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SYNCHROTRON-BASED X-RAY FLUORESCENCE ANALYSIS OF BYZANTINE PLASTER FIGURINES FROM JORDAN MUSEUM

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ABSTRACT

Non-destructive analysis techniques became an important approach for the characterisation of cultural heritage and conservation science. In this study, for the first time, Synchrotron-based X-ray Fluorescence (SR-XRF) analysis, at the Synchrotron-light for Experimental Science and Applications in the Middle East (SESAME), was utilized to examine and analyze three Byzantine plaster figurines from Jordan Museum. Analysis was applied to identify the main composition of the body, the black drawings on the figurines and the mirror fragment on one of the objects. The results showed that the matrix of the body is composed of Calcium carbonate (plaster) and other inclusions were added to facilitate setting of the lime during hydrating. The black drawing on the objects was identified as wooden charcoal. Finally, results showed that the mirror is not a silicon based but a highly polished surface material composed of manganese (Mn), galium (Ga) and lead (Pb).

KEYWORDS: Synchrotron-based X-ray Fluorescence, Jordan, SESAME, plaster figurines.

1. INTRODUCTION

In Jordan, figurine production has been known since Neolithic times. They were found in different archaeological sites (Kuijt & Insoll, 2017). Figurines are a three-dimensional anthropomorphic or zoomorphic representation (Badre, 1995; Kuijt & Insoll, 2017; Mahasneh & Bienert, 1999). Human figurine objects might be an exact representation of human body, or resemblance to some parts of it (Bailey 2005; Joyce 1993). Most studies of the figurines objects were to interpret and understand the meaning and the function of those objects (Ucko 1968; Voigt 2000; Bailey 2005; Kujit, 2008; Twiss, 2001). Other studies were undertaken to explore when they were produced, the manufacturing technology and the material that was used in their production. There are different types of materials that were used to produce the figurines objects. For example, terracotta figurines are very familiar and largely produced in Jordan (Petit, 2009; Petit & Kafai, 2016; Tuttle, 2009; Vieweger and Haser, 2009; Uhlenbrock 2015; Hunziker-Rodewald 2021). Other types were made mainly of plaster material, such as the statues of 'Ain Ghazal and Khirbet es-Samrā figurines (Kafafi, 2000; Rollefson, 1983; Grisson, 2000; Nabulsi, 2019). In very few cases, flint figurines were also found in Jordan (e.g. Ibáñez et al., 2020).

Despite the archaeological importance of those objects, few analytical studies have been performed to identify their chemical and mineralogical composition. This is mainly because most of the analytical methods are destructive techniques in which probe sampling is required. This usually is not allowed to such objects which are unique in nature. Therefore, analysis was performed to either broken or incomplete fragments of the figurines (Hunziker-Rodewald & Fornaro, 2019; Bennallack, 2012). However, the recent developments in analytical instrumentation have allowed performing chemical and physical characterisation for archaeological objects without intervention, or with the least possible intervention to the artefacts.

The most common instrumentation now is the portable XRF (pXRF), with hand-held systems, that are used for non-destructive, non-invasive and in insitu characterization (Madariaga et al. 2014; Liritzis & Zacharias, 2011; Shugar, 2013; Shackley, 2012). The pXRF has been a primary tool for elemental compositional analysis of stone and ceramic artefacts, pigments in paintings and glazes, and metal alloy content (Zacharias et al. 2009; Liritzis & Zacharias, 2011).

Alternatively, Synchrotron photon-based methods are increasingly and widely used now for archaeological investigation and conservation science (Bertrand et al., 2012; Bertrand, 2007; Cotte et al., 2010; Creagh et al., 2007).

The Synchrotron analysis provide invaluable information of the chemical and physical structure of the objects, conservation state and manufacturing technology for the objects (Paterson & Daryl, 2019; Dooryhée et al., 2004). The brightness of the Synchrotron beam increases the accuracy of the obtained data, especially for elements present only in small concentrations.

