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Assessment of Metal Pollution in Umm el-Surab Archaeological Layers, Northeast Jordan

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Abstract

This study aims to assess the extent of the anthropogenic pollution of Area A at the Umm el-Surab archaeological site, Jordan. It examines the concentrations of 21 metals from four Roman, Byzantine, and early and late Ayyubid-Mamluk layers. The site, located near Bosra and Umm el-Jimal, was continuously settled from the Roman till the Islamic periods. The chemical analysis and pollution indices (enrichment and contamination factors (EF, CF), and geo-accumulation index Igeo) show low enrichment in Zn, K, Sr, Na, Pb, Ba, Li, Mn, Mg, Mo, and Cu, and moderate enrichment in Cd, Ag, Ca, As, and Ni. Co is significantly enriched in one sample and moderately enriched in the other ones. The results show pollutant metals more enriched in the Byzantine layer than the rest of the layers. A likely primary source of the limited pollution is organic fuels and their firing product of ash, while a likely secondary source might be the pottery fragments spread in the layers. The results agree with the fact that the site was rural with limited industrial activities, especially metal production, like iron and copper, that would have increased the concentration of the analyzed metals.

Keywords: Archaeological layers, enrichment and contamination factors, geo-accumulation index, ash, pollution, Umm el-Surab, Jordan.

1. INTRODUCTION

Ancient human activities at archaeological sites represent the main source of pollution that is inherited in archaeological layers and adjacent soils. Pollutant heavy metals that accumulate in these layers and soils transmit, through plants, to humans and affect their health (Al-Taani and Al-Qudah 2013, Rasmussen et al. 2020). Human activities motivate the release of heavy metals from the earth's crust (their natural source) to the environment, and consequently their concentration levels increase in the archaeological layers, soils and other components of the environment (Middleton and Price 1996). Therefore, chemical analysis of archaeological layers has been used to determine ancient anthropogenic pollution and compare the level of pollution between different sites or different periods (Manzanilla 1996; Parnell et al. 2002; Zhang et al. 2005; Oonk et al. 2009; Knabb et al. 2016; Holdridge et al. 2021).

The Umm el-Surab site provides a singular opportunity to examine the pollution of an unbroken comparative stratigraphy. This research investigates the level of pollution of Roman, Byzantine and Islamic layers by chemical analysis of a set of 21 heavy metals, then use the pollution levels to evaluate the extent of human activities that occurred at the

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site during these periods.

2. SITE SETTING

The archaeological site of Umm el-Surab is located in the Hauran Plain of northeastern Jordan, close to the Bosra and Umm el-Jimal archaeological sites, and within the massive Harrat Ash Shaam basaltic volcanic field (Fig. 1), which extends from Syria through Jordan into northern Saudi Arabia and covers about 11,000 km^2 (Bender 1974; Kwatli et al. 2015). The Umm el-Surab area has an arid to semi-arid climate, where the temperature and rainfall vary seasonally, recorded maximum and minimum temperatures are 46.4 and -6.8 ^{o}C, while the minimum and maximum amounts of rainfall are 50 and 250 mm/year; therefore, the land is restricted to grazing during the spring season. The sediments in the area include alluvial, wadi sediments, alluvial fans, alluvial mudflats and soils of Holocene to recent age (Fig. 1). The soil (Aridisol) in the Umm el-Surab area is shallow and hardly developed because of low precipitation, wind erosion and restricted chemical weathering (Bender 1974). In general, the soil of the region is medium to moderately fine-textured, which can be classified as silty loam. The most part of the soil-forming materials are locally derived from the underlying formations of Harrat Al-Shaam basalt, in addition to secondary carbonates and windblown fine-grained quartz (Allison et al. 2000).

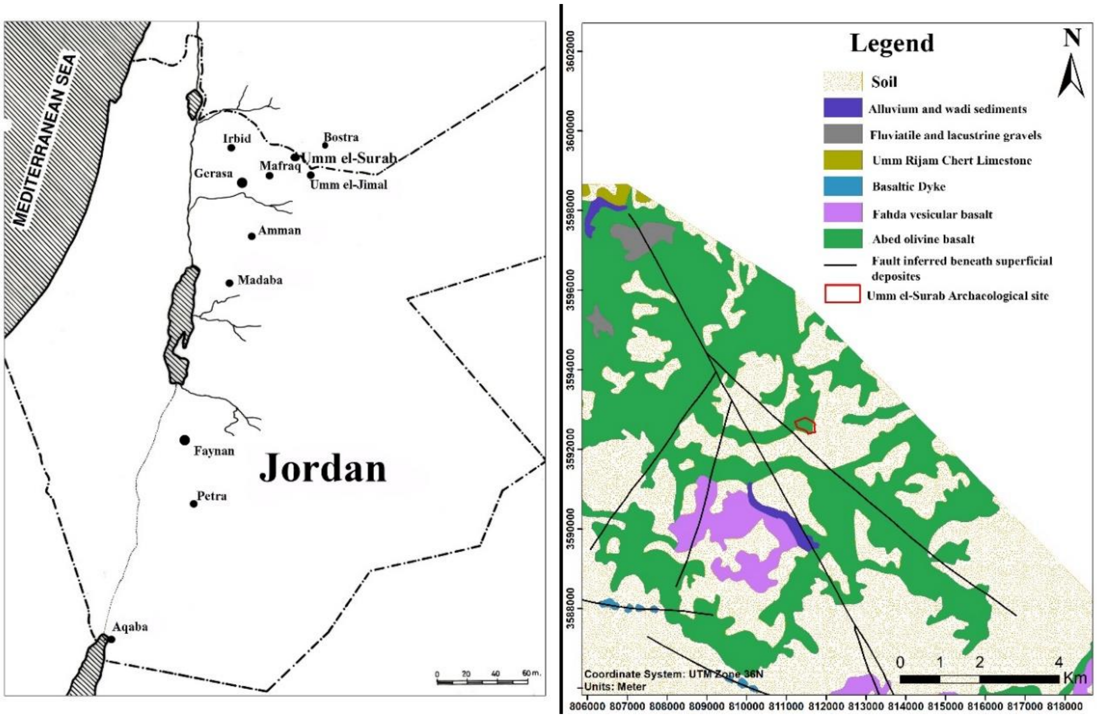


Figure 1. The location of the Umm el-Surab archaeological site (left), and the geological map of its area (right: modified after al-Bashaireh et al. 2021).

