STRUCTURAL AND LABORATORY STUDY OF A FEW GRAY PAINTED POTTERIES FROM SHAHR-I SOKHTA

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Abstract: Shahr-i Sokhta is one of Iran's biggest prehistoric ancient sites, located in the southeast of Iran, near Zabol. Early settlements of the site date to the 4th millennium BC. Archaeological excavations over the past few decades in this area have revealed the existence of four different settlement periods from 3200 BC to 1800 BC, including eleven cultural layers. The continuation of excavations over the decades has led to thousands of valuable ancient finds with fantastic variety. Valuable cultural findings and discoveries from Shahr-i Sokhta caused the registration of the site as a UNESCO World Heritage in 2014, the 17th Iranian historical piece. Historical pottery is the most numerous findings from the site, consisting of three main groups of buff, red, and grey paste wares. Grey wares are one of the most important pottery findings of the site, which itself classifies into two plain and painted subclasses. In this study, three samples of gray painted pottery discovered from the burned city were studied experimentally using various laboratory methods, such as X-ray diffraction (XRD) and X-ray fluorescence (XRF), to identify the structure and mineralogical composition of these valuable pottery samples, to be determined the importance and impact of the elements and compounds that make up the pottery body in metamorphosis and change their color during the firing stage to gray. Structural study and analysis of the chemical composition of gray pottery samples in this study showed that the presence of silicate phases and the use of clay soils with a high percentage of iron oxide, along with the use of reducing atmosphere and high furnace temperature during firing, are the most important factors in durability and creation of gray color in this group of pottery of the Shahr-i Sokhta has been.
Keywords: Shahr-i Sokhta, grey ware, XRD, XRF.

چکیده: محوطه باستانی شهر سوخته یکی از بزرگترین محوطه‌های باستانی دوران آغاز تاریخی ایران است که در جنوب شرق کشور در استان سیستان و بلوچستان و در نزدیکی شهرستان زابل واقع شده است. تاریخ آغاز سکونت در این محوطه باستانی به هزاره چهارم قبل از میلاد بازمی‌گردد. کاوش‌های باستان‌شناختی در طی چند دهه گذشته در این محوطه، حاکی از وجود چهار دوره استقراری مختلف از ۳۲۰۰ ق.م تا ۱۸۰۰ ق.م است که در مجموع شامل یازده لایه فرهنگی است. تداوم کاوش‌های باستان‌شناسی در این محوطه در طول چند دهه، منجر به کشف هزاران یافته ارزشمند باستانی با تنوع فوق‌العاده شده است. کشفیات و یافته‌های ارزشمند فرهنگی که در طول کاوش‌های باستان شناختی شهر سوخته بدست آمده است، موجب شد که در سال ۱۳۹۳ این محوطه باستانی به عنوان هفدهمین اثر تاریخی ایران در فهرست میراث جهانی یونسکو به ثبت برسد. فراوان‌ترین یافته‌های فرهنگی که از این شهر باستانی در طول کاوش‌های باستان‌شناسی بدست آمده، سفالینه‌های تاریخی است که شامل سه گروه اصلی: نخودی، قرمز و خاکستری است. یکی از مهمترین انواع سفال‌های بدست آمده از شهرسوخته، سفالینه‌های خاکستری است که می‌توان آنها را به دو گروه ساده و منقوش طبقه‌بندی نمود. در این پژوهش سه نمونه از ظروف سفالی خاکستری منقوش مکشوفه از شهرسوخته به روش تجربی و با استفاده از روش‌های آزمایشگاهی مختلف، همچون: پراش پرتوی ایکس، فلورسانس اشعه ایکس، مورد بررسی و مطالعه قرار گرفته است تا ضمن شناخت ساختار و ترکیب کانی شناختی این نمونه‌های ارزشمند سفالی، اهمیت و تاثیر عناصر و ترکیبات تشکیل دهنده بدنه سفال در دگرگونی و تغییر رنگ بدنه در طی مرحله پخت به رنگ خاکستری روشن و مشخص گردد. مطالعه ساختاری و آنالیز ترکیب شیمیایی نمونه سفال‌های خاکستری در این پژوهش، نشان داد که وجود فازهای سیلیکاتی و استفاده از خاک‌های رسی با درصد بالایی از اکسید آهن به همراه استفاده توامان از اتمسفر احیاء و دمای بالای کوره در هنگام پخت، مهمترین عوامل موثر در دوام و ایجاد رنگ خاکستری در این گروه از سفال‌های شهر سوخته بوده است.
کلمات کلیدی: شهرسوخته، سفال خاکستری، آنالیز سفال، پراش پرتوی ایکس (XRD)، فلورسانس اشعه ایکس (XRF).