X-ray fluorescence (XRF) analysis is one of the analytical techniques used in beamlines at Synchrotron facilities. The technique can deliver elemental analysis by identifying the presence of more than 80 elements (Mantler & Schreiner, 2000; Koval'chuk et al., 2016; Brand et al., 2019; Janssens et al., 2000; Pantos, 2005; Aloupi et al., 2000). It provides reliable quantitative analysis for the detected elements.

In this study, SR-XRF analysis technique is used to characterize Byzantian plaster figurines that are found in Khirbet es-Samrā archaeological site in Jordan and displayed in Jordan museum since 2014. The study aims to identify the main elemental composition of the body context and black painting.

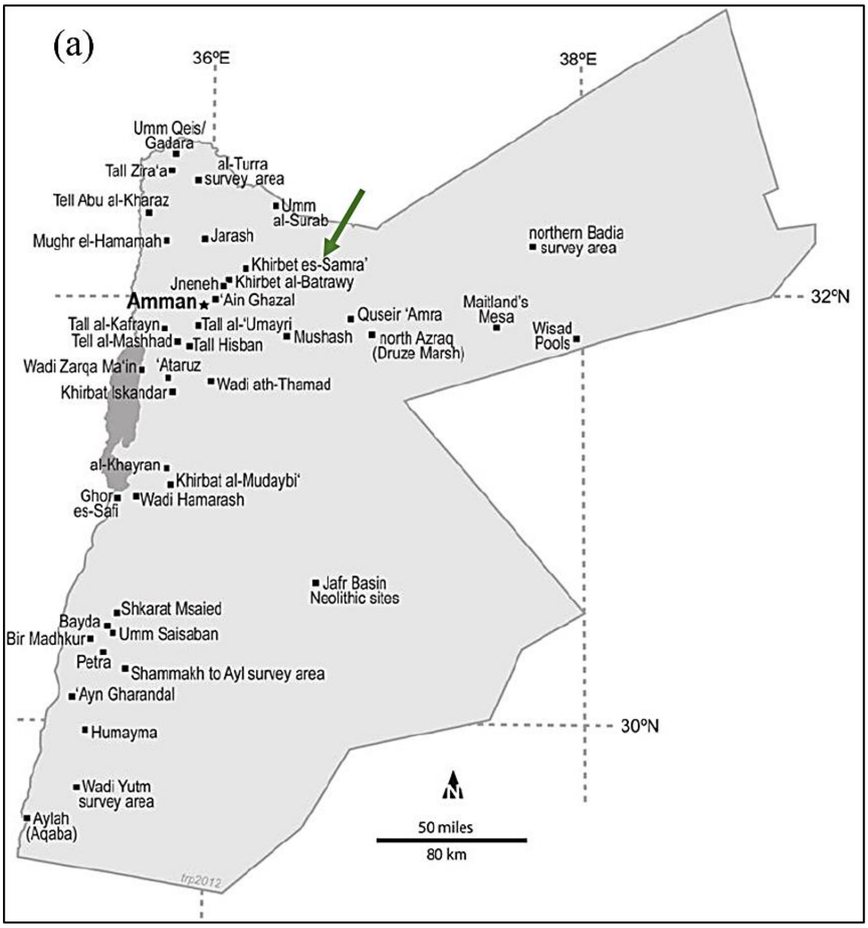

Figure 1. Jordan map, green arrow showing the location of Khirbet es-Samrā archaeological site in north-eastern Jordan (after Keller et al., 2012).

2. MATERIAL AND METHOD

2.1 The figurines

Several small size plaster figurines are displayed in Jordan Museum. The objects were excavated from the Byzantian archaeological site Khirbet es-Samrā. The site is located about 50 km northwest Amman (Fig. 1) (Nabulsi 2007). During the excavation works, several figurines were found in the ancient cemetery. The objects have been displayed in the Jordan Museum since 2014, and until now, no analytical or elemental analysis was performed to any of those figurines.

