cases, the thickness was found as 200 μm in rims or highly curved fragments (Palamara et al., 2016). For this purpose, in this section, the thicknesses of both slip and glaze layers are measured in order to group the sherds before microanalytical studies.

The thicknesses of slip and glaze layers were measured from 5 different areas in the body fragments and in each area a minimum of 10 measurements were taken for good statistics. The average data for the thickness measurements are given in Fig. 4 and the variation in the obtained data can be attributed to the lack of craft standardization. For the ZW sherds (Fig. 4a), the glaze thicknesses of OZW-2 (252 μm) and OZW-1 (193 μm) are about three times thicker than those of OZW-4 (85 μm) and OZW-3 (77 μm). The data indicate that the ZW sherds can be divided into three groups: (i) almost equal slip and glaze thickness (OZW-1), (ii) a thin layer of slip with a thick glaze layer (OZW-2) and (iii) moderate slip thickness covered with a thin layer of glaze (OZW-3 and OZW-4). According to the classification by Megaw (1968), the OZW-1 sherd belongs to the ZW-1A group, which has a pale green glaze. The OZW-2 sherd has yellow glaze, corresponding with the ZW-1B group. The sherds with dark green glaze are similar to Megaw's sherd ZW-1C group. The results of the LM examination provide an accurate match with the classification based on the macroscopic examinations since the thickness of the glaze and slip determines the optical features such as transparency level, refractive index, absorption coefficient of the sherds. The range of the glaze thickness of ZW is quite broad and the ratio between the maximum and minimum thickness levels exceed 4 (Fig. 4a). On the other hand, this ratio for PSSW is below 2 (Fig. 4b). The similar methodology used for grouping OZW is used for the OPW sherds, as well and three groups can also be defined as (i) OPW-1 and OPW-5, (ii) OPW-2, (iii) OPW-3 and OPW-4. With regard to the thicknesses of slip layers, no correlation can be detected between slip and glaze (Fig. 4b). As discussed above, both Lane (1938) and Day (1939) evaluated the PSSW sherds based on the nature of the clays and the stylistic features of the decoration. Our results on the OPW sherds do not overlap with any established classification.

3.2. SEM/EDX examinations

The ceramic body, slip and glaze of all samples were examined by SEM utilizing both secondary (SE) and backscattered electrons (BSE) and shown in Figs. 5 and 6.

The compositions of the ceramic bodies of OZW and OPW were determined by SEM/EDX and the obtained data are listed in Table 3. The ceramic body of all samples has a calcareous body due to their higher CaO content (above 6 wt%) (Maniatis, et al., 1981). In this section, the clay compositions of ZW sherds are compared with the data given by Waksman and Françoise (WF) for ZW coded samples which are from Acre (Waksman and François, 2004). In their grouping of clay compositions (Group A to I), except for Group H (ZW-WF-H), all groups have higher SiO_2 content (above 55 wt%) than that of our findings

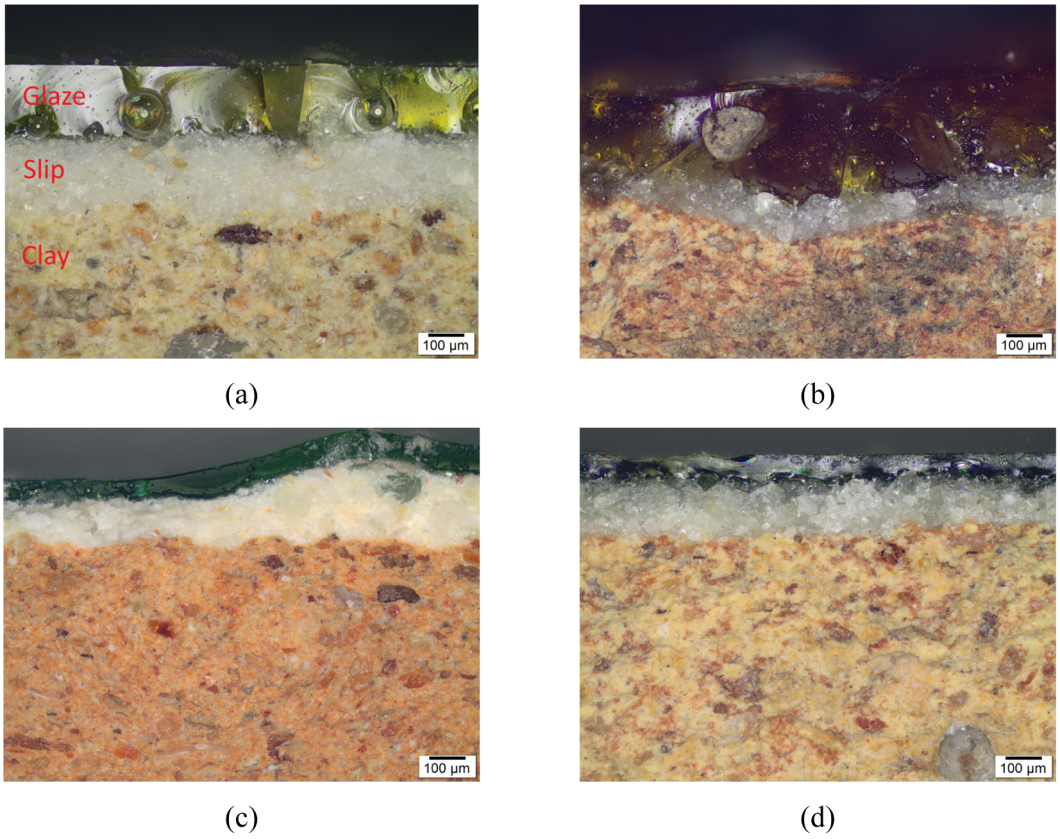


Fig. 2. LM images of ZW sherds; (a) OZW-1, (b) OZW-2, (c) OZW-3 and (d) OZW-4.

(lower than 55 wt%). In our comparison, content of the major oxide SiO_2 is taken as the main criterion in order to decide whether our results fit ZW-WF-H. For this purpose, in order to detect similarities between the OZW and ZW-WF-H, all oxide (Al_2O_3, CaO, Fe_2O_3, MgO, Na_2O, K_2O, TiO_2) compositions are given with respect to SiO_2 in Fig. 7. When the major components (SiO_2 and Al_2O_3) of clay are compared (Fig. 7a), although SiO_2 content (48,0–53,0 wt%) lies in the same range, the Al_2O_3 content slightly differs. The amount Al_2O_3 lies about at 15,0 wt% in OZW, while that of ZW-WF-H lies at about 20 wt%. In Fig. 7b, the calcerous character of the clay is compared by CaO versus SiO_2 diagram and it shows that OZW has slightly higher CaO content (14,0–20,0 wt%) compared to ZW-WF-H (10,0–15,0 wt%). Both diagrams given in Fig. 7a and b indicate that clay compositions of all OZW sherds are in close range with ZW-WF-H. The similarities between clay composition for OZW and ZW-WF-H are more obvious when rest of the oxides are considered with respect to SiO_2 as given in Fig. 7c–f.

