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Studies

ARCHAEOLOGY

COMPOSITIONAL ANALYSIS ANALYSIS OF THE POTTERY SHARDS FROM KUH-I KHAWJA HISTORICAL SITE, SISTAN, EAST OF IRAN

Abstract: The aim of this study was to determine whether pottery shards from Kuh-i Khawja were locally made or imported. Kuh-I Khawja is one of the most ancient settlements in Iran's Sistan during the Sassanid period. The study shows that the antiquity of the site goes back to the 3rd to 8th centuries CE and the earthenware found in Kuh-I Khawja can be categorized into four groups which are i) Plain unglazed pottery; ii) Unglazed painted pottery; iii) Plain glazed pottery; iv) Glazed and painted pottery. Archaeologists believe that most of the pottery shards are locally made; hence, to test this hypothesis, a scientific analysis was done to determine the chemical composition of the pottery shards. X-Rays Fluorescence (XRF) was applied to determine the major and trace elements of the pottery shards. The results demonstrate that most of the pottery shards are in the same group and this strongly suggests that they are local products. Additionally, based on the major and trace elements, it can be suggested that some of the samples were imported items.
Keywords: *Kuh-I Khawja, Sistan, Sasanian, X-Rays Fluorescence (XRF).*

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INTRODUCTION

Sistan is located in a vast territory in the south east of Iran, and a large part of it today is in Afghanistan. The Sistan region, along with Baluchestan, constitutes the Sistan and Baluchestan Province of Iran[1]. Kuh-I Khawja is the one and only elevated condition in Sistan plains, located 30 kilometers away from the city of Zabol (Map 1). The complex consists of Islamic and pre-Islamic structures, which are considered to be Iran's most prominent adobe monuments[2].

Like other ancient Iranian Lands, the foundation of Sistan is also rooted in myth and history. Kuh-I Khawja is only mountain on the extensive plain of Siatan that has had a special sanctity for at least three religions, Zoroastriannism, Christianity and Islam. On the other hand, the natural attractions of Hamun Lake, with its green grass, favorable weather and the possibility of hunting different kinds of birds and aquatic animals has attracted the attention of rulers and powerful people. The complex consists of Islamic and pre-Islamic structures, which are considered to be Iran's most prominent adobe monuments.

[1] MORADI *et alii* 2013, MORADI *et alii* 2014; SARHADDI-DADIAN 2015; SARHADDI-DADIAN 2017a; SARHADDI-DADIAN 2017b; SARHADDI-DADIAN 2020; MUTIN 2017.
[2] SARHADDI-DADIAN 2013.

ARCHEOLOGICAL BACKGROUND

The first studies conducted on Kuh-I Khawja were mostly geological surveys, which was conducted by a British military officer, Goldsmid in the second half of the 19th century. But the first serious step in identifying was taken by the Hungarin-English archaeologist Aurel Stein in 1915, he included the results of his studies and excavation in his well-known work, Innermost Asia[3]. After that, Ernst Herzfeld, German archaeologist continued to explore the Kuh-I Khawja in 1925, he visited the site and, four years later, returned with a board to study and survey this place. His final work was published in 1941 in the book Iran in the Ancient East[4]. The third scientific work, after Herzfeld"s work at Kuh-I Khawja, was conducted by Italian board supervised by Giorgio Gullini in 1961, whose results were published in a book Kuh-I Khawja[5].

The fourth scientific archaeological work was undertaken by an Iranian team , and since then, Iranians have been able to bring the exploration on Kuh-I Khawja under own control. Initially three short-tern exploratory excavation 1982-1987 under the supervision of Seyyed Mahmoud Mousavi were conducted with the aim of training students in archaeology. In 1997 the archaeological team of Shahri Sukhta, let by Syyed Mansour Seyyed Sajjadi worked on this complex but unfortunately, the related reports have not been published yet. Then Sorur Ghanimati studied and researched the ancient palace of this mountain, and the latest research has been done in August 2018 by authors.

According to architectural evidence, Herzfeld assumes that Kuh-I Khawja palace at least two construction phases. He argues that the first stage relates to in the 1th century CE, and the second phase relates to the 3th century CE, the early Sasanid Dynasty. After more than three decades, Gollini started to work in this complex and introduced six consecutive layers of settlement from the Achaemind period to the Islamic era. Of cource, archaeologists did not accept Gollini's dating and he was faced with a wave of criticism.

