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# Study on Late Bronze Age Potteries of Shahrak-e Firouzeh, Neyshabur, Iran by XRD and XRF

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#### ABSTRACT

During excavations of the Shahrake Firouzeh site, much evidence of the Bactria Margiana Archaeological Complex or Amu Darya Civilization has been found. Such evidence is spread across northern Iran, northern Afghanistan, eastern Turkmenistan, southern Uzbekistan, and western Tajikistan. In the present study, pottery samples from the Firoozeh town area were studied using XRD and XRF methods. Using the XRF method and Dplot Software, 15 samples were studied and the data evaluated by analyzing the ratio of various components. The results of the study show that the dominant clay source of the pottery samples existed in the same region, while two other clay subsources from adjoining regions were also identified.

## 1. Introduction

Because of its continuous and abundant production, pottery has always been associated with the alterations and innovations of its shapes and patterns. Such changes happen partly due to the internal intricacies of cultures over time and partly due to the cultural and economic ties with neighbouring and distant societies. On the other hand, for various reasons, such as being a merchandise that can be exported as well as its use in transporting other goods, pottery can be considered as the best cultural data for the recognition of ethnic groups and societies. Pottery is so important in studying the relationship between the different cultural units of a region on the one hand, and trans-regional cultures on the other, that no other cultural data in archaeology could reach the same level of significance. Thus pottery should be studied not only from various aspects but also from different perspectives. The samples that were discovered across the ancient sites of Iran show the connection of this art to culture, economy, and religion; moreover, they display the taste and virtue of the potters (Majidzadeh, 1991).

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Using chemical and physical methods, archaeometry is a great help to archaeologists and art historians. Its use in determining the origins of archaeological findings is one of the most modern approaches in the field of archaeometry. This field of science presents archaeological data, or related phenomena, as quantitative, measurable or qualitative data. Among the laboratory methods, two relatively quick and easy ones that have helped the fields of archaeology and art history are the XRF and XRD methods. XRF (X-ray fluorescence) involves a device for measuring the wavelength and intensity of fluorescence waves released from the atoms of a sample through which the amounts of the sample's constituent elements can be recognized (Talai et al., 2009). As a rapid mechanical analysis tool, this device is widely used in many laboratories around the world (Sabzali et al., 2010). The XRD (X-ray diffraction) test is also used to measure atomic plate spacing; in other words, to identify the material structure and ultimately determine the minerals within the pottery. For the XRD test, the pottery sample must be crushed into powder and then placed in the device. The device automatically analyzes the sample and records the analysis results in the computer connected to it (Talai et al., 2009).



Figure 1. Geographic position of Neyshabur city, northeast of Iran (map by authors).

In this regard, such use of laboratory methods and techniques enables more accurate and reliable results to be obtained. There exist several techniques for the identification of the materials and elements that form archaeological findings and through which a broader range of knowledge and understanding of the rocks, pottery, their material, application, history, age, style, identity, origin, manufacturing process and technology used, source, and trade routes utilised, can be gained. Laboratory research on the physical characteristics of prehistoric pottery is one of the efficient non-descriptive methods that aim to identify such technical features as method of production, the presence or absence of vegetation, type of soil grading, type of tempering, and the quality of baking; moreover, the empty spaces in the body of the clay, which is related to the general quality of the clay, can be determined (Talayi, Yari, and Taqizoqi, 2009).

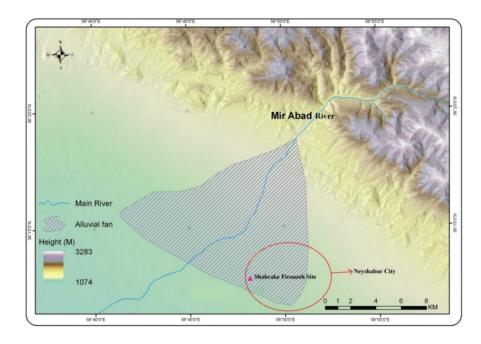
### 2. Area under Study

Neyshabur town is located in the Neyshabur plain in the Razavi Khorasan Province in northeast Iran (Figure 1).