The basalt, 478m thick, is subdivided into five groups (from bottom to top, Fig.1): Wisad (10.5-9.37Ma), Safawi (9.3-8.45Ma), Asfar (3.96-1.41Ma), Rimah (2.94-2.01Ma) and Bishriyya (1.45-0.1Ma) (Batayneh et al. 2001; Ibrahim 1997). The chemical composition (averages) of the Bishriyya (Abed olivine) basalt that covers the region is mainly composed of SiO_2 (47.5%), Al_2O_3 (14.5%), TiO_2 (2.2%), Fe_2O_3 (11.4%), MnO

(0.17%), MgO (8.4%), CaO (9.7%), Na_2O (3.0%), K_2O (0.8%) (Tarawneh et al. 2020). Some metal analysis of the basalt from the Dhlail area (west Mafraq) showed contents of Sr (438-663 ppm), Zr (194-237 ppm), Rb (20-27 ppm), Nb (44 ppm), Y (9-26 ppm), Ba (315-325 ppm) (Abed 2000).

Although the Harrat Ash-Shaam region suffers a harsh environment for human inhabitation, surveys and excavations showed that it has a rich well-preserved archaeological record dating from Palaeolithic to recent times (Huigens 2018; Richter 2017, Betts et al. 2013). In the Umm el-Surab area, lithic artifacts found on the banks of the surrounding ancient Wadis witnessed Iron Age occupations (King 1989). After a period of abandonment, the Umm el-Surab site was continuously settled during the Nabatean, Roman, Byzantine and Islamic periods (Parenti and Gilento 2010; al-Bashaireh et al. 2021). Little evidence of inscriptions confirms the Nabatean period, more evidence of ceramics, columns and features attests the Roman period, and large evidence of findings, houses, churches, a mosque and a minaret confirm the flourishing Byzantine and Muslim periods (Butler 1909; King 1983). Historical and archaeological data propose a subsistence strategy based on agriculture and animal husbandry in the eastern Badia since Roman times (Kirk 1998). The Druze who inhabited the site at beginning of the 20th century transformed or replicated ancient building techniques using stones of fallen structures (Gilento 2015).

The site was surveyed by several missions; the Princeton Expedition in Syria leaded by Butler (Butler 1909) is considered the first systematic study of the site and of the Hauran area in general. The Jordan Hawrān Archaeological Survey and excavations at Umm el-Surab have recently started in 2018 in collaboration between Paris-1 (Panthéon–Sorbonne) and Yarmouk Universities; for more details on the studies and surveys of the site see Al-Bashaireh (2021), Bartoccini (1941), Mittmann (1966), King (1983), Bucarelli (2007), Parenti and Gilento (2010).

The structures of the site were built with local black greyish basalt blocks. Its water system harvested the rainwater that precipitated on the slopes of Jabal Druze mountain on the east, and ran in Wadis passing close to the site. The flowing water was diverted and captured in small dams, pulled into canals, and stored in a castellum, reservoirs and cisterns distributed in different parts of the site and some houses.

Al-Bashaireh et al. (2021) showed that Byzantine, Umayyad, Ayyubid-Mamluk ceramic samples were probably produced locally. Gilento et al. (2019) showed the use of gypsum and lime mortars in the construction of the tower and cistern of the Sergius and Bacchus Church, the main church of the site. Excavations were carried out in 2018 and 2019, under the direction of the Université Paris 1 Panthéon–Sorbonne and co-direction of Yarmouk University and in collaboration with the Department of Antiquities of Jordan, in Area A. The excavations revealed the presence of two large ovens (tannours), where the second oven, dated by pottery fragments to the Umayyad-Abbasid periods, was placed on top of the older Umayyad one.

3. MATERIALS AND METHODS

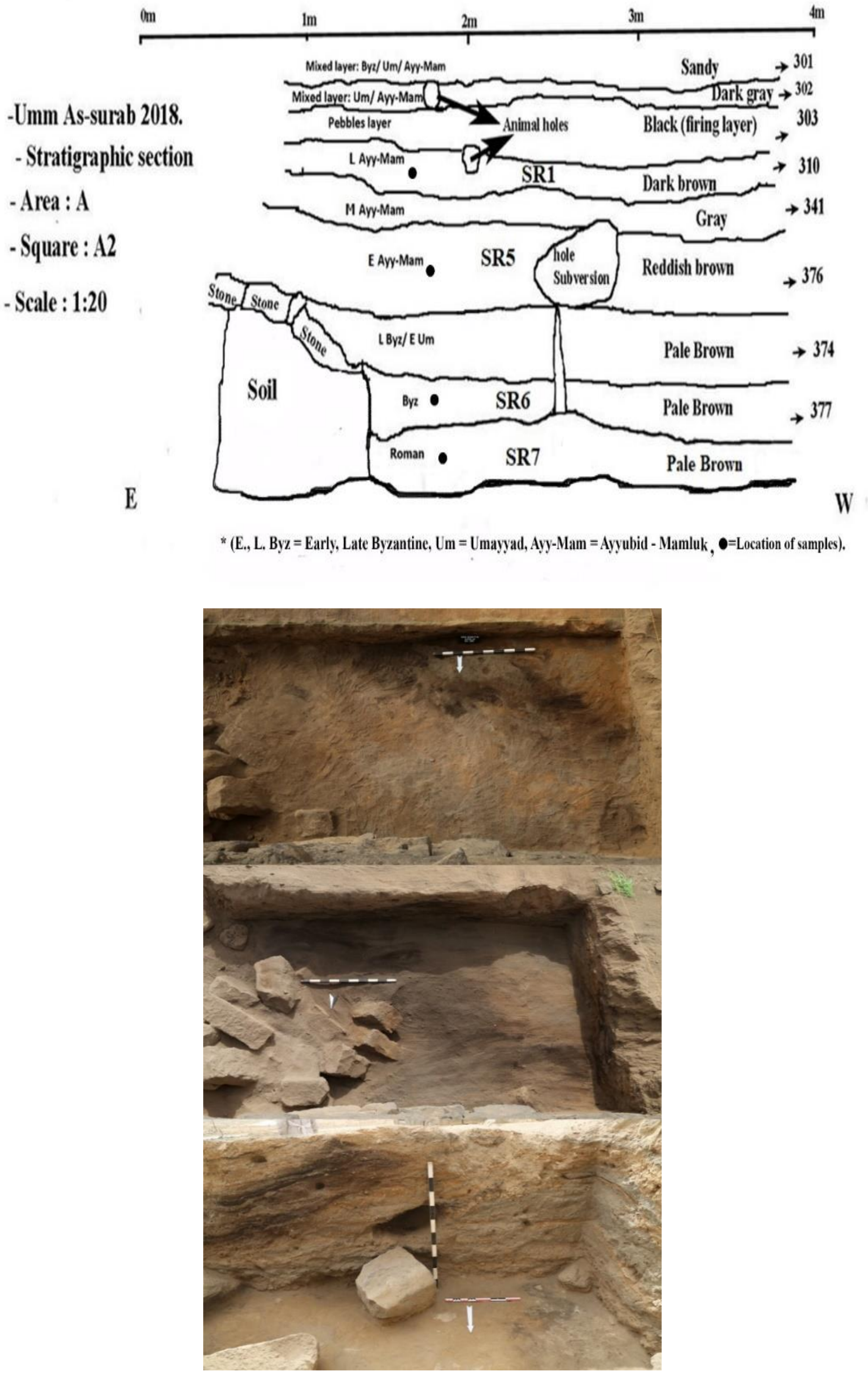
In the field, four core samples were collected from the sequence of excavated archaeological layers of Area A (zone A2), located between two houses called units TU24 and TU29. The layers were dated by ceramics to periods from Roman till Ayyubid-Mamluk times: SR1 late Ayyubid Mamluk (the youngest), SR5 early Ayyubid-Mamluk, SR6 late