This characterization of oxide compositions provides values of close range indicating that the production centers of OZW are within the close geographic proximity pointing to the East Mediterranean region; yet archaeological works have not yet provided evidence to locate any production center of ZW in East Plain Clicia. East Plain Cilicia as an alluvial plain is silted with thick layers of sediments due to the fluvial dynamics, that are the Ceyhan River and its branches. Principal geomorphological characteristics of the lower part of the Plain Cilicia were well examined with regard to the coastal belt, lower parts of the Ceyhan River, and the areas surrounding the two major cities, Adana and Mersin, whereas the upper, the eastern part of the Plain is less studied (Isola et al., 2017). The Cilicia-Adana basin complex, the Eastern Mediterranean is a Neogene evolution (Aksu et al., 2005) while the Misis- Andırın Complex is a Mid-Tertiary mélange (Robertson et al., 2004). The Plio-Quaternary non-marine sediments flood the northern parts of the plain, East Plain Cilicia with fine-grained pyroclastics of basalt volcanism (Tülek and Yılmaz, 2011). Besides, lacustrine clay layers are numerous as potential clay resources in the region. However, yet there are no archaeogical investigations of the pottery kilns and production workshops in East Plain Cilicia, thus it cannot be asserted whether the OZW sherds are local production or not.

When Port St. Symeon ceramics (OPW) are considered, data from present work are compared with Armstrong et al., (1997), (PW-A) and Capelli et al., (2005), (PW-C). The amounts of oxide with respect to SiO_2 are given in Fig. 8. Since the compositions given by Capelli et al., (2005) are for the content of SiO_2, Al_2O_3 and MgO + CaO, thus in the current work only those oxides could be compared. The literature data indicate that SiO_2 content of PW-A and PW-C lie within the range 45,0–65,0 wt% and is in agreement with the findings of this study. Fig. 8a shows that Al_2O_3 content of these sherds are between 6,0–20,0 wt% and the data of current work fall within this range. The total content of MgO and CaO appears to be within 17,0–38,0 wt% for all samples.

Slip compositions are also studied and given in Table 4. The major oxides of the slip compositions are SiO_2, Al_2O_3, and PbO, which are also the basic oxides observed in both clay and glaze. Since slip is placed between clay and glaze, diffusion occurs from both layers and the slip acts as an adhesion layer between them (Molera et al., 2001). Slip compositions of ZW of Anaia-Kadıkalesi excavations are also studied by Kırmızı and similar major and minor elements were detected by SEM/EDX (Kırmızı, 2013).

The glaze layers and interface between glaze and slip are studied by SEM/EDX. BSE-SEM images at higher magnification show that the glaze layers contain gas bubbles and a concentration of crystals at the

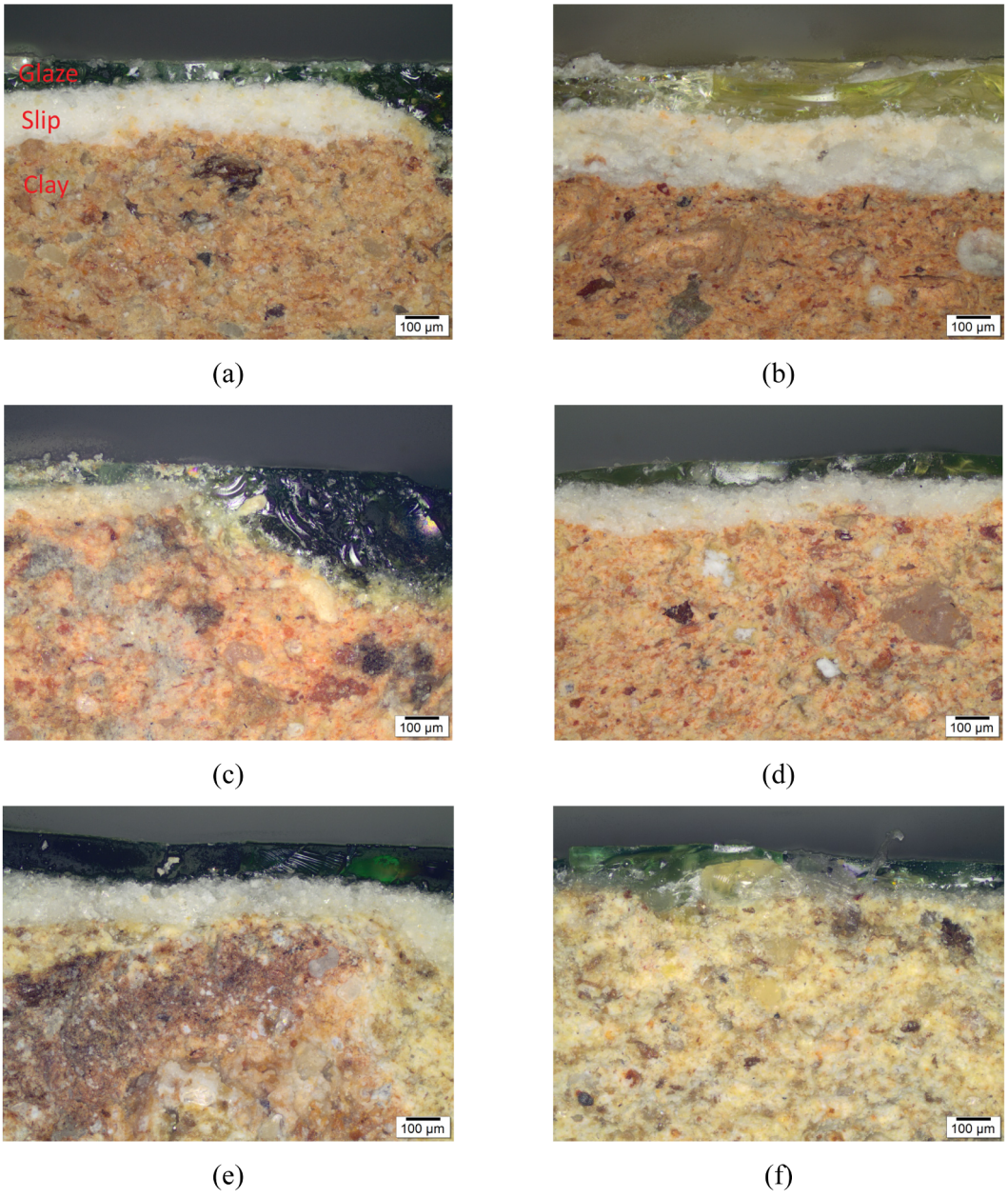
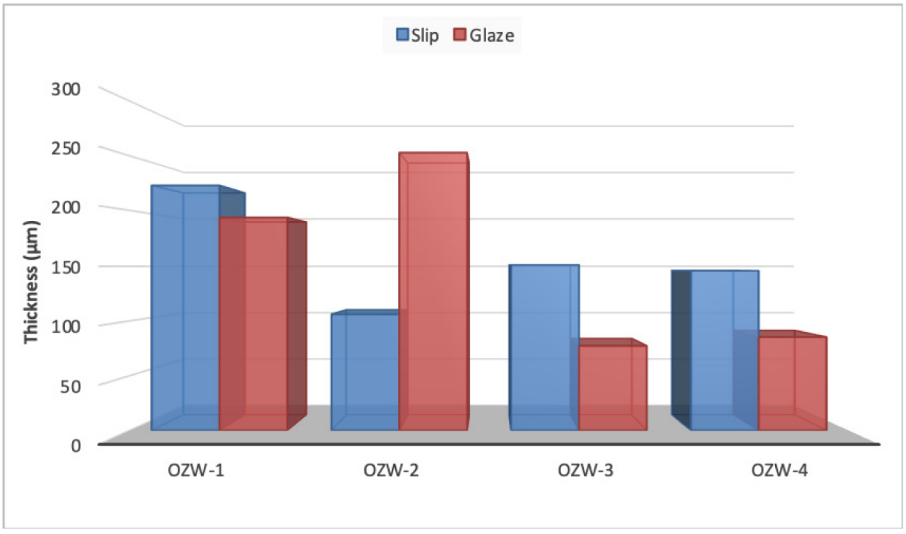


