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An investigation of Hellenistic period pottery production technology from Harabebezikan/Turkey

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Abstract

Upper ancient Mesopotamia is a part of Southeastern Anatolia and irrigated by the Euphrates and Tigris rivers. It has several sites, ruins and monuments belonging to ancient civilizations. Among these, an ancient site namely Harabebezikan, is on the east bank of Euphrates (Fırat) river and located 17 km south of Birecik in Şanlıurfa/Turkey. It is left under water by the reservoir lake of Karkamış (Carchemish) dam today. An archaeological salvage project was carried out in 1999 shortly before the reservoir filling. One of the most important results of the excavations was to discover a ceramic workshop of the Hellenistic period. In this study, some selected Hellenistic period potsherds were studied in order to investigate the technological parameters of pottery production such as raw materials used in manufacturing, firing temperatures and conditions. The selected potsherds are probably production of this workshop. Wavelength dispersive X-ray fluorescence (WDXRF) and X-ray diffraction (XRD) analysis were performed for chemical and phase analysis, respectively. Scanning electron microscopy (SEM) and energy dispersive X-ray spectrometry (EDX) were further performed for microstructural and microchemical characterization. Micro-Raman analysis was performed for assessing the mineralogical components of the slip layers. Finally, thermal behavior of the potsherds was characterized by thermal gravimetric-differential thermal analysis (TG-DTA) techniques. The obtained results showed that calcareous materials including clays were used for pottery production and the potsherds were fired in the temperatures from 600 to 1000 °C. Furthermore, iron rich slip layers in different colors suggest that Hellenistic period potters at Harabebezikan were aware of adjusting the redox conditions of firing to obtain the desired color.

Keywords: Archaeometrical characterization; Hellenistic ceramics; Harabebezikan (Turkey)

1. Introduction

Euphrates (Fırat) and Tigris (Dicle) Rivers are still the main source of life as they had been in the past for Mesopotamia and southeastern Anatolia where the most important advances of the history of civilizations were made. On the other hand, the dams which are constructed over the rivers because of the technological, industrial and agricultural needs of our times submerge a number of archeological sites in an irrecoverable way. One of the many sites lost in the last fifties is Harabebezikan mound in the influence zone of Karkamış Dam, last ring of the chain of dams on the Euphrates River (Fig. 1). The most important result of the short-term salvage excavations held in the site was the discovery of a ceramic workshop dating to Hellenistic Period. Clay storage and levigation pits, slags, lots of faulty produced ceramics were found in workshop area, but kiln was not recovered. It was understood that the main production of the workshop, probably active between the beginning and middle of the second century B.C., was narrow necked amphorae without handle. It seems possible that the workshop also produced jugs and bowls with incurved rim, called as ‘‘fish plate’’ [1].

Archaeometric studies cover wide varieties of characterization techniques related with the archeological/historical matter. Amongst those, X-ray based techniques such as X-ray diffraction (XRD) and X-ray fluorescence (XRF), Fourier transform infrared (FTIR) and Raman spectroscopy, scanning electron microscopy (SEM) with energy or wavelength dispersive X-ray spectrometry (EDX/WDX) are conventional characterization techniques to enlighten constitutional characteristics of ancient ceramics [2–6]. This study focuses on

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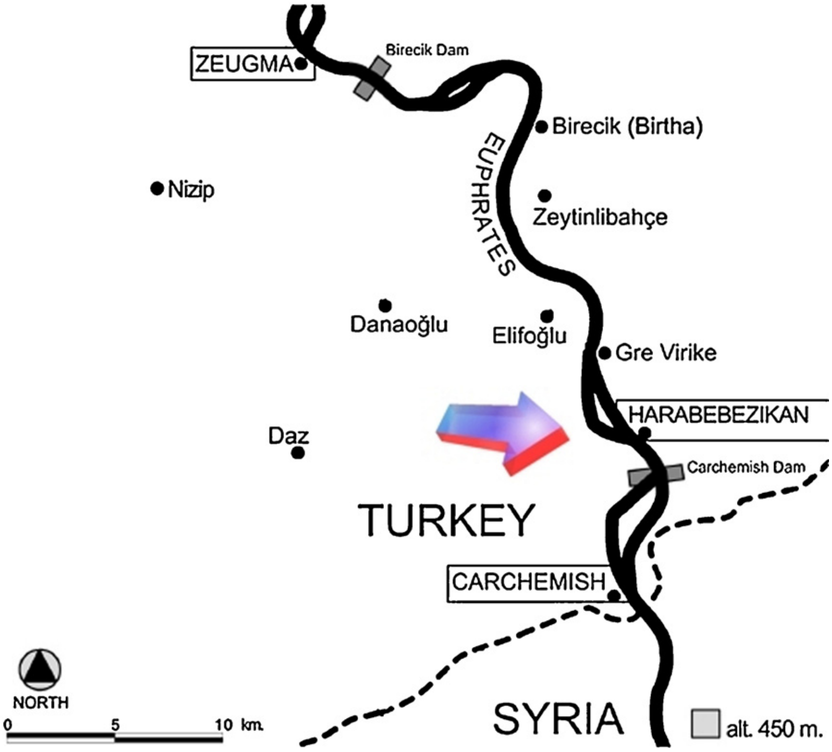