The objects are small and almost flat, about 10-35 cm high and 1 mm thick. They do not represent realistic human body proportion. The hands and feet usually short compared to the body. In some figurines, the hands are extended to the sides, while other figurines, the hands are raised to the head level (see Fig. 2).

From the visual inspection of the objects, the figurines are white in colour, with thin black lines used to draw the eyes, eyebrows and decoration around the hands, neck, dress and head (see Fig. 2). The objects show no marks of deterioration or weathering damage. However, some objects have been subjected to restoration works by adhering the broken parts.

Because of the fragility of the figurines, only three objects we could transfer from Jordan Museum to SESAME Synchrotron facility.

The three objects selected for this study are KS-0030, KS-1048 and KS-1051. The objects are shown in Fig. 3 a-c. Analysis was performed on different single points on the figurines body, as shown in Fig. 3(a-c), in order to characterise the elemental composition of the body and the black painting used for the drawings of facial features and decorations.

Figure 2. Example of female plaster figurines from Khirbet es-Samrā site which are displayed in Jordan Museum (Photo by Sahar al Khasawneh)

2.2 Experimental technique

The objects were subject to elemental composition analysis via SR-XRF technique at the BM08-XAFS/XRF beamline (Harfouche et al., 2022). The use of the tenability energy allowed separating overlapping fluorescence emission lines such as the K_a of As and La of Pb by changing the monochromatic photon between energy before and after the excitation energy of each element. The figurines were placed carefully in the photon beam path with an angle of 45° with respect to the incoming beam and the fluorescence detector. The latter was an energy selective 64-element array detector presenting an energy resolution of ~145eV @ 5.9 keV (Rachevski et al., 2019). Several points were chosen on each object for measurements points are shown in Fig. 3 (a-c). For each point, SR-XRF data were collected for 300 seconds exposure time, in parallel the distance between the point and the detector as well as the incoming photon flux were recorded. Moreover, SR-XRF spectra were collected on certified reference samples (Ore Research & Exploration Pty Ltd) for quantification Analysis. We aimed with these measurements to identify the elemental composition of the following;

1. Body of the figurines, plain white, no colours or stains (KS-0030-a & b, KS-1048-a, and KS-1051-a).
2. The black lines that decorated the body or used for drawing eyes (KS-0030-c, KS-1048-c, and KS-1051-b).
3. The mirror on the head (KS-0030-a.)

Collected SR-XRF spectra were analysed using PyMCA software package, an open-source developed by the Software Group of the European Synchrotron Radiation Facility (ESRF) in Grenoble (Solé et al., 2007). The program can be used to identify the elements composition in the sample by assigning the energy of the peaks in the spectra. Knowing the experimental setup geometry such as the distance between the sample and the detector and the absorbing matrix, the counting time and the deadtime of count rats as well as the detector specifications, PyMCA al-lows measuring the concentration of each element present in the sample matrix. The standard deviation (SD) from the counting statistics of an individual measurement is defined as the square root of the gross counts for the target peak.

