A Micro-Analytical Study of Roman Glazed Pottery from Hayyan al–Mushref, Yasila, Tell al-Husn, El- Bediyeh and Khirbet edh–Dharih in Jordan (a Comparative Study)

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Abstract: *This research aimed to evaluate the interdisciplinary approach of several investigative methods (Optical Microscopy, XRF, XRD, SEM-EDX, ATR-FTIR) in the analysis of archaeological objects. Specifically, it explored the morphology of various types of glazed pottery and their corrosion patinas from different sites in Jordan, including Hayyan al-Mushref, Yasila, Tell al-Husn, El-Bediyeh, and Khirbet edh-Dharih. The patinas result from environmental and burial factors that have affected the pottery. Therefore, the glazed pottery patina plays an active role in the formation of the artifact's characteristics. Elemental analysis methods, such as X-ray fluorescence (XRF) and scanning electron microscopy (SEM-EDX), were selected to explore methodological diversity in this study. Additionally, compositional analyses using X-ray diffraction (XRD) and Attenuated Total Reflection-Fourier Transform Infrared Spectroscopy (ATR-FTIR) were employed to examine the crystalline composition of the patinas on the glazed pottery and their molecular structure. The results indicated that the artifacts required restoration to preserve the original material.*

Keywords: Khirbet edh-Dharih, glaze; pottery, corrosion; ATR-FTIR; SEM-EDX, XRD; XRF 1.Introduction.

Analytical techniques have been employed to explore archaeological information by examining the chemical composition of artifacts using advanced methods in archaeological science, such as X-Ray Fluorescence (XRF), Fourier Transform Infrared Spectroscopy (FTIR), and SEM-EDX (Măruţoiu et al., 2018; Papakosta et al., 2020). The study focused on the microstructure and chemical properties of pottery, enabling archaeologists to determine the conditions under which these objects were used. Microstructure refers to the crystal sizes of the pottery, where the crystal alignment of silicate materials is linked to the organic residue in the pottery objects. Additionally, atomic structures and the distribution of trace elements in the fragments provide valuable information about the technology used in producing the objects. New applications of ATR-FTIR and XRF mapping have been utilized to study the mechanical properties of the glazed pottery, including stress and strain in ceramic materials from all sites (Dey et al., 2020). Several ceramic objects excavated from Hayyan al-Mushref, Yasila, Tell al-Husn, and El-Bediyeh in Jordan were analyzed to determine Roman techniques and the composite materials used during that period. In this paper, we present the pottery fragments that are the subjects of the study, which have been dated based on archaeological stratigraphy and context, in addition to typology as identified by the author. The focus is on their material composition, obtained through macroscopic descriptions. Based on the information gathered, we proceeded with the sampling procedure, producing powders and cross-sections according to international standards. Additionally, technical descriptions of the diagnostic methodologies employed have been presented.

The sites of Hayyan al-Mushref, Yasila, Tell al-Husn, and El-Bediyeh are located in

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the northern part of Jordan, no more than 20 km from Irbid, while Khirbet edh-Dharih is situated in southern Jordan (Fig. 1). Modern excavations at these ancient sites on the northern Jordanian plateau have revealed numerous Roman artifacts, including pottery, metals, glass, pigments, and structural pottery. These findings have been studied to enhance our understanding of the development of firing technologies during that period (Al-Muheisen and Piraud-Fournet, 2013; Al-Muheisen and Villeneuve, 2005; Al-Shorman et al., 2023).

Due to the trading nature of Decapolis in Jordan, artifacts such as pottery and glaze have been analytically studied to gain more insight into the people of that period. The study focused on the microstructure and chemical properties of the glaze, which can help archaeometrists and archaeologists determine the conditions under which these artifacts were used. Microstructure refers to the crystal sizes of the pigments, with the crystal alignment of metal oxides used in glaze technology being associated with the deterioration of the inorganic components of pottery. This aspect has both positive and negative consequences. On the one hand, artifacts with scattered provenance make it difficult to identify trends in manufacturing over time at a single production site. In other words, it might be interesting to observe how people at a particular site altered their production processes and techniques—whether for better or worse—over time. However, it would be impossible to use data from our artifacts to draw conclusions about production in these cities themselves. On the other hand, having a relatively random sampling of artifacts from across the Roman and Byzantine Empires and beyond provides a valuable indication of the general state of archaeological materials production in Jordan.