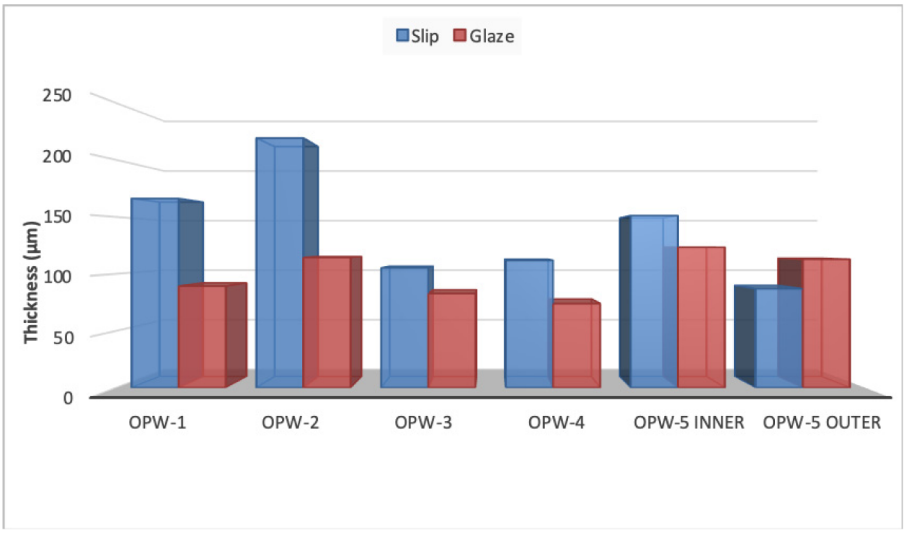
Fig. 3. LM images of PSSW sherds; (a) OPW-1, (b) OPW-2, (c) OPW-3, (d) OPW-4, (e) OPW-5 (inner) and (f) OPW-5(outer).

interface between the glaze and slip (Fig. 9). In medieveal ceramics, the underlying slip has low CaO concentration and the slipped ceramic bodies were biscuit-fired. The bubbles in the glaze layer may be due to low firing temperatures or not long enough soaking time (Tite et al., 1998). The interface zone that exists between the sections (glaze/slip or glaze/clay) is, in fact, a layer structure formed between them due to the chemical diffusion of the elements (Molera et al., 2001 Walton and Tite, 2010). The laboratory examinations of Molera et al., 2001 revealed an interface zone formed by clay and lead glaze due to different thermal paths. Any firing at about 750–1000 °C has shown that various crystallites (K-Pb feldspar, Ca-rich pyroxene, etc.) may be present in the interface zone depending on the glaze and clay compositions. The existence of crystals like quartz, wollastonite, and lead-feldspars at the interface zone of glaze and slip depends on whether the clay is calcareous or non-calcareous (Walton and Tite, 2010; Lyubomirova et al., 2017). On the other hand, during firing, elements such as aluminum, iron, potassium, calcium, and magnesium diffuse from the body into the glaze and, as the concentration of these elements in the glaze increases, crystals of potassium-lead-aluminum–silicate formed at the body-glaze interface (Tite et al., 1998). In Fig. 10a, a crystalline structure formed due to chemical diffusion of elements between slip and glaze is shown and the EDX data (Fig. 10b) shows that the structure is typical potassium-lead-aluminum–silicate.

The glaze composition consists of several components like silica, alumina, lead or alkali-based flux, and some colorants. Within the glaze composition, silica may not always be present and the presence of alumina may be associated with the used silica source. Among these components, flux materials are used in order to decrease the melting point of silica. The fluxing oxides in a ceramic glaze contain lead, sodium, potassium, lithium, calcium, magnesium, barium, zinc, strontium, and manganese. Both transparent high lead glazes and transparent alkali glazes were used in several ancient ceramics (Tite et al.,



(a)



(b)

Fig. 4. Thickness values for slip and glaze of ZW (a) and PSSW sherds (b).

1998; Maniatis and Tite, 1981; Walton and Tite, 2010). Transparent high lead types contain 45–60 wt% PbO, with an alkali content normally less than 2 wt% $Na_2O + K_2O$ and an alumina content typically in the range 2–7 wt%. This normalized composition makes the material more wettable at its firing procedure and more reflective. Tite et al. summarized the several methods to produce transparent high lead glazes and the firing temperature was within the range of 800–1050 °C (Tite et. al, 1998). However, lead-alkali glazes contain typically 20–40 wt% PbO and 5–12 wt% alkali and their firing temperatures can be 950–1000 °C.

