Mediterranean Archaeology and Archaeometry, Vol. 14, No 1, pp. 261-271

ARCHAEOMETRIC ANALYSES OF HASANKEYF UNGLAZED CERAMICS

Nurşen Özkul Fındık[1], Ali Akın Akyol[2], Nurşen Sarı[3]

[1]Nurşen Özkul Fındık, Gazi University, Faculty of Art, Department of Art History, TR-06500, Beşevler, Ankara, Turkey
[2]Ali Akın Akyol, Gazi University, Faculty of Fine Arts, Department of Conservation and Restoration of Cultural Properties, Material Research and Conservation Laboratory (MAKLAB), TR-06830, Gölbaşı, Ankara, Turkey
[3]Nurşen Özkul Fındık, Gazi University, Faculty of Sciences, Department of Inorganic Chemistry, TR-06500, Beşevler, Ankara, Turkey

Received: 02/12/2013
Accepted: 07/05/2014
Corresponding author: Nurşen Özkul Fındık (nursenf@gazi.edu.tr)

ABSTRACT

Ceramic and soil/clay samples obtained from three different areas of the Hasankeyf (Batman, Turkey) archaeological site and the near vicinity of river bank Dicle were investigated archaeometrically. The petrographic characteristics of the samples were determined by thin section optical microscopy analysis, furthermore the chemical structures were determined by XRF, SEM-EDS and TGA analyses. According to their matrix/aggregate content, aggregate type/distribution/dimension, porosity, and the properties of clay (matrix) structure of ceramics were classified. The clay types of the samples are mainly illite, smectite and kaolinite. The samples have similarities with the soil of the region and they are also similar to each other in terms of both chemical compositions and petrographic characteristics. According to their Sr and Zr content, the samples must have been manufactured mainly by using local clay with terrigenous origin as raw materials.

KEYWORDS: Hasankeyf, Medieval ceramics, ceramic analysis, petrographic analysis, SEM-EDX, PED-XRF

1. INTRODUCTION

Most of the ceramic objects found in archaeological excavations in Anatolia are made of unglazed ceramics. The excavations which revealed a great number of glazed or unglazed ceramic pieces in addition to architectural ruins have necessitated both archaeological and archaeometric analyses.

Hasankeyf is an important medieval town located in the southeast of Turkey (Fig.1). Its existence is currently threatened by the Ilısu Dam, whose construction is continuing in the region. Excavations for the documentation, conservation and protection of the natural and historical assets in Hasankeyf, most of which will be flooded by the dam when completed, has been going on systematically since 1998. Defining the ceramics in terms of history and region, and determining their mutual or different physical structures will surely provide valuable knowledge about the daily lives and production technologies in the medieval Hasankeyf and its near vicinity bank of river Dicle (ancient named Tigris).

Hasankeyf, which is situated in the area called Al Jazeera, or Mesopotamia, (between the Tigris and the Euphrates rivers), is a medieval town constructed on both banks of the Tigris River and that remains within the borders of the province of Batman in the Southeast Anatolia Region of Turkey today. The town has been a settlement area since prehistoric times. It functioned as a strategically important castle throughout the rule of the Roman Empire (IInd Century, AD). Starting from the VIIth Century AD, the Ayyubids, the Abbasids, the Hamdanis and the Mervanis, all with Muslim conquerors, ruled the area. In 1101 AD, the Hısn-ı Keyfa Artukids State was established by Artukoğlu Sökmen, one of the commanders of the Seljuq Dynasty. After being ruled by the Ayyubids in 1232 AD, and then by the Akkoyunlu State (1461-1482 AD), the region was included in the Ottoman property in 1517 AD.

The excavations and research in the area have been continuing since 2004 within the scope of the Research, Excavation and Rescue Project for the Hasankeyf Historical and Archaeological Protection Area (Uluçam, 2007: 681-710). In excavations carried out at different regions of the town, many architectural works of art, such as mosques, tombs, madrasahs, palaces, houses as well as ceramics, metal objects with gypsum ornaments and coins from the Roman Empire, the Artukids, the Ayyubids, the Akkoyunlus and the Ottoman Empire were obtained. Furthermore, two ceramic ateliers in the region, namely, the Salahiye Gardens (Salahiye Bahçeleri) and the Beach Palace (Sahil Sarayı) in the east of the town, were also found in the excavations (Fig.1). The findings show that ruins from the Roman period are present in the foundations of the Beach Palace, followed by a civilian building used as a palace or a mansion in the Artukids era, and finally an Ottoman cemetery is found in the same area. In the Beach Palace excavations performed between 2008 and 2010, many pieces of kiln materials, broken ceramic pieces, and kiln ruins were revealed. The kilns found in the other atelier in the Salahiye Gardens, which was revealed between 2001 and 2003 in the southwest of Hasankeyf, have an oval or circular plan (Uluçam, 2007: 681-710; Göçmez, et al, 2004: 2407-2410; Çeken, 2007: 469-488). Glazed and/or unglazed ceramics were manufactured in this atelier in the XIVth and XVth centuries AD.