I. Introduction

Shahr-i Sokhta archaeological site, which is one of the largest archaeological sites in the southeastern part of Iran, is located in Sistan and Baluchestan province, near the city of Zabol, which is a large site with an irregular design (Tosi, 1976, 1983). At the end of the Chalcolithic Age, between the years 3200-4000 BC, in this region, thanks to the Helmand River and due to the favorable climatic conditions, the Indo-European people founded a city with an area of about 5 hectares that we know today as Shahr-i Sokhta (Costantini, 1979: 88).

Archaeological excavations over the past few decades in this area showed that the area of this ancient city is 151 hectares (Seyed Sajjadi, 1995: 169) and has four different settlement periods from 3200 BC to 1800 BC. It includes a total of eleven cultural layers (Tosi, 1973: 68-80). Continued archaeological excavations in this archaeological site over several decades have led to the discovery of thousands of valuable archaeological finds of great diversity, which the discovery of large volumes of pottery and stone artifacts and pieces of marble and Lapis lazuli indicate that this ancient city was the center for the construction and distribution of such works. The geographical location of Shahr-i Sokhta on the route of trade and commercial routes (Tosi, 1978: 55), has caused a tremendous impact of this important ancient site on the ancient areas around it.

The commercial and cultural relations of Shahr-i Sokhteh with its neighbors in different areas can be well observed and studied according to the archaeological artifacts and findings obtained in these areas. These discoveries indicate that Shahr-i Sokhta has connections with its foreign neighbors, in southern Turkmenistan southern Afghanistan, and Pakistan (Biscioneh, 1974: 134). It is also communicating with its internal neighbors, such as Bampur in Baluchistan, the ancient site of Tappe Yahya (Lamberg-Karlovsky and Tosi, 1989: 21), and Shahdad.

Valuable cultural discoveries and findings were obtained during the archaeological excavations of Shahr-i Sokhta. In 2014, this ancient site was registered as the seventeenth historical monument of Iran on the UNESCO World Heritage List. Historical pottery is the most abundant culture found in this ancient city during archaeological excavations, which includes three main groups: buff, red, and grey. One of the most important types of pottery obtained from Shahr-i Sokhta is grey ware, which can be classified into two groups: plain ware and painted ware.

Buff-colored ware and grey ware can be found in all settlement periods of Shahr-i Sokhta, usually in various forms, including cups, bowls, and jars, but redware, to a limited extent, is sometimes found in excavations. (Seyed Sajjadi, 2009: 192)

One of the most important indicators of the Old and Middle Bronze Ages in the plateau of Iran in the field of pottery is the emergence and popularity of grey ware in the historical geography of Iran (Talaei, 2009: 49). Grey ware, based on the historical period, can be classified and divided into two different groups as follows: The first group: is grey ware of the third millennium BC, which is called Yanighi grey ware, and the second group: is grey ware of the first millennium BC, which is known as the grey ware of the Iron Age (Karimi Mansoub and Mohammadifar, 2019: 40).

Shahr-i Sokhta grey wares are a special type of pottery mostly made in bowls, small cups, and horned vessels. The grey wares of this ancient site are sometimes severely burned and blackened. In Shahr-i Sokhta, almost all the grey wares made in the shape of a bowl are painted, while among cups of this group of pottery, there are also unpainted vessels. In the group of grey bowls, usually inside and outside bowls were decorated with beautiful designs (Seyed Sajjadi, 2009: 192). There are various theories about the method of making grey wares and how to process them, which can be mentioned in a general summary of these theories as follows. The reason for the grey color of the body of these wares is their different firing method compared to buff and red wares. In other words, the firing process of these wares is in the atmosphere of reduction condition, which causes the color of the body of this ware to change from light grey to black .