Table 5 shows the glaze composition of the sherd samples. According to the data, the glaze is of the high lead type (45–60 wt% PbO). As seen in Table 5, glaze compositions of all sherds contain PbO and SiO_2 as major oxides, alkali oxides ($Na_2O + K_2O$), Al_2O_3, CaO and other oxides (MgO, Fe_2O_3, TiO_2, CuO). PbO, one of the major oxides, acts as a flux like alkali oxides and SiO_2 is responsible for vitrification of the glaze. The presence of both Al_2O_3 and CaO can be also attributed to the interaction with the underlying slip. The effect of coloring is provided by oxides of Fe, Ti, Mn, Ni, Zn, Cr and Cu (Khamseh et al., 2011). The observed colors in the studied sherds (Tables 1 and 2) may be due to the presence of iron oxides (green, yellow, brown), titanium oxide (yellow), copper oxide (green, blue), although the amounts of these oxides appear to be low in the compositions (Kırmızı, 2013; Burleson, 2003). Except for OPW-3, the major component in the glaze is PbO and varies between 43,0 and 68,1 wt%. OPW-3 has approximately 50,0 wt% SiO_2 as a major component. It has the lowest PbO (~27,0 wt%) and the highest Al_2O_3 content (~8,0 wt%) among the studied sherds. OPW-3 also has CuO which does not exist in the others. According to the study of Özçatal et al., glazes can be classified based on their PbO, alkali oxides and Al_2O_3 content and those having 45,0–60,0 wt% PbO, less than 2,0 wt% alkali oxides and 2,0–7,0 wt% Al_2O_3 are classified as transparent high lead glazes (Özçatal et al., 2014). OPW-1, OPW-2 and OPW-4 sherds have colorless transparent glazes as given in Table 2 and this observation supports their chemical composition according to Özçatal et al., (Özçatal et al., 2014). Such type of glaze was extensively used in Islamic and Byzantine worlds, as well as elsewhere in medieval Europe (Tite et al., 1998). LM images are given in Figs. 2 and 3 indicate that all glazes are transparent, colorless or colored. However,

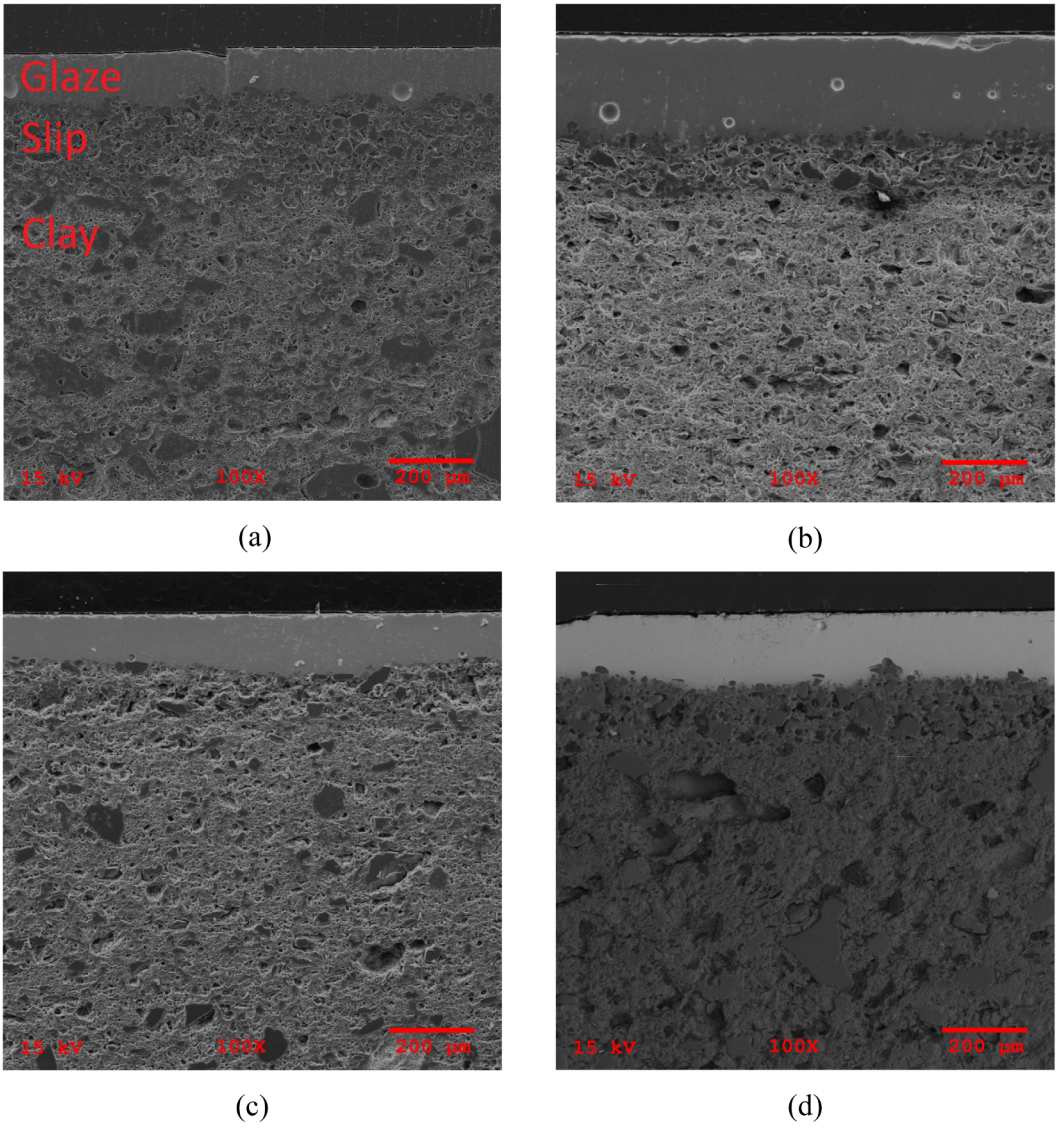


Fig. 5. SEM images of ZW sherds; (a) OZW-1, (b) OZW-2, (c) OZW-3 and (d) OZW-4.

classifying all of them as transparent glazes according to the macroscopic examinations is not easy due to both color addition and weathering conditions. The dominant color of the glaze in the studied sherds is green although brown and yellowish colors also exist. Fe^{+2} and Fe^{+3} give yellow and green colors (Molera et al., 2018).

In order to show the inverse relationship between the amount of PbO and alkali oxides data in study and also those from the published data are given in Fig. 11. Fig. 11 shows PbO versus alkali oxides of the glazes of Zeuxippus group (OZW) and Port St. Symeon group (OPW) together with the data from the literature on Zeuxippus ceramics (ZW-K) (Kırmızı, 2013) and Port St. Symeon ceramics (PW-A) (Armstrong et al, 1997). OZW samples are in the same range as the literature data (ZW-K) for both high and low PbO and alkali oxide content (Kırmızı, 2013). On the other hand, present data show that total value of MgO and K_2O are even higher than the total value of Na_2O and K_2O suggesting the use of plant ash. Since literature data of Port St. Symeon ceramics are very limited only the OPW with high PbO content could be compared with them and are very consistent with PW-A.

As discussed in Section 3.1 and given in Fig. 4, the OZW sherds can be grouped into two based on their glaze thicknesses. Those with thick glaze (OZW-1 and OZW-2) have low PbO content (40–50 wt%), alkali oxides are > 2 wt% and their silica content is between 40 and 45 wt%. Those with thin glaze (OZW-3 and OZW-4) have high PbO content (50–70 wt%), alkali oxides are < 1 wt% and their silica content is ~30 wt%. Thus, the sherds having high PbO have low alkali oxides and silica, whereas the ones with low PbO have higher alkali oxides and silica. A similar grouping can not easily be made for the OPW sherds based on their glaze thicknesses. On the other hand, they can also be grouped according to their chemical compositions; OPW-3, OPW-4, and OPW-5 that have low PbO (20–50 wt%) have high alkali oxides (> 1 wt %) and high silica content (40–50 wt%). Quite similar to the OZW group with high PbO, OPW-1, and OPW-2 have > 60 wt% PbO in their composition and their alkali oxides are less than 1 wt% with ~30 wt% silica.