Based on ^{14}C tests, Sorur Ghanimati attempted to provide an absolute date for the entire sites. She collected the samples from two distinct areas; the first sample was obtained from mortar and other structural materials belonging to the ceiling of the gallery, and the second sample was taken from a wooden nail used in bas-relief in the southern façade of the fire abattoir. These samples determined the dates of 240-80 ± 50 CE and 450-550 ± CE respectively[6].

In the last decade and after the systematic study of Sistan and consequently, the Kuh-I Khawja site, based on extensive studies on pottery data, dealt with classification and the collected pottery samples. According to the obtained results Kuh-I Khawja palace has three different settlement periods. The first period includes the Parthian era, the second period is related to the Sasanid period, and the third period is attributed to the 6th- 8th centuries CE, which are related to the Islamic era.

In the last season of the exploration on external wall of the site has been done by authors in this part 4 Cone-shaped tower were identified throughout the fence. Although it seems that the total number of the watchtowers had been more that the identified ones, only the remnants of these fourth towers were discovered and the rest of towers were destroyed in the course of time. The structure of these towers is uniform; they have been built not based on a determinate order in the external part of the wall with a semicircular plan and in a conical shape. The structure of these towers is uniform, and they are located on the external part of the wall in not so orderly form[7]. The thickness of the tower in the base was almost 2 and half meter and as the height of the tower increases this thickness gets lower in so far as in the highest part. The general form of the tower was semicircular and like the structure of the fort a clay wall has been used on the foundation and in higher levels of the tower, adobes might have been used. Inside this clay wall has been filled with mud, dust and other stuffs. In other words, the tower itself was not built fully of the clay wall rather its external body as well as its lower part has been built from the pure clay wall and then the clay and adobe have been used as the thick crust of the tower and its inside has been filled. These towers had been used as supporting walls for the fort. there was an entrance with narrow width of 50 to 60 cm from the top of the wall into the small internal space of the tower and the remnants of this entrance can only be found in the tower number 1, i.e. the far eastern tower in the fort. Tower number 1 is enumerated as the most complete discovered tower from the point of view of the structure and from the other third towers only part of the remnants and structure exists that generally includes the lower parts. But

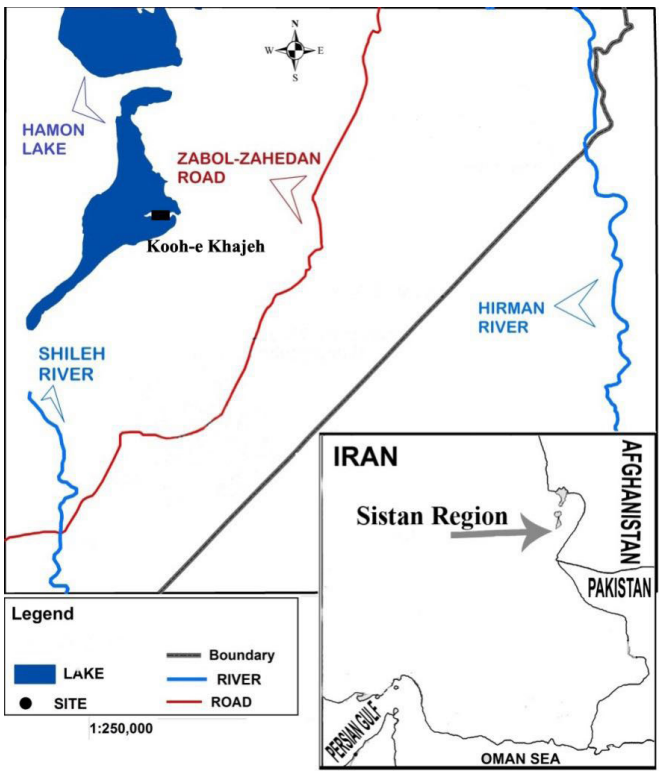

Fig. 1. Location of the Site in Sistan Region (SARHADDI-DADIAN 2017)

[3] STEIN 1937.
[4] HERZFELD 1941.
[5] GULLINI 1964.
[6] GHANIMATI 2001, 62.
[7] SARHADDI-DADIAN *et alii* 2019.

we can reconstruct the plan and body of other towers based on the structure of the eastern tower number1[8].