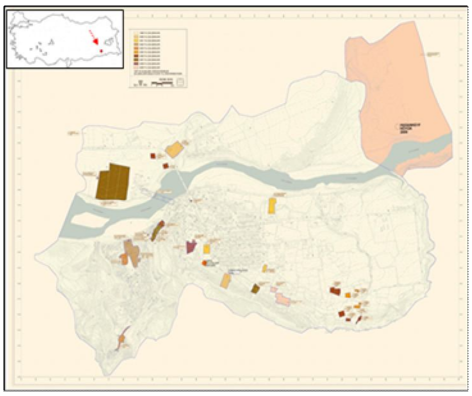

Figure 1. Hasankeyf archaeological area and excavation regions

Most of the unglazed ceramics found in Hasankeyf were produced by using a lathe and some were manufactured by means of mould and press techniques. Earthen pitchers are most intensely found objects in the group, followed by pieces of jugs, large earthen jugs, canteens, mugs, bowls, jars, pots, plates and other pottery. Large earthen containers, which are deep, have semi-spherical bodies and which have narrow or wide mouths, were used for the storage of food such as butter, oil, molasses, jam and cheese. Jugs acquired usually have one handle and a cylindrical neck (Fig.2).

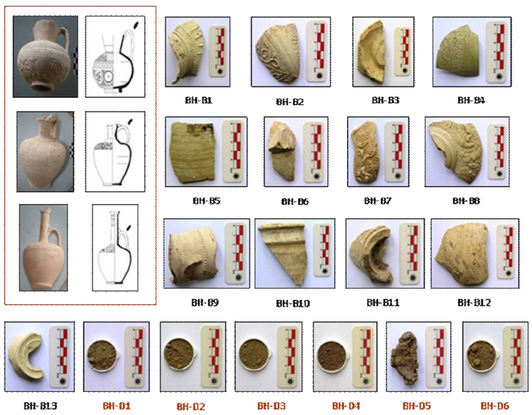

Figure 2. Hasankeyf excavations ceramic and soil/sediment samples.

The body section is commonly frustoconical at the bottom and half spherical at the top. Few half spherical body forms both at the bottom and top were determined (Fındık, 2009: 109-110). The molds used in the production of the containers formed by means of the printing and molding technique were found as whole or nearly-whole pieces. Animal figures and floral and calligraphic compositions consisting of palmet and rumi motifs, which were made by means of the printing and molding technique, are notable. The containers were shaped by means of lathes, and then decorated by different techniques such as scraping, dredge printing, roulettes, etc. Few body pieces involving the barbotine technique were also found.

The purpose of this study is to conduct archaeometric analyses of the ceramics found in the Hasankeyf excavations (Fig.2) and the clay specific to the region in order to determine whether they have been produced locally or not. Thus, the historical and cultural assets of Hasankeyf, most of which will stay under the Ilısu Dam waters when it is completed, will have been documented.

2. MATERIALS AND ANALYSES

Ceramic pieces obtained from different areas of the Hasankeyf archaeological site and soil/clay samples taken from the near vicinity (south hill cult place and the river bed) were investigated archaeometrically. The samples were first evaluated visually and grouped, and accordingly, they were photographed (Canon Digital IXUS 870 IS 10 Mp), documented and coded. The physical properties (their color and thickness) and the dynamic characteristics of the samples were determined by means of an ultrasonic velocity (SV) measurement test (Table 1). The clay color of the ceramic pieces was defined by means of a portable Chroma Meter that involved the ColorQA Pro System III software. Because colors could not be defined exactly as the visible colors were only defined as primary/secondary or light/dark, in determining the colors, various color systems were formed for different areas. CEI L*a*b* (Commission Internationale de L'Eclairage) color system is one of the most used and most detailed standard color systems. The (L), (+a), (-a), (+b) and (-b) values represent the lightness/darkness, the intensity of red, the intensity of green, the intensity of yellow and the intensity of blue, respectively. The dynamic characteristics of materials were determined by using a Matest C372N Model High Performance Ultrasonic Test Instrument. Ultrasonic velocity (SV) measurements were performed on the ceramics (Table 1). The petrographic texture (matrix) and aggregate (rock and mineral content) characteristics of the ceramic and clay samples were determined by thin section optical microscope analysis. To make optical microscope analysis possible, samples were cut by using a proper cutter, placed on a slide, thinned and their thin cross sections were prepared (Whitbread, 1995: 365).

The thin cross sections of the samples were examined by using a LEICA Research Polarizan Microscope DMLP Model optical microscope with light from beneath and above. Photographs were taken by the digital camera Leica DFC280, connected to the microscope with proper magnifying and assessments were made by using the "Leica Qwin Digital Imaging Program". In the samples, the matrix and the clay, rock and minerals that make up the matrix were defined by using the "Point Counting Method" (Table 2a-2c and Fig.3).

X-rays work in the Polarized Energy Dispersive (PED-XRF) system that is used in Fluorescence analysis. In this study, a SPECTRO X-Lab 2000 PEDX model spectrometer was used to determine the chemical compositions of ceramic and clay samples (Table 3 and Fig.4). This spectrometer can analyze elements from sodium (Na) with atom number 11 to uranium (U) with atom number 92, and it can accurately measure heavy elements up to 0.5 ppm and light elements up to 10 ppm (Shackley, 2011, 7-44; 127).