Majidzadeh considers the cause of the greying of these wares as the presence of elements such as oxygen, iron, and carbon in the structure of pottery (Majidzadeh, 1991: 7-9), while the presence of these elements in all pottery is obvious because the source and the raw material for making all pottery is clay, which is rich in these elements. However, Kambakhsh Fard considers the main reason for the greying of the body of these wares to be the firing of pottery in the conditions of reduction (Kambakhsh Fard, 2010: 296).

Other studies on greyware have shown that Bronze Age grey ware is very different from Iron Age specimens. The study of grey ware of these two groups in comparison with each other shows that the greyware of the Iron Age in the first millennium BC in terms of firing was more developed in terms of reduction and had better and more complete firing in the atmosphere of furnace reduction than Bronze Age grey ware in the third millennium BC, which was the first examples and early experiences of potters in achieving this type of pottery (Karimi Mansoub and Mohammadifar, 2019: 41).

II. Materials and methods

In this applied research development, experimental-analytical research methods have been used, and research data collection has been collected using various techniques, such as library study, field study, and laboratory study. In this study, three samples of gray painted pottery discovered from the burnt city (Fig. 1-6), experimentally and using various laboratory methods, such as X-ray diffraction (XRD) and X-ray fluorescence (XRF), were examined and studied to identify the structure and mineralogical composition of these valuable pottery specimens, to be determined of the importance and impact of the elements and compounds that make up the pottery body in metamorphosis and their color changes during the baking stage in the kiln in the conditions of reduction in gray color.

III. Results

The results of examining samples of greyware discovered from Shahr-i Sokhta by various laboratory methods and analyzing the structure and chemical composition of these potteries in various instrumental methods are as follows:

IV. External observations and sampling

The pottery studied in this research included three samples of greyware discovered from Shahre-i Sokhta, two of which were in the shape of a semicircular bowl and one in the shape of a small jar (Fig. 1-6). The motifs of these wares are geometric and symbolic, and their use was probably ritual. The color used to decorate the pottery with the mentioned patterns is dark brown. Due to the discovery of these pottery wares from the area of Shahr-i Sokhta cemetery and their location under the soil for thousands of years, as well as environmental conditions, burial in the cemetery, the soil pressure resulting from burial, and the presence of soluble salts in the soil of the burial environment, over time and the effect of various environmental damaging factors on these wares, various damages in various forms are observed, including ware fractures, deficiencies, surface paleness of the ware, and salt deposition. In Table 1, the specifications of the pottery wares are written. To study the pottery in the laboratory, very small samples were taken from its different parts to be studied and examined with the help of device analysis methods. To study the structure and mineralogical composition of the pottery body, as well as elemental analysis of the chemical composition of the pottery, a small amount of sampling was selected. The body color of gray pottery number one and number two was slightly lighter, but the third gray pottery was darker. The texture and structure of the body in all three pottery were perfectly cohesive and very hard, and this hardness was slightly higher in the third sample. All three grey ware were made by wheel-making. In the body of all three samples, soft and fine-grained sand was used as a filler or temper, and the thickness of the body in all three pottery samples, especially in bowls number two and three, was very low.

Figure 1 and 2. Greyware number one and details of geometric patterns on the shoulder of the vessel.

Figure 3 and 4. Side view of the grey bowl number two and its upper view and the designs inside the ware.

Figures 5 and 6. Side view of the grey bowl number three and its upper view and the designs inside the ware.

Table 1. Characteristics of the studied grey ware, discovered from Shahr-i Sokhta.