KUH-I KHAWJA POTTERIES

In Kuh-I Khawja Site; According to coats, being plain or painted, pottery can be categorized into four groups: 1-. Plain unglazed pottery. They are with no design and decoration. In the category, shreds clay is red, red brown, buff, brown, orange, and gray. The slip is buff-colored, red brown, brown, orange, or grey as well. 2-. Unglazed painted pottery. In the category, slip is clayey and the clay itself is red, orange, grey, dark brown, or buff. The pottery of this category can be subdivided into three groups: a. Incised designed pottery b. Burnished designed pottery c. Polychrome decorated pottery. The decoration themes in this category are of geometrical type. 3-. Plain glazed pottery. In this category, clay is red brown and buff while potteries are glazed in green, blue, and transparent. The potteries are kilned enough while the clay is dense and solid. 4-. Glazed and painted pottery. The category is subdivided into two subcategories, according to decoration: a. pottery with under glazed incised decoration b. Pottery with under glazed paintings meanwhile, according to color it is subdivided into green and turquoise groups. In this category, decoration themes are of geometrical and floral type[9].

SAMPLE PREPARATION

For the analysis (picture 1), in order to determine the chemical composition of the pottery, each sample of weight 0.7 g was pulverized, heated up at a temperature of 105°C for one hour and mixed until homogenous with the flux powder, a type of Spectroflux 110 (product of Johnson & Mathey).

These mixtures were baked for one hour in a furnace with a temperature of 1100°C. The homogenous molten material was molded in a container and cooled gradually into pieces of fused glass with a thickness of 2 mm and a diameter of 32 mm. The samples were of 1:10 dilution. Press pallet samples were prepared by mixing 1.0 g of samples together with 6.0 g of boric acid powder; then, a pressure of 20 psi (137.895 kPa) was applied using hydraulic pressure equipment. The samples of fused pallets and pressed pallets were analyzed by wavelength-dispersive X-Ray Fluorescence (WD-XRF). A Philips PW1480 sequential spectrometer fitted with a rhodium-anode X-Ray tube (3kW 60kV) was used for the analysis of major and trace elements. The spectrometers were controlled using Philips X40 application software package version 3.2 and 4.01 running under the DEC VMS operating system.

Scatter plot diagrams of CaO versus K_2O and Rb versus Sr, were then performed to demonstrate the differences among the groups and was analyzed using Microsoft Excel software. The main purpose was to see the distribution of the samples in the group. Hierarchical Cluster Analysis (HCA) was applied to the chemical data from the three components, namely calcium oxide (CaO), potassium oxide (K_2O), rubidium (Rb) and strontium (Sr). All the 17 pottery shards were sampled in order to verify the presence of compositional groups of pottery shards differentiated by their probable major element sources. The measurement of distance used in the assignment rule was based on Ward's Linkage and the Squared Euclidean Distance algorithm. The results are presented in the form of a dendrogram (Figures 5 and 6), showing in graphical form the distance between the pottery samples on the basis of their CaO and K_2O percentage and Rb and Sr concentration. The applicability of the analytical methods for the multi-elemental analysis of the pottery shards by XRF was evaluated using an analysis of certified reference material, 315 Fire Brick (Calibration: G_FBVac28mm) for major elements.

RESULTS AND DISCUSSION

XRF

The X-ray fluorescence analysis of the 17 pottery shards samples was conducted to determine the major elements content in the pottery shards from Kooh-e Khajeh (Figure 2). In the compositional analysis, each compositional

Fig. 2. Sample of potsherds

[8] SARHADDI-DADIAN *et alii* 2019; MIRI *et alii* 2020.
[9] BANIJAMALI *et alii* 2016, 46-47.