Fig. 1. Map showing the location of Harabebezikan with important ancient sites of southeastern Turkey.

enlightening of the technological parameters of pottery production such as raw materials used in manufacturing, firing temperatures and conditions for the selected Hellenistic potsherds of Harabebezikan. Wavelength dispersive X-ray fluorescence (WDXRF) and X-ray diffraction (XRD) analysis were performed for chemical and mineralogical/phase contents, respectively. Scanning electron microscopy (SEM) in combination with energy dispersive X-ray spectrometry (EDX) was further performed for microstructural and microchemical characterization. Micro-Raman analysis was performed for assessing the mineralogical components of the slip layers. Thermal behavior of the potsherds was characterized by thermal gravimetric-differential thermal analysis (TG-DTA) techniques in order to estimate the firing temperatures.

2. Materials and Methods

Representative images of selected Hellenistic potsherds investigated in the study are given in Fig. 2. Potsherd types are the bottom and the rim of the wares. Body layers have buff color with fine and medium grains in the texture. Almost all the slip layers have been nearly worn-out. They are in reddish, light brown and black color.

Fine powders were prepared in an agate mortar in order to be analyzed by wavelength dispersive X-ray fluorescence (WDXRF) and X-ray diffraction (XRD) techniques. Rigaku ZSX primus instrument was used for chemical analysis of major and minor elements. Glass tablets were prepared by fluxing for the measurement with a ratio of 1:10 powder:-$Li_2B_4O_7$ in weight. Rigaku Rint 2200 powder diffractometer with Cu Kα radiation was used for the mineralogical analysis. XRD patterns were obtained by scanning 5° to 55° 2θ, with a goniometer speed of 2 °/min, operating at 40 kV and 30 mA. Zeiss Evo 50EP SEM attached with Oxford instruments EDX was used for the characterization of vacuum impregnated bulk samples of the potsherds. The samples were polished and coated with Au/Pd target material to obtain flat and conductive surfaces prior to SEM investigation. The Raman spectra were recorded in situ using Horiba Jobin-Yvon, LabRam 300 micro-Raman spectrometer, equipped with double-frequency Nd:YAG laser line operating at 632.81 nm with the excitation

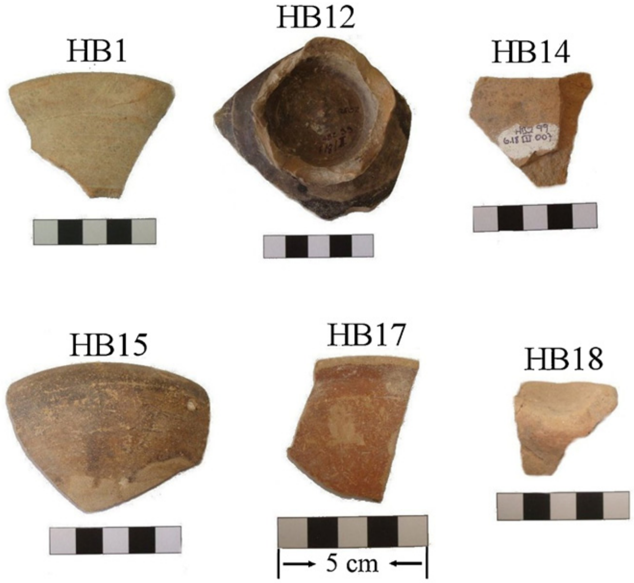


Fig. 2. Representative images of some selected potsherds.

power of 6.7 mW at the sample. An Olympus MPlan microscope with magnification of the objective x50 was used to focus the laser. The backscattered light was detected on a multi-channel air-cooled CCD detector. The exposure time was between 10 and 60 seconds with 5–10 scans. The instrumental resolution was around 2 cm^{-1}. LabSpec package was used for spectra acquisition and Grams 32 for spectra manipulation. All the spectra were baseline corrected with the LabSpec software in order to remove background fluorescence. Thermal analyses (TG-DTA) were performed with the powders of the potsherds from room temperature to 1200 °C in oxidative atmosphere. The firing temperature was increased with a slope of 10 °C/min. Netzsch 409 Lux instrument was used for the analyses.

