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ARTICLE

Semi-Quantitative Analysis for Pottery Fragments Excavated at Udhruh Site, Jordan Using Non-destructive SR-XRF Analysis Employing Multivariate Statistical Methods

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Abstract: This paper presents a multielement analysis of fifteen Ayyubid-Mamluk glazed pottery sherds for determining the chemical composition in order to study their provenance. The tested fragments in this work belong to the historical site of Udhruh in southern Jordan. The chemical analysis for samples has been carried out by using Synchrotron Radiation X-ray Fluorescence Spectrometry (SR-XRF) technique. The semi-quantitative analysis of the elements Fe, Cu, Zn, Br, Rb, Sr, Y, Zr, Nb, Mo, Pd, Ag, Cd and Pb has been applied to the samples using SR-XRF technique. The data were analyzed by using Principal Component Analysis (PCA) and Hierarchical Cluster Analysis (HCA) in order to define groups of different glazed pottery sherds by obtaining information about their similarity and clustering. The results provide persuasive evidence that the Udhruh pottery fragments have at least three different sources of provenance.
Keywords: SR-XRF, Semi-quantitative, Udhruh, Ayyubid-Mamluk pottery, PCA.

Introduction

The application of interdisciplinary analytical methods to archaeology is well established to date. The employment of various chemical, geological and physical analytical techniques in order to study archaeological artefacts (e.g. ceramics, mortars, slags, marbles … etc.) is also a common practice nowadays. The chemical, mineralogical and structural characterization of ancient pottery can shed light on the provenance of raw materials used for ceramic production and determine the technological processes related to pottery manufacture. During the last years, several studies have been carried out concerning the analysis of archaeological ceramics employing various techniques. Most studies deal with the determination of chemical composition. The identification of specific chemical elements in high concentration could be related to the geological profile of the region of study and thus one can distinguish between local potteries and imported ones [1, 2]. Therefore, one of the most important questions asked by archaeologists can be answered. The question is related to the provenance of the excavated objects, since any ancient item could have been produced locally at the place where it was found or transported to the site from a location where it was originally manufactured [3].

Analysis of cultural objects by X-Ray Fluorescence (XRF) spectroscopy is the most widely used technique due to a number of favorable analytical characteristics. It is a multi-element, non-destructive and high sensitivity technique. In addition, it is applicable to a wide

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range of samples (solids, liquids and gases). These features have made the XRF a very popular analytical technique in several archaeometric studies [4,5]. Regarding the possibility of getting quantitative analysis in archaeometric applications, when using the energy dispersive detector for X-ray spectroscopy, the problems arising from the limited detector sensitivity to detect low Z elements and the irregular shape or the non-homogeneous composition of the sample have generated a widespread opinion that only semi-quantitative analyses are possible in XRF applications to archaeometry such as glass and ceramic samples. The problem of quantitative XRF analysis does not arise normally from the irregular shape of the tested object, but because of the impossibility of evaluating the auto absorption correction due to matrix low Z (<11) elements which in most cases cannot be detected by standard solid-state detectors [6, 7].

Semi-quantitative analytical procedures [8] are becoming more and more popular among researchers and using such procedures makes the question of the accuracy of results more profound. The accuracy of any analytical procedure depends to a great extent on the spectral resolution, counting statistics, matrix correction and analytical procedure, which are especially optimized to provide fast analysis of alloy composition [9]. The energy and intensity results of the XRF spectra are not only in accordance with a given element, but also with its abundance in the tested sample(s) [10].

According to the analyzing capability, the Synchrotron Radiation X-ray Fluorescence Spectrometry (SR-XRF) has gained an increasing interest in the field of elemental analysis. Several works showing the advent of technique based on XRF can be found in literature during the past decade [11, 12].

In the present study, two multivariate statistical methods, Principal Component Analysis (PCA) and Hierarchical Cluster Analysis (HCA) have been applied to ancient Ayyubid-Mamluk glazed pottery from Udhruh historical site in southern Jordan. Similar technique, experimental conditions and statistical methods were provided for Karak Castle site [13]. The main goal was to determine similarities and correlation between the selected samples based on their elemental composition. The extracted information will help to know more about the provenance sources of Ayyubid-Mamluk ceramic in Udhruh historical site. The samples were kindly borrowed from the Department of Archaeology at Al-Hussein bin Talal University.