Sample	SiO2	Al2O3	Fe2O3	CaO	Na2O	MgO	K2O	TiO2	MnO	P2O5	LOI
	Dry Weight (%)										
Kh.1	59.35	14.47	7.56	5.41	2.04	4.06	2.82	0.69	0.10	0.33	3.17
Kh.2	57.98	13.90	6.94	8.06	1.43	6.05	2.50	0.65	0.09	0.13	2.29
Kh.3	56.61	13.34	6.71	10.27	1.78	5.19	2.26	0.61	0.10	0.13	3.01
Kh.4	60.12	13.50	6.08	6.94	2.47	5.92	2.11	0.59	0.10	0.13	2.03
Kh.5	59.07	13.63	7.52	7.25	1.27	4.08	2.64	0.66	0.11	0.22	3.56
Kh.6	54.32	13.93	5.91	9.30	3.03	4.85	2.39	0.55	0.09	0.21	5.41
Kh.7	57.48	14.63	6.68	7.45	2.11	5.28	2.57	0.66	0.10	0.12	2.92
Kh.8	57.24	13.68	6.73	6.10	2.51	5.68	2.71	0.63	0.09	0.18	4.44
Kh.9	56.93	15.08	7.29	5.75	2.40	4.48	2.89	0.68	0.10	0.43	3.98
Kh.10	55.14	14.33	7.70	10.40	1.91	5.30	2.51	0.59	0.11	0.15	1.82
Kh.11	55.55	14.05	6.45	7.81	2.11	4.70	2.58	0.65	0.09	0.13	5.86
Kh.12	54.82	15.21	6.42	11.45	2.51	5.57	1.27	0.60	0.09	0.11	1.94
Kh.13	58.81	15.06	6.27	7.85	1.38	5.03	2.55	0.67	0.09	0.12	2.18
Kh.14	58.30	14.61	7.31	7.27	1.26	4.25	2.75	0.69	0.09	0.11	3.36
Kh.15	59.03	14.63	7.14	5.46	1.83	4.01	2.62	0.67	0.11	0.59	3.90
Kh.16	56.46	14.77	7.49	7.65	1.60	4.86	2.59	0.67	0.11	0.12	3.68
Kh.17	54.73	14.47	8.73	7.46	1.56	4.85	2.50	0.63	0.12	0.11	4.81

Fig. 3. Major elements of pottery shards from Kuh-i Khawja

group was taken to represent locally manufactured pottery based on the assumptions of the Provenance Postulate and the Criterion of Abundance. The Provenance Postulate assumes that compositional differences among different sources are greater than differences within a single source, and that these differences can be recognized by the analytical or compositional approach[10]. The Criterion of Abundance, on the other hand, holds that a ceramic unit (in this case, elemental abundance) strongly represented at a site, can be presumed to be a local manufacturer, while those scarcely represented are non-local[11].

Table 1 shows the content of major element of the pottery shards taken from several potential archaeological sites in Kuh-I Khawja. The pottery samples show quite homogeneous composition except for three samples Kh.3, Kh.10 and Kh. 12 which had higher content of calcium. The range of the silica dry weight percentage for shards categories as prehistoric and historical period is from ~54 to 60%. Content of aluminum is from ~13 to ~15%. Content of calcium and iron are from 4 to 11% and 6 to 8%, respectively. Alkaline element such as magnesium, sodium and potassium shows content of dry weight percentage from 4 to 67, 1 to 3 and 1 to 2.89%, respectively. The percentage of P_2O_5 which is average in every shard indicates that none of the shards have been used as a container for some organic materials. The high percentage of CaO in the shards shows that potters in Kuh-I Khawja site used calcareous clays as their main resources.

The content of trace elements is shown in Table 2. The result clearly shows that the pottery samples have quite homogeneous composition of trace elements except for three samples Kh.11 ans Kh.12 which had lower content of copper. The amount of lead in the pottery shards also low when compared with Shahr-I-Sokhta pottery shards which one of the pottery shard has a high content of lead [12]. Lead is usually added as a coloring agent into the pottery by ancient potters and archaeological research confirm that the ancient community in Indus Valley always used lead as a colorant[13]. Figure 2 show a scatter plot of Rb and Sr which shows that there were also two major group of pottery shards. One group is considering a local production pottery or came from the same resources. One group is considering non-local pottery shards or came from different raw material.