3. Results

3.1. Chemical and Mineralogical Analysis

Chemical composition of a potsherd has a unique importance in terms of assessing the raw materials used for production. The relations between the elements are also important for the indication of mineralogical assemblages in the raw materials. XRF results showed that all of the samples belonging to body of the potsherds have high amounts of CaO. CaO quantities change between from 9.92 to 25.43 wt. %. Some results of the samples representing lower and upper limits of CaO are given in Table 1. CaO has a drastic change but MgO quantities are in moderate range of 4.93-5.95 wt. %. It may be considered that the source of CaO is not only from dolomitic materials but predominantly from the calcareous materials. Harabebezikan is surrounded with rich calcareous raw material deposits which are still used for mining today [7,8]. Therefore, it is not surprising that the clays used for production contain high amount of CaO. Na_2O and K_2O quantities are also in moderate range of 1.20-2.38 and 2.15-3.84 wt. % such as typical clay contents, respectively. Na_2O and K_2O should have been supplied principally from the feldspathic contents (K-feldspars and plagioclases) and clay minerals (illite or muscovite). The iron oxides were in similar quantities in the sources of clay and calcareous material of the used deposits.

Table 1
WDXRF results of the samples designated as HB12, HB14, HB15 and HB18.

	Sample code			
Oxide, wt. %	HB12	HB15	HB14	HB18
SiO_2	56.12	53.65	44.99	44.66
Al_2O_3	14.98	13.97	11.78	10.49
Fe_2O_3	7.39	8.52	7.03	7.19
MgO	4.93	5.95	5.43	5.17
Na_2O	1.20	2.38	1.74	1.58
K_2O	3.84	2.15	2.63	3.28
CaO	9.92	11.70	24.22	25.43
TiO_2	0.87	1.02	0.86	0.92
P_2O_5	0.25	0.21	0.25	0.43
SO_3	0.10	0.04	0.29	0.39
Cl	n.d.	n.d.	n.d.	0.04
Cr_2O_3	n.d.	0.11	0.13	0.10
MnO	0.19	0.15	0.13	0.15
NiO	0.04	0.04	0.05	0.04
CuO	0.03	0.02	0.02	n.d.
ZnO	0.06	0.01	0.02	0.02
Rb_2O	n.d.	n.d.	0.05	n.d.
SrO	0.04	0.04	0.07	0.08
ZrO_2	0.03	0.02	0.02	0.02
BaO	n.d.	n.d.	0.29	n.d.

n.d.: not detected or below the detection limits.

XRD results showed different mineralogical and phase contents. Quartz (SiO_2), clay minerals: illite/muscovite $[(K,H_3O)Al_2Si_3AlO_{10}(OH)_2]/[(K,Na)(Al,Mg,Fe)_2(Si_{3.1}Al_{0.9})O_{10}(OH)_2]$, feldspars: [K-feldspar ($KAlSi_3O_8$), and plagioclase $[(Na,Ca)AlSi_3O_8]$], carbonated minerals: [calcite ($CaCO_3$) and dolomite$[(CaMg(CO_3)_2]$, pyroxenes: diopside

Table 2
XRD results and estimated firing temperatures of the potsherds.

Sample	Minerals/Phases	Estimated firing temperature (°C)
HB1	Quartz, calcite, K-feldspar, plagioclase, illite/muscovite, magnesiohornblende, hematite	600-700
HB2	Quartz, calcite, plagioclase, diopside/augite, gehlenite, hematite	900-1000
HB3	Quartz, calcite, plagioclase, gehlenite, illite/muscovite, magnesiohornblende, maghemite, hematite	850-900
HB4	Quartz, calcite, K-feldspar, plagioclase, illite/muscovite, magnesiohornblende, hematite	600-700
HB5	Quartz, calcite, plagioclase, diopside/augite, gehlenite, illite/muscovite, magnesiohornblende, hematite	850-900
HB6	Quartz, calcite, K-feldspar, plagioclase, diopside/augite, gehlenite, illite/muscovite, crossite, hematite	850-900
HB7	Quartz, calcite, plagioclase, diopside/augite, gehlenite, illite/muscovite, crossite, hematite	850-900
HB8	Quartz, plagioclase, diopside/augite, hematite	900-1000
HB9	Quartz, calcite, plagioclase, diopside/augite, gehlenite, hematite	900-1000
HB10	Quartz, calcite, dolomite, plagioclase, diopside/augite, gehlenite, illite/muscovite, crossite, hematite	800-850
HB11	Quartz, calcite, plagioclase, diopside/augite, gehlenite, illite/muscovite, magnesiohornblende, maghemite, hematite	850-900
HB12	Quartz, K-feldspar, plagioclase, diopside/augite, gehlenite, maghemite, hematite	900-1000
HB13	Quartz, calcite, plagioclase, illite/muscovite, magnesiohornblende, hematite	600-700
HB14	Quartz, calcite, plagioclase, illite/muscovite, magnesiohornblende, hematite	600-700
HB15	Quartz, plagioclase, diopside/augite, gehlenite, hematite	900-1000
HB16	Quartz, calcite, plagioclase, gehlenite, illite/muscovite, crossite, maghemite, hematite	850-900
HB17	Quartz, calcite, plagioclase, gehlenite, illite/muscovite, crossite, maghemite, hematite	850-900
HB18	Quartz, calcite, K-feldspar, plagioclase, illite/muscovite, crossite, hematite	600-700
HB19	Quartz, calcite, K-feldspar, plagioclase, ilite/muscovite, crossite, maghemite, hematite	600-700