Site, Samples and Methodology

Udhruh Historical Site

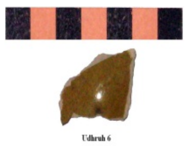
Udhruh is located at the eastern foot of the Al-Shara mountain series to the east of the well-known historical site of Petra, (30° 19' 40" N, 35° 35' 55" E), as shown in Fig. 1. The site draws its importance from its strategic location on the crossroads of the ancient trade routes in addition to the fact that water was always available to the site from its domestic, perennial springs. Udhruh is referred to, in historical literature and sources, as early as the second century A.D. Ptolemy, in the second century A.D, mentioned Udhruh only as a town in Arabia Petraea [14-18]. The site appears more often in Byzantine and Early Islamic sources and documents, where, for example, the Byzantine tax document known as the Beersheba Edict lists Udhruh among the towns of PalestinaTertia as does Stephan of Byzantium. Udhruh is also thought to be the Augustopolis mentioned by George of Cyprus and Hierocles [14], [19- 21]. This belief also is supported by data, which have been recently revealed from the Petra Papyri [22]. Two bishops from Augustopolis have been mentioned attending two church councils in the region during the 5th and 6th centuries AD [23, 24]. Udhruh was also often mentioned in early Islamic sources, as the town's inhabitants agreed to pay the poll tax to Prophet Muhammad in A.D. 630 [14, 25].

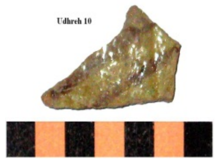
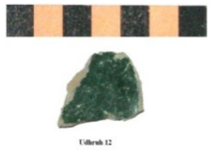
The major archaeological remain at Udhruh is the Roman fortress, but there are other significant monuments referred to other times. Outside the curtain wall of the fortress and about 20 m south of the south-western corner tower, a Byzantine church was built to serve a community who lived within the fortress at the time. An Ottoman fort was constructed, against the northern side of the curtain wall of the Roman fortress, since the site might have been a station on the pilgrimage route [26].

Samples under Investigation

Fifteen pottery samples have been investigated and reported in this work. The macroscopic description of these samples is presented in Table 1. The name of the sample, the hypothesized types, the characterizations of the ceramic, core fabric and color are given in the table. The sherds have been found in the Udhruh archaeological site (Fig. 1). These ceramic samples were found during an archaeological excavation campaign in 2008 and 2009 by the Department of Archaeology at Al-Hussein bin Talal University in cooperation with the Department of Antiquities, Ministry of Tourism and Antiquities, Jordan.

TABLE 1. The description of ceramic pottery samples (Udhruh site).

Sample Name (Object)	**Object Type**	**(Characterizations of the ceramic object)** - **Glaze color*, interior/ exterior.** - **Core fabric types and color***	**Photo (camera)**
Udhruh 1	Plain-body	- Glazed bowl (wheelthrown). - Lead glazed color = (5G 5/4) / undecorated. - Medium- hard, friable, dense fabric. Fire pink (5YR 8/6).	
Udhruh 2	Body	- Glazed bowl (wheelthrown). - Lead glazed color = (7.5G 6/6), (10GY 4/3) / (2.5GY 4/4). - Medium-hard, dense fabric with regular voids (10Y 8/2).	
Udhruh 3	Body	- Glazed bowl (wheelthrown). - Lead glazed color = (2.5GY 8/10). - Comments: Thin mottled glaze with regular intrusions and speckling. - Medium-hard, friable, dense fabric with regular voids (2.5YR 7/8).	
Udhruh 4	Plain-body	- Glazed bowl (wheelthrown). - Lead glazed color = (2.5G 8/12) / undecorated. - Medium-hard, friable, dense fabric with regular voids (5YR 7/6).	
Udhruh 5	Plain-body	- Glazed bowl (wheelthrown). - Lead glazed color = (5Y 4/6). - Hard, dense fabric with small voids (5YR 7/6).	
Udhruh 6	Plain-body (thin)	- Glazed bowl (wheelthrown). - Lead glazed color = (7.5Y 5/6). - Medium, dense fabric with regular voids (10Y 8/6).	
Udhruh 7	Plain-body	- Glazed bowl (wheelthrown). - Lead glazed color = (5 GY 4/4). - Medium, friable, dense fabric (2.5YR 7/8).	