This area has a special geographic and climatic situation and, due to its location on the Silk Road, has attracted so many people throughout history that much archaeological evidence has been found from the prehistoric, historical, and Islamic era in this region. Neyshabur plain is located on the slopes of the Alborz Mountains and Binaloud Heights that provide rich water sources to this plain. Water abundance has created numerous habitats in different parts of the slopes of these mountains and the Shahrake Firouzeh site is one of the most significant ones (Figure 2). This area with a latitude of 36 degrees and 12 minutes, longitude of 58 degrees and 47 minutes, and with an average height of 1250 metres above sea level is located in the Neyshabur plain (Basafa et al., 2014). This area is named after the newly-constructed residential complex in the western part of the present-day Neyshabur city which is located in the eastern part of the Faroub Ruman River. As the most important water stream of the city, this river originates from the highlands of the Binaloud Heights. It is also one of the independent rivers of the Markazi Desert sub-areas that floods in the rainy season. Because of the slopes of the plain, with the flood of the Farub



**Figure 2.** Position of Shahrake Firouzeh in Neyshabur plain (map by authors).



Ruman and other rivers, a high volume of alluvium has been deposited in the plain in a long-term process. The dimension of the Shahrake Firouzeh site is unclear because it is buried under alluvial deposits and according to preliminary studies, a large part of it lies under the residential areas of Neyshabur (Basafa et al., 2014). Generally, based on the excavations, the Shahrake Firouzeh site area represents Bronze and probably Iron Age cultures in the region; besides, the findings about the cultural materials point to the existence of a culture called the Bacteria Margiana Archaeological Complex (BMAC) (Basafa, 2014). This culture appeared in the second half of the 2<sup>nd</sup> millennium BC, in the Merv region, which is located in the southeast of today's Turkmenistan, changes occurring in the human population during this period (Sarianidi, 1998). The time span starts from 2300 or 2200 BC and continues until 1700 BC. The most important cultural materials were prestigious goods made of chlorite or steatite, metal objects made with casting techniques, marble columns, steatite stamps, plates made in Late Serie style, and special types of pottery whose illustrations related specifically to this culture. The obtained artefacts show the regional and trans-regional interactions of Shahrake Firouzeh with its neighbouring areas, such as Merv, Balkh, southern Turkmenistan, and Iran. Findings such as pieces of slag, metals, furnace waste, pottery furnaces, etc., show a dynamic and stable settlement with extensive industrial activities (Basafa, 2014). The BMAC potteries are related to the Late Bronze Age, which are often found in a burial context in the Shahrake Firouzeh site.

Neyshabur's geological zone consists of the three eras, starting with the Paleozoic, then Mesozoic and Cenozoic. The oldest geological formations of the region is related to the Precambrian era that consists of calc, schist and phyllite (Shorm) and dolomite crystalline (Soltanieh) that outcrops in the north eastern parts. Formations of the first

geological period are located in the northern parts of the plain in the northern Binaloud Heights that include quartzite (Lalun) sandstones, dolomite, dolomitic limestone, and shale (Mila), marl limestone (Niur), limestone and dolomite (Bahram), and crystallized quartzite. The second geological formations consist of Mashhad phyllite, light grey limestone, pale golden (Jurassic) limestone, and coloured mélanges ('mixture of colours'). The Jurassic black schist has spread across the 250 km<sup>2</sup> of the Binaloud Heights to the Mashhad area. In the northwestern and southern heights, the tuffs and conglomeration formations can be seen in green. The igneous rocks of the area have plenty of gaps due to the decomposition of the feldspars, which provide a source for groundwater storage. Miocene formations exist in the form of marl-gypsum and salty sediments along with sandstone with a variety of colours. These formations have spread across the northern margins and partly onto the southern parts of the plain. The deposits of the third era of geology that are widely distributed in the region outcrop in the slopes of the central and southern heights. These formations consist of sandstones, shale, conglomerate, marl, and igneous (often exterior) rocks (Figure 3).