Excavation work was carried out at the archaeological sites of Khirbet edh-Dharih

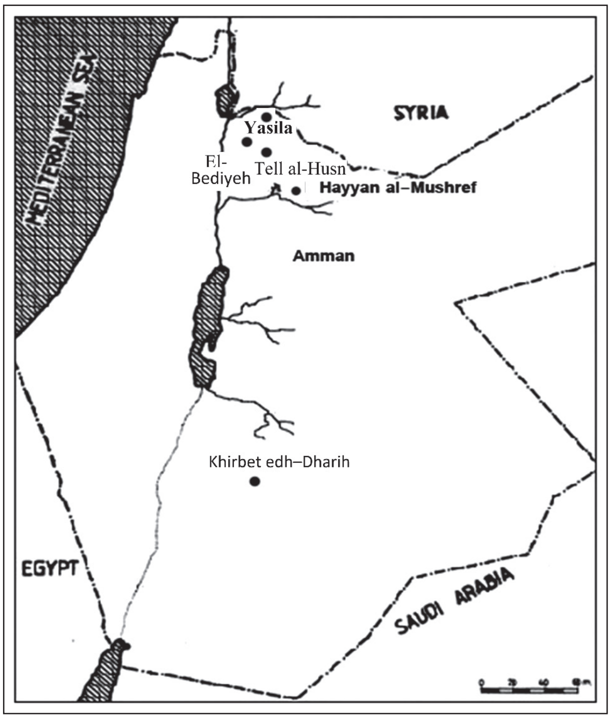

Fig. 1: Map of Jordan showing the locations of studied archaeological sites.

and Hayyan al-Mushref by the Institute of Archaeology and Anthropology at Yarmouk University, in cooperation with the Department of Antiquities, under the supervision of Zeidoun Al-Muheisen. Both sites feature significant architectural structures, including churches, wine presses, public buildings, necropolises, and other notable features. Pottery sherds and objects are the most common archaeological materials found at these sites. The earliest pottery sherds from these sites show signs of settlement dating back to the late Hellenistic period and continuing through the Islamic periods (Al-Shorman et al., 2023; Gregg et al., 2009).

The site of Khirbet edh-Dharih is located 130 km from the Mediterranean coast and approximately 100 km north of Petra (Fig. 1). This site was discovered in 1818 by the British explorers Irby and Mangles, who identified the temple without knowing the name of the site. In the early 20th century, it was briefly reexamined by some explorers and then further investigated in 1930. In 1979, a Canadian team led by Prof. MacDonald began a detailed survey of all the sites in the Wadi El Hesa area. In 1983, brief investigations were conducted by Al-Muheisen and Villeneuve. Thirteen field seasons of excavations (1984–2007) were carried out by the Institute/Faculty of Archaeology and Anthropology at Yarmouk University and the French Institute of Oriental Archaeology, under the direction of Zeidoun Al-Muheisen, in cooperation with the Department of Antiquities of Jordan. Numerous architectural structures were uncovered at the site, along with a significant collection of pottery objects of various types and functions, as well as many finds of glass, metals, and mosaics. According to archaeologists, the site has been continuously inhabited throughout the Lithic, Bronze, Iron, Roman, Byzantine, Nabataean, and Islamic periods (Mayyas et al., 2023; Al-Sekhaneh et al., 2020; Villeneuve and Al-Muheisen, 2008; Lenoble et al., 2001).

The site of Hayyan al-Mushref is located in the northeast of Jordan, approximately 8 km southwest of Mafraq city. Archaeological surveys were conducted by Siegfried Metman in 1970, making him the first to identify the site's location in terms of geography and the periods during which it was inhabited. The site was extensively surveyed and excavated over three seasons from 1995 to 1997, under the direction of Zeidoun Al-Muheisen. The excavation results indicated that the site was occupied from the late Roman period through the Byzantine and up to the Umayyad and Abbasid periods. During these seasons, significant architectural structures, pottery sherds and objects, glass, mosaic floors, and various other materials were uncovered (Abd-Allah and al-Howadi, 2010). Among these findings, a considerable collection of pottery pots was uncovered, particularly in the domestic areas (Al-Muheisen, 1997).