		Grey ware number one	Grey ware number two	Grey ware number three
Ware Code		86 Trench NHF surface (2)	76 Trench 2703(2)	81 Trench 3400(6)
Place of discovery		Shahr-i Sokhta Cemetery	Shahr-i Sokhta Cemetery	Shahr-i Sokhta Cemetery
Year of discovery		2007	1997	2002
Trench number and code		Trench NHF	Trench HRT	Trench NFH
Shape of the dish		Small jar	Bowl	Bowl
Type of ware		Painted grey ware	Painted grey ware	Painted grey ware
Type of motif		Geometric	Geometric and symbolic	Geometric and symbolic
Place of motif		The rim and shoulder of the vessel	Inside and outside the bowl	Inside and outside the bowl
Dimensions of the vessel in millimeters	height	142	90	95
	Rim diameter	101	186	185
	Maximum diameter	165	180	181
	Base diameter	65	64	53
Pottery shaping method		Wheel maker	Wheel maker	Wheel maker

V. Structural study of pottery body by X-ray diffraction (XRD) method

To identify the mineralogical composition and phases in the body of grey wares studied in this research and to analyze their structure, samples taken from the body of each grey ware discovered from Shahr-i Sokhta were tested and analyzed by X-ray diffraction (XRD). In Fig 7-9 and Table 2, the results of the analysis of the pottery body are presented by using this method of instrumental analysis and diffraction spectra of the samples.

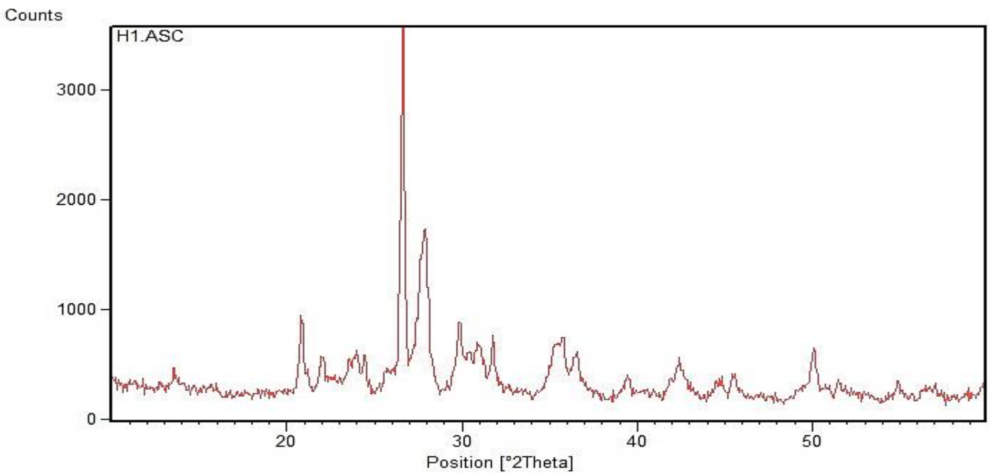

Figure 7. X-ray diffraction spectroscopy (XRD) of grey ware number one.

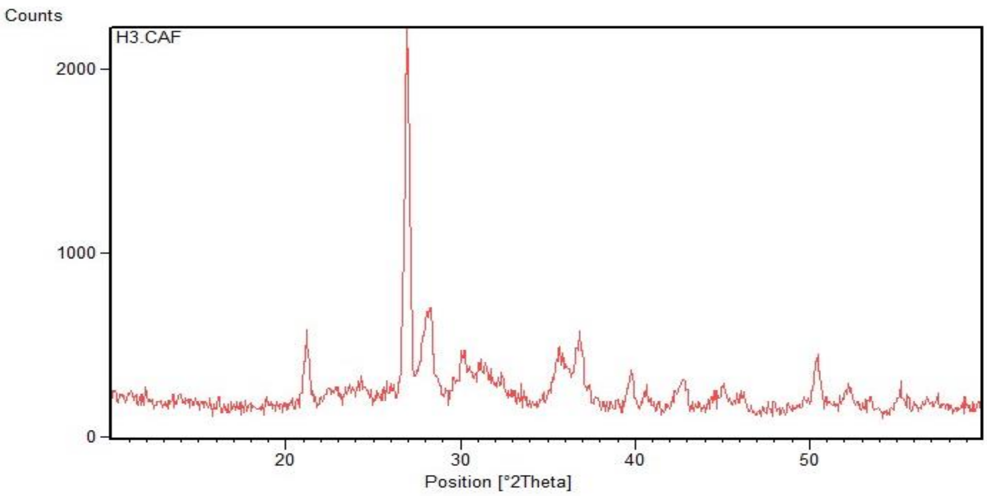

Figure 8. X-ray diffraction spectroscopy (XRD) of grey ware number two.