Sample Name (Object)	Object Type	(Characterizations of the ceramic object) - Glaze color*, interior/ exterior. - Core fabric types and color*	Photo (camera)
Udhruh 8	Plain-body	- Glazed bowl (wheelthrown). - Lead glazed color = (7.5 YR 5/6). - Medium, dense fabric with regular voids (10YR 8/6).	
Udhruh 9	Plain-body	- Glazed bowl (wheelthrown). - Lead glazed color = (2.5Y 8/10), (5YR 2/4) decorated lines/ undecorated. - Hard, dense fabric (2.5YR 3/4).	
Udhruh 10	Plain-body	- Glazed bowl (wheelthrown). - Lead glazed color = (2.5Y 6/6), (2.5YR 2/4) decorated lines. - Medium-hard, friable, dense fabric with regular voids (2.5YR 6/8).	
Udhruh 11	Plain-body	- Glazed bowl (wheelthrown). - Lead glazed color = (5YR 2/6), (2.5YR 5/8) / undecorated. - Medium, friable, dense fabric with regular voids (5YR 3/4).	
Udhruh 12	Plain-body	- Glazed bowl (wheelthrown). - Lead glazed color = (5G 4/6). - Medium, dense fabric with regular small voids. Fire pink-orange (2.5YR 7/10).	
Udhruh 13	Body	- Blue under turquoise bowl (wheelthrown). - Alkaline glazed color = (7.5 BG 7/6). - Medium, dense, friable fabric. Fire pale pink (10YR 9/3).	
Udhruh 14	Rim	-Black under turquoise bowl (wheelthrown). - Alkaline glazed color = (10Y 9/2), (10YR 1/2). - Medium, dense, friable fabric (7.5 Y 8/4).	
Udhruh 15		- Blue under turquoise bowl (wheelthrown). - Alkaline glazed color = (10 BG 6/8), (2.5 B 3/4). - Medium- hard, dense, friable fabric (7.5 Y 8/4).	

*Colors according to Munsell Book of Colour [26, 27].

FIG. 1. Map of Jordan showing Udhruh site.

Measurements and Analysis

To perform the analysis of the samples using SR-XRF, the inner part (core) of the samples was exposed to synchrotron X-ray beam. All measurements were carried out in BAM Line at BESSY II Synchrotron light source in Berlin-Germany using 40 keV incident energy X-ray beam, focused to a (0.8x0.8) mm^2 spot size. For each tested sample, the data have been acquired using a Si (Li) detector from two selected points on the core to be representative of the sample. The measurement time was 60 seconds for each sample. All measurements have been performed at room temperature. The spectra were processed by the AXIL-PC computer software program [29] based on IAEA standard program package QXAS. In order to be able to perform a semi-quantitative analysis, the elemental composition of the samples has to be identified. This task could be achieved by calculating the net peak area of the XRF spectrum for which the used software is capable. The importance of the semi-quantitative estimation stems from the fact that it can be useful for determining the elemental concentrations because the latter are proportional to the weight concentrations that can be measured using in the technique. Therefore, comparisons between the content of one chemical element in different samples with similar composition can be directly made through the energy and the area under the measured peaks. The intensity of emission from each element can lead to classify the elements. The net area under the peak (counts) for certain element in an XRF spectrum is used to calculate the abundance of the element in the sample, since the peak intensities are proportional to the concentrations of the elements present in the sample.

Statistical Methods

In the archaeological field, cluster analysis together with Principal Component Analysis (PCA) represent the most commonly used methods to classify items in subgroups such that individuals within a group are similar to each other. PCA is a mathematical technique that allows n-dimensional identification of sample groups and hence is a very powerful method of exploratory multivariate statistical analysis. Therefore, it is often used in the interpretation of XRF artifact characterization for helping archaeologists to identify discrete compositional groups within a data set. This information may then be applied to formulate and test hypotheses on trade and exchange routes and socio-economic relations. Hierarchical Cluster Analysis is a multivariate method, which allows providing evidence for groups of objects within the data set. The results are commonly presented as dendrograms showing the order and levels of clustering as well as the distances between individual samples [30, 31, 32].

In this work, the Bray-Curtis coefficient [33] was used to calculate similarity matrices for analysis. The Bray-Curtis coefficient is widely used in the study of provenance of cultural heritage artefacts. The dendrograms have been constructed using a group-average linkage hierarchical cluster analysis [34] and the data have been processed by a PC computer software package Unscrambler X Ver.10.1 (Camo ASA software, Oslo, Norway).