$[Ca(Mg,Al)\ (Si,Al)_2O_6]$ and augite $[Ca(Mg,Fe)Si_2O_6]$, a melilite: [gehlenite $(Ca_2Al_2SiO_7)$], calcic and sodic amphiboles: magnesiohornblende $[(Ca,Na)_{2.26}(Mg,Fe,Al)_{5.15}(Si,Al)_8O_{22}(OH)_2]$ and crossite $[(Na,Ca)_2(Fe,Mg,Al)_5(Si,Al)_8O_{22}(OH)_2]$, iron minerals: hematite (α-Fe_2O_3) and maghemite (γ-Fe_2O_3) were identified.

The present mineralogical and phase contents enable to estimate firing temperatures. This issue has previously been discussed in several studies. For example, illite/muscovite or mica structure breaks down in the range of 900-1000 °C [9–12]. If the firing temperature does not exceed this range, these minerals still continue to exist as the components of the potsherds. For further stages of firing temperature, from the destruction of illite, an intermediate phase between spinel ($MgO.Al_2O_3$) and hercynite ($FeO.Al_2O_3$) originates [13]. Calcite decomposition into CaO and CO_2 begins around 650 °C and it is completed up to temperature of 900 °C. Dolomite has the decomposition with two stages beginning at around 650 °C. After the decomposition of dolomite into calcite and magnesia, decarbonation of calcite again continues up to 900 °C [9,11,14]. Trindade et al. concluded that the decarbonation of calcite may extend to 1100 °C for calcite rich systems [15]. However, secondary calcite may occur in ceramics as a result of post-burial deposition processes due to recarbonation of lime [9]. Quartz and feldspars can persist up to 1000 °C [16]. Pyroxenes like diopside and augite may be generated from dolomite and silica reactions at 800-900 °C. Gehlenite may be formed at 850 °C with the reaction of CaO and illite structure [17]. Moreover, these neo-mineral formation temperatures may be affected by firing type such as pit or kiln firing. For instance, calcite decomposition ends around 825 °C in kiln firing but it tends to 875 °C in pit firing conditions [18]. These reactions, of course, are related with peak firing temperature, soaking time, abundance and the type of minerals/phases present, firing atmosphere, pressure, specific surface area of components, etc. Mineralogical/phase contents and estimated firing temperatures of the potsherds are given in Table 2.