The Quaternary formations consist of terraces, alluvial fans, shifting sands, and alluvial deposits. In addition, scientists believe that some of the volcanic formations of this region are also of the Pleistocene Age. In terms of tectonics, the Khorasan Province is generally divided into four distinct regions: the Kopet-Dagh area, the Eastern region of Central Iran, the Lut region, and finally the Eastern part of the Iran mountains. Neyshabur city is located in the tectonic region of the eastern part of Central Iran.

The Neyshabur plain has special natural and geographical features and one of the reasons for its importance is its connective role as a result of its city's location on the commerce route of the Silk Road from earliest times.

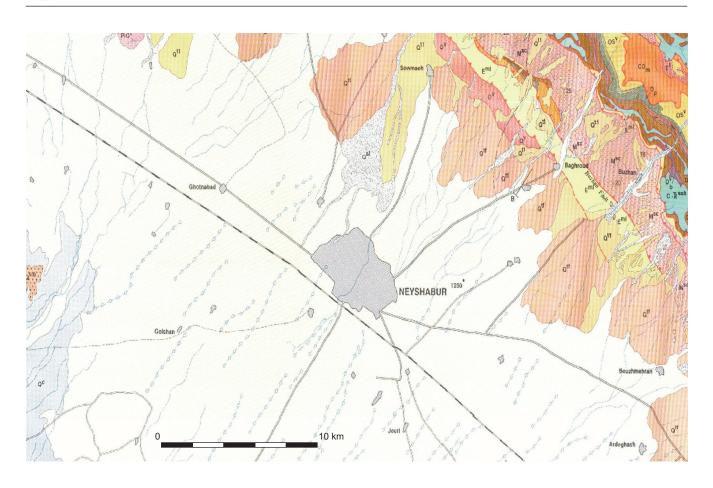




Figure 3. Geological map of the region under study (map from Iranian geology org).



However, due to the active and dynamic geomorphological conditions of the region and continuous flood flows, the prehistoric settlements of the area were displaced and buried under alluvial deposits. The Shahrake Firouzeh site is one of the prehistoric areas dating back to the Late Bronze Age that was established on the sidelines of an alluvial fan. Generally, alluvial fans have provided appropriate conditions for the establishment of human settlements (Figure 3) around the world from prehistoric times, which is due to their underground water-rich aquifers, the alluvial fan's gentle slopes, the fertility of the soil due to sedimentation, and the existence of water streams that provide the water needed for drinking, farming, and such craft industries as making pottery (Gillmor *et al.*, 2009; 2011).

#### 3. Aims and Research Method

This study uses the XRF and XRD laboratory methods in order to determine the composition of the potteries found in the Shahrake Firouzeh site. These potteries relate to the Late Bronze Age (BMAC Culture) and they were often obtained from the burial areas. This research investigates the New Bronze Age potteries of the Shahrake Firouzeh site based on laboratory methods for the first time: a step towards recognizing the clay sources used for pottery production at the Shahrake Firouzeh site and determining the cultural and commercial ties of this town. Ultimately, it should be pointed out that the purpose of these experiments on the samples is to understand if the potteries were produced locally or were imported into the Shahrake Firouzeh through trans-regional transactions.

With the XRF and XRD laboratory methods, 15 pottery samples were analyzed from trenches X & XII. The research

samples were selected from a set of potteries with a specified chronological and stratigraphic context. Three samples were analyzed by the XRD method and 15 samples by XRF. The statistical analysis of the laboratory findings were based on the spectroscopy of the chemical elements and their quality and quantity. After the sampling and performing of relevant tests, the data were analyzed so as to ascertain if the potteries were of local production or imported.

Fragments of about 1cm<sup>2</sup> were removed from each shard and abraded using a silicon carbide burr in order to remove surface treatments (*e.g.* glaze, slip, paint) and adhering soil, thereby reducing the risk of measuring contamination. The specimens were washed in deionized water and allowed to dry in the laboratory.