Results and Discussion

The SR-XRF results of Udhruh site samples are shown in Table 2. In the table, the net peak area data (in counts) for selected elements: Fe, Cu, Zn, Br, Rb, Sr, Y, Zr, Nb, Mo, Pd, Ag, Cd and Pb of the XRF spectra are given. In order to perform a semi-quantitative analysis, 210 data entries (15 samples ×14 elements), the PC computer software package Unscrambler X Ver.10.1 (Camo ASA software, Oslo, Norway) has been employed. The multivariate statistical analysis has been carried out to verify the similarity of the samples.

PCA and Hierarchical Cluster Analysis are routinely applied to the data with the purpose of identifying groups of chemically similar sherds, which can be interpreted as representing sherds made from the same raw materials or mixtures of raw materials and therefore presumably made in the same place, although not necessarily at the same time. Fig. 2 and Fig. 4 show the Principal Component Analysis (PCA) and Cluster Analysis (CA) for the data presented in Table 2.

The analyzed data have been applied to the raw data matrix of dimensions 15×14 (samples × chemical parameters). Fig. 2 represents the scatter score plot of the readings PC1 versus PC2 data and the loading plot is reported in Fig. 3. The corresponding score plot shows that the data are confined in two large groups except sample UDR15, which does not follow either of the other large groups. It should be mentioned that each group contains the samples belonging to the same pottery provenance source.