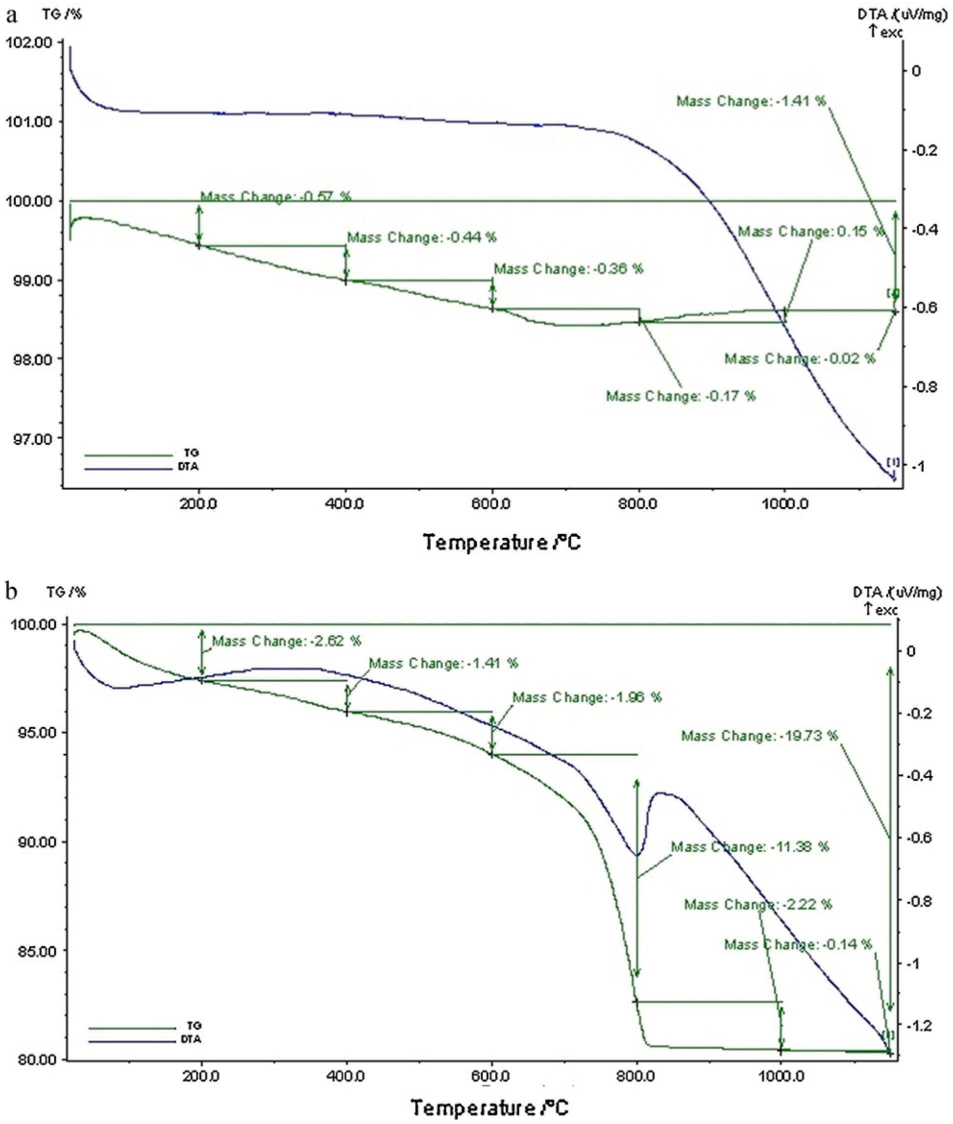


Fig. 3. TG-DTA curves of the sample (a) HB8 and (b) HB18.

The samples of HB2, HB8, HB9, HB12 and HB15 do not include clay minerals. The samples of HB8, HB12 and HB15 do not include calcite. Therefore, they can be considered as the potsherds which were fired in the highest temperature around 1000 °C. The firing temperature for the samples including both clay minerals and calcite such as samples of HB1, HB13 and HB18 should not exceed 700 °C. Representative TG-DTA curves of the samples HB8 and HB18 are given in Fig. 3. Corresponding endothermic peak around 800 °C indicates the decarbonation of the sample HB18.

Iron minerals found in the samples may help to assess the firing atmosphere [19,20]. All the samples include hematite. It may be estimated that firing cycle may be ended in oxidative condition. Besides, maghemite in the samples of HB3, HB11, HB17 and HB19 may be an indication of reducing atmosphere. Probably, these potsherds should have been exposed to both oxidative and reducing atmosphere during firing. Although the presence of the maghemite in the raw materials cannot be excluded, since its existence in the natural raw materials is scanty, most probably it was obtained during firing [21]. It is also known that monochrome and bichrome coatings were produced through a single firing cycle consisting of a sequence of oxidizing and reducing steps obtained by varying the firing atmosphere for ancient Greek ceramic wares [20,22]. Considering the results, it may be thought that similar procedure for firing should have been performed for these wares.

Clay deposits may include some non-plastic materials such as quartz, feldspars and iron compounds. Non-plastic materials may be natural compounds of the clays or they may be used as additional materials. The addition of non-plastic (temper) materials such as siliceous raw materials, shells or crushed potsherds may be seen in several artifacts [16]. Temper materials may improve the mechanical properties of the pots during both shaping and firing processes. Especially, calcareous materials may contribute to preserve the desired shape after forming [23]. Since all the samples are rich in calcareous materials, potters should have not needed to add any kind of temper materials.

3.2. Microstructural Analysis

Representative BSE images of the lowest and the highest CaO containing samples HB12 and HB18 were given in Fig. 4 a and b, respectively.

The size of grains occasionally may reach to 500 μm. BSE images of the samples conclude that the microstructure of the potsherds include different sized particles, mainly quartz, calcite and feldspars (Fig. 5).

These fine and medium grained microstructure of the potsherds have different characteristics related to the firing temperature. With increasing temperatures some interactions occur in the structure of clay based systems. Some of these interactions are welding between clay matrix and mineral grains, shape changes of mineral phases, an increase of the aggregation rate within the clay matrix with the formation of secondary porosity and intergranular bridges [24]. Especially welding behavior and intergranular bridges from the representative BSE images given in Fig. 6 for the potsherds of HB12 and HB15 may easily be distinguished.