Byzantine, SR7 Roman (oldest) (Fig. 2). Securely dated layers were sampled; while layers comprising ceramics from two successive periods and the upper mixed layers were avoided. The color of the early Ayyubid-Mamluk layers is grey-brown and have substantial amounts of ash, charcoal and fragments of a tabun, while the late Ayyubid-Mamluk layer is dark grey-brown, with more amounts of ash and charcoal, some animal bones and a few fragments of glass and mortar. The Byzantine and Roman layers are grey–pale brown and comprise ash and charcoal. The degree of the spread of ash and charcoal in some layers can be noted visually in figure 2.

The samples were obtained by inserting a labeled plastic core into each layer. The outer surface material of each core was removed and then small amounts of the soils of the inner cores were collected in small clean plastic containers. The samples were sent for chemical analysis to the Arizona Laboratory for Emerging Contaminants (ALEC), the University of Arizona, Tucson. The sample contents of a set of 21 metals (Li, Be, Na, Mg, Al, K, Ca, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, As, Rb, Sr, Mo, Ag, Cd, In, Sn, Sb, Cs, Ba, Tl, Pb, Bi, U) were analyzed to assess their pollution using an Inductively Coupled Plasma Mass Spectrometer ICP-MS, model Agilent 7700 Technologies, Santa Clara, CA, (Ramirez-Andreotta et al. 2013; U. S. EPA 2014).

The samples were analyzed according to the laboratory procedure discussed elsewhere; see (Ramirez-Andreotta et al. 2013; Manjón et al. 2020). The samples were air dried until they reached a constant mass, sieved to ≤ 63mm diameter, then digested by nitric acid (HNO3). A NIST standard was included for quality control. The analytical quality assurance/quality control (QA/QC) protocol was adapted from US EPA Method 200.8 for analysis by ICP-MS. Calibration standards were prepared from multi-element stock solution (SPEX

CertiPrep, Metuchen, NJ) diluted in 2% HNO3 from AriStar-Plus grade acid (VWR Scientific). Calibration curves include at least 7 points with correlation coefficients > 0.995. The QC protocol includes a continuing calibration blank (CCB), a continuing calibration verification (CCV) solution and at least one quality control sample (QCS) to be analyzed just after calibration and again after every 12 samples and at the completion of the run. The QCS solutions are from an independent source, such as NIST SRM 1643e - Trace Elements in Water, or QCS solutions from High Purity Standards (Charleston, SC). Acceptable QC responses must be between 90 and 110% of the certified value. An internal standard (Ir for Hg, Rh or In for other analytes) is added via on-line addition into the sample line using a mixing tee. Scans for standards and samples are performed with three readings with 50 sweeps each. The concentration values are reported as the mean of the triplicate scans. The relative standard deviation of the three readings is typically between 1 and 5%.



* (E., L. Byz = Early, Late Byzantine, Um = Umayyad, Ayy-Mam = Ayyubid - Mamluk, ● = Location of samples).

Figure 2. Stratigraphic section of Area A2 and samples' locations (above): drawn by Waseem Jaradat and modified by the author, distribution and spread of ash in the layers (below): photos by Yousef al-Zou'bi.

Three pollution indices were used for pollution assessment: the Enrichment Factor (EF) was used to assess the heavy metal pollution of the layers (Sinex and Helz, 1981, Ergin et al., 1991), EF was calculated using the formula: EF = (M/Fe) sample / (M/Fe) UCC, where UCC is the upper continental crust, (M/Fe) sample is the ratio of the metal and Fe concentrations in the sample, and (M/Fe) UCC is the ratio of the metal and Fe concentrations in the upper continental crust (Kabata-Pendias and Pendias 2001, Clemente et al. 2005). EF value < 1.5 indicates that the metal originated totally by natural processes, while EF value > 1.5 indicates that the metal originated by anthropogenic processes (Sinex and Helz 1981; Zhang and Liu 2002).

The intensity of anthropogenic pollutants and extent of heavy metal pollution of the layers were assessed using the geo-accumulation index (Igeo) (Müller 1969, 1981), which was calculated according the following formula: $Igeo = \log_2 [Ms / 1.5Mr]$, where Ms is the concentration of the metal in the sample, Mr is the concentration of the same metal in reference value (UCC), and 1.5 is a background matrix correction factor for lithogenic effects. Based on Müller (1981)'s scale for the Igeo, its values indicate that: $Igeo \leq 0$ represents an uncontaminated sample, $0 < Igeo < 1$ represents an uncontaminated/ moderately contaminated sample, $1 < Igeo < 2$ represents a moderately contaminated sample, $2 < Igeo < 3$ represents a moderately/strongly contaminated sample, $3 < Igeo < 4$ represents a strongly contaminated sample, $4 < Igeo < 5$ represents strongly/extremely contaminated sample, and $Igeo > 5$ represents an extremely contaminated sample.