SCATTER PLOT AND CLUSTER ANALYSIS

Figure 1 show a scatter plot of CaO and K_2O which shows that there were two major group of pottery shards.

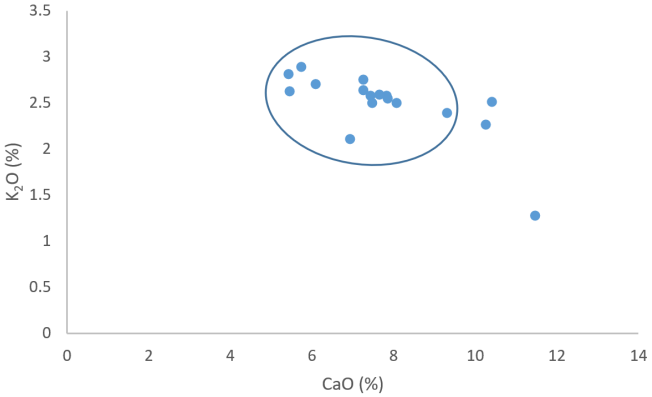

Fig. 4. Scatter plot graph of CaO and K_2O percentage of pottery shards from Kuh-i Khawja

[10] WEIGAND *et alii* 1977.
[11] BISHOP *et alii* 1982; HOSSEIN *et alii* 2015, 2017; RAMLI *et alii* 2017, 2018; RAHMAN *et alii* 2019
[12] MORADI *et alii* 2013.
[13] CALEB 1991.

Sample	Cl	S	As	Ba	Ce	Co	Cr	Cu	Ni	Pb	Rb	Sr	V	Y	Zr	Zn
	Concentration (ppm)															
Kh.1	3803	1527	13	350	97	19	127	736	141	75	120	339	84	46	175	562
Kh.2	1459	3568	13	381	N	20	108	866	144	97	112	307	77	52	166	686
Kh.3	1332	704	10	314	14	15	120	561	148	54	102	398	75	38	200	441
Kh.4	5561	854	27	373	N	17	142	505	124	60	78	321	74	38	177	448
Kh.5	879	1752	N	362	51	21	131	382	168	46	118	461	84	48	196	389
Kh.6	10773	8091	11	260	42	17	109	760	134	64	79	515	69	41	189	575
Kh.7	6021	1076	21	351	59	18	123	210	131	36	97	227	77	43	135	345
Kh.8	7746	3088	31	390	33	16	124	332	126	51	96	341	73	44	165	355
Kh.9	3819	1729	3	390	67	14	124	411	134	45	111	353	80	49	168	412
Kh.10	4482	1840	16	311	34	21	108	415	157	39	106	537	82	40	211	306
Kh.11	7005	2323	21	366	56	16	123	73	135	32	99	296	75	47	148	248
Kh.12	1032	1364	17	337	33	17	120	43	130	22	88	460	69	42	170	253
Kh.13	586	1880	10	365	65	14	129	169	137	47	92	297	75	39	138	335
Kh.14	894	1657	8	341	N	16	121	619	148	54	109	232	80	49	148	524
Kh.15	3893	2498	13	318	41	22	131	528	156	53	106	268	78	43	148	470
Kh.16	4418	1345	13	315	87	19	125	383	134	43	114	212	81	45	154	381
Kh.17	4239	1137	N	297	34	26	121	679	177	68	137	260	94	45	195	427

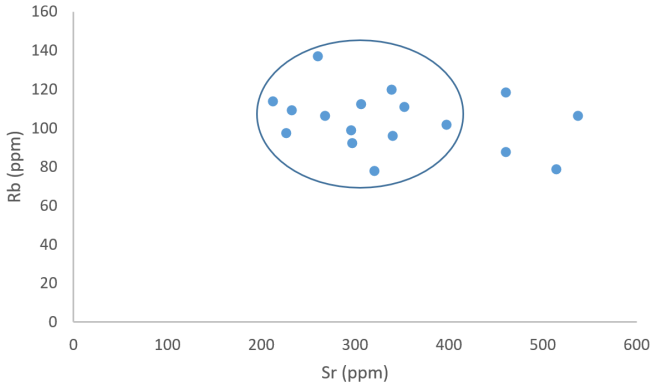

Fig. 5. Scatter plot graph of Sr and Rb (ppm) in pottery shards from Kuh-i Khawja.