#### 4. Results

#### **4.1 XRD**

The X-ray diffractometer was originally designed for examining powder samples. However, the diffractometer is today more often used for examining samples of crystalline aggregates other than powder. Also polycrystalline solid samples and even liquids can be examined. Importantly, a sample should contain a large number of tiny crystals (or grains) which randomly orient in three-dimensional space because standard X-ray diffraction data are obtained from powder samples of perfectly random orientation (Leng, 2008, p.61). The key characteristic of XRD is its ability to identify crystalline minerals. The primary use of the powder XRD method has been the identification of clay minerals in pottery in order to characterize pottery types and to investigate sources for raw materials (Pollard *et al.*, 2007, p.120). XRD also allows the examination of firing

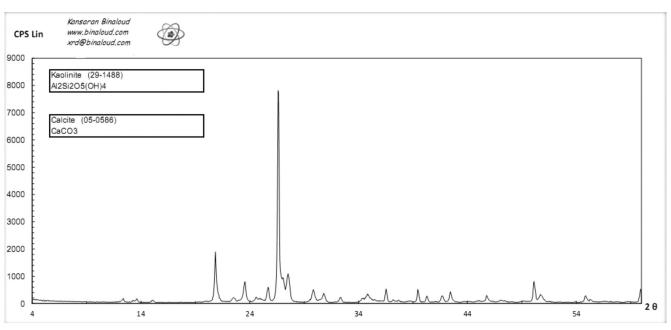


Figure 4. Sample No.1 XRD spectra diagram, showing clay and calcite minerals.

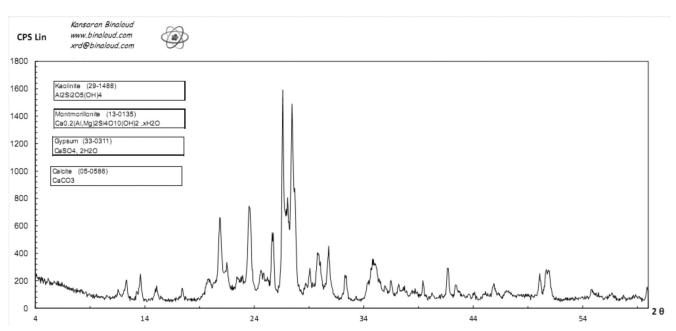


Figure 5. Sample No.1 XRD spectra diagram, showing clay, chalk and calcite minerals.

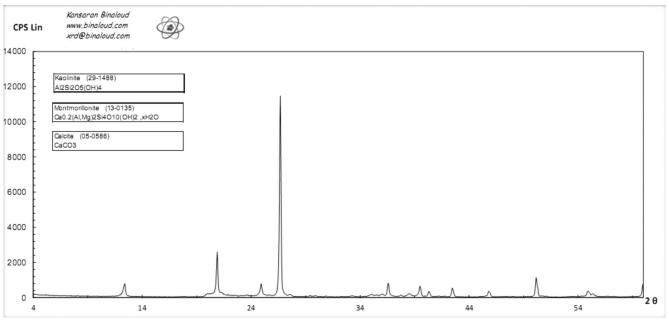


Figure 6. Sample No.2 XRD spectra diagram, showing kaolinite, montmorillonite and calcite minerals.

temperatures, as different minerals are destroyed or begin to form at different temperatures. XRD is also commonly used in the study of corrosion products formed on the surface of metals, including iron and copper alloys.

Three samples were sent to the Kansaran Binaloud laboratory for analysis. When pottery is exposed to different temperatures, it shows different and distinct physical and chemical changes. Normally, the water and moisture of the pottery evaporates at a temperature of 100 to 200°C. At a temperature of 250 to 450°C, the organic materials in the pottery burn. Finally, at a temperature of 700 to 800°C, the calcium carbonate in the clay decomposes and turns into calcium oxide and carbon dioxide. Conventionally, this is

the proper temperature for producing good quality pottery. At 850°C, the pottery only contains quartz and feldspar. At temperatures above 850 to 950°C, a baking process occurs which leads to the emergence of pyroxene besides the quartz and feldspar in the pottery. In these samples, no sign of calcium carbonate or calcium oxide are found.