The Contamination Factor (CF) was calculated to assess the level of contamination of the samples using the following formula of Martin and Meybeck (1979): CF= Concentration of the Sample's metal/ Concentration of the UCC's metal. Based on Hakanson (1980), $CF < 1$ indicates low contamination, $1 < CF < 3$ indicates moderate contamination, $3 < CF < 6$ indicates considerable contamination and $CF > 6$ indicates high contamination.

Umm el-Surab at present is a small town, located in an arid area of low precipitation, and far from large industrial plants that could contribute to the pollution of the site's soil or leaching processes; therefore, the results of the samples' metals concentrations were compared to the averages of these metals measured in the upper continental crust UCC (Taylor 1964; Fortescue 1992; Kabata-Pendias and Pendias 2001).

4. RESULTS AND DISCUSSION

The results of the ICP-MS analysis of the 21 metals are listed and compared to their corresponding background average values measured in the upper continental crust in Table 1 and presented in Fig. 3.

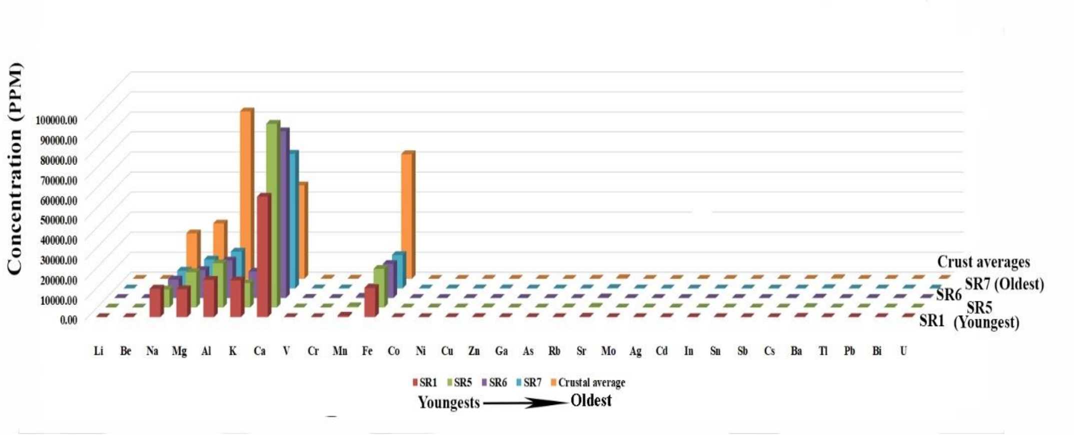


Figure 3. Concentrations of the metals compared to those of crustal average values.

The results show that most of the measured values of the metals of the samples listed in Table 1 are below their corresponding background average values except Ca, Co, Ni, As, Ag and Cd in all samples, Zn, Ga and Sr in sample SR6, and Zn in sample SR7 (Figs. 3, 4). The samples show significant differences in the concentrations of some metals such as Ca, Fe, Mg, Na, Al, and Na. However, the average of all measured metals of the late Byzantine sample SR6 is the highest; while that of the late Ayyubid-Mamluk sample SR7 is the lowest; see Table 1. The measured values of the key metals are lower than those of reference values for soils suggested by World Health Organization (WHO), for instance, As=10, Pb=10, Cd=0.3, Zn=50, Cu=20, Mn=2000, Cr=100, Ni=50, Co=8.

Table 1. The concentrations of the examined metals, contamination and enrichment factors in the samples, and their average concentrations in the crust of the earth; S.= sample, El.= Element, ppm= Part per million, C= Concentration (ppm), Cr. Av.= Crust averages, CF= Contamination Factor, EF= Enrichment Factor, Igeo= Geoaccumulation index.

S/ El	SR1		SR5		SR6		SR7		Cr. Av.	SR1				SR5				SR6				SR7			
	C ppm	CF	C ppm	CF	C ppm	CF	C ppm	CF		El.	EF	El.	Igeo	El.	EF	El.	Igeo	El.	EF	El.	Igeo	El.	EF	El.	Igeo
Li	14.50	0.69	15.91	0.94	18.76	0.80	17.33	0.87	20	Cd	11.11	Cd	0.80	Cd	10.62	Cd	1.14	Cd	12.16	Cd	1.15	As	15.51	As	1.47
Be	0.76	0.57	0.78	0.30	0.91	0.26	0.78	0.26	3	As	8.68	As	0.44	As	8.98	As	0.89	As	8.7	As	0.66	Cd	12.38	Cd	1.14
Na	14107	0.62	9193	0.40	8998	0.40	8856	0.39	22700	Ag	7.02	Ag	0.13	Ag	7.17	Ag	0.57	Ag	6.65	Ag	0.27	Ni	6.38	Ni	0.19
Mg	13892	0.50	13953	0.64	17653	0.50	14416	0.52	27640	Ni	6.26	Ni	-0.03	Ca	6.34	Ca	0.39	Ca	6.54	Ca	0.25	Ag	5.77	Ag	0.04
Al	18617	0.22	18667	0.26	22094	0.22	18397	0.22	83600	Ca	5.5	Ca	-0.22	Ni	6.05	Ni	0.33	Ni	6.1	Ni	0.15	Ca	5.39	Ca	-0.06