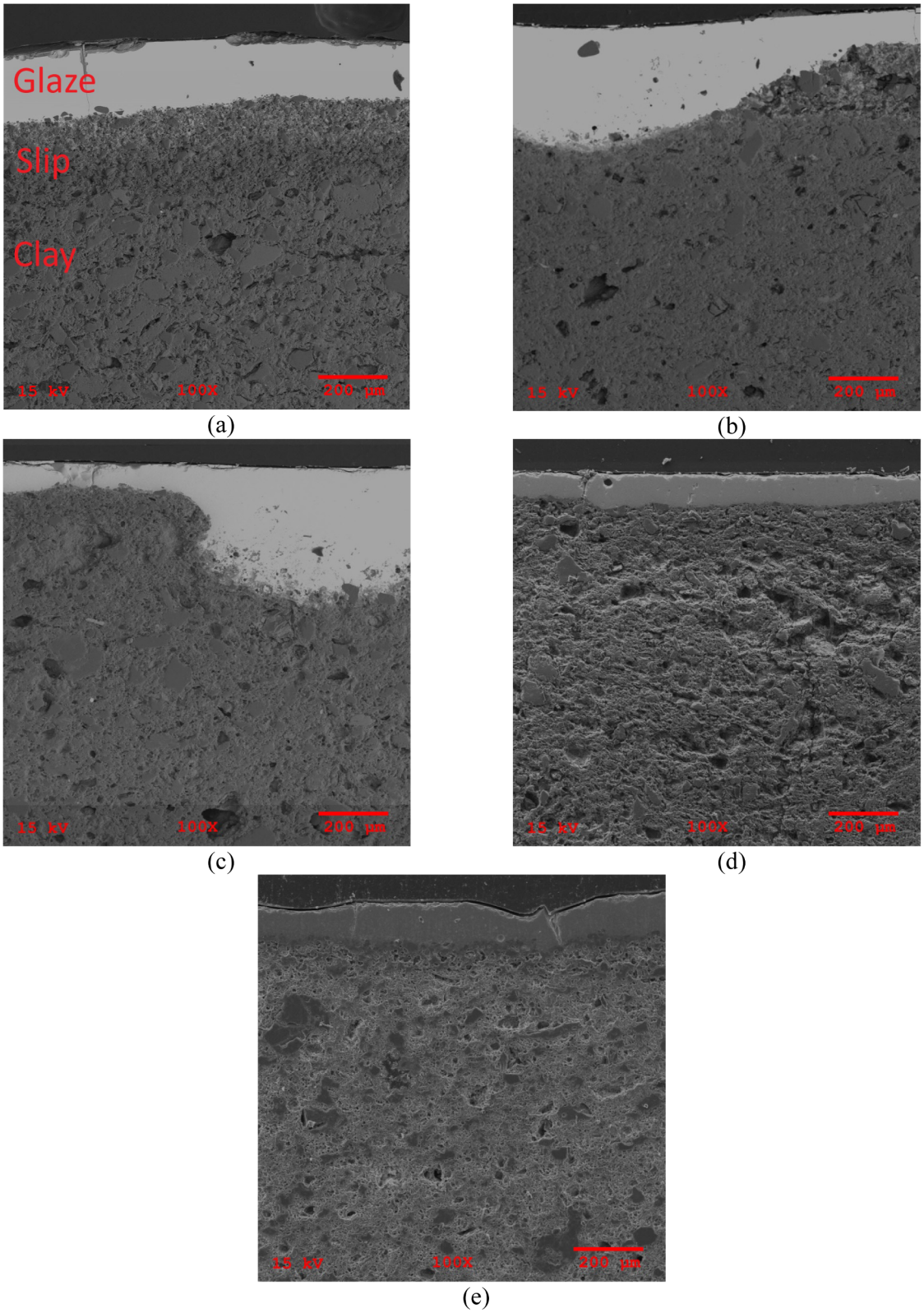


Fig. 6. SEM images of PSSW sherds; (a) OPW-1, (b) OPW-2, (c) OPW-3, (d) OPW-4, OPW-5.

Table 3
The compositions of clay (wt%).

Sherd	Sample	SiO_2	Al_2O_3	Fe_2O_3	Na_2O	K_2O	CaO	MgO	TiO_2
ZW	OZW-1	52,87	13,98	6,51	0,65	2,53	19,96	2,74	0,70
	OZW-2	50,79	15,85	7,26	0,40	2,30	19,14	3,44	0,79
	OZW-3	47,98	10,77	9,49	0,54	1,20	19,42	9,92	0,64
	OZW-4	51,60	14,54	8,02	1,87	2,10	13,24	7,99	0,60
PSSW	OPW-1	47,73	11,46	7,37	0,54	1,40	24,44	6,37	0,68
	OPW-2	52,75	8,90	7,61	0,60	1,00	8,78	19,73	0,62
	OPW-3	56,99	15,20	6,21	0,54	2,35	13,07	5,00	0,65
	OPW-4	48,28	14,55	11,37	0,96	1,35	17,57	5,03	0,89
	OPW-5	45,11	13,95	7,34	0,65	2,19	14,06	2,42	0,82