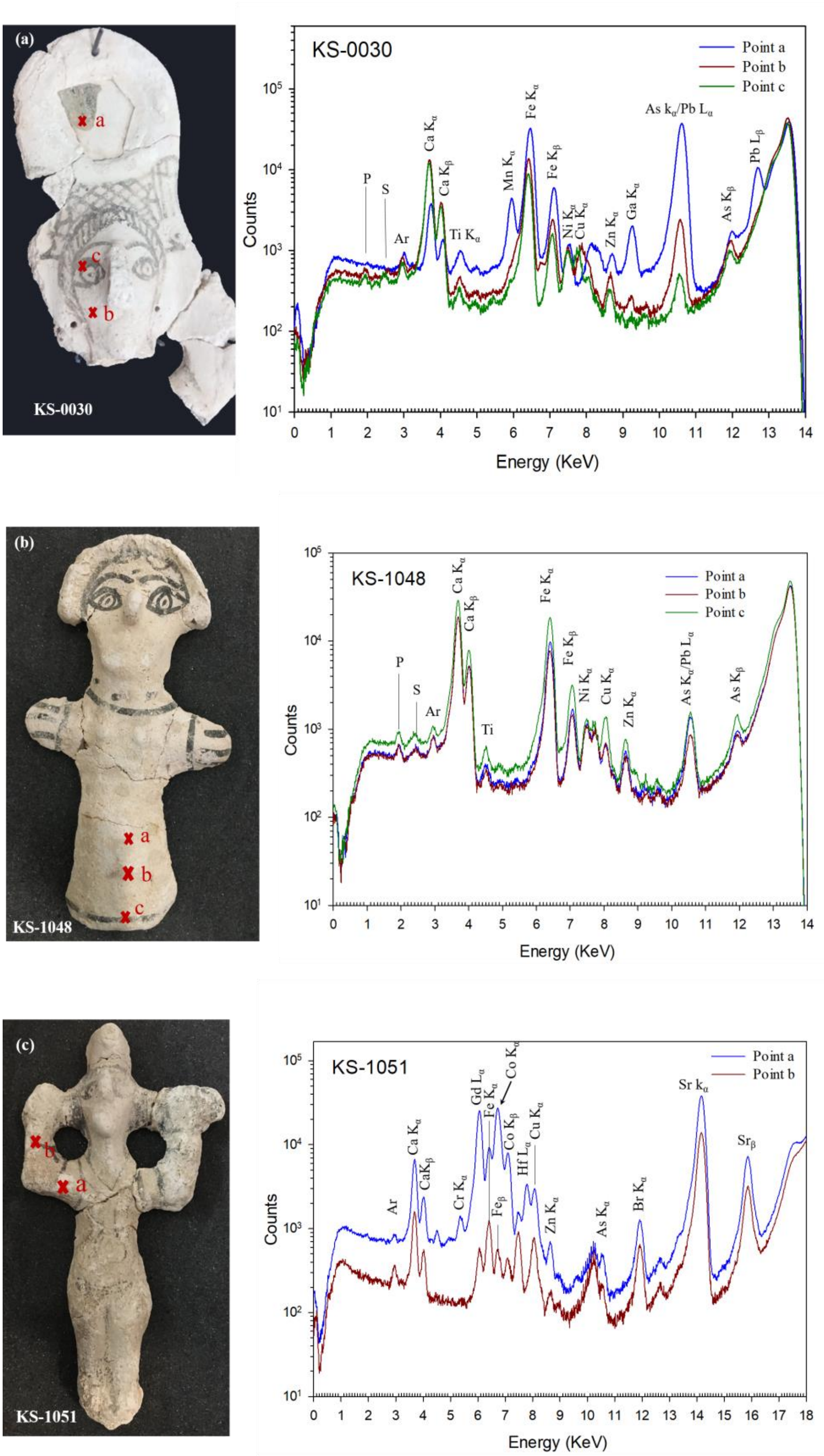

Figure 3. photograph of each sample (left) together with its corresponding XRF spectrum of the analysed points (right). We used the same code that the objects are registered in the museum records. (Photo by Sahar al Khasawneh)

3. RESULTS AND DISCUSSION

Graphs of Fig. 3 (a-c) show, on a logarithmic scale, the SR-XRF spectra of each point excited with X-rays of 13.5 keV except for object KS-1051, which we used high excitation energy at 18.2 keV. We considered in our analysis to identify elements, that the peaks are centred at energies within about ±0.05 keV to their reference line energies.

The peak of calcium element (Ca) presents the highest fluorescence intensity in all the spectra of all measured points (Table 1). Fe is the second element that has high intensity in all the measured points.

Table 1. Concentration (ppm) of all elements obtained by XAFS/XRF beamline.