S/El	SR1		SR5		SR6		SR7		Cr. Av.	SR1				SR5				SR6				SR7			
	C ppm	**CF**	**C ppm**	**CF**	**C ppm**	**CF**	**C ppm**	**CF**		**El.**	**EF**	**El.**	**Igeo**	**El.**	**EF**	**El.**	**Igeo**	**El.**	**EF**	**El.**	**Igeo**	**El.**	**EF**	**El.**	**Igeo**
K	18340	1.00	13140	0.66	12223	0.71	11159	0.61	18400	Co	5.26	Co	-0.28	Co	4.36	Co	-0.15	Co	4.08	Co	-0.43	Co	4.78	Co	-0.23
Ca	60075	1.29	83164	1.97	91708	1.78	67189	1.44	46600	K	4.26	K	-0.59	Zn	4.03	Zn	-0.26	Ga	3.6	Ga	-0.61	Zn	4.3	Zn	-0.38
V	39.76	0.29	44.35	0.40	54.40	0.33	44.65	0.33	136.0	Ga	4.13	Ga	-0.63	Ga	3.8	Ga	-0.35	Zn	3.25	Zn	-0.76	Ga	3.73	Ga	-0.59
Cr	29.46	0.29	33.78	0.39	38.97	0.34	35.34	0.35	100	Zn	3.56	Zn	-0.85	Sr	3.32	Sr	-0.54	Li	2.91	Li	-0.91	Li	3.24	Li	-0.79
Mn	731	0.81	612	0.73	659	0.68	583	0.65	900	Mn	3.47	Mn	-0.88	Li	3.02	Li	-0.68	Sr	2.8	Sr	-0.97	Sr	2.81	Sr	-0.99
Fe	14570	0.23	16976	0.31	19312	0.27	16648	0.27	62200	Li	3.1	Li	-1.05	Mn	2.36	Mn	-1.03	K	2.62	K	-1.07	Mn	2.42	Mn	-1.21
Co	12.31	1.23	11.13	1.35	13.54	1.11	12.80	1.28	10	Sr	2.97	Sr	-1.11	K	2.14	K	-1.18	Mn	2.49	Mn	-1.14	K	2.27	Pb	-1.30
Ni	29.31	1.47	33.30	1.88	37.59	1.66	34.17	1.71	20	Na	2.65	Na	-1.27	Mg	2.06	Mg	-1.23	Pb	2.13	Pb	-1.37	Pb	2.27	K	-1.31
Cu	24.18	0.44	23.05	0.52	28.79	0.42	24.84	0.45	55	Mg	2.15	Mg	-1.58	Mo	2.01	Mo	-1.27	Mg	1.85	Mg	-1.57	Mg	1.95	Mg	-1.52
Zn	58.45	0.84	62.05	1.25	87.59	0.89	80.56	1.15	70	Ba	1.93	Ba	-1.73	Pb	1.91	Pb2	-1.34	Ba	1.66	Ba	-1.73	Mo	1.88	Mo	-1.58
Ga	14.50	0.97	14.76	1.18	17.67	0.98	14.97	1.00	15	Cu	1.88	Cu	-1.77	Ba	1.73	Ba	-1.48	Cu	1.54	Cu	-1.84	Ba	1.77	Ba	-1.67
As	3.66	2.03	4.28	2.79	5.02	2.38	7.47	4.15	1.8	Pb	1.84	Pb	-1.80	Cu	1.69	Cu	-1.52	Na	1.48	Cs	-1.89	Cu	1.69	Cu	-1.73
Rb	18.32	0.20	23.35	0.29	26.26	0.26	23.90	0.27	90	Mo	1.52	Mo	-2.07	Cs	1.39	Cs	-1.79	Cs	1.48	Na	-1.89	Na	1.46	Na	-1.94
Sr	260.79	0.70	286.81	1.03	386.26	0.76	282.45	0.75	375	Cs	1.32	Cs	-2.28	U	1.37	U	-1.82	Mo	1.34	Mo	-2.03	Cs	1.34	Cs	-2.07

S/El	SR1		SR5		SR6		SR7		Cr. Av.	SR1				SR5				SR6				SR7			
	C ppm	**CF**	**C ppm**	**CF**	**C ppm**	**CF**	**C ppm**	**CF**		**El.**	**EF**	**El.**	**Igeo**	**El.**	**EF**	**El.**	**Igeo**	**El.**	**EF**	**El.**	**Igeo**	**El.**	**EF**	**El.**	**Igeo**
Mo	0.54	0.36	0.55	0.62	0.93	0.37	0.75	0.50	1.5	V	1.26	V	-2.35	V	1.3	V	-1.90	U	1.28	U	-2.10	Cr	1.32	Cr	-2.09
Ag	0.10	1.65	0.11	2.23	0.13	1.81	0.09	1.55	0.06	Cr	1.26	Cr	-2.35	Na	1.28	Na	-1.92	Cr	1.24	Cr	-2.15	U	1.26	U	-2.16
Cd	0.26	2.60	0.33	3.30	0.33	3.32	0.33	3.31	0.1	U	1.13	U	-2.50	Cr	1.26	Cr	-1.94	V	1.2	V	-2.19	V	1.24	V	-2.18
In	0.02	0.18	0.03	0.27	0.03	0.24	0.03	0.23	0.11	Be	1.09	Be	-2.56	Fe	1	Fe	-2.27	Bi	1.01	Bi	-2.45	Fe	1	Fe	-2.49
Sn	0.42	0.17	0.39	0.20	0.50	0.16	0.26	0.11	2.5	Bi	1.03	Bi	-2.63	Be	0.97	Be	-2.31	Fe	1	Fe	-2.46	Rb	0.99	Rb	-2.50
Sb	0.00	0.01	0.01	0.02	0.00	0.04	0.00	0.01	0.2	Fe	1	Fe	-2.68	Rb	0.94	Rb	-2.36	Be	0.96	Be	-2.52	Bi	0.98	Bi	-2.52
Cs	0.93	0.31	1.22	0.43	1.30	0.41	1.07	0.36	3.00	Tl	0.98	Tl	-2.71	Bi	0.91	Bi	-2.40	Rb	0.95	Rb	-2.53	Be	0.97	Be	-2.53
Ba	180.53	0.45	181.46	0.54	215.05	0.45	189.04	0.47	400	Al	0.95	Al	-2.75	In	0.88	In	-2.46	In	0.88	In	-2.64	In	0.86	In	-2.71
Tl	0.11	0.23	0.11	0.25	0.13	0.21	0.11	0.22	0.5	Rb	0.87	Rb	-2.88	Al	0.85	Al	-2.50	Al	0.82	Al	-2.75	Al	0.82	Tl	-2.77
Pb	6.48	0.43	8.71	0.59	8.88	0.58	9.13	0.61	15	In	0.78	In	-3.03	Tl	0.82	Tl	-2.57	Tl	0.78	Tl	-2.81	Tl	0.82	Al	-2.77
Bi	0.05	0.24	0.06	0.28	0.06	0.28	0.05	0.26	0.2	Sn	0.72	Sn	-3.14	Sn	0.64	Sn	-2.92	Sn	0.57	Sn	-3.27	Sn	0.39	Sn	-3.83
U	0.53	0.26	0.70	0.43	0.85	0.35	0.67	0.34	2.0	Sb	0.04	Sb	-7.51	Sb	0.06	Sb	-6.29	Sb	0.15	Sb	-5.20	Sb	0.05	Sb	-6.95
Av.	4549.3		5046.8		5599.7		4452.5		8495.5																