To determine thermal deterioration and mass losses of the samples, their thermal changes were investigated by using a Setaram model Simultaneous TG/DTA device at an interval of 25°C - 900°C, at 10°C/minute heating speed and $N_{2(g)}$ atmosphere (Table 3 and Fig.5). Furthermore, surface morphologies and element analyses were determined by means of a SEM-EDX analysis by using a QUNTA 400F Frield Emission SEM device (Fig.6).

3. RESULTS OF ANALYSES & DISCUSSION

In the ceramic samples, the (L) color code values range between 43.33 and 73.45; the (a) color code values range between -8.62 and 11.73; and the (b) color code values range between 20.90 and 43.86. In the clay samples, the (L) color code values range between 5.68 and 12.52; the (a) color code values range between 6.07 and 8.90; and the (b) color code values range between 5.38 and 13.74 (Table 1).

The body thickness of the ceramic pieces that make up the sampling is between 2.67 and 7.18 mm (mean 5.10) (Table 1). The SV values of the ceramic samples vary between 5.31 and 12.43 km/s (mean 7.70 km/s) (Table 1). Factors such as deterioration, disintegration and porous/cavernous structure are important parameters that affect the SV value. Higher SV values indicate that ceramics have a more durable and more homogenous petrographic structure.

The petrographic texture and aggregate characteristics of the Hasankeyf ceramic samples – i.e. clay matrix type, matrix aggregate content, aggregate type, aggregate distribution, and aggregate dimensions – were determined by thin cross section optical microscope analysis (Table 2a-2c and Fig.3). The samples that have been investigated petrographically were classified into 7 different groups considering their matrix (clay) structure, aggregate type and distribution, firing temperature and porosity. Accordingly, it was determined that the samples that were taken from different areas of the Hasankeyf archaeological site were produced by using different construction technologies (Table 2a-2c). The firing temperature of the ceramic samples from different areas of the Hasankeyf archaeological site was estimated to be between 850°C - 950°C when assessed in terms of their matrix limestone/calcitecontent,deterioration/vitrification of the clay structure, matrix porosity ratio and aggregate type phase changes. During firing at and above 900°C, the clay structure of ceramics deteriorate and vitrification starts (Table 2a and Fig.3). Vitrification is also supported by SEM photographs (Fig.6).Moreover, the carbonate, calcite and limestone in the structure are indicators of firing temperature (Rice, 1987, 54) (Table2a, Fig.4).

Table 1.Hasankeyf Excavation ceramic and soil samples descriptions and physical properties.

Sample Codes	Descriptions	Material Type	Thickness (mm)	Colour Codes		
BH-B1	Neck part of printed patterned jug	Ceramic	2,98 (4,79)*	**L**	**a**	**b**
BH-B2	Body of printed patterned jug	Ceramic	4,50 (5,15)	65,81	-8,62	28,12
BH-B3	Bottom part of wheel-made jug	Ceramic	6,16 (14,65)	57,83	-1,31	31,17
BH-B4	Neck part of jug	Ceramic	2,67 (3,80)	43,33	8,68	29,12
BH-B5	Body part of jug	Ceramic	6,06	61,56	-5,75	33,79
BH-B6	Fragment of handled jug	Ceramic	3,61 (14,83)	51,97	-4,25	20,90
BH-B7	Body part of animal patterned jug	Ceramic	4,02	64,35	-2,04	42,18
BH-B8	Shoulder pice of animal fight scene patterned jug	Ceramic	6,14 (10,69)	60,47	3,37	34,80
BH-B9	Neck part of handled jug	Ceramic	5,04 (6,70)	64,18	-2,10	30,95
BH-B10	Neck part of wide mouthed pottery or jug	Ceramic	6,60	46,74	7,49	30,69
BH-B11	Neck part of jug (from ceramic kiln)	Ceramic	7,18 (9,52)	55,23	8,74	43,86
BH-B12	Body part of wave shape printed patterned jug (from ceramic kiln)	Ceramic	6,70	50,54	3,09	31,04
BH-B13	Bottom part of jug (from ceramic kiln)	Ceramic	4,68 (7,61)	40,92	11,73	39,37
BH-D1	Ankenki (water reservoir) soil deposit	Soil/Clay		73,45	-2,37	30,15
BH-D2	Hevsel region river bank	Soil/Clay		8,70	7,00	9,89
BH-D3	Cami-u Mardinike region river bank	Soil/Clay		8,07	6,61	8,93
BH-D4	Hevsel region river bank	Soil/Clay		11,23	8,55	13,65
BH-D5	Hevsel region river bank	Soil/Clay		5,68	8,90	5,38
BH-D6	Zeynel Bey region river bank	Soil/Clay		12,52	6,07	13,74

Sample Codes: BH (= **B**atman **H**asankeyf Excavation); **BH-B1**; ceramic sample number 1
(*) Maximum thickness of amorphous sample

Table 2a. Petrographical thin section optical microscopy analysis of ceramic samples