Khirbet edh-Dharih

Khirbet edh-Dharih is situated on the King's Highway, in the Wadi al-Laaban, a southern tributary of Wadi al-Hesa, 12 km north of Tafileh and 7 km south of the high-altitude sanctuary at Khirbet et-Tannour, with which Dharih is closely associated. Dharih is a medium-sized site in the countryside, located 100 km north of Petra (Fig. 1). It is situated near the King's Highway, the most important caravan route linking the North and South of the Near East, and the area is not lacking in abundant water. With the help of the Department of Antiquities and the Governorate of Tafileh, excavations at the site began in 1983 as a joint project between Yarmouk University, IFAPO, and Sorbonne University (Al-Muheisen and Al-Shorman, 2004; Villeneuve and Al-Muheisen, 2004; Lenoble et al., 2001: 89-151; Al-Muheisen and Villeneuve, 1994: 41-45; 1994: 735-57; 1990).

That beautiful site, located within a large uninhabited valley, has become an attractive spot for both local visitors and foreign tourists due to its position on the main tourist route in Jordan. On the other hand, Dharih, along with Humayma (located south of Petra and excavated by a North American team), is one of the only sites in the Nabataean world east of the Jordan River to be thoroughly studied. Its remains are more diversified and better preserved than those at Humayma.

Although such sites may be excavated over many decades, this is not our objective. We have already completed the excavations step by step within the cemeteries, the village, and the surrounding remains—areas that are now well documented. Our focus is now on finishing the excavations within the sanctuary area and its annexes as soon as possible, with the goal of presenting the site to the public in its final form.

2. Materials and Methods

The study focuses on the chemical and physical properties of pottery and glazed pottery using various methods, including ATR-FTIR, X-ray diffraction (XRD), and EDX. It examines specific artifacts with archaeometric potential, employing interdisciplinary methods to analyze the corrosion patina on glazed artifacts. The glazed sherds have complex corrosion patinas on their surfaces, resulting from interactions during the processes that occurred in the archaeological soil or from changes that took place after the objects were excavated (Papakosta et al., 2020; Teoh et al., 2014).

This study presents the pottery sherds that are the focus of our investigation, emphasizing their material composition, which was obtained through macroscopic descriptions. The methods were applied to study the morphology of the complex-structured patina on several pieces of glazed sherds, formed during the underground residence of ancient objects dated from the 1st to the 5th century A.D. (Moon et al., 2021). The microstructure of glaze corrosion products was investigated and characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM) combined with energy dispersive X-ray spectrometry (EDS) at IPHT, Jena, Germany. Clay minerals were not detected, as they decomposed and vitrified during the firing process (Holakooei et al., 2014).

SEM combined with EDS was used to examine the elemental composition of the patina products and core metal. This technique provided additional information about the nature of the minor or amorphous corrosion products that would not have appeared in the XRD spectrum (Annamalai et al., 2014; Weymouth, 1973). Cross-sections were taken from the edge of each artifact using a fine piercing saw. The mineralogical composition of the corrosion products was determined using a Shimadzu LabX XRD-6000 X-ray diffractometer at Yarmouk University, Jordan. A small amount of corrosion product was scraped from the surface of each artifact, mixed with ethanol, and spread on a microscopic glass slide. After the ethanol evaporated, the slide was analyzed using the XRD instrument. Copper Kα radiation (λ = 1.54178 Å) was used for the investigation, with the spectrum acquired at 40 kV, 30 mA, a scale of 2000 counts/min, and a scanning angle range (2θ) from 10° to 60°. The identification of minerals was carried out using PANalytical's X'Pert HighScore software.

SEM is typically a qualitative and morphological technique that allows the study of details not visible to the naked eye. In our study, SEM was chosen because it is indispensable for the preliminary observation of the samples (Gliozzo, 2020). SEM images provided information about the large-scale microstructures present in the samples, as well as the composite minerals or trace elements, using suitable methods (Velraj et al., 2015). Since the fragments of colored glazed pottery are not transparent to visible light, the light beam is not transmitted but reflected, producing an image with magnification up to 1000x and a lateral resolution of 0.5 µm. This enables the morphological and structural study of the stratification of the glaze on the pottery.