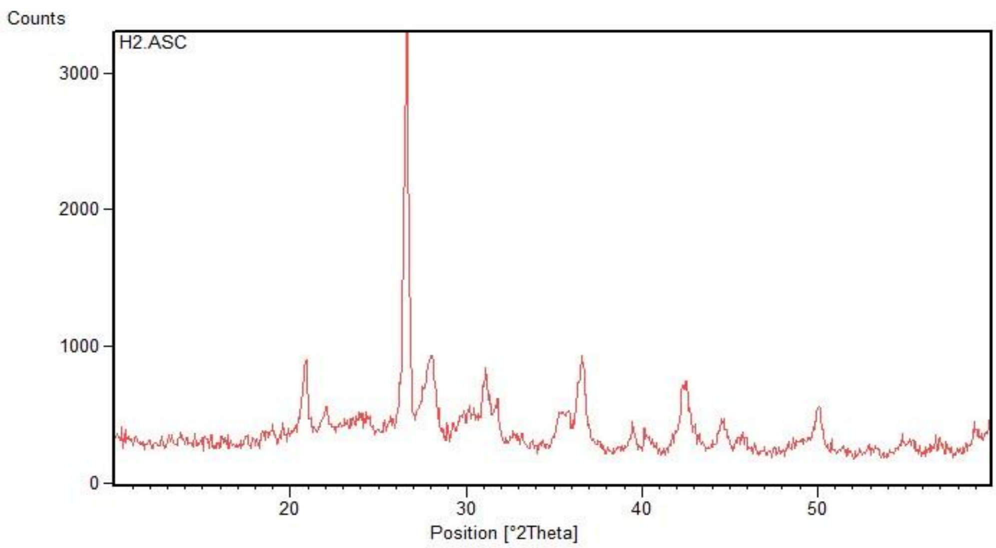

Figure 9. X-ray diffraction spectroscopy (XRD) of grey ware number three.

Table 2- Results of a structural study of grey ware body of Shahr-i Sokhta by X-ray diffraction (XRD) method.

Pottery number	Mineralogical composition and identified phases	Chemical properties of phases
Grey ware number one	Quartz	SiO_2
	Albite	Na Al Si_3O_8
	Anorthite	$CaAl_2$ Si_3O_8
Grey ware number two	Quartz	SiO_2
	Anorthite	$CaAl_2Si_2O_8$
	Augite	$CaMg(Si_2O_6)$
	Albite	$CaAl_2Si_3O_8$
Grey ware number three	Quartz	SiO_2
	Enstatite	$Mg_2(Si_2O_6)$
	Ferrosilite	$FeSiO_3$

The study of the mineralogical structure of three samples of grey ware discovered from Shahr-i Sokhta by the X-ray diffraction method shows that the body structure of these three types of pottery is very similar to each other such that, in all three samples, according to the results obtained in Table 2, common silicate phases were observed. The most important phases identified in Pottery Number One are Quartz, Albite, and Anorthite, and in Pottery Number Two, the Augite phase has been added to these three previous phases. In pottery number three, in addition to quartz, we encounter phases of Enstatite and Ferrosilite, which also have a silicate structure (Table 2).

VI. Analysis of the chemical composition of pottery body elements by X-ray fluorescence (XRF) method

To identify and analyze the qualitative and quantitative composition of the elements of the body of grey wares discovered in Shahr-i Sokhta, studied in this paper, and to study their chemical composition, by X-ray fluorescence (XRF), samples taken from the body of each grey ware were analyzed. Tables 3 and 4 present the results of the pottery body analysis with this device element analysis method.

Table 3. Results of elemental analysis of the body of grey wares of Shahr-I Sokhta by X-ray fluorescence (XRF) method (wt. % oxides).

wt.%	Grey ware number one	Grey ware number two	Grey ware number three
SiO_2	56/635%	59/539%	60/361%
Al_2O_3	12/34%	12/182%	14/584%
Fe_2O_3	7/259%	7/105%	7/697%
CaO	4/949%	3/022%	5/594%
Na_2O	3/185%	1/972%	2/025%
MgO	3/88%	3/755%	4/441%
K_2O	2/275%	2/562%	2/14%
TiO_2	0/7%	0/722%	0/698%
MnO	0/074%	0/053%	0/107%
P_2O_5	0/102%	0/117%	0/109%

Table 4. Results of elemental analysis of trace elements in the body of grey wares of Shahr-i Sokhta by X-ray fluorescence (XRF) method.