Examination of these samples showed that the calcite mineral (CaCO<sub>3</sub>) did not decompose when heated and did not convert to CaO and CO<sub>2</sub>. Therefore, the maximum identified temperature is around 700°C. There exist a number of clay minerals in the texture of all the samples, each one indicating a different temperature. The presence of clay minerals usually depends on the type of soil used to make the pottery.



Figure 7. Analysed samples.

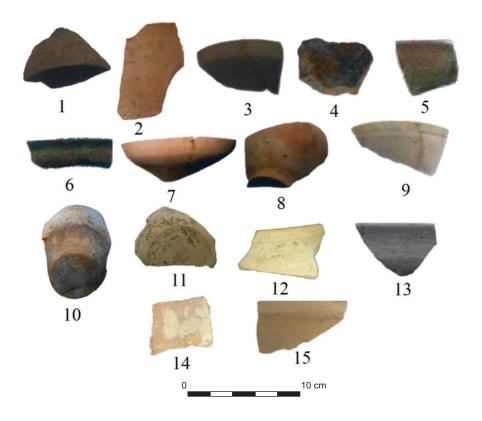


Table 1. General data of samples.

Sample	Section	Technique	Heating	Interior	Exterior	Paste	Design	Height	Thick	Diametr	Temper
No.				colour	colour	colour		cm	mm	cm	
1	base	wheel made	complete	red	buff	red	_	3.5	17	9	mineral
2	lip	wheel made	complete	buff	buff	red	_	9.0	7	10	mineral
3	lip	wheel made	complete	buff	buff	red	_	3.7	4	13	mineral
4	lip	wheel made	complete	buff	buff	red	_	5.1	4	16	mineral
5	body	wheel made	complete	red	red	red	geometric	3.3	14	_	mineral
6	lip	wheel made	complete	buff	buff	red	_	7.0	5	20	mineral
7	base	wheel made	complete	red	red	buff	_	7.0	10	9	mineral
8	lip	wheel made	complete	red	red	red	_	4.0	6	29	mineral
9	lip	wheel made	complete	buff	buff	red	_	4.0	9	21	mineral
10	lip	wheel made	complete	buff	buff	red	_	8.0	4	24	mineral
11	base	wheel made	complete	buff	buff	red	_	17.5	10	12	mineral
12	base	wheel made	complete	buff	buff	buff	_	50.0	8	8	mineral
13	lip	wheel made	complete	red	buff	red	_	67.0	6	8	mineral
14	lip	wheel made	complete	buff	buff	red	_	45.0	6	15	mineral
15	lip	wheel made	complete	red	red	red	_	37.0	5	20	mineral

Clay minerals are derived from the chemical decomposition of rocks containing high levels of feldspar, a phenomenon that is called argillic alteration in geology. The result of these changes is the formation of clay deposits that are important sources depending on their type of minerals, presence of impurities, etc. Probably the soil used for the pottery production contained a mixture of lime and clay with impurities such as gypsum. Due to the presence of clay minerals in the pottery's soil and the temperature for baking the pottery, the existent clay in the soil is sufficient.

# 4.2 XRF Test

XRF analysis has become a routine technique in investigations of elemental composition for a wide variety of archaeological materials. In archaeology, the main application of EDXRF is for the rapid identification and semi-quantitative analysis of a wide range of materials including metals and their alloys, ceramics, glass, jet, faience, pigments, glazes, gemstones, and industrial debris (Pollard *et al.*, 2007, p.118).

For this method, 15 samples were sent to the XRF laboratory of Tarbiat Modares University and analyzed by a



Table 2. Chemical composition (%) of the samples under study (sum of elements is not 100%).