Fourier transform infrared spectroscopy (ATR-FTIR) was carried out with a Bruker Tensor 27 FTIR in the range of 4000-400 cm^{-1} with a resolution of 4 cm^{-1}. The samples were prepared as pellets (12 mm in diameter and 4 mm thickness) pressed with potassium bromide (KBr) at a ratio of 1 mg sample/100 mg KBr under a pressure of 5 tons. The second series of experiments used the attenuated total reflectance technique (ATR-FTIR) with 20 scans between 400 and 4000 cm^{-1}, maintaining a resolution of 4 cm^{-1}.

The deposited dirt was mainly composed of calcite, clay minerals, and quartz (calcareous soil remains). Since calcite is destroyed when fired to a temperature above 750°C, the detected calcite in pottery objects is primarily from before the firing process took place.

Figures (2, 3, 9, and 11) show green glazed pottery from Tell al-Husn (Figures 2 and 3) and El-Bediyeh (Figures 9 and 11), depicting ancient pottery sherds made from clay, molded into a desired shape, and coated with a transparent green glaze before being fired at high temperatures. The glaze provides the object with a glossy surface, enhances its visual appeal, and adds a layer of durability by sealing the porous ceramic surface.

Figure (4) shows a glazed pottery sherd from

Fig. 2: shows the patina on the green glaze object from Tell al-Husn Site in Jordan

Fig. 3: shows the patina on the green glaze object from Tell al-Husn Site in Jordan

Fig. 4: shows the patina on the green glaze object from El- Bediyeh Site in Jordan

Fig. 5: Brown glaze, from Yasila, before being fired at high temperatures.

Fig. 6: shows the glazed pottery of an ancient pottery sherd from al Yasila.

Fig. 7: shows an ancient glazed pottery sherd from Tell-al-Husn.

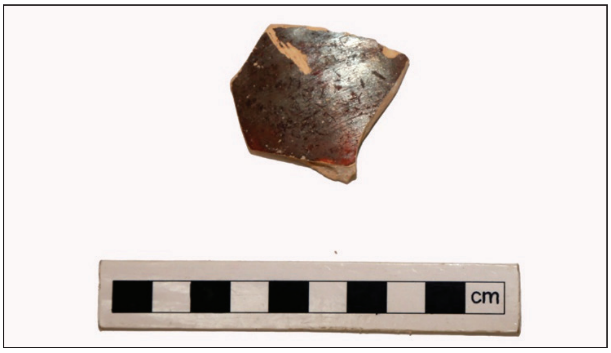

Fig. 8: shows the glazed pottery of an ancient pottery sherd from Al-Yasila.

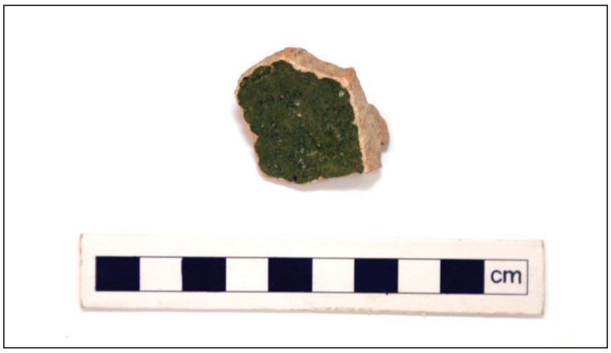

Fig. 9: shows an ancient glazed pottery sherd from El- Bediyeh.

El-Bediyeh, which notably has a patina with an olive-brown colored glaze. A similar patina can be observed in Figure (12), which features a yellow glaze.

Figure (5) shows how the glaze gives the object a glossy surface, enhances its visual appeal, and adds durability by sealing the porous ceramic surface. In contrast, Figure (6) presents an ancient glazed pottery sherd from Al Yasila. The sherd, made of clay, is coated with a brown-line glaze on the base of the bowl and fired. The glaze enhances the object's visual appeal and adds durability.

The sherds in Figures (7 and 8) are made of clay, shaped into the desired form, and coated with a brown glaze before being fired at high temperatures. The glaze provides the object with a glossy surface, enhances its visual appeal, and adds durability by sealing the porous ceramic surface. Figure (10) presents another glazed pottery sherd from Al-Yasila, featuring a blue-colored glaze.