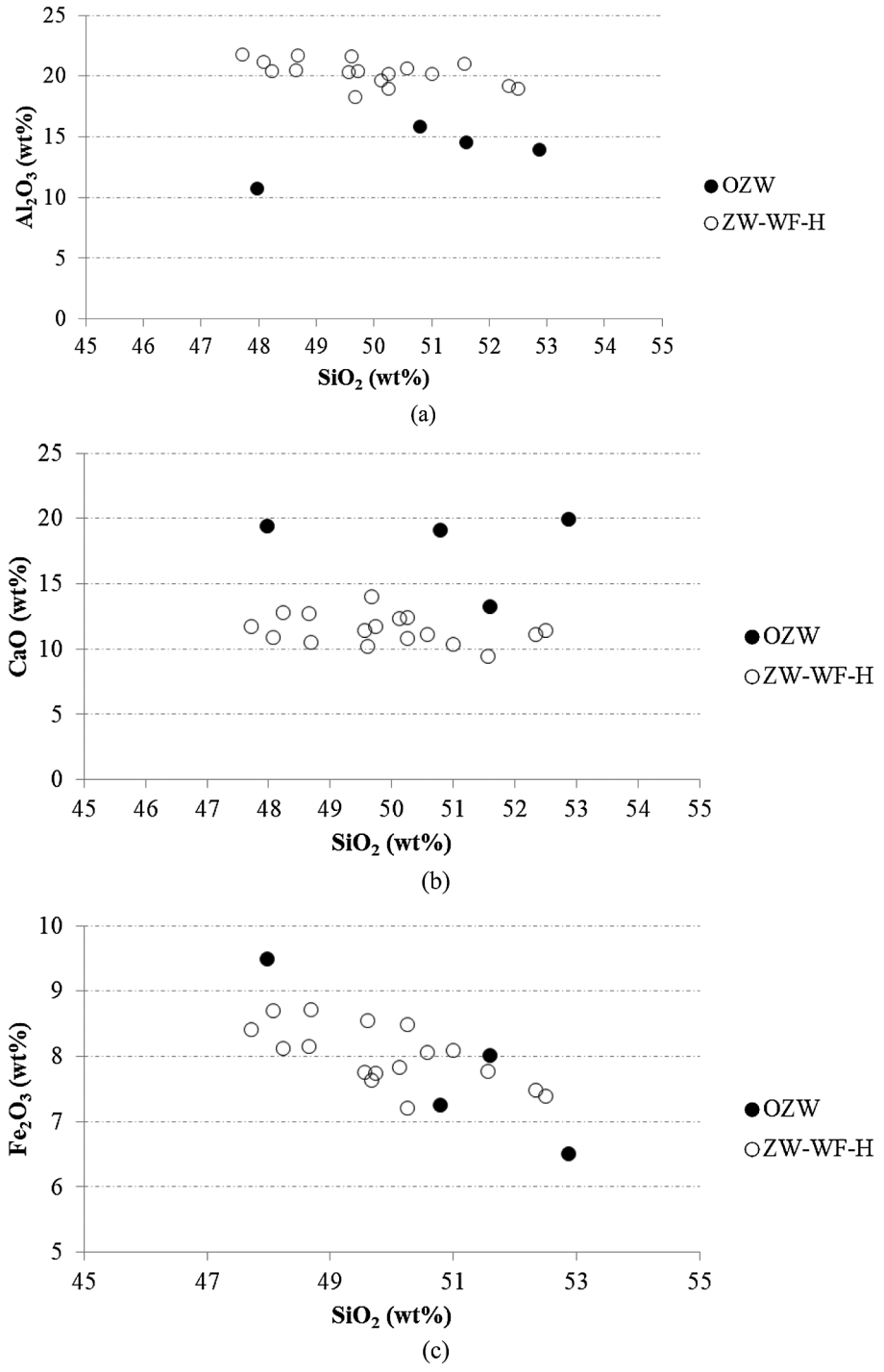


Fig. 7. Oxide compositions of OZW and ZW-WF-H with respect to SiO_2 content; (a) Al_2O_3, (b) CaO, (c) Fe_2O_3, (d) MgO, (e) total alkali oxides and (f) TiO_2.

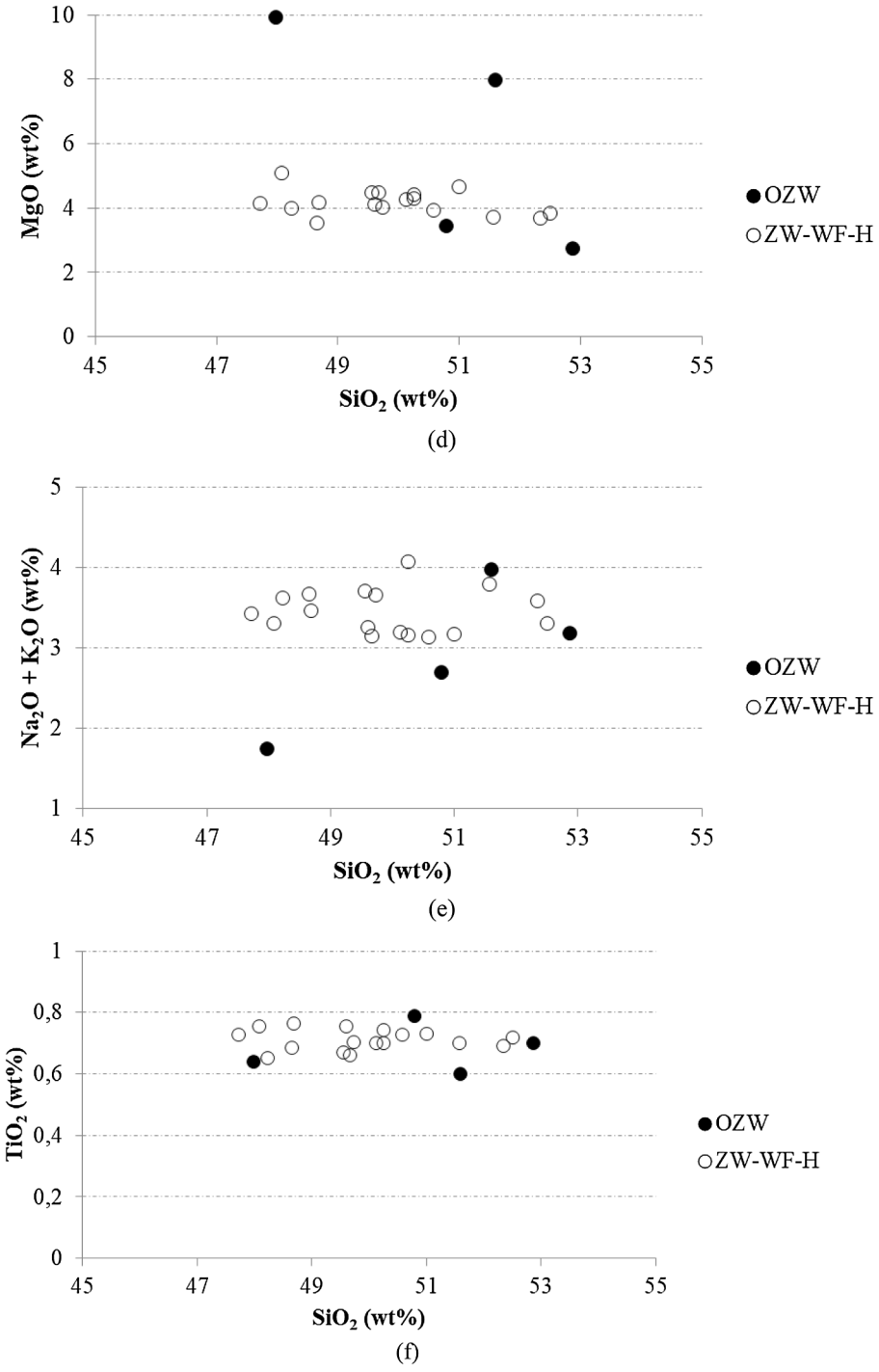


Fig. 7. (*continued*)