One group is considering a local production pottery or came from the same resources. One group is considering non-local pottery shards or came from different raw material. Figure 2 show a scatter plot of Rb and Sr which shows that there were also two major group of pottery shards. One group is considering a local production pottery or came from the same resources. One group is considering non-local pottery shards or came from different raw material.

Hierarchinal agglomerative clustering of CaO and K_2O percentage shows that there are three component of groups which are group A that is considered a local production of pottery shards and has significance value below 5; group B is also considered as a local production pottery shard which has significance value below 10 and group C is considered as non-local pottery shards (see figure 3).

Hierarchinal agglomerative clustering of Rb and Sr concentration (ppm) shows that there are three component of groups which are group A that is considered a local production of pottery shards and has significance value below 5; group B is also considered as a local production pottery shard which has significance value below 10 and group C is considered as non-local pottery shards (see figure 3).

CONCLUSION

It can be suggested based on recent studies the site of Kuh-i Khawja goes back to the 3rd to 8th centuries CE and the earthenware found in Kuh-i Khawja can be categorized into four groups which are i) Plain unglazed pottery; ii) Unglazed painted pottery; iii) Plain glazed pottery; iv) Glazed and painted pottery. Based on compositional analysis most of the pottery shards are locally made. A scientific analysis was done to determine the chemical composition of the pottery shards was done X-Rays Fluorescence (XRF) technique and applied to determine the major and trace elements of the pottery shards. The results demonstrate that most of the pottery shards are in the same group and this strongly suggests that they are local products. Additionally, based on the Hierarchical Agglomerative Clustering shows that there was three group of pottery signature, it can be suggested that one of the group was imported pottery items.