	Grey ware number three		Grey ware number two		Grey ware number one	
Cl	-------	ppm	720	ppm	939	ppm
S	364	ppm	N	ppm	707	ppm
As	9	ppm	17	ppm	8	ppm
Ba	290	ppm	193	ppm	356	ppm
Ce	83	ppm	63	ppm	110	ppm
Co	15	ppm	17	ppm	18	ppm
Cr	95	ppm	110	ppm	153	ppm
Cu	747	ppm	3179	ppm	N	ppm
Nb	N	ppm	N	ppm	N	ppm
Ni	94	ppm	153	ppm	140	ppm
Pb	82	ppm	178	ppm	56	ppm
Rb	84	ppm	88	ppm	75	ppm
Sr	240	ppm	95	ppm	158	ppm
V	76	ppm	72	ppm	78	ppm

The results of elemental analysis of the chemical composition of the body of grey wares tested by X-ray fluorescence (XRF) showed that results of elemental analysis of the chemical composition of the body of grey wares tested by X-ray fluorescence (XRF) showed that the clay soil used to make the body of these gray pottery was of very high quality and the amount of impurity low. As the results of the analysis show, the average amount of aluminum oxide ($Al2O3$) in the body of all three samples of grey pottery wares is above 12% (Table 3). On the other hand, the presence of a high percentage of silica ($SiO2$), 56 to 60% in the body of this pottery, has been caused by baking the body of gray pottery in a high-temperature furnace, by sintering and creating a phase Glass, gray pottery body finds great strength, hardness, and durability. The results of qualitative and quantitative analysis of chemical elements in the grey wares of Shahr-i Sokhta indicate that to reduce the firing temperature, calcium oxide, sodium oxide, magnesium oxide, and potassium oxide have been used in the composition of the body of these pottery wares. Calcium oxides with 3 to 5 weight percent and sodium and potassium oxides with 2 to 3 weight percent, respectively, have the highest and lowest levels (Table 3). Calcium oxide, in addition to being present in the pottery body as a flux, in combination with silica oxide in the body of gray pottery, has increased the strength and durability of the pottery body. Also, the presence of about 7% by weight of iron oxide indicates a high percentage of iron cation III in the body of grey ware (Table 3).

The importance of a high percentage of iron cation III in the structure and chemical composition of gray pottery is very important and key due to the important role of this element in the process of darkening and blackening the pottery color during firing in the kiln, which is due to the placement of pottery paste in the reduction in atmospheric conditions as well as the conversion of red iron oxide III ($Fe2O3$) to black iron oxide II (FeO).

VII. Discussion

Quartz mineral is observed in the structure of all three pottery samples. It is one of the most abundant minerals that can be seen in the texture of historical pottery, which is either in the form of gravel or sand in the soil used to build the pottery body. Its presence in the pottery body may be accidental or intentional and conscious as an additive to improve the composition of the pottery body (Sterba *et al.*, 2009: 1583). Due to the high percentage of silica in the body of all three studied gray pottery, based on the analysis of the chemical composition of their body, it seems that the use of a high percentage of silica in the body of these gray pottery, intentionally and consciously to strengthen and their body has been more durable after firing. On the other hand, the study of the mineralogical composition of three samples of grey wares pottery shows that due to the absence of calcite phase in their structure, the firing temperature of these samples should be more than 800 degrees Celsius. Calcite mineral is very important in the "study" of historical pottery, as it disappears at 800 ° C (Emami and Trettin 2010; Rathossi and Pontikes 2010). Other high-temperature phases in the composition of the body of these pottery wares can be referred to as the anorthite phase, having an index peak of 2θ between 27 to 30 degrees in the graph, which is observed in the diffraction spectrum of samples of grey wares number one and two.