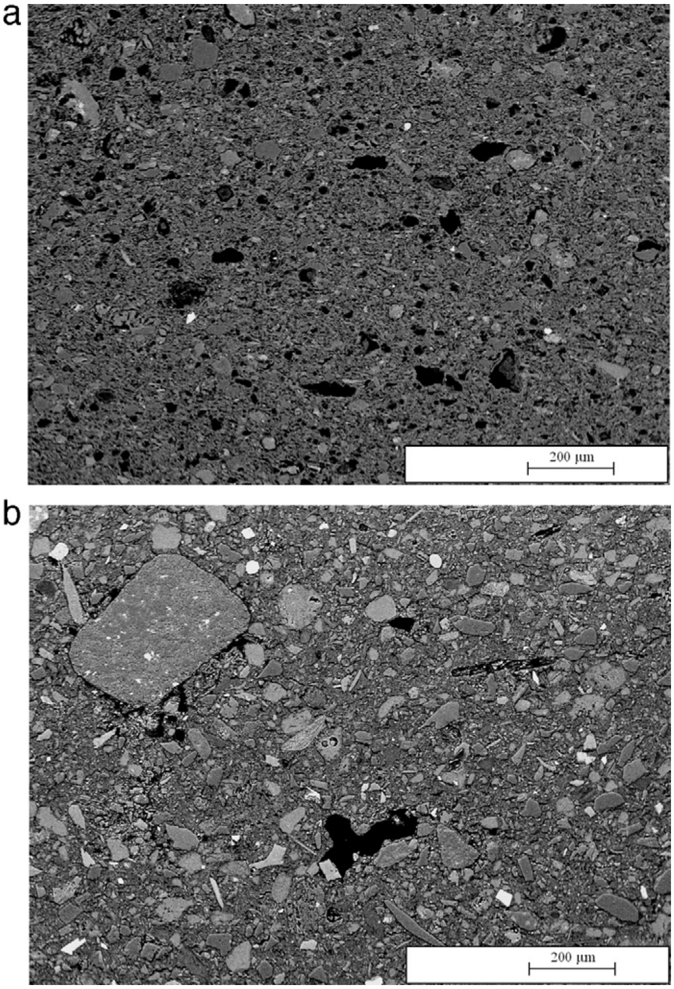


Fig. 4. Representative BSE images of the lowest and the highest CaO containing samples HB12 and (b) HB18.

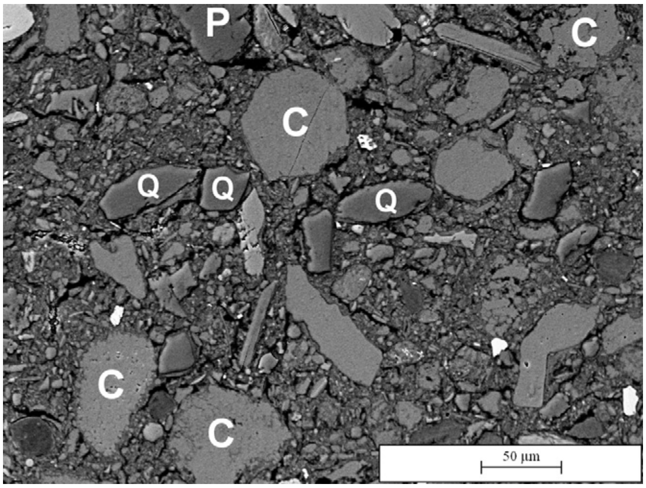


Fig. 5. Representative BSE image of sample HB18 (Q: quartz, C: calcite, P: plagioclase).