KS-0030	**point a**	**Point b**	**Point c**
Element	**(ppm)**	**(ppm)**	**(ppm)**
P	1340	1407	1543
S	970	963	1162
Ca	387612.03 (38.76 wt.%)	550222.4 (55.02 wt.%)	472110.5 (47.20 wt.%)
Ti	77.09	80.58	51.03
Cr	12.26	7.79	28.41
Mn	718.33	46.62	38.30
Fe	8820.05 (0.80 wt.%)	75582.34 (0.75 wt.%)	4891.93 (0.49 wt.%)
Ni	63.50	98.29	106.96
Cu	42.99	50.93	24.29
Zn	5.22	17.78	10.64
As	4.49	0.006	0.08
Pb	3113.01 (0.03 wt.%)	373.30	53.51

KS-1048	**Point a**	**Point b**	**Point c**
Element	**(ppm)**	**(ppm)**	**(ppm)**
P	-	1019	1348
S	-	814	1019
Ca	472601.70 (47.26 wt.%)	471089.4 (47.10 wt.%)	493726.4 (49.40 wt.%)
Ti	46.37	30.91	72.19
Cr	19.17	2.39	6.15
Mn	14.68	1.20	5.25
Fe	4697.58 (0.47 wt.%)	3510.64 (0.35 wt.%)	5361.15 (0.53 wt.%)
Ni	74.083	77.77	56.55
Cu	21.13	26.47	49.71
Zn	14.80	11.95	19.99
As	1.41	1.33	2.54
Pb	220.00	101.00	247.1

KS-1051	**Point a**	**Point b**
Element	**(ppm)**	**(ppm)**
Ca	596644.92 (59.66 wt.%)	123488.68 (12.35 wt.%)
Fe	220.80	238.92
Ni	11.1	60.12
Cu	4.29	4.93
Zn	2.12	4.16
As	1.51	0.077
Ga	231.3	38.07
Ho	-	22.03
Pb	-	94.82

Other major elements are also detected Cu and Mn. High peak around 10.5 keV is observed almost at all measured points. The analytical software identified it as the emission line for Pb (*La*, ~10.55 keV) and combined it with the peak around 12.7 keV as Pb (L_β) line. However, knowing that the ratio between L_α and L_β is always about 1:1 (Van Grieken & Markowicz, 2002), which is not the case for those two peaks, we ruled out that this peak is Pb. However, the Pb-*La* (~10.50 keV) and the As-K_α (~10.52 keV) emission lines are overlapping when considering the detector resolution

(130 eV). To work around and overcome these overlapping lines issue, we used the advantage of the energy tenability offered by the beamline to collect SR-XRF data at two different energies that are 12.5 and 13.5 keV. As shown in Fig. 4, at 12.5 keV we allow just As-K_α to be excited (binding energy = 11.867 keV) and at 13.5 keV we excite both As and Pb (binding energy = 13.035 keV). As mentioned above, fit is considered within ±0.05 keV to the reference line. This is also shown in Fig. 3c, where an excitation at 18.2 KeV is used. Some other elements detected in most of the points are Co, Ni, Ti and Zn. Peaks with lowest intensities at some points were P, S, Cl and Ar.

Comparing the intensities of the elements with each other, we can correlate elements to identify mineralogical composition of the objects. The presence of Ca in high concentrations and the low concentration of Si and absence of Al, exclude the existence of calcium-silicate nor calcium- aluminate. On the other hand, the absence of the S, or existence in a very low concentration, rule out the use of calcium sulphate (gypsum plaster), and confirms the using of the calcium carbonate $CaCO_3$ as the principal component of the figurines body.