3. Results and Discussion

The glaze initially forms on the surface of the pottery, but over time, it penetrates the interior and may also create thick layers of corrosion products on top of the original pottery surface. If the corrosion has not entirely consumed the glaze, the core of uncorroded material may remain, surrounded by layers of corrosion products. XRD analysis was conducted on the diffractograms of the patina products formed on the artifacts during their long-term burial in ancient soil. Various corrosion products of pottery were identified through XRD analysis after prolonged exposure to the soil.

The analysis of corrosion products from a sample of El-Bediyeh (Fig. 13) and a sample from Al Yasila (Fig. 14) reveals the following minerals and compounds commonly associated with the deterioration of glazed pottery:

- **Calcium magnesium phosphate ($CaMg(PO_4)_2$):**
  - o Formation cause: Interaction of the pottery material with phosphate-rich soil or burial environment.
  - o Implication: May indicate exposure to organic matter or fertilizers in the surrounding soil.
- **Wollastonite (calcium silicate, $CaSiO_3$):**
  - o Formation cause: High-temperature phases from the original firing process or alteration due to prolonged burial in a silicate-rich environment.
  - o Implication: Found in the glaze layer, suggesting firing temperatures and environmental stability.
- **Quartz (silicon dioxide, SiO2):**
  - o Formation cause: Part of the original

pottery matrix or naturally occurring sand inclusions.

- o Implication: Stable and resistant to corrosion; serves as a baseline material for the pottery's integrity.

- **Enstatite (magnesium silicate, $MgSiO_3$):**
  - o Formation cause: High-temperature firing or metamorphic transformation during burial in magnesium-rich environments.
  - o Implication: Indicates potential high-temperature exposure or post-depositional alteration.

The significance of the findings regarding corrosion layer formation lies in the presence of calcium magnesium phosphate, which suggests an interaction with the burial soil, highlighting phosphate's role in forming stable corrosion layers. Wollastonite and enstatite point to the thermal history and environmental interactions, possibly reinforcing the glaze structure over time. The mineral composition indicates that the artifacts were likely buried in slightly alkaline or silicate-rich soil with moderate magnesium content. Stable quartz phases and alteration minerals suggest a long burial period in a relatively stable environment with periodic wet and dry cycles.

The EDX spectrum (Fig. 16) shows the elemental composition of the artifact, including Ca, Sn, Fe, Mg, Ba, C, P, Sn, and Pb, along with a significant proportion of S. These elements are formed during the processing of the glazed surface of the object. Additionally, P and a portion of S originate from the corrosion crust in the inner profile of the sherd edge, resulting from its interaction as a submerged object in wet soil.

The EDX spectra of the pottery corrosion products were analyzed at two different points: from the border (orange) and from the interior

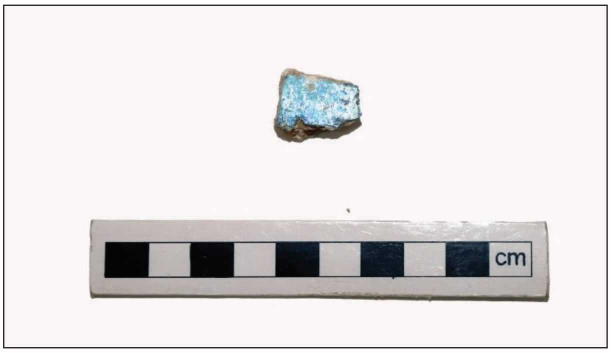

Fig. 10: shows an ancient blue glazed pottery sherd from Al-Yasila.

Fig. 11: shows an ancient glazed pottery sherd from El- Bediyeh.

Fig. 12: shows an ancient glazed pottery sherd from El- Bediyeh

(red) (Figs. 15, 16). Typical observations in the EDX analysis of pottery corrosion products show the following:

Border (orange) composition:

- o Higher presence of iron oxides (Fe_2O_3), suggesting environmental interaction, especially with oxygen or water, leading to the orange hue of the patina.
- o Silicon oxides (SiO_2), indicative of the

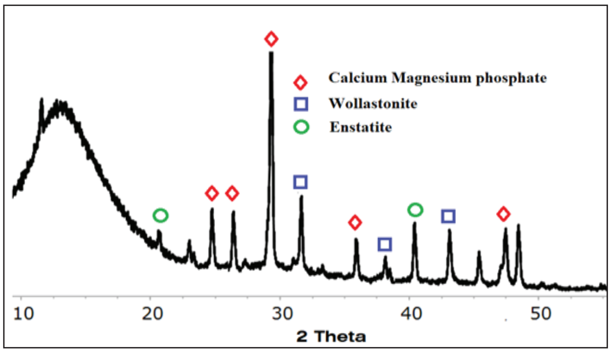

Fig. 13: shows the ancient pottery corrosion products identified by XRD on the surface of a sample from El- Bediyeh.