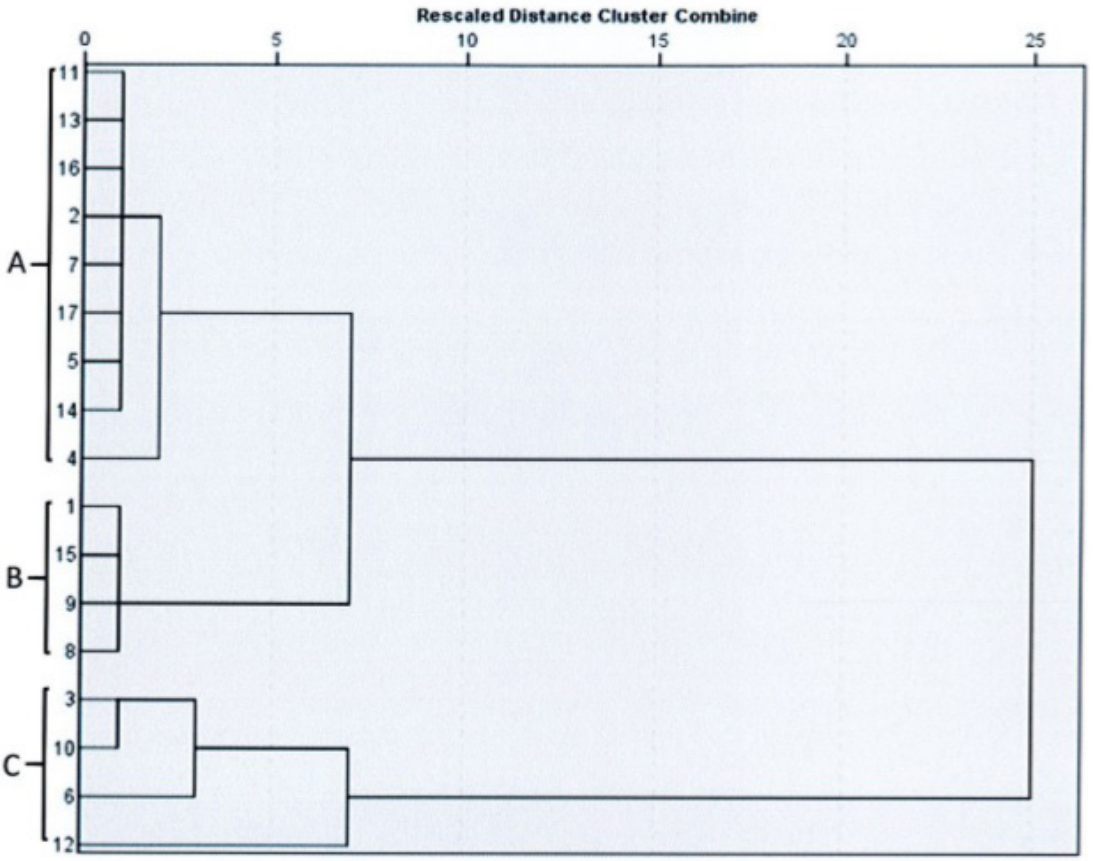

Fig. 6. Hierarchical agglomerative clustering of the CaO and K2O percentage of the pottery shards from site Kuh-i Khawja

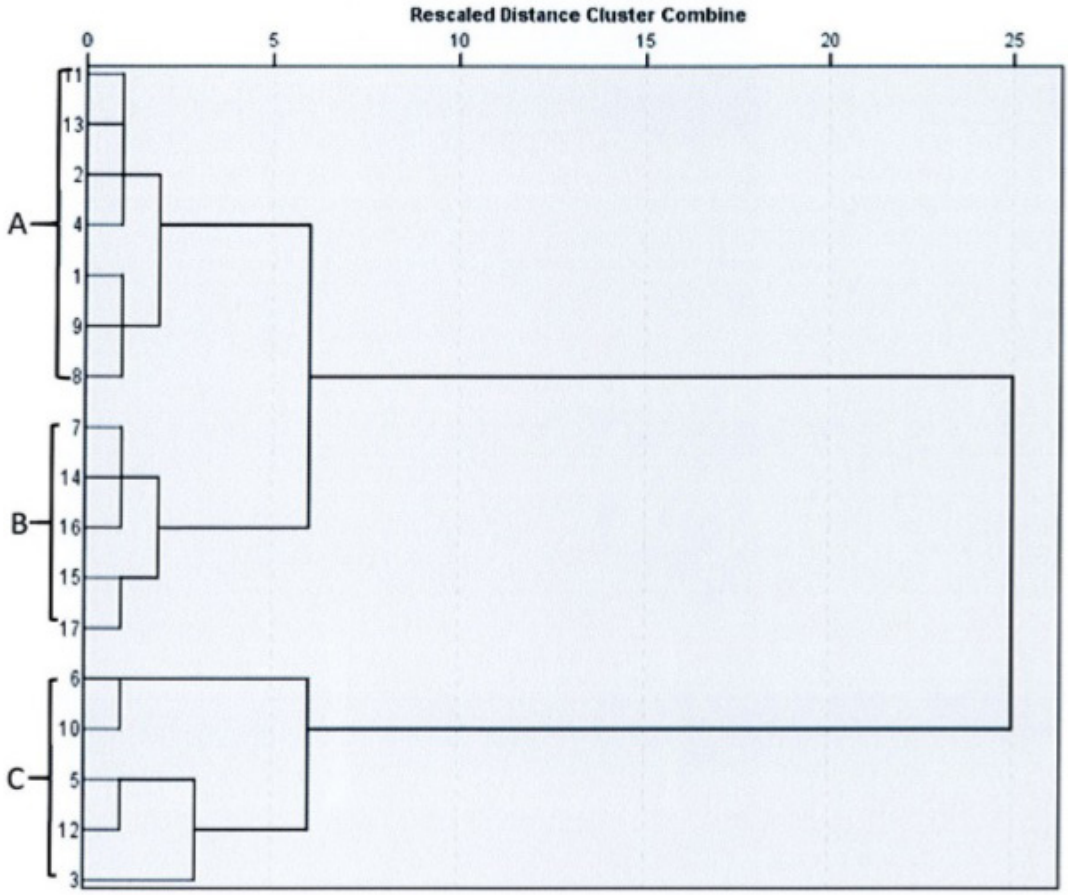

Fig. 7. Major elements of pottery shards from Kuh-i Khawja Hierarchical agglomerative clustering of the Rb and Sr concentration (ppm) of the pottery shards from site Kuh-i Khawja

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