Ceramic Samples	T (°C)	P (%)	MA (%)	Clay Type	Rocks & Minerals*
BH-B1	~950	4	13	illite+smectite	Q,Pl,Ç,By,Op
BH-B2, BH-B3	850-900	4	14	illite	Q,Pl,Op,A
BH-B4	~950	1	2,5	kaolinite	Q,By,Op,Ç
BH-B5, BH-B6, BH-B7, BH-B10, BH-B12, BH-B13	~950	4	25	illite+smectite	Q,Pl,By,TK(%1)
BH-B8	850-900	6	25	smectite	Q,Pl,Ç,By,Py,Op
BH-B9	~950	5	14	illite	Q,Pl,By,Op,TK(%1)
BH-B11	850-900	5	8	illite	Q,Pl,By

(*) **A:** Andesite, **By:** Biotite, **C:** Calcite, **Ç:** Chert, **G:** Granite, **K:** Limestone, **MA:** Matrix Total Aggregate Ratio, **Op:** Opaque Minerals, **P:** Total Porosity, **Pl:** Plagioclase, **Py:** Pyroxene, **Q:** Quartz, **T:** Firing Temperature, **TK:** Brick Particles Ratio

Table 2b. Petrographical thin section optical microscopy analysis of soil / clay samples

Soil / Clay Sample Group	Soil / Clay Samples	Particle Size (%)			MA (%)	Rocks & Minerals*
		Clay	**Silt**	**Sand**		
Group 1	BH-D1, BH-D2, BH-D3,BH-D4,BH-D6	20	45	35	12	Q,K,C,A,G,Ç
Group 2	BH-D5	20	30	50	10	Q,Pl,Op,By,Ç,Py

Table 2c. Matching of the groups of ceramic and soil samples by petrographical thin section optical microscopy analysis.

Ceramic Samples	Clay Group	Rock Type	Descriptions
BH-B1	Group 1	Andesitic	
BH-B2, BH-B3	Group 1		Ceramics fired undried (oriented aggregate in matrix)
BH-B4	Group 2	Andesitic	Ceramics fired undried (oriented aggregate in matrix)
BH-B5, BH-B6, BH-B7, BH-B10, BH-B12, BH-B13	Group 1	Andesitic	
BH-B8	Group 1	Andesitic	Having coarser and mixed sized particle
BH-B9	Group 1	Andesitic	Ceramics fired undried (oriented aggregate in matrix)
BH-B11	Group 1		

Table 3. Hasankeyf Excavation ceramic and soil sample [a]PED-XRF and [b]EDX analyses.

Element	MgO	Al_2O_3	SiO_2	K_2O	CaO	Fe_2O_3	LOI*
BH-B1	[a]4,36 [b]6,90	10,58 13,25	41,97 39,76	1,36 0,96	16,54 15,32	6,48 6,50	17,47
BH-B2	4,79 6,70	10,99 13,59	44,58 46,60	1,68 1,56	15,17 14,09	6,96 6,97	12,58
BH-B3	4,79 5,30	11,43 11,60	45,90 36,42	1,81 1,54	13,69 12,36	7,07 6,12	12,68
BH-B4	5,00 4,98	11,17 10,07	44,74 31,50	1,29 0,64	16,16 13,44	7,09 5,26	12,86
BH-B5	4,37 5,38	11,17 7,08	45,00 27,52	1,31 1,44	13,98 21,69	6,90 7,18	15,79
BH-B6	4,73 4,43	10,96 10,22	45,13 39,48	1,94 1,45	12,56 16,93	6,88 5,28	14,86
BH-B7	4,41 5,11	11,02 13,38	43,68 46,54	1,23 0,79	16,47 12,53	6,81 5,43	12,22
BH-B8	4,10 7,58	11,24 10,90	45,42 40,08	1,42 1,21	17,05 13,42	6,95 5,21	8,74
BH-B9	4,11 4,93	10,41 12,39	44,09 40,44	2,44 1,79	11,89 12,10	6,45 5,28	18,75
BH-B10	5,78 4,56	10,75 6,79	44,30 22,86	2,60 1,96	16,28 14,31	7,09 7,73	11,47
BH-B11	5,39 5,19	11,22 0,52	45,87 43,68	1,64 1,43	13,75 12,56	7,11 7,11	13,57
BH-B12	4,05 5,66	10,28 12,12	40,99 36,20	1,40 1,23	17,11 13,33	6,57 6,03	12,55
BH-B13	4,63 4,33	10,95 12,73	44,62 44,94	1,49 1,39	16,03 15,73	6,82 5,97	13,55
Average	**4,66**	**10,94**	**44,33**	**1,66**	**15,13**	**6,86**	**13,62**
SD**	**0,51**	**0,34**	**1,43**	**0,44**	**1,76**	**0,23**	**2,61**
BH-D1	5,08	11,84	44,94	1,90	7,20	7,14	19,44
BH-D2	4,57	10,60	43,30	1,56	8,49	6,49	23,68
BH-D3	4,68	10,91	45,36	1,64	7,07	7,03	21,57
BH-D4	4,02	9,46	37,89	1,36	13,83	5,89	26,97
BH-D5	3,98	9,67	38,03	1,40	13,28	5,81	26,85
BH-D6	3,53	8,73	36,29	1,16	12,42	5,59	31,47
Average	**4,31**	**10,20**	**40,97**	**1,50**	**10,38**	**6,33**	**25,00**
SD**	**0,56**	**1,13**	**4,01**	**0,26**	**3,13**	**0,66**	**4,33**

(*) LOI: Loss on Ignition at 950ºC, (**) Standart Deviation

This situation is also supported by SEM photographs.