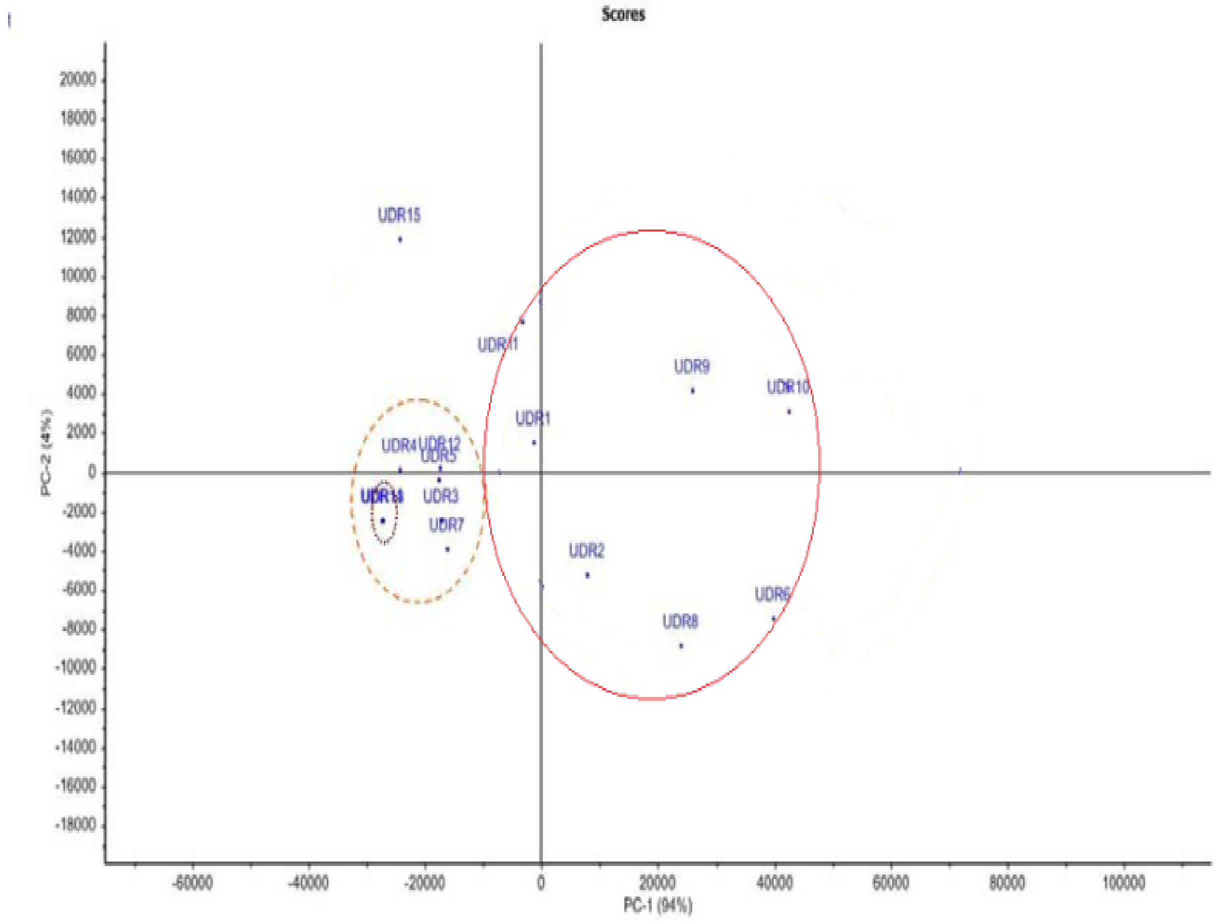


FIG. 2. Score plot of PCA of the normalized area under the peaks of SR-XRF result for Udhruh site samples. PC1 vs. PC2 data matrix of dimensions 15×14 (15= number of samples; 14= number of elements).

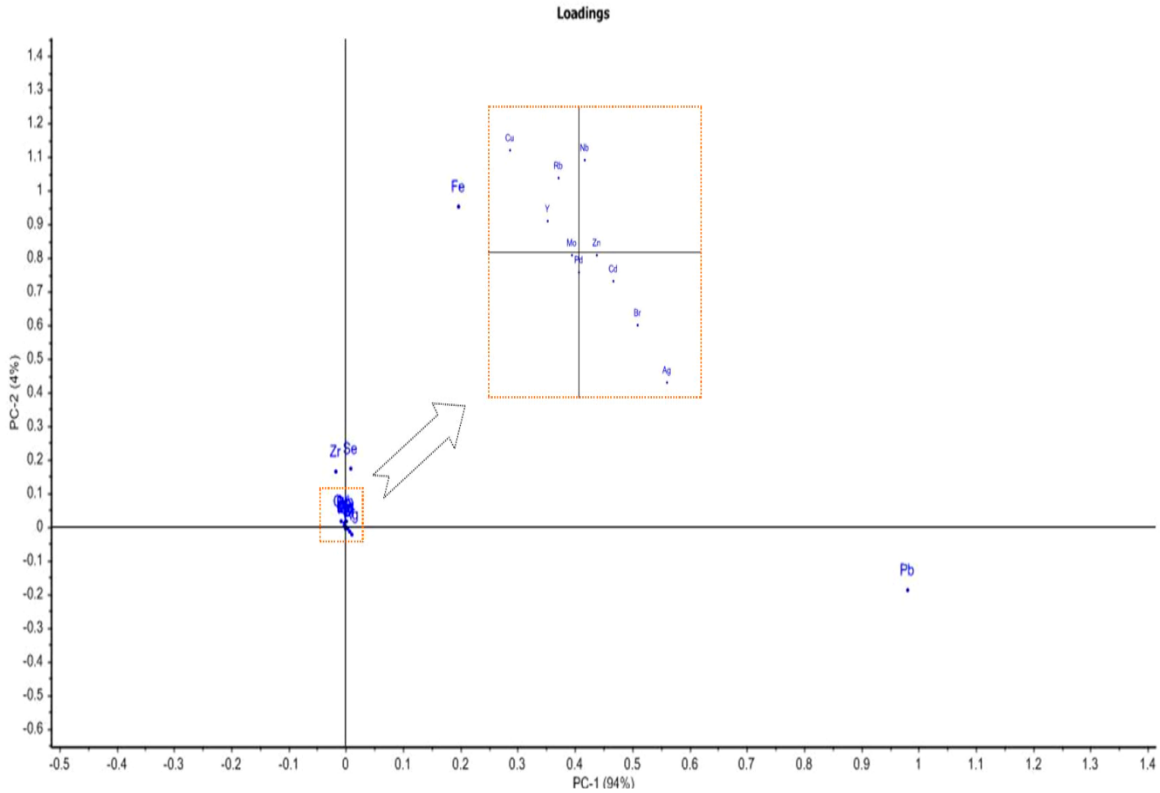


FIG. 3. Loading plot of PCA of the normalized area under the peaks of SR-XRF result for Udhruh site samples. PC1 vs. PC2 data matrix of dimensions 15x14 (15= number of samples; 14= number of elements).