SampleNo	SiO,	Al,O,	Fe,O,	CaO	Na,O	K,O	O MgO TiO,		MnO	$P_{2}O_{5}$	$\mathbf{S}$	Loss on ignition		
	% -	%	%	%	%	%	%	% -	%	%	%	%		
1	54.01	5.9	15.11	8.24	1.28	2.83	1.20	1.701	0.070	0.701	0.001	5.98		
2	52.65	5.51.	13.17	10.51	0.31	1.48	1.83	2.383	0.143	0.251	0.002	7.10		
3	53.11	5.26	15.76	9.32	1.03	1.21	1.61	1.231	0.094	0.652	0.004	5.77		
4	50.98	3.72	14.43	13.72	1.89	1.03	1.42	1.201	0.120	0.45	0.003	7.75		
5	55.11	5.6	14.13	7.24	1.08	2.12	1.07	1.60	0.021	0.311	0.001	6.38		
6	51.55	5.33.	14.17	11.82	0.11	1.65	1.43	2.435	0.109	0.437	0.004	7.10		
7	52.21	4.12	13.9	10.15	0.27	1.30	1.63	2.215	0.136	0.532	0.003	6.96		
8	52.33	3.95	12.73	12.80	0.81	1.27	1.12	1.63	0.120	0.52	0.011	7.62		
9	53.12	5.1	14.98	9.78	1.48	1.83	1.65	1.130	0.062	0.661	0.002	5.63		
10	54.01	6.12	14.21	8.24	1.28	2.83	1.20	1.701	0.061	0.621	0.001	6.82		
11	53.68	2.73	13.33	12.72	1.19	1.04	1.32	1.26	0.131	0.361	0.003	7.52		
12	53.01	5.76	13.45	11.32	0.87	1.34	1.19	1.613	0.068	0.751	0.002	5.68		
13	54.78	3.72	13.02	13.72	0.74	1.14	0.96	1.52	0.342	0.861	0.001	7.58		
14	53.28	4.76	13.31	12.74	0.75	1.13	1.81	1.181	0.203	0.371	0.003	6.56		
15	56.01	3.39	15.19	10.64	1.36	2.09	1.73	1.126	0.172	0.598	0.001	5.63		

Table 3. Elements in samples under study (ppm).

Sample No.	Cl	Ba	Sr	Cu	Zn	Pb	Ni	Cr	V	Ce	La	W	Zr		Rb	Co	As	U	Th	Mo	Ga	Nb
1	61	175	243	13	110	11	40	14	18	53	19	7	203	32	49	2	8	1	3	26	17	10
2	101	152	178	29	102	16	42	7	22	46	17	4	219	42	56	2	9	2	1	16	18	13
3	73	178	240	16	108	15	48	18	20	48	21	5	213	30	29	3	7	2	3	23	16	13
4	79	310	160	27	130	24	26	3	70	113	38	1	289	69	119	2	83	1	2	8	19	9
5	85	173	249	19	114	15	52	15	18	58	17	9	231	42	50	2	7	2	3	22	17	12
6	109	129	182	33	114	14	48	1	24	42	16	4	227	39	52	1	9	1	4	15	16	12
7	93	202	170	30	128	19	31	8	42	61	25	5	251	52	72	2	32	2	3	5	19	18
8	92	167	213	15	118	8	39	17	19	34	15	7	180	32	48	3	9	3	5	18	15	11
9	97	190	177	14	118	9	33	9	46	64	21	4	240	59	69	2	29	3	2	4	17	8
10	96	128	190	27	119	10	40	2	21	50	18	6	210	43	52	3	8	1	2	16	12	13
11	80	309	158	30	140	28	24	2	73	110	42	2	293	72	114	3	79	2	1	9	18	8
12	105	137	183	36	120	16	47	10	19	39	19	5	217	41	56	2	12	1	1	15	16	13
13	74	183	236	19	119	12	39	9	20	54	21	3	210	39	53	2	10	2	3	19	19	14
14	98	149	182	366	115	13	41	8	22	43	17	4	213	49	59	1	14	2	2	17	18	12
15	83	316	157	26	138	24	19	2	80	98	39	3	279	81	135	2	81	1	2	9	17	7

Phillips XRF machine (Model 2404). This machine has the ability to detect elements up to a one millionth part (ppm). The samples were crushed into powder and then placed in the machine for testing. The results of the experiments are listed in Tables 2 & 3 below.