The results of TGA analysis reveal that samples contain limestone and calcite. In the thermal analysis conducted, the TGA curves were examined and a distinct difference was observed in the structure of 4 findings between 107°C - 112°C (Fig.3). As it is known, gypsum ($CaSO_4 . 2H_2O$) is a common compound used in making ceramics. At the temperatures between 100 °C - 115°C, H_2O is released and $CaSO_4$ is formed. This change is clearly observed in TGA curves. The TGA curves of the findings indicate such a change clearly. Therefore, it can be established that the structure of the BH-B3, BH-B6, BH-B8 and BH-B11 samples include gypsum.

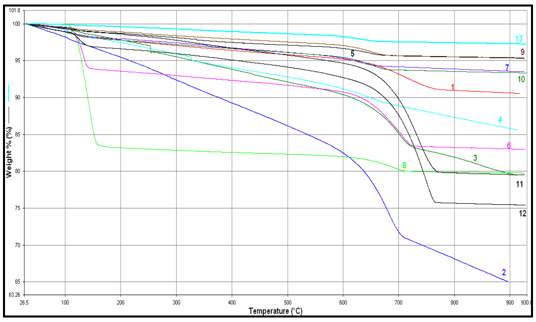

Figure 3.Thermogravimetric analysis (TGA) of ceramic samples (Numbers 1-13 in graphs represents the samples from BH-B1 to BH-B13).

It can be asserted that BH-B1, BH-B2, BH-B4, BH-B5, BH-B7, BH-B9, BH-B12, and BH-B13 samples contain Calcite ($CaCO_3$) because these findings have TGA curves that indicate a decrease in mass and then a clear mass loss between 700°C - 800°C. It is known that calcite ($CaCO_3$) is a common compound used in making ceramics. At temperatures between 700°C - 800°C, CO_2 is released and CaO is formed (Fig.5).

The Hasankeyf ceramic samples have a porosity that vary between the ratios 1% - 6%. In the samples, the clay group is generally represented by illite and smectite (Pollard and Heron, 1996: 121) and some (BH-B4) contain kaolinite (Table 2a). When the samples are assessed in terms of aggregate/matrix ratios, the matrix aggregate content is found to be between 2.5% and 25% (Table 2a). All the ceramic samples are original. Their raw material contains crushed pieces of local rock used without any screening (broken pieces of angular aggregates) at different sizes and that have a heterogenic distribution (Fig.3). Some samples contain aggregates with quite fine-sized grains (silt-sized; <63 µm) with homogenous distribution while other samples include large sand-sized (>1000 µm) aggregates (Table 3a and Fig.3). The clay samples were grouped into two (Table 2b). Except for one of the ceramic samples (BH-B4), all other samples have a petrographic structure similar to the clay content of the same group (Group 1) (Table 2c).

The aggregate structure of the two of the seven groups of ceramic samples included some broken bricks (1% of the total aggregate ratio), which requires questioning the originality of adding broken bricks in ceramic clay. It is known that broken bricks have been knowingly added into ceramic clay in ateliers with the purpose of increasing its fragility and regulating the moisture of the paste (Table 2a and Fig.4).

The chemical compounds of 13 ceramic and 6 clay samples were examined by means of a PED-XRF analysis (Table 3 and Fig.4).

When the chemical compositions used in ceramics production are evaluated in accordance with the TGA analyses, the main structure can be described by the presence of SiO_2, and carbonate (soil alkalis) and clay play the role of melting down and increasing durability. The chemical and petrographic characteristics of clay which are obtained from local riverbeds in a refined form and provide durability through their various properties – such as plasticity, firing/production ease, coloring properties, etc. – are important for ceramic production. The aggregates that form the clay structure provide important information about the origin of the manufacturing centers as they naturally involve the local rock compounds in them (Hodges, 1964: 19-41; Demirci et al, 1999: 53-62; Akyol, 2007: 99-114; Tekkök et al, 2009: 101-121; Aygün et al, 2010: 411-429). Among all the samples, BH-B4 has a different petrographic aggregate content.