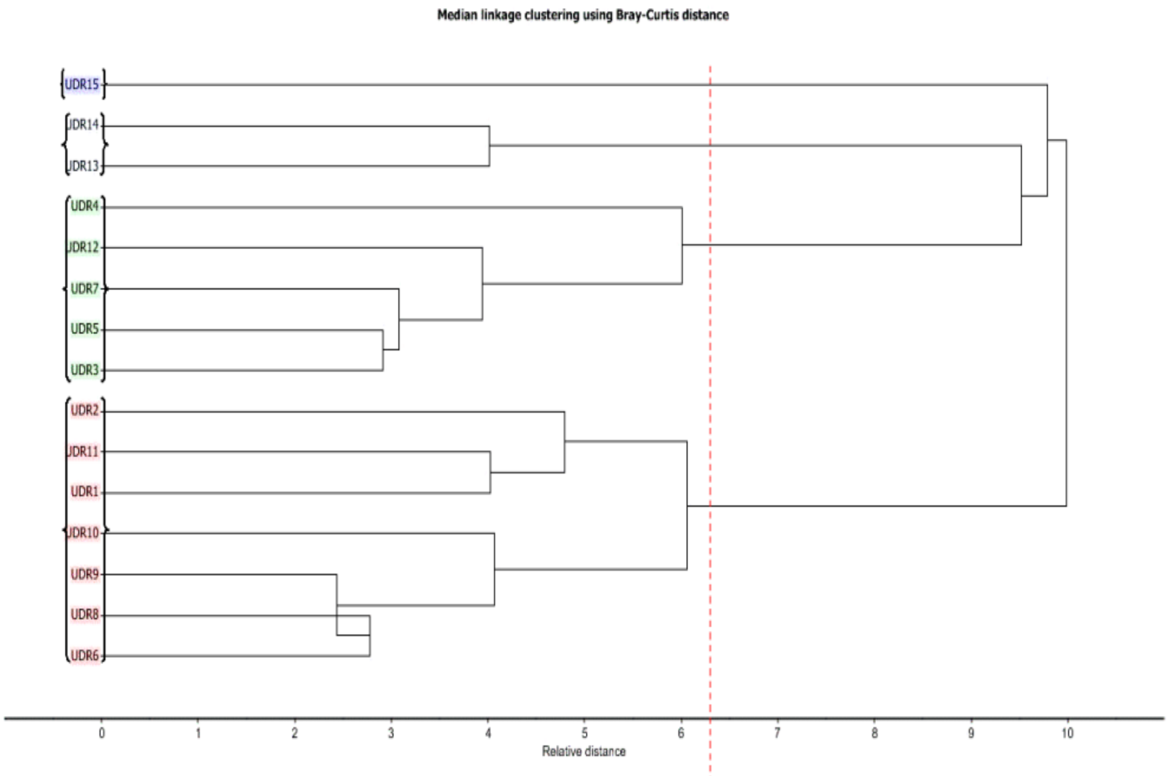


FIG. 4. Hierarchical clustering dendrograms by median linking and Bary-Curtis distance for the 15 ceramic sherd samples from Udhruh site (normalized area under the peaks of SR-XRF result).

TABLE 2. Net peak area (in counts) for selected elements of SR-XRF result for Udhruh site samples.