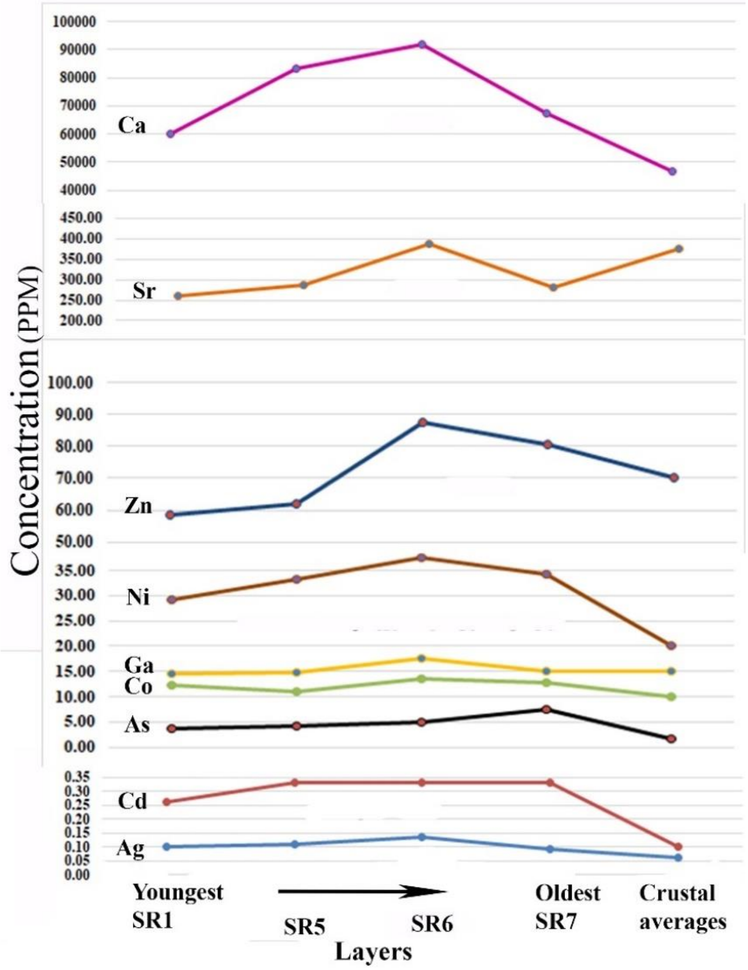


Figure 4. Comparison of metal concentrations in the samples to those of the earth's crust.

The enrichment factor (EF) (Fig. 5) and the geo-accumulation index (Igeo) (Fig. 6) values are presented in a descending order in Table 1. Nearly, all the samples comprise the same enriched metals that likely originated by anthropogenic processes (EF>1.5), for example the least number of enriched metals (16 metals) is present in sample SR6 (Cd, As, Ag, Ca, Ni, Co, Ga, Zn, Li, Sr, K, Mn, Pb, Mg, Ba, Cu), while SR1 comprises two more enriched metals: Na and Mo (total = 18 metals). Five metals Cd, Ag, Ca, As and Ni show significant enrichment; Co shows significant enrichment in sample SR1 and moderate enrichment in samples SR5, SR6 and SR7. The metals (Zn, K, Sr, Na, Pb, Ba, Li, Mn, Mg, Mo, Cu) show low enrichment in all the samples, while the rest of metals show no enrichment; see Table 1 and Fig. 5 for more details. Few elements present clear anomalies in their concentrations in one sample compared to the other ones; for instance, As in sample SR7, K and Mn in sample SR1 (Fig. 5).

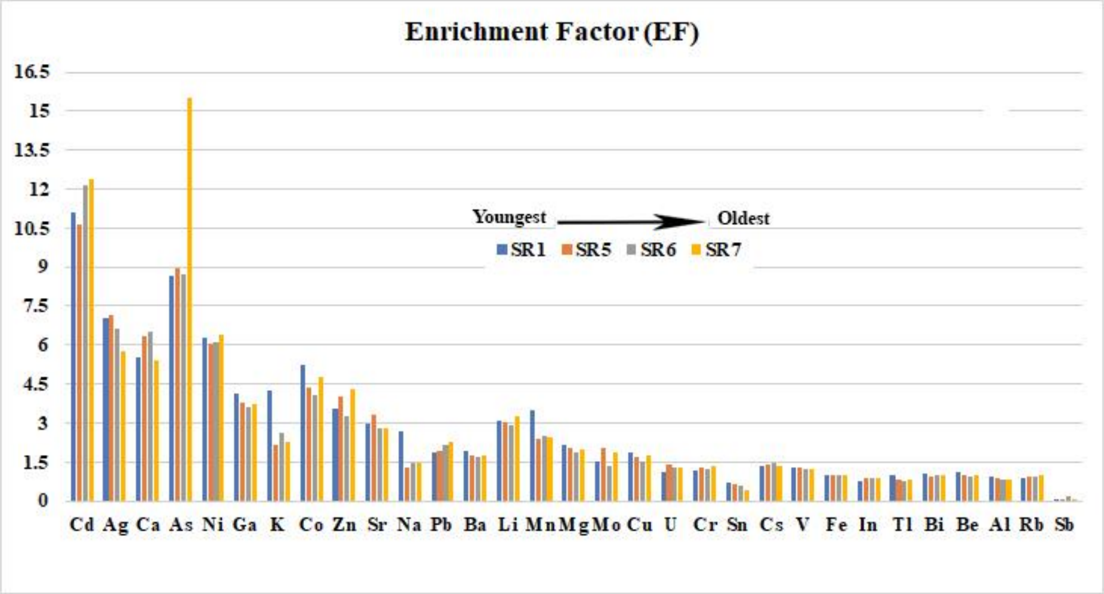


Figure 5. Enrichment Factor of the metals in the samples.

The Igeo index was used to assess the quality of the samples and the extent of their metal contamination. As shown in Fig. 6, Igeo values (Igeo< 0) indicate unpolluted samples by most of the metals, Igeo values (0 <Igeo< 1) indicate unpolluted to moderately polluted samples by Ni, Cd, Ag, Ca, and As, while 1 < Igeo< 2 values of Ni in sample SR7 and Cd in samples SR5, SR6, and SR7 indicate moderate pollution. The average Igeo values of the above metals were ranked as: Cd> As> Ag> Ni> Ca.