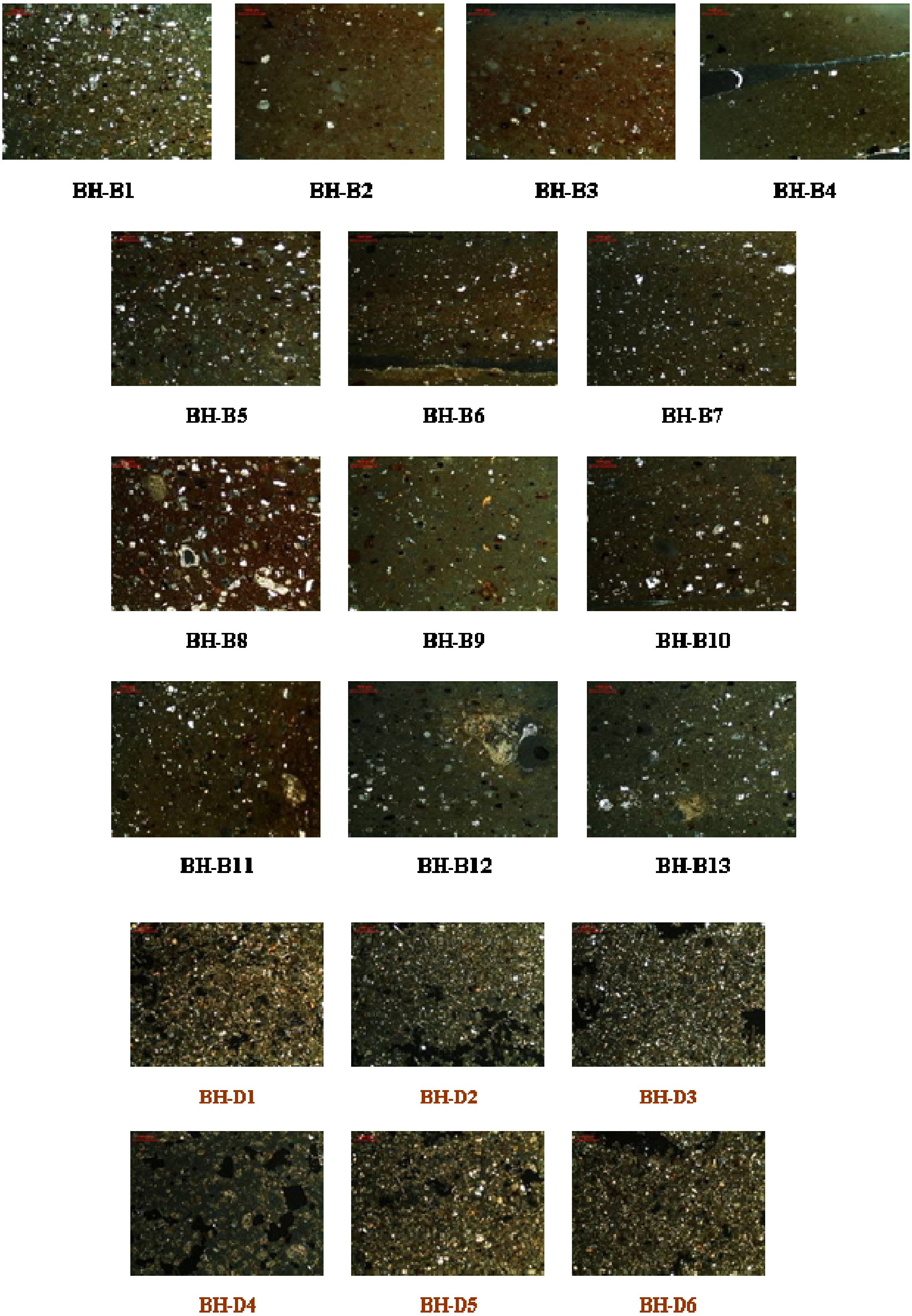

Figure 4. Thin section optical microscopy micro-photographs of ceramic and soil samples

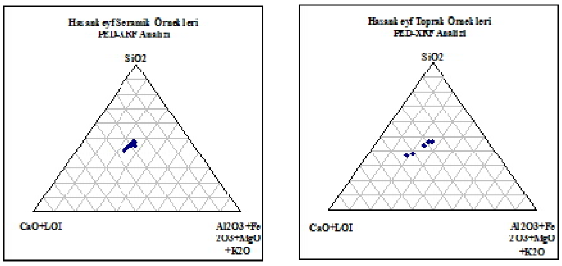

Figure 5. Hasankeyf Excavation ceramic (on left) and soil samples PED-XRF analysis -Triangle Plotting (SiO_2-CaO+LOI - Al2O3+Fe2O3+MgO+K2O)

Both optical microscope analysis and SEM-EDX analyses comparatively determined that (Table 3 and Fig.6) Hasankeyf ceramic samples contained SiO_2 (44.33%), CaO (15.13%), Al_2O_3 (10.94%), Fe_2O_3 (6.86%), MgO (4.66%), K_2O (1.66%) and total carbonate content (13.62% LOI) (Tables 3,5).

The main element (>1%) contents of the examined soil/clay samples consisted of SiO_2, CaO, Al_2O_3, Fe_2O_3, K_2O and total carbonate content (LOI) (Table 4). The main element contents of the clay samples form two groups (first group: BH-B1, BH-B2 and BH-B3 and second group: BH-B4, BH-B5 and BH-B6 samples). The first group samples (BH-B1, BH-B2 and BH-B3) contain silica (SiO_2) more densely, and other group samples have a content of richer lime (CaO) and carbonate (LOI). The element contents of the ceramic samples that are similar with clay samples can provide the address for the probable clay bed (Table 3).