The spectra obtained from diffraction analysis in all samples of grey wares studied from Shahr-i Sokhta indicate the presence of a quartz phase that, according to the quartz phase exchanges at different temperatures, namely alpha quartz and beta quartz, the former is stable below 573° C and the latter between 573° C and 870 ° C. Due to the absence of high-temperature phases of quartz such as tridymite and cristobalite, it can be said that the firing temperature of these samples of grey wares should be 850 degrees Celsius. On the other hand, in the body of pottery, there exist albite and anorthite feldspar minerals. Considering the stability of the albite phase in the monoclinic system at 980 ° C and also the presence of the enstatite phase that is stable up to 985 ° C, it can be said that the firing temperature of this sample of grey ware is probably about 950 ° C.

Qualitative and quantitative analysis of the composition of the body elements of grey wares by X-ray fluorescence method shows a high percentage of silica and alumina with important flux compounds such as calcium, magnesium, potassium, and sodium oxides in the body composition of three samples of grey wares discovered from Shahr-i Sokhta. However, iron oxide III plays a very key and important role among all the elements identified in the body composition of the studied grey wares. The presence of a high percentage of iron oxide III in the composition of the body of all samples that is above 7%, along with the above-mentioned flux agents, at a temperature above 900 ° C, hard and solid structure in the form of grey wares in the conditions of reducing firing in the kiln, has created. In addition to the grey to black color of the pottery body due to chemical reactions caused by the conversion of red iron oxide at a temperature above 850 ° C to black iron oxide II, the presence of iron causes the durability and strength of such pottery.

VIII. Conclusion

The study of the mineralogical structure and chemical composition of the elements of the body of three samples of gray pottery discovered from Shahr-i Sokhta, by various methods of instrumental analysis based on the use of X-rays, showed that the structure and crystal composition of minerals in all three samples of gray pottery studied in this research is similar to each other and has similar phases. This similarity indicates the use of the same resources and earth mines in supplying the necessary raw materials for constructing this ancient pottery in Shahr-i Sokhta. The most important minerals identified in the gray pottery samples studied in this research are silicate phases that can be justified according to the geological structure of the Sistan Plain and alluvial sediments from water resources in the region. The results of elemental analysis of the chemical composition of the body of grey ware have good coordination and compliance with the results of diffraction analysis and minerals identified in the structure of the pottery, and according to the percentage of different elements in the body of samples, it can be said that the soil used as a raw material for making these gray pottery had very few impurities and had a good quality for preparing the pottery produced in this ancient city. Considering the structural study of the pottery body Sokhta by the X-ray diffraction method and the presence of high-temperature phases in the structure of pottery, such as albite feldspar minerals next to quartz and the absence of calcite phase, it seems that the firing temperature of these samples of grey wares was probably 950 degrees Celsius. Therefore, according to the results of laboratory studies performed on the body of the samples of grey ware discovered from Shahr-i Sokhta, it was clear that firing this pottery at such a high temperature has led to the high strength and durability of the grey ware body that due to the presence of effective flux compounds, such as calcium oxide, sodium oxide, magnesium oxide, and potassium oxide in the structure of the body of grey wares, has been possible. Laboratory studies have also shown that the presence of iron compounds, such as iron oxide III in high amounts (above 7% by weight) in this pottery body during firing in the kiln, has been a key point in their manufacturing and processing techniques. The potters of Shahr-i Sokhta fired this pottery in special pottery kilns with a reduction atmosphere at a temperature above 900 degrees Celsius. During firing, chemical exchanges and reactions under reduction conditions in the kiln were completed by converting red iron oxide III to black iron oxide II, the process of gray and blackening of the paste of this pottery. Although in the process of making gray pottery, such as discovered samples from Shahr-I Sokhta, the atmospheric conditions of the pottery kiln are very important, this study showed that in addition to the reduction conditions during firing in the kiln, the presence of a high percentage of iron compounds in the body of pottery products also seems necessary to make gray pottery. It can be said that in the Bronze Age in Shahr-i Sokhta, due to the existence of large and numerous industrial workshops that were engaged in the production of pottery products, the local potters of Shahr-i Sokhta, based on their experiences over the years, had found out for what purpose to use each of the rich clay mines and deposits in the Sistan Plain to produce what kind of pottery products.

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