4. Conclusions

In this study, nine ceramic sherds from the same area – East Plain Cilicia, Osmaniye – but from different ceramic traditions were macroscopically and microscopically examined in order to detect the distinctive features in their glaze components and manufacturing techniques. The macroscopic examination identified the samples to two established ceramic traditions: four of them are of ZW and five are of PSSW ceramics. In cross-sectional examinations, the clay, slip, and glaze components were observed. In order to classify the glaze thickness and composition of sherds both of ZW and PSSW, PbO versus total alkali oxides (Na_2O + K_2O) and (Mg_2O + K_2O) variations were

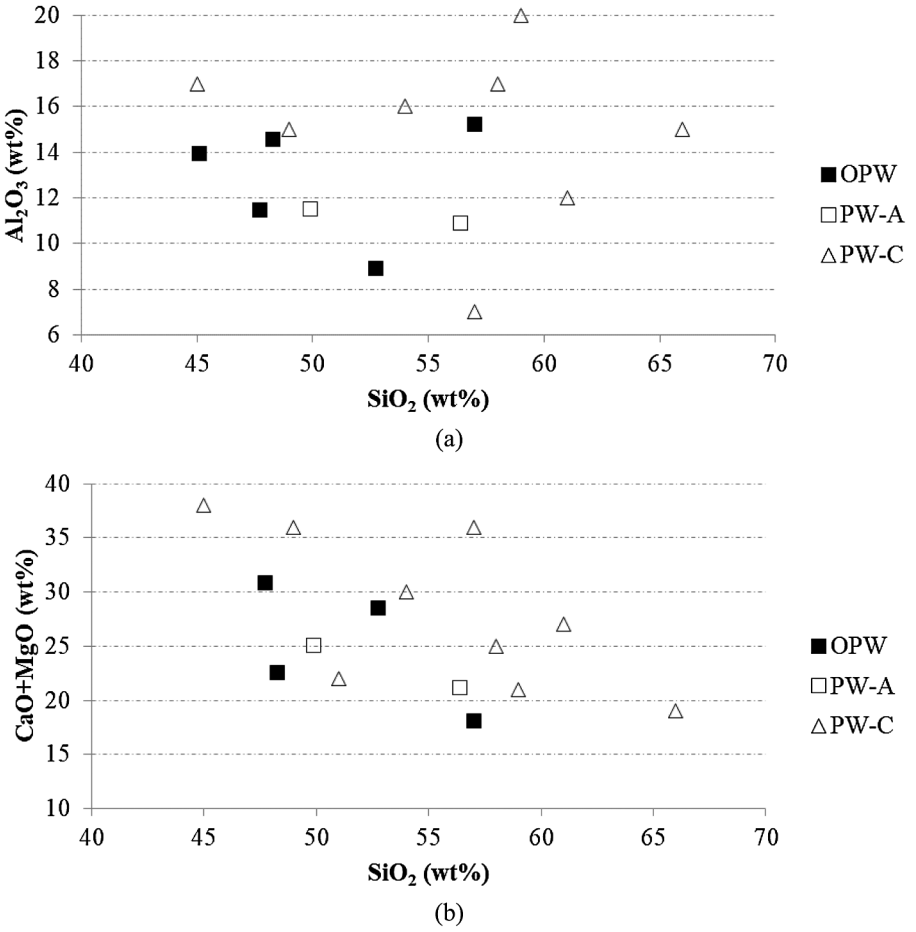


Fig. 8. Oxide compositions of OPW, PW-A and PW-C with respect to SiO_2 content; (a) Al_2O and (b) CaO + MgO.

Table 4
The compositions of slip (wt%).

Sherd	Sample	SiO_2	Al_2O_3	PbO	Na_2O	K_2O	CaO	MgO	Fe_2O_3	TiO_2
ZW	OZW-1	64,49	11,01	10,66	0,34	6,46	3,41	2,06	1,23	0,35
	OZW-2	63,79	9,41	17,14	0,33	6,49	1,03	0,63	0,99	0,19
	OZW-3	51,95	16,38	15,58	0,36	4,70	1,26	1,45	1,54	6,78
	OZW-4	64,33	9,73	18,27	0,47	4,58	0,34	0,85	0,69	0,75
PSSW	OPW-1	58,17	9,97	24,71	0,38	3,23	1,97	0,88	0,51	0,20
	OPW-2	49,93	8,73	11,10	0,62	1,70	6,35	15,19	5,92	0,46
	OPW-3	44,45	15,33	14,49	0,40	1,77	11,64	5,27	5,96	0,70
	OPW-4	60,77	13,37	18,30	0,43	3,66	0,88	1,09	0,91	0,59
	OPW-5	49,93	8,73	11,10	0,62	1,70	6,35	15,19	5,92	0,46

considered and results showed that (i) two ZW sherds (OZW-3 and OZW-4) and two PSSW sherds (OPW-1 and OPW-2) are within the same group having high PbO content (50–70 wt%), low alkali oxides (< 1 wt %) and SiO_2 about 30 wt%, (ii) OPW-3, OPW-4 and OPW-5 within the PSSW had low level PbO content (20–50 wt%), high level alkali oxides (> 2 wt%) and high SiO_2 (40–50 wt%) and (iii) although PbO content is within the range of low level (40–50 wt%), both OZW-1 and OZW-2 have high level of alkali oxides (> 2 wt%) and high SiO_2 (40–45 wt%). These results show that the samples, despite belonging to different ceramic traditions, have similar glaze composition. However, some variations exist, in which the OZW sherds can be grouped based on their glaze composition and thickness, the ones with high PbO content have a thin glaze, whereas similar grouping cannot be easily made for the OPW sherds based on their glaze thicknesses.

SEM-EDX analysis was also carried out for clay materials and the obtained data showed that when the major components like SiO_2, Al_2O_3, Fe_2O_3 and alkali oxides are considered, it is difficult to classify the studied sherds. However, both MgO and CaO content could be a helpful feature in order to classify the sherds. Indeed, OPW-2 as a PSSW sherd contains the highest MgO content and the lowest CaO content,

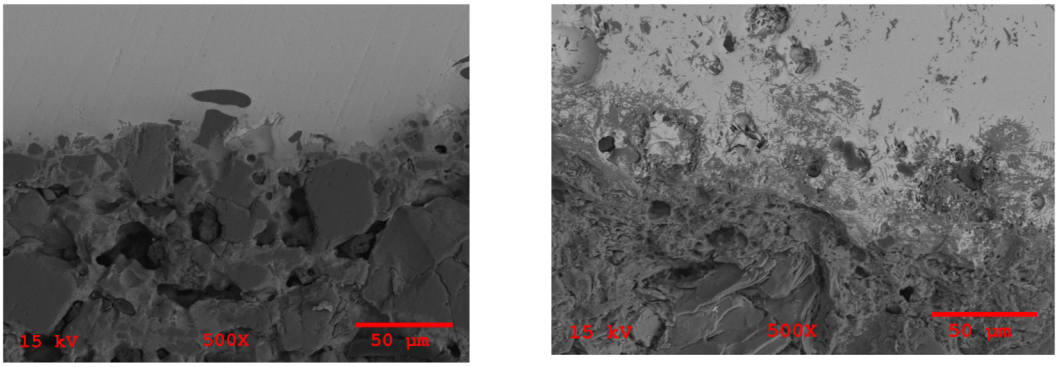


Fig. 9. BSE-SEM images showing the gas bubbles and crystals within both glaze and interface layers; (a) OZW-4 and (b) OPW-3.