Strontium (Sr) is similar to Ca geochemically and it is found in substances that contain lime (seashells, limestone, etc.). As the sand used in the making of the ceramics contains more than 400 ppm Sr, it can be asserted that clay obtained from marine sediments was mostly used in production. In addition, in inland sand that contains limestone, the amount of Sr is usually less than 150 ppm. What's more, if inland sand had been used in production, Zirconium (Zr) would be expected to be more than 160 ppm (Freestone et al, 2003: 19-32). Of the soil/clay samples, the Sr and Zr contents of BH-D1 sample are 247.6 and 87.6 ppm, and those of BH-D2 sample are 130.9 and 266.9 ppm, respectively. Whereas marine effects are partially observed in the BH-D1 sample, the BH-D2 sample has intense inland properties (Table 3). The Sr content of two of the 16 ceramic samples taken from three regions (BH-B3a and BH-B3b) is below 150 ppm (inland origin), and the Sr content of two samples (BH-B2a and BH-B3f) is above 400 ppm so they must have a marine origin (Table 3). Similarly, the Zr value of seven of the ceramic samples (BH-B2a, BH-B2b, BH-B2c, BH-B2d, BH-B2f, BH-B3d, BH-B3e) is above 150 ppm so they should most probably have an inland origin (Table 3). When the samples are evaluated generally, it can be asserted that they are produced by using inland origin clay, except for the BH-B3f sample. The BH-B2a sample must have been produced by using a mixture of inland and marine clays; not one of them alone (Table 3). The fact that clay from inland, i.e. belonging to a riverbed in the near vicinity, was used is also supported by the thin cross section analyses which did not reveal any fossil contents.

4. CONCLUSION

Thirteen ceramic and 6 soil/clay samples acquired from different regions of the Hasankeyf archaeological site were examined archaeometrically.

The matrix/aggregate content, aggregate type, aggregate distribution, and aggregate dimensions, porosity and clay structure of the samples were determined by thin cross section optical microscope analysis and the samples were classified into 7 different groups. The firing temperature of the ceramic pieces must be mainly between 800–950°C. Moreover, TGA curves indicate deterioration temperatures between 750–850°C, which support the suggested temperature range.

The clay types of the samples are mostly illite and smectite, as well as kaolin. Broken brick pieces (1%) were observed in the aggregate contents of most of the samples (7 samples). The petrographic characteristics of the ceramic samples comply with local formation. The raw material content of the ceramic samples, all of which are original, contains crushed pieces of local rock at different sizes, used without any screening and has a heterogenic distribution.

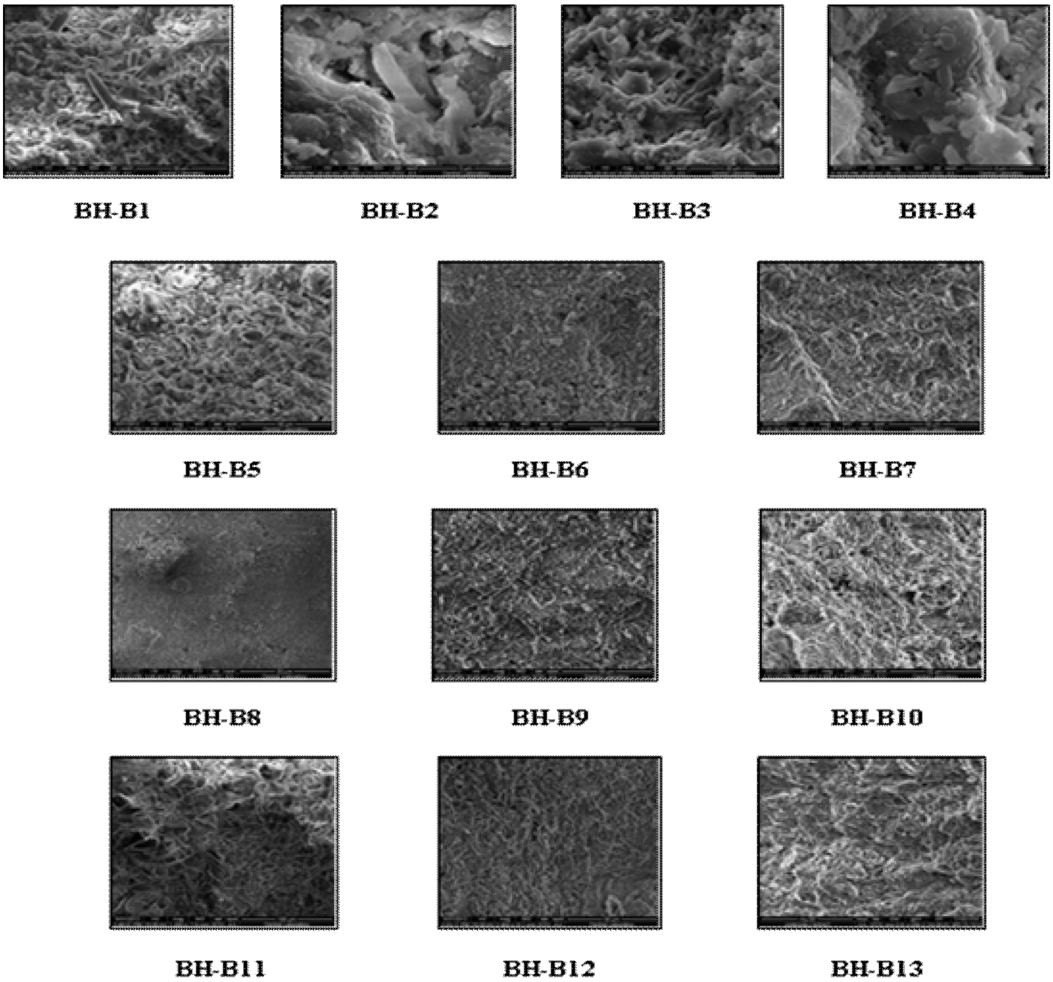

Figure 6. SEM analysis microphotographs of ceramic samples

The samples have similarities with the soil of the region and they are also similar to each other in terms of both chemical compositions and petrographic characteristics. The source of raw material must usually have been clay from inland soil and riverbeds in the near vicinity.