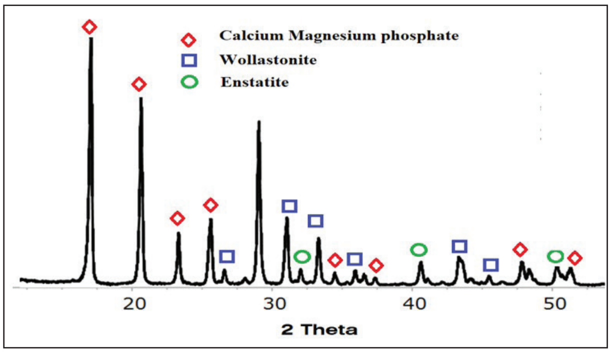

Fig. 14: shows the ancient pottery corrosion products identified by XRD on the surface of a sample from Al Yasila.

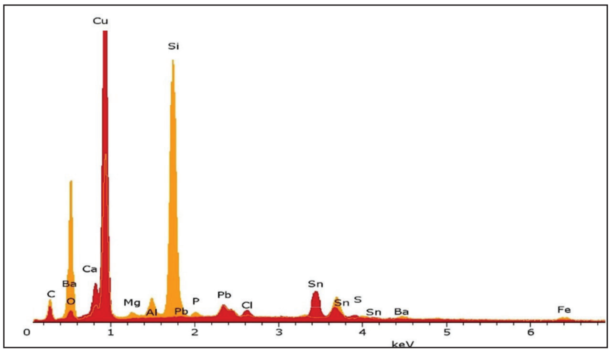

Fig. 15: shows the EDX spectrum of elemental composition of the artifact of figures 11 and 12

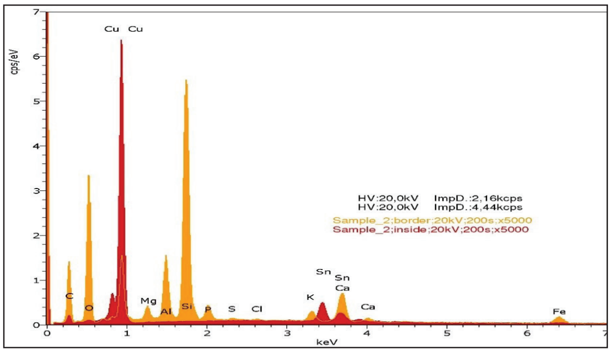

Fig. 16: Results of EDX analysis pottery corrosion products of in two different points, (from the border, orange and from inside, red)

glaze or surface material of the pottery.

o Chlorides (Cl), which may indicate exposure to salty or marine environments or burial in chloride-rich soil.

o Aluminum oxides (Al2O3), likely originating from the underlying clay or glaze matrix.

· Interior (red) composition:

o Predominance of silicon oxides (SiO2) and aluminum oxides (Al2O3), typical for the ceramic body.

o The absence of chlorides suggests minimal interaction with external chloride sources.

o Iron content (Fe) is present but not as oxidized as in the border composition, indicating lower exposure to environmental agents.

The EDX spectra of the sample primarily consist of SiO_2 with a significant amount of PbO. Other detected oxides include Al_2O_3, MgO, CaO, K_2O, Fe_2O_3 (?), and TiO_2.

Scanning electron microscopy (SEM) analysis of ancient glazed pottery, such as those from the Yasila site in Jordan, often reveals detailed information about manufacturing techniques, material composition, and wear patterns. In studies of glazed pottery, energy dispersive spectroscopy (SEM-EDS) has been used to analyze glaze microstructures, including the distribution of opaque materials like lead stannate or other elements that affect glaze coloration and durability.