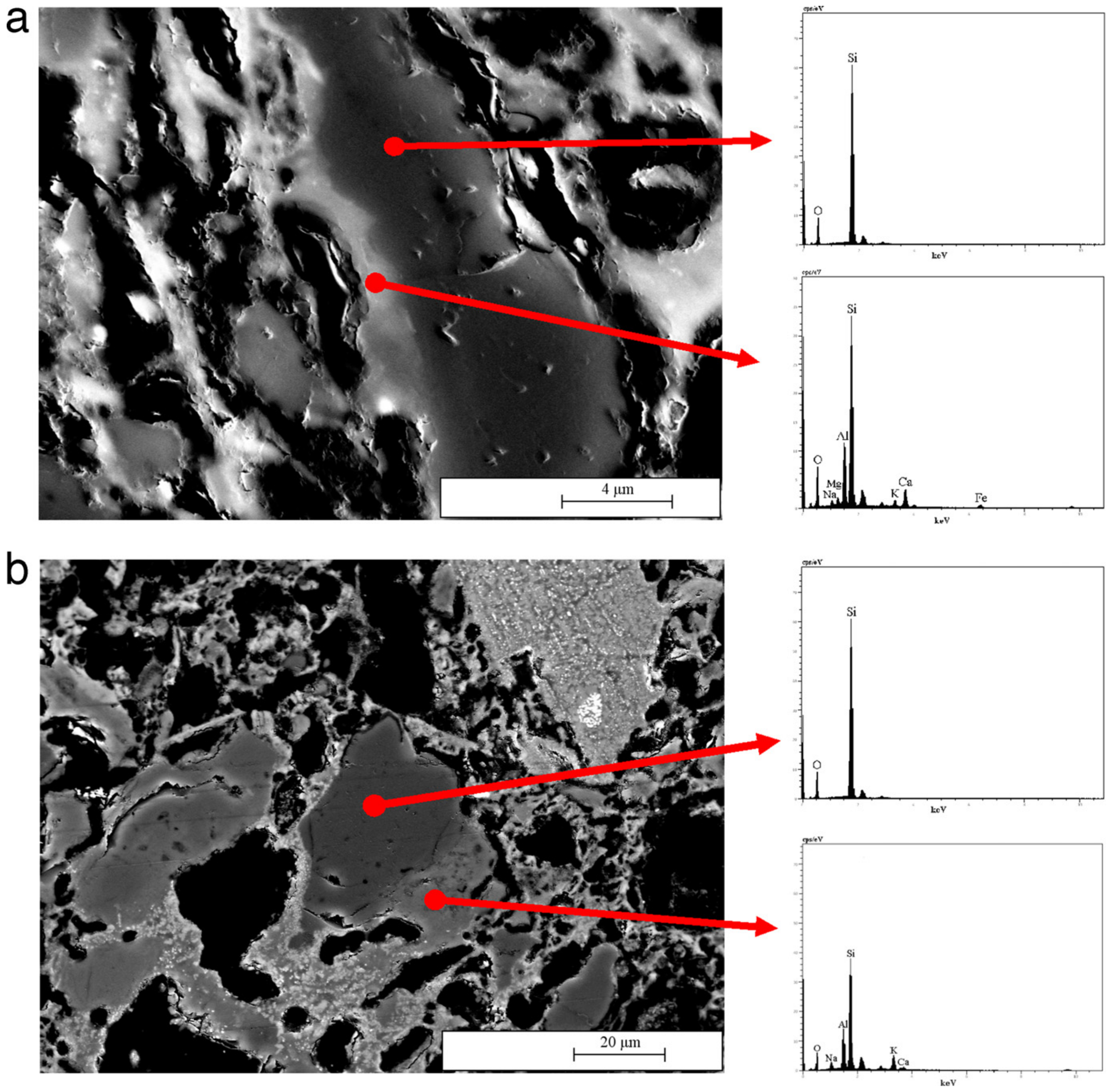
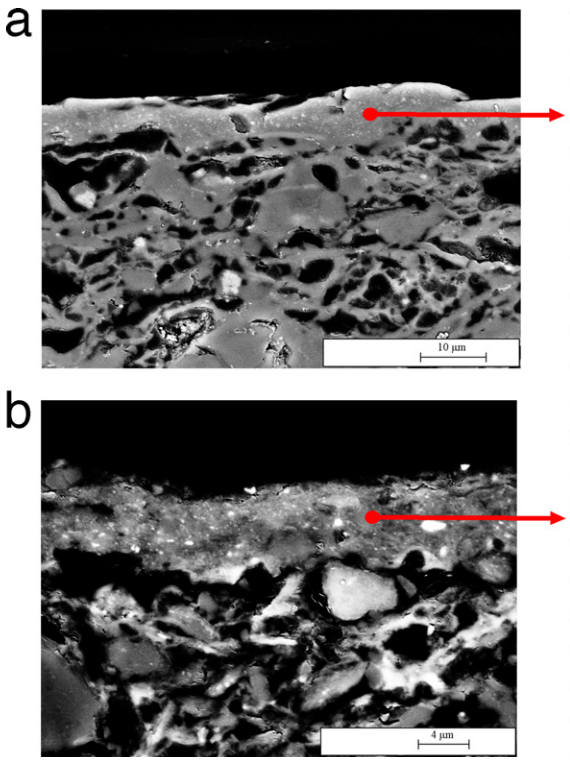


Fig. 6. Representative BSE images showing welding behavior and intergranular bridges from the potsherds (a) HB12 and (b) HB15.

Most of the potsherds have eroded slip layers or decorations. The thickness of the slip layers change from 5 to 20 µm. BSE images and semi-quantitative EDX analysis of the slip layers of HB12 (black in color) and HB17 (reddish in color) are given in Fig. 7.

Elemental composition of the slip layers are important for the assumption of the raw materials used for production. Slip layers have similar elemental diversity but in different ratios. They are rich in iron. K_2O and CaO ratios are higher than the other alkali and earth alkali elements. It suggests a typical iron and calcium rich clayey batch for the slip preparation. Additional iron or iron rich materials should have been needed for the enrichment of the clayey batch. Some traditional approaches require selecting specific clay; richer in iron oxides of decanted clay or adjustment of composition of a clay with specific compounds [25]. Nodari et al. concluded that total amount of iron in the raw material and redox conditions of firing were generally the main factors determining the color of the potsherds [26]. Difference in mass ratio of the elements present in the slip layers and the redox conditions of firing were the most effective parameters to adjust to obtain the desired color [19,20,27,28]. Hematite and feldspars (albite/microcline) were detected from the micro-Raman spectrum that belongs to the slip layer of HB17 (Fig. 8a) [29,30].