	^{26}Fe	^{28}Cu	^{30}Zn	^{35}Br	^{37}Rb	^{38}Sr	^{39}Y	^{40}Zr	^{41}Nb	^{42}Mo	^{46}Pd	^{48}Cd	^{47}Ag	^{82}Pb
UDR 1	7660.36	1107.77	210.07	195.76	BDL	5968.74	BDL	12064.11	910.65	260.59	BDL	412.97	BDL	25242.55
UDR 2	5063.89	2046.31	567.19	202.45	BDL	2077.62	BDL	1924.17	101.87	BDL	BDL	443.90	231.74	35146.14
UDR 3	2849.74	181.16	30.17	BDL	BDL	3089.02	BDL	1695.07	98.729	BDL	BDL	BDL	73.85	9978.72
UDR 4	3014.93	38.30	57.35	BDL	121.97	1941.72	337.57	9385.33	407.71	184.34	BDL	472.15	66.11	2822.70
UDR 5	4894.98	36.97	32.79	BDL	225.64	1639.84	523.73	2978.97	213.24	BDL	BDL	BDL	BDL	9057.06
UDR 6	7358.94	633.79	122.17	815.11	BDL	1944.37	BDL	550.09	99.87	BDL	494.49	1538.11	94.69	70318.04
UDR 7	1626.87	211.29	42.93	113.50	BDL	1132.03	BDL	3189.92	199.81	115.83	BDL	125.46	BDL	11287.01
UDR 8	4861.81	379.71	63.46	368.49	BDL	1361.19	BDL	939.23	86.67	BDL	83.61	293.37	BDL	51558.53
UDR 9	21565.12	38.27	125.42	337.73	BDL	839.52	BDL	5399.74	550.49	BDL	124.98	335.97	BDL	59550.83
UDR 10	28657.99	147.59	275.53	359.68	BDL	6845.27	BDL	3306.58	282.75	BDL	253.52	607.63	BDL	67796.03
UDR 11	15440.33	41.49	73.39	BDL	274.68	2348.81	BDL	3998.37	317.48	BDL	BDL	BDL	84.49	22954.89
UDR 12	4514.92	2915.73	BDL	BDL	240.39	2290.93	721.21	7967.36	373.66	BDL	BDL	BDL	BDL	9542.79
UDR 13	1042.65	1081.62	BDL	BDL	108.93	2232.71	BDL	1613.44	74.66	94.44	BDL	BDL	105.59	104.59
UDR 14	834.39	43.76	165.32	80.57	BDL	3280.46	119.67	1708.10	71.45	BDL	BDL	BDL	BDL	BDL
UDR 15	15255.33	2564.09	103.91	42.27	480.63	6208.33	288.64	2363.13	284.77	BDL	BDL	BDL	BDL	217.87

* BDL: Below Detection Limit.

The two large groups are characterized as follows:

- Group A (UDR1, UDR2, UDR6, UDR8, UDR9, UDR10 and UDR11): This group is characterized by large concentrations of lead. It is worth mentioning that the samples in this group do not appear close to each other like the other groups in the PCA.
- Group B (UDR3, UDR4, UDR5, UDR7, UDR12, UDR13 and UDR14): The samples belonging to this group are characterized by large average concentrations of iron except UDR13 and UDR14.

Sample UDR15 does not belong to any of the identified groups. Moreover, this sample shows very high concentration of iron and very low concentration of lead, which does not appear in the other samples of groups A and B.

Hierarchical cluster analysis was employed based on median linking and Bary-Curtis distances. The resulting dendrograms of the 15 ceramic sherd samples, investigating the area under the peak data, from Udhruh site are presented in Fig. (4). The dendrograms suggest that the data fall in two large groups: the first group consists of (UDR1, UDR2, UDR6, UDR8, UDR9, UDR10 and UDR11) and the second group consists of (UDR3, UDR4, UDR5, UDR7 and UDR12) while samples UDR13 and UDR14 appear together in one small group; whereas sample UDR15 still appears alone with no affiliation to any of the groups

Conclusions

This study demonstrates the growing importance of interdisciplinary research. More specifically, it demonstrates the importance of using SR-XRF in archaeological investigations. The increased need for non-destructive investigations has become a major issue in archaeometry. The work performs a chemical characterization of fifteen glazed pottery sherds dated to Ayyubid-Mamluk period from Udhruh archaeological site in southern Jordan in order to extract information about their provenance. Multivariate statistical methods display how objects from various groups can be differentiated according to their elemental composition to obtain information on their provenance by showing similarity and clustering. The analysis presents good results concerning the fabric characterization of clay (matrix and color) and results from this report are in good agreement with these presented in Table 1. Hence, it is evident that the technique is powerful to confirm the usefulness of chemical characterization of pottery sherds and statistical techniques of data handling to complete and integrate the work of archaeologists in provenance studies.

The ceramic sherds from Udhruh show two large groups representing provenance region, while sample UDR 15 appears apart from these two groups. The latter also appears to contain very high concentration of iron, which is not observed in the other site samples. It is worth noting that the samples in group two are apart of each other, which means that they do not belong to one provenance region. Another interpretation of the variance of chemical composition of the samples of this group is that several kinds of raw material have been brought from different parts around the site to manufacture the sherds. Further analysis of this sample may clarify this point.

The significance of the present work comes from the scarcity of the studies on the Udhruh site. Therefore, the study will help archaeologists to explain the cultural contacts and behaviors during the Islamic period and contribute to better understanding of human behaviors, particularly when integrated with typological, technological and other studies.

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