The ceramic samples that were grouped according to their petrographic structures were examined in more details by means of a PED-XRF analysis. The ceramic samples reflect local rock formation; the ceramic samples and clay samples from the nearby riverbed have similar chemical structures. Furthermore, the results of the SEM-EDX and PED-XRF analyses are generally compliant, which is important evidence that assessments can be made based on EDX analyses on various ceramic samples.

The archaeometric studies on a limited number of ceramic samples acquired from different regions of the Hasankeyf archaeological site have revealed important information. By considering the Hasankeyf ceramics in a local and regional context, and increasing the number of studied pieces and diversifying them, it will be possible to provide more comprehensive knowledge. Thus, Hasankeyf can be studied as a local ceramics manufacturing center, and data such as production diversity, production technologies, relations with other manufacturing centers, trade paths, etc. can be acquired.

ACKNOWLEDGEMENT

The authors wish to thank Gazi University for the support of research project, Hasankeyf Excavation director Prof. Dr. Abdüsselam Uluçam, Prof. Dr. Yusuf Kağan Kadıoğlu and Kıymet Deniz from Ankara University Earth Sciences Application and Research Center (YEBIM), Gülşen Albuz Gazi University Historical Material Research and Conservation Laboratory (MAKLAB) for laboratorial processes and Orhan Karaman for thin section preparations.

REFERENCES

Akyol, A.A., Tekkök, B., Kadıoğlu, Y.K., Demirci, Ş. (2007) Tarsus, Gözlükule Erken Roma Dönemi Seramikleri Arkeometrik Çalışmaları, 22. *Arkeometri Sonuçları Toplantısı,* Ankara, p. 99-114.

Arık, O., (2003) *Üç Dünyanın Buluştuğu Kent*, İstanbul.

ASTM - American Society for Testing and Materials (1984) Standard Test Method for Unconfined Compressive Strength of Intact Core Specimens. Soil and Rock, Building Stones: *Annual Book of ASTM Standards* 4.08, Philadeplhia, Pennsylvania.

Aygün, Ç.Ö., Kadıoğlu, Y.K., Akyol, A.A. (2010) Elazığ, Hazar Gölü Altındaki Batık Yerleşimlere Ait Seramiklerin Petrografik İncelemeleri, *25. Arkeometri Sonuçları Toplantısı*, Ankara, p. 411-429.

Çeken, M. (2007) Anadolu'da Bir Seramik Üretim Merkezi: Hasankeyf (Fırın ve Atölyeler), Byzas, 7, p. 469-488.

Göçmez, H., Çeken, M., Yanık, G. (2004) The Characterization of Ancient Ceramics from Hasankeyf (Turkey), *Key Engineering Materials*, Vol. 264-268, p. 2407-2410.

Demirci, S., Türkmenoglu, A.G., Akyol, A.A. (1999) Characterization of the Ceramics Obtained from Antic Amphora Workshops in Sinop Region, *Caesaraugusta* 73, p. 141-148.

Freestone, I.C., Leslie, K. A., Thirlwall, M., Gorin-Rosen, Y. (2003) Strontium Isotopes in the Investigation of Early Glass Production: Byzantine and Early Islamic Glass from the *Near East, Archaeometry. Volume 45, Issue 1, p. 19-32.*

Hodges, H. (1964) Artifacts An Introduction to Primitive Technology, New York.

Özkul-Fındık, N. (2009) Hasankeyf Seramikleri (2004-2006), Ankara.

Pollard, A.M., Heron, C. (1996) *Archaeological Chemistry*, The Royal Society of Chemistry, Cambridge.

Rice, P.M. (1987) *Pottery Analysis, A Source Book,* Chicago.

Shackley, M.S (Ed). (2011) An Introduction to X-Ray Fluorescence (XRF) Analysis in Archaeology. In*: X-Ray Fluorescence Spectrometry (XRF) in Geoarchaeology*, Springer, New York.

Uluçam, A. (2007) Başlangıcından Bugüne Hasankeyf Kazıları, Konya Kitabı X. Özel Sayı, Rüçhan Arık - M. Oluş Arık'a Armağan, 681-710.

Tekkök, B., Akyol, A.A., Kadıoğlu, Y.K., Demirci, Ş. (2009) The Importance of Archaeometric Analysis on Ceramics from Archaeological Excavations: The Example of Early Roman Glazed Ware from Tarsus and Troia (Ilion), *Proceedings of The 1st International Ceramic, Glass, Porcelain, Enamel, Glaze and Pigment Congress-*SERES'09. Eskişehir, Turkey, p. 101-121.

Whitbread, I.K. (1995) Grek Transport Amphorae: A Petrological and Archaeological Study, *Fitch Laboratory Occasional Paper 4, British School at Athens*: Oxford.