For example, SEM scans identified distinct glaze layers ranging in thickness from 160 to 600 microns, with evidence of quartz inclusions and minimal interaction between the glaze and the ceramic body. These analyses also revealed chemical compositions, such as high concentrations of lead oxide (PbO), copper oxide or carbonate for green coloring, and variations in alkali contents, which provide insights into the technological innovations of the time.

Further analysis of the surface effects also identified similar chemical compounds with particle structures (Figs. 15 and 16). The elements detected by the EDX spectrum are associated with base materials (Ca, Fe), soil impurities (Si, Al, Ca), and structurally reformed chemical compounds (Cl, O) (Figs. 15 and 16). The SEM images (Figs. 17 and 18) reveal the corrosion crust on a glazed pottery fragment, showing the location of chemical compounds resulting from the recrystallization process. Among the identified elements are Ca, Si, Fe, Cl, Mg, and S, which are part of the glaze base, along with Si, Al, and Mg from soil contamination (as shown in the EDX spectrum in Fig. 15).

FTIR spectra also indicated that all samples contained calcite in varying amounts. Calcite, a common impurity in local clays, was observed in the spectra. The bands associated with calcite ($CaCO_3$) allow us to infer the firing temperature, which must have been below the decomposition temperature of calcite, typically between 700–900°C. FTIR spectra (Figs. 19 and 20) revealed a broad peak at 992 cm^{-1}, attributed to sulphate compounds, and a peak at 1098 cm^{-1}. Additionally, the peak at 1375 cm^{-1} suggests the possible presence of organic compounds. These bands could also be related to carbonates, as evidenced by the 1415 cm^{-1} band observed in the FTIR spectrum. Furthermore, considering that the fountain water contained small amounts of chloride compounds, the presence of chloride patina cannot be ruled out. The bands at 226 cm^{-1}, 398 cm^{-1}, and 986 cm^{-1} may indicate chloride compounds, such as atacamite.

Key Observations from FTIR Analysis:

o Calcite (CaCO3) Detection: Bands associated with calcite are visible in the FTIR spectra, indicating its presence as an impurity in the local clays used for pottery production.

o Firing Temperature Determination: The presence of calcite bands suggests that the firing temperature did not exceed its decomposition range of 700–900°C. At temperatures above 700°C–900°C, calcite

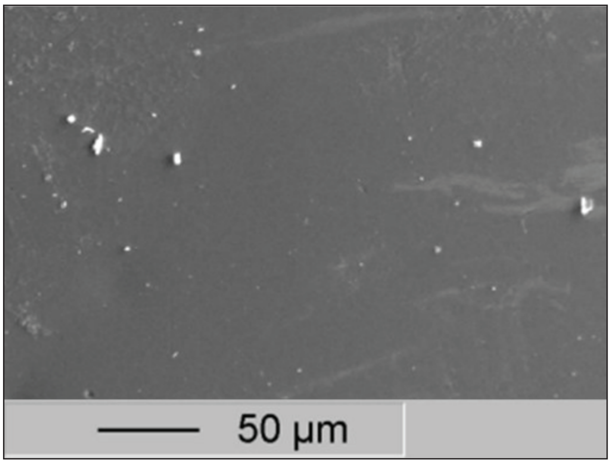

Fig. 17: SEM of glazed pottery from Yasila Results analysis pottery corrosion surface products.