#### 5. Discussion

Although our collection for XRD (3 samples) is very small and we could not interpret its results as a reliable and definitive outcome, the existence of montmorillonite in Sample 3 can be considered as an indication of the local production of this pottery. Montmorillonite is the main constituent of bentonite that was a hair-cleaner clay used in traditional healthcare that is also referred to as Neyshaburi or Khorasani clay in the Persian Language. Based on some historical sources, this clay was used not only for cleaning but also for eating (Nuiri, 2007).

As Alikhani, Shamanian and Jafari Zanglanlo have reported in their article, montmorillonite is found south of Neyshabour at Tajroud's geological section (Alikhani, Shamanian and Jafari Zanglanlo, 2014). They also reported that the calcite in this region was created due to hydrothermal alteration. The presence of calcite shows the temperate of the kiln was not above 800°C.

The XRF analysis prepared comprehensive data about the main chemical elements in the pottery samples. This data does not sum to one hundred percent. In the XRF method, the main constitutive elements and their ratios were selected for a better understanding. One of these groups is the proportion of nickel/vanadium and barium/rubidium; these ratios were then compared in the software. The results were put in a chart using the Dplot software (Figure 8). As can be seen, the findings on this diagram show at least one main source (A) and two other possible sub-sources. For more certainty, the results were compared to the ratio of cerium/



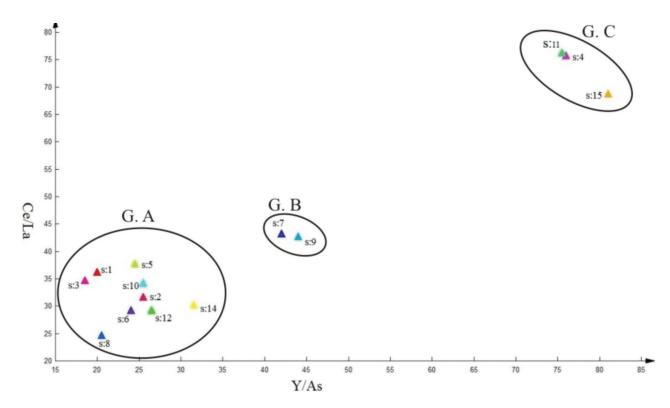


Figure 8. Ratio of Ni/V to Br/Rb elements.

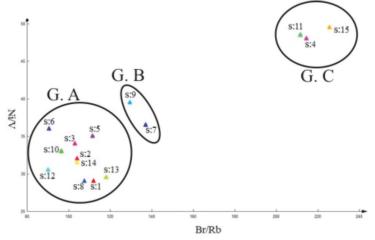


Figure 9. Ratio of Ce/La to Y/As elements.

lanthanum to yttrium/arsenic and again were estimated using the Dplot software which confirmed the results of the previous comparison (Figure 9). The result shows that the same samples that are in group A in Figure 8 are also in group A in Figure 9. This situation is repeated for the other groups.

The results show that the main source was probably located near the site (not to mention inside the site) that was the major supply of the pottery found in the area. However, different clay qualities were also identified in several pottery pieces indicating that some of the pottery was imported as a result of commercial interactions (at the moment, nothing more can be said about them being distant or close interactions).

#### 6. Conclusion

Based on the experiments carried out on the clay samples, the presence of clay mines around the site, and the geological structure of Neyshabur, most of the potteries were local productions. The presence of montmorillonite (bentonite) in Sample 3 that was identified with the XRD testing shows that the sample originated from the Neyshabur geological region.

Assuming the locality of the BMAC potteries as being found in the Shahrake Firouzeh site, one can argue that the significant differences that were found in the composition of samples B and C, though very small, could indicate different sources of clay that were imported from other areas. It is

possible that based on the texture and nature of the area, which is a cemetery, the samples were burial gifts that were placed in the graves by relatives who lived in neighbouring areas. However, the authors consider this idea only a possibility. In future studies, along with the analysis of more BMAC samples, soil samples from the area should be analyzed with the same methods used in this study in order to obtain a more accurate pattern of the possible clay extraction sources. Also, samples should be studied using petrography and thin-section preparation to compare the minerals in the pottery with the geological structure of the area.

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