Most of the CF values are less than 1 indicating low levels of contamination. The CF values between 1 and 3 indicate moderate level of pollution by Ca, Co, Ni, and Ag in all the four samples, Zn in samples SR5 and SR7, Ga in sample SR5, As in sample SR1, SR5, SR6, and Cd in SR1, while CF> 3 indicates considerable level of pollution by As in sample SR7 and Cd in samples SR5, SR6 and SR7. These results concord with the results of EF and Igeo.

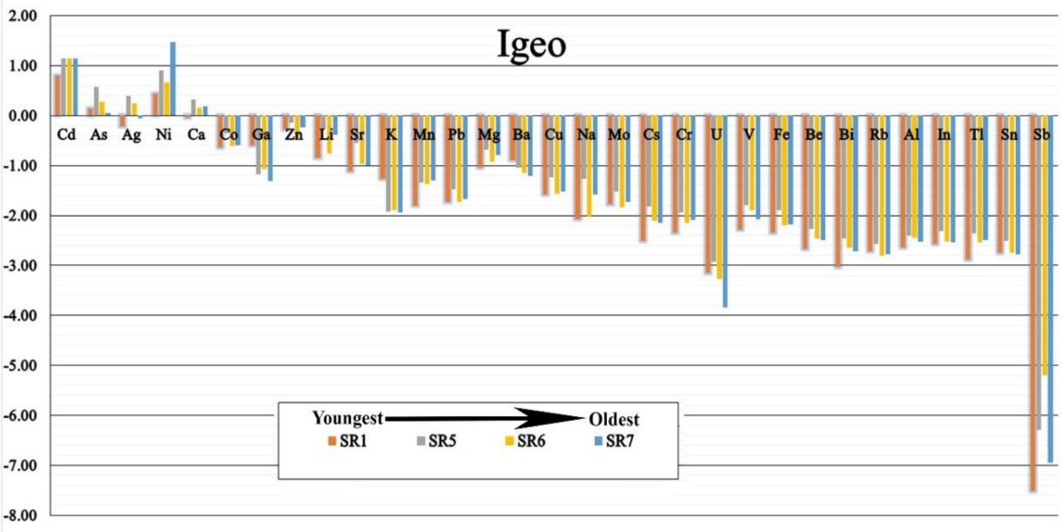


Fig. 6. The Igeo values of the samples.

The results show that the late Ayyubid-Mamluk period has the minimal concentration values of the enriched elements except for Co and Ag. The variations of the concentrations of the metals in the archaeological layers show different trends (Fig. 4). The main trend presents an increase from the late Ayyubid-Mamluk layer to the early Ayyubid-Mamluk layer, to the Byzantine layer, then a decrease towards the Roman layer; Ca, Sr, Zn, Ni, Ga, and Ag follow this trend. Cd has a similar trend but its values in SR5, SR6 and SR7 are the same (0.33). Sr, Zn, and Ga have a similar trend, but their values exceed the background average values only in the early or/and the late Ayyubid-Mamluk layers (SR5, SR6); Table 1.

The second trend shows a decrease in the metal concentrations from the late Ayyubid-Mamluk layer to the early Ayyubid-Mamluk layer, an increase to the Byzantine layer, then a decrease to Roman layer; Co follows this trend. The third trend shows a continuous increase in the values from the late Ayyubid-Mamluk layer to early Ayyubid-Mamluk layer, to the Byzantine layer, to the Roman layer; As demonstrates this trend.

In this study, the heavy metals of the enriched values (EF> 1.5) are considered to be anthropogenic, while the rest of the measured metals are natural or geogenic. The likely major source of anthropogenic metals are organic fuels and their firing product of ash, while the archaeological materials especially ceramics might be a secondary source for the enriched metals.

Long-term ancient human activities of baking, cooking, and heating that depended on burning different kinds of fuel mainly wood and dung, produced substantial quantities of ash and charcoals, which were clearly observed in the archaeological layers of Area A, see fig. 2. The quantities of charcoal (seen by the naked eye), and ash largely spread in layers (visually evaluated) most likely indicate house activities of a large community (Fig. 2). As said above, the excavations at Area A uncovered broken Tabun fragments, and two *in situ* ovens (Tabuns); the new one (95cm diameter) built on the stone base of the older one (100cm diameter). Pottery findings indicate their use during the early Islamic periods.

The results support the archaeological data about the site, in contrast to the Roman period, the site largely developed during the Byzantine period and to a less extent during the Islamic periods. The larger community of the Byzantine period most probably had sizeable house-based activities of firing, animal husbandry, and food processing where their waste materials and residues were probably the direct source of the examined heavy metal concentrations.

Wood, brushes, charcoal, olive oil pressing waste and animal and poultry manures were the most common fuels used for combustion in antiquity. Combustion of these fuels (mainly wood) in various activities would produce, accumulate and distribute the ash enriched in many metals along the layers (Pierce et al. 1998; Zhang et al. 2005). These metals originate from the normal chemical composition of plants used as a fuel (Aston et al. 1998).

Similar results were presented by previous studies. Monge et al. (2015) found that guano and fly and wood ashes caused enrichments of Zn, Ni and Cu in archaeological layers of four caves from the Iberian Peninsula. Zhang et al. (2005) suggested that the high accumulation of Cu, Zn, and Pb in cultural layers of Nanjing city (China) reflected a long-lasting input of pollutants from metal processing, paints, sludge and other anthropogenic sources. Theden-Ringl and Gadd (2017) presented anthropogenic enrichments of Cu and Zn, and biological enrichment of Br and P from a stratified archaeological cave deposit at Wee Jasper, Australia. Ottaway and Matthews (1988) found that the values of Cu and Zn at the site of Gomolava in Eastern Europe are low compared to expected high values from metal working, meanwhile they match phosphate values from excretion.

Previous studies showed the association of Co, Zn, Pb, Ni, Mg and Cu with human and animal wastes (Bintliff and Degryse 2022). While Wilson et al. (2005) and Wilson et al. (2008:423) showed high concentrations of Ca, P, Ba, Sr, P, Pb, and Zn in archaeological hearths from six abandoned farms in the UK, Aston et al. (1998:467) concluded that P, Cd, Mn, Zn, Cu, Pb, Mo, Ni, Co, and Cr (phosphorus >> cadmium > manganese > zinc >> other metals) were enriched in archaeological soils in a rural site adjacent to the village of Shapwick in central Somerset (UK) by faeces and combustion of woody materials.