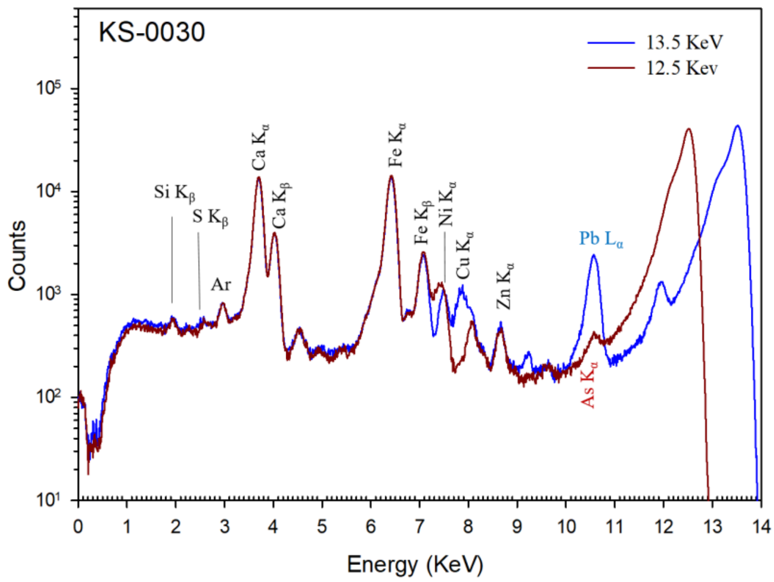

Figure 4. XRF spectra collected at two different energies (12.5 and 13.5 keV) to distinguish the contributions from As and Pb in the same sample (KS-1048-point a)

To identify the black pigment used for drawing the lines, we compared the SR-XRF spectrum of the unpainted body and the black lines on the body. Both spectra were almost identical. The spectra showed no marked increases in Mn content or Fe. This excludes the using of the Fe ores (magnetite Fe_2O_3), nor Mn-based pigments for the black drawing as detected in other painted materials (Castañeda et al., 2019; Koenig et al., 2014; Hyman et al., 1996; Zolensky, 1982), and indicate to the use of carbon-based pigments (Angeli et al., 2018; Rifkin et al., 2016; López-Montalvo et al., 2014). Alternatively, the absence of P (phosphorous) excludes the use of black pigments extracted from animal bones. This suggests that the black drawings have been applied using wooden charcoal (Roldán et al., 2013; Aloupi et al., 2000).

In the other hand, the XRF spectra from the body and the mirror (point a&b in figurine object KS-0030) showed a higher concentration of Mn and Pb in addition to the appearance of Ga fluorescence emission lines. In addition to the absence of Si element, this might suggest that the mirror is not a Si based object but a highly polished material containing Pb/As, Mn and Gd components.

The significance presence of Fe and those of Cu, K, Co, Cr, and Ti in most of measured points indicates the presence of other ores, such as hematite, cuprite, and other impurities when forming the aggregate, or impurities in the original source of the limestone. Nevertheless, reviewing the archaeological reports related to the uncovering of these objects, there were signs of mending and ancient restoration works that were applied to the broken parts of the figurines using lime plaster (Nabulsi 2022, 2019), which could be replicating the process of producing the figurines. Aggregate could be added to facilitate setting of the plaster during hydrating. Despite synchrotron XRF analysis are non-destructive technique and safe for archaeological objects like the figurines, however, there is some limitations to the technique. Such as that XRF delivers only information about element-specific and not the chemical state. Therefore, introducing a multi analytical approach will provide detailed and complementary information about the composition of the objects that would be useful for conservation works. In the case of the plaster figurines, FTIR and XRD analysis would be useful to differentiate between limestone, chalk and lime plaster (De Samber et al., 2000; Liritzis & Zacharias, 2011).

4. CONCLUSIONS

The SR-XRF analysis for three figurines from Khirbet es-Samrā results indicated that the matrix of the body is composed of calcium carbonate. Weather calcium carbonate existed as natural limestone or as manufactured plaster can only be proved by further analytical approaches, (e.g., FTIR or petrographic analysis).

The black drawing on the objects was identified as wooden charcoal which carbon-based painting.

The mirror fragments id found to be not a Si based material but a highly polished material.

Despite the limitation of this study, the present work represents the first archaeometric characterization of Khirbet es-Samra figurines objects using X-ray Fluorescence. The study draws the attention to the importance of using the advanced non-destructive analytical instrumentation for the characterization of museums' collection for future conservation works.

Author Contributions: Conceptualization, S.K.; methodology, S.K, M.H., A.A.; software, K.A and M.H.; writing—original draft preparation, S.K. All authors have read and agreed to the published version of the manuscript.

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