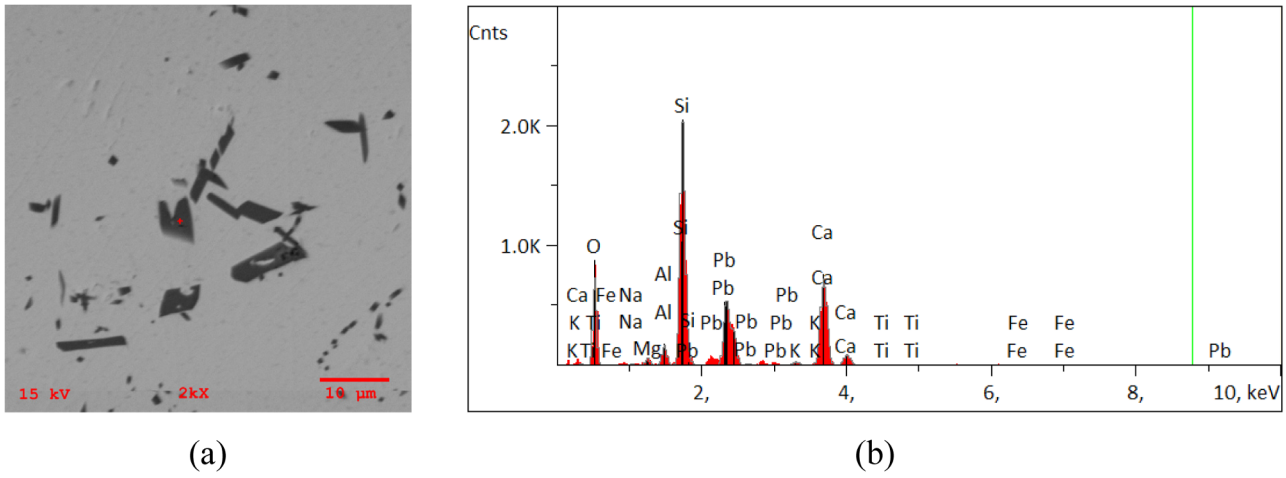


Fig. 10. (a) BSE/SEM image, (b) EDX data of OPW-3.

Table 5
The compositions of glaze (wt%).

Sherd	Observed color	Sample	SiO_2	Al_2O_3	PbO	Na_2O	K_2O	CaO	MgO	Fe_2O_3	TiO_2	CuO
ZW	Pale green	OZW-1	40,86	4,28	48,50	0,26	1,90	2,32	0,77	0,69	0,42	–
	Yellow	OZW-2	45,24	4,20	42,95	0,23	1,85	3,37	0,82	0,61	0,73	–
	Green	OZW-3	27,36	2,18	68,07	0,16	0,27	0,52	0,51	0,59	0,33	–
	Green	OZW-4	31,38	2,55	62,50	0,36	0,39	0,57	0,65	1,15	0,45	–
PSSW	Colorless transparent	OPW-1	29,38	1,64	66,14	0,23	0,29	0,92	0,53	0,49	0,39	–
	Colorless transparent	OPW-2	30,77	1,86	63,11	0,24	0,26	0,92	0,81	1,61	0,43	–
	Green	OPW-3	50,52	8,11	27,23	0,63	1,04	6,72	1,35	1,95	0,69	1,75
	Colorless transparent	OPW-4	37,40	5,03	47,70	0,29	0,92	2,90	0,83	4,45	0,48	–
	Green	OPW-5	48,91	4,57	37,82	0,42	1,87	4,00	1,30	0,75	0,34	–

which is a significant feature to distinguish the clay of ceramic sherds from the others. This could be attributed to the variation of minerals within the clay that might have been obtained from a different clay source. This feature denotes that OPW-2 sherd must have been produced in a different workshop other than the rest. Another distinct feature can be SiO_2 content within the clay composition, thus OPW-3 (PSSW) having the highest SiO_2 content and OZW-3 (ZW) having the lowest SiO_2 content among the studied clays denote that they might be also from a different workshop than the others. In addition to the clay and glaze analysis, the slip composition and thickness were also studied, it was observed that the composition includes the major oxides of clay, SiO_2, and Al_2O_3, as well as, the major oxide of the glaze, PbO.

These results indicate that among the medieval ceramic bulk of East Plain Cilicia, local productions and import ware coexisted, since Plain Cilicia is the junction of military and trade routes, various cultures coexisted, as well. Port Saint Symeon as the genuine production center of PSSW is geographically close to the East Plain Cilicia, thus present work considered it as local production compared to ZW, of which yet no local workshops are discovered.

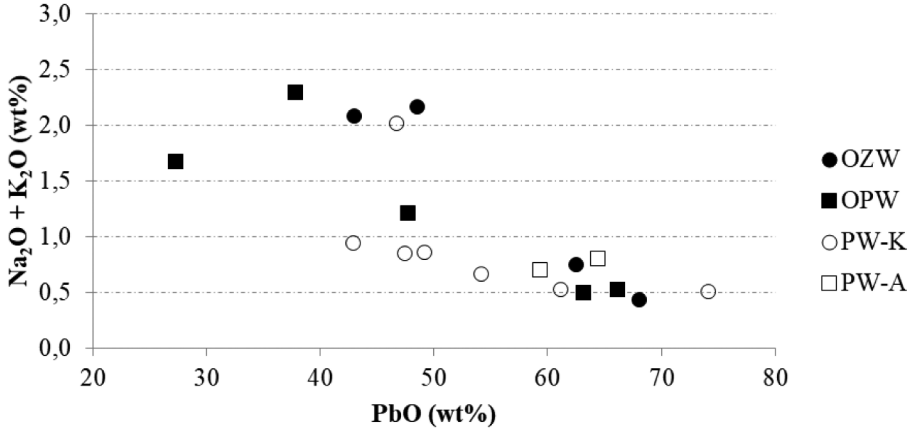


Fig. 11. The plot of PbO versus alkali oxides in the sherds.

Acknowledgements

Sample ceramics that are the subject of present study are obtained during the Osmaniye Archaeological Survey between the years 2005 and 2014, which were conducted under the permission letter issued by the General Directorate of Cultural Heritage and Museums of Turkish Republic. The authors acknowledge the financial supports granted by the Scientific Research Projects Coordination Unit of Kocaeli University under the projects no BAP2008/18 and BAP2012/73, the Governorship of Osmaniye Province and General Director Özgür AKIN, PhD. of BERİCAP A.Ş., Kocaeli.

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