Hematite should be the principal coloring agent for the slip layer. Magnetite and carbon black were also detected from the



HB12

Oxide	wt. %
SiO_2	41.41
Al_2O_3	28.30
Fe_2O_3	16.39
MgO	1.71
Na_2O	1.84
K_2O	5.02
CaO	5.23
TiO_2	0.11

HB17

Oxide	wt. %
SiO_2	48.20
Al_2O_3	27.81
Fe_2O_3	14.05
MgO	2.10
Na_2O	0.91
K_2O	4.32
CaO	1.93
TiO_2	0.67

Fig. 7. Representative BSE images of cross-sections and corresponding semi-quantitative EDX results of the slip layers of (a) HB12 (black in color) and (b) HB17 (reddish in color).

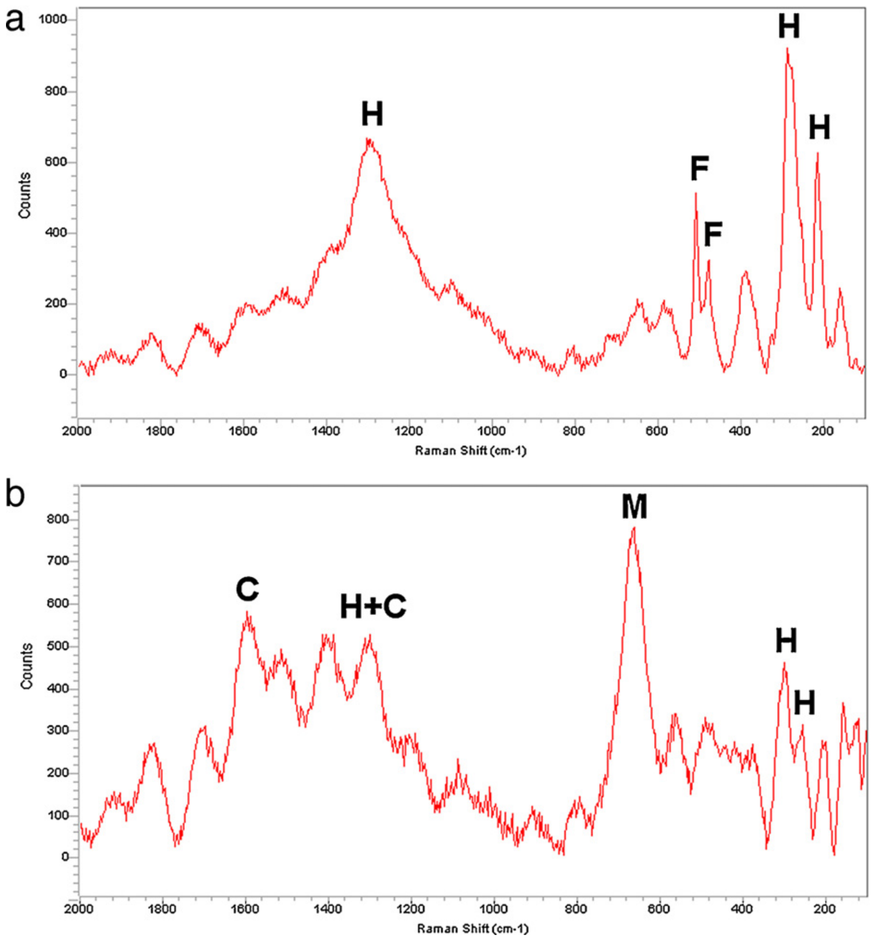


Fig. 8. Representative micro-Raman spectra for (a) HB17 (reddish in color) (b) HB12 (black in color) H: hematite, F: feldspars, M: magnetite, C: carbon black.

Raman spectrum of HB12 slip layer (Fig. 8b) [29,30]. These materials should be the reason of black color for the slip layer. The results of micro-Raman analysis suggest that firing atmosphere was oxidative for HB17 and reductive for HB12. Considering the lots of reddish and darker (brownish and black) colored potsherds, it may be stated that the potters had the experience of obtaining the desired color.

4. Conclusions

The obtained results indicate that the potsherds from Harabebezikan were mainly produced from the clay deposits which are rich in calcareous materials and including two types of sodic and calcic amphiboles; crossite and magnesiohornblende, respectively. The potsherds should have been exposed to different firing temperatures. The mineralogical/phase contents lead to conclude that the range of the firing changes from 600 to 1000 °C. It may be inferred from the estimated firing temperatures that either a uniform flame distribution could have not been set up during firing or individually it could have not been reached the higher temperatures required for pottery production. The slip layers of the potsherds show similar elemental diversity but they are different in colors. The redox conditions of firing should have been adjusted by the potters. Finally, it may be concluded that pottery production technology for Harabebezikan has similar characteristics of the ancient Greek style pottery.

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