It is not improbable that pottery fragments represent a secondary factor to enrich the layers with some of these metals especially those used in their painting i.e. Fe, Mn, Cu, As etc. (James 1999). The excavated area produced a substantial number of pottery fragments, where the Ayyubid-Mamluk fragments were decorated with black, brown or dark red geometric motifs; see Al-Bashaireh et al. (2021) for more details. Other studies showed the effect of the spread of ceramics on the concentration of some metals in soils. James (1999) showed that chemical elements, including Pb, Cu, Zn, Mn, Ca, K, and clay content, have high concentrations in areas of artefacts spread, mainly ceramics. Wells et al. (2000) interpreted the high concentrations of Fe, Hg, Mn, and Cu associated with archaeological soils at bases of some structures in Piedras Negreas, Guatemala, as probable residues of paint used in the decoration of stucco using ochre and other minerals; i.e hematite, cinnabar, pyrolusite, malachite, and azurite. The main sources of Arsenic anthropological pollution are mining and burning of fossil fuels (Patel et al. 2023: 8804). However, Arsenic (As) was used in the production of pigments (Gliozzo and Burgio 2022), so it is probable that pottery paints and pigments are its source.

The site is rural, small and of limited sources. It is unlikely that metal production activities contributed to the concentrations of the studied metals. Surveys and excavations (although limited) did not uncover signs of metal production i.e. hearths and smelting or shaping wastes. It is likely that the inhabitants of Umm el-Surab depended on neighboring sites like Jerash or Bosra (Holdridge et al. 2021) to import metal products.

In contrast to the results of the non-industrial Umm el-Surab nsite, heavy metal analysis of sediments from Wadi Faynan (an ancient copper industrial area, southern Jordan; Fig. 1) showed very high concentrations of Cu, Pb, Tl, far above typical crustal concentrations (Grattan et al., 2007; Grattan et al., 2013; Grattan et al., 2016; Knabb et al., 2016). Copper concentrations in the sediments of the Khirbat Faynan barrage between the Iron Age and Byzantine period have several hundred times the average of the crustal value, typical of heavily-polluted industrial environments, and still have significant environmental pollution impacts (Barker et al. 2000; Gilbertson et al. 2007). It is likely that the low thallium concentrations in the Umm el-Surab archaeological layers were of natural origin. Higher concentration of thallium of anthropogenic origin results from its condensation, during smelting, onto the surfaces of ash and charcoal particles which precipitate in the sediments (Grattan et al. 2007:87). These results confirm the archaeological record of the absence of industrial features at Umm el-Surab. Research of heavy metals in sediments of Faynan showed strong positive correlation between the presence of ash and charcoal (and their dust) and high concentrations of heavy metals (Grattan et al. 2007: 88; 2013; 2016: 253). This evidence strongly supports the interpretation of the results of this study showing the effect of these materials on increasing the concentrations of some metals.

Recently, at Gerash, Holdridge et al. (2021) examined the heavy metals pollution inside and outside the city center from the early Roman period until the end of the Umayyad period and found high concentrations of Pb, Cu, Sn and As. They concluded that the major pollution sources were associated with the residues of common, cumulative artisanal and daily activities in the Roman provincial city; mainly the wastes from the household, agriculture, metal working, pigment production, and sewage systems. Their conclusions support the results of this study that household and wastes from daily activities are considered sources of heavy metals pollution.

5. CONCLUSIONS

Assessment studies of pollution of archaeological sites in north Jordan by chemical analysis of metals in archaeological layers are rare compared to those in the south, specifically Wadi Faynan. The results presented moderate pollution by Ca, Co, Ni in all samples, and Zn and As in some samples, while Cd and As levels in some samples are considerable. The rest of the studied metals in all the samples showed low pollution level. Firing of dung and wood, and use of ovens for food preparation, heating and other activities produced ash, and distributed it within the layers, which was the main cause for the enrichment of the metals. The substantial number of pottery fragments uncovered from the layers, especially those painted Ayyubid-Mamluk wares, probably contributed to the enrichment of these metals. Although the number of analyzed samples is small, the results were able to show the difference in pollution between the Roman, late Byzantine and early/late Ayyubid-Mamluk layers.

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تقييم التلوث المعدني في طبقات أثرية من موقع أم السرب، شمال شرق الأردن

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ملخص

في هذه الدراسة، تم فحص تركيز 21 فلزا لأربع طبقات رومانية وبيزنطية وأيوبية مملوكية مبكرة ومتأخرة من المنطقة أ في موقع أم السرب الأثري، الأردن، لتقييم مدى تلوثها بشريا. سُكن الموقع، الواقع بالقرب من بصرى وأم الجمال، باستمرار منذ العصر الروماني حتى العصور الإسلامية. تظهر نتائج التحليل الكيميائي ومؤشرات التلوث (معاملات الإثراء والتلوث، ومؤشر التراكم الجيولوجي) انخفاض الإثراء في عناصر ال *Zn*، *K*، *Sr*، *Na*، *Pb*، *Ba*، *Li*، *Mn*، *Mg*، *Mo*، و*Cu*، وإثراء معتدل في عناصر ال *Cd*، *Ag*، *Ca*، *As*، و *Ni*. إثراء الكوبالت يظهر بشكل واضح في عينة واحدة وبشكل معتدل في العينات الأخرى. تظهر النتائج إثراء أكثر للفلزات الملوثة في الطبقة البيزنطية من بقية الطبقات. المصدر الرئيسي المحتمل للتلوث البسيط في الطبقات هو الوقود العضوي والرماد الناتج عن حرقه، في حين أن المصدر الثانوي المحتمل قد يكون محتويات كسر الفخار المنتشرة في الطبقات. تتفق النتائج مع حقيقة أن الموقع كان ريفيا بأنشطة صناعية محدودة، وخاصة صناعة الفلزات كالحديد والنحاس الذي كان سيزيد من تركيز الملوثات الفلزية التي تم تحليلها.

الكلمات الدالة: طبقات أثرية، معاملا الإثراء والتلوث، مؤشر التراكم الأرضي، رماد، تلوث، أم السرب، الأردن.

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