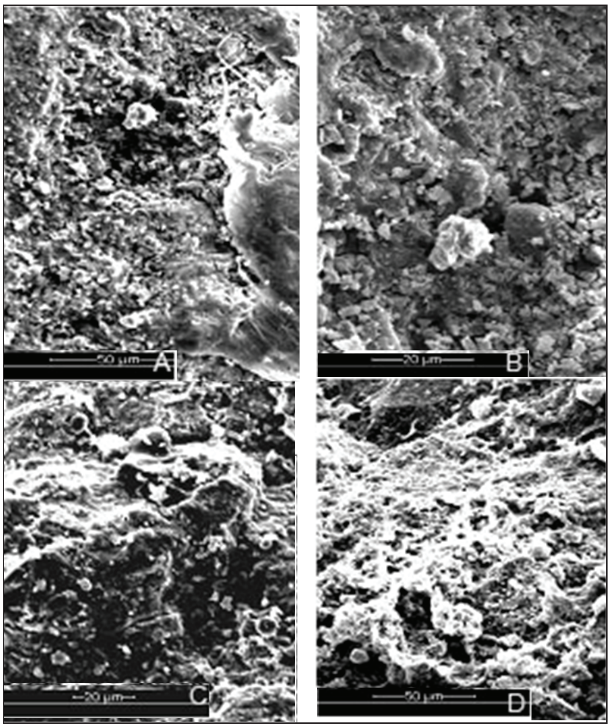

Fig. 18: Shows SEM of glazed pottery from from Hayyan al-Mushref

decomposes into calcium oxide (CaO) and carbon dioxide (CO3). The intact calcite bands imply that the firing temperature was below this range, which likely points to a low-temperature firing process.

- Environmental and Thermal Implications: Calcite's retention in the pottery contributes to the porosity and thermal resistance of the ceramic material. Its presence suggests either intentional low-temperature firing or a limitation in the technological access to high-temperature kilns during pottery production.
- FTIR Spectral Features for Calcite:
  - Around 1400 cm-1: Asymmetric stretching of CO_3^{2-} ions.
  - Around 870 cm-1: Bending vibration of CO_3^{2-} ions.
  - Around 711 cm^{-1}: Out-of-plane bending vibration of CO_3^{2-} ions.
- Quantitative Variation: Differences in the intensity of the calcite bands suggest variations in the clay source or firing methods among the samples, providing insights into regional practices and technological differences.

4. Conclusions

A collection of ancient glazed pottery sherds excavated from the archaeological sites of Khirbet edh–Dharih, Hayyan al–Mushref, Yasila, Tell al-Husn, and El-Bediyeh in Jordan was analyzed. Based on the archaeological context, these artifacts date back to the 1st century B.C. The following conclusions can be drawn from the collected data:

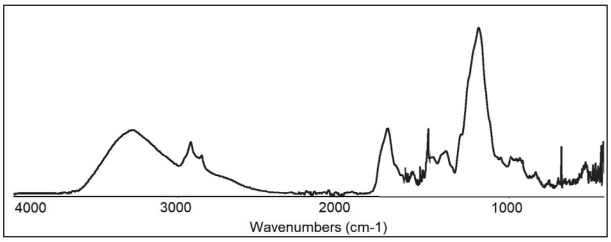

Fig. 19: ATR-FTIR spectra collected on pottery samples

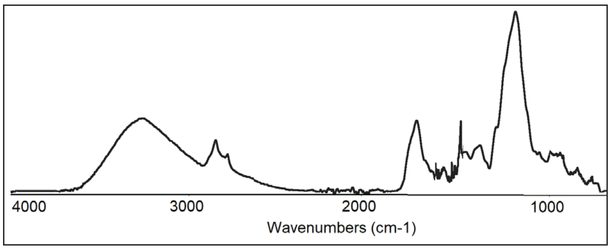

Fig. 20: ATR-FTIR spectra collected on pottery samples

During its underground burial, the artifact interacted with its environment, leading to partial or complete mineralization. SEM-EDX analysis identified the basic components of the glaze, such as Si (former), Ca (stabilizer), Sn (opacifier), and K (modifier). Additionally, several metal oxides used as colorants, such as Cu and Fe, were detected, along with S and P in trace amounts, which were likely derived from soil interaction. The corrosion products also revealed Cl, Al, and Mg, which originated directly from the corrosion layers. These elements, particularly in the contamination microstructures, were incorporated into the corrosion crust as various reformed compounds from the surrounding soil.

The XRD results further revealed the presence of calcite in the samples, suggesting that the firing temperatures did not exceed 900°C.

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ملخّص: يهدف هذا البحث إلى تقييم المنهج متعدد التخصصات لعدة مناهج بحثية (المجهر الضوئي، XRF, XRD، SEM-EDX, ATR-FTIR) في تحليل القطع الأثرية. وتناول البحث، على وجه التحديد، مورفولوجيا أنواع مختلفة من الفخار المزجج وآثار الباتينا عليه من مواقع مختلفة في الأردن، بما في ذلك حيان المشرف، واليصيلة، وتل الحصن، والبدية، وخربة الذريح. وتنتج هذه الآثار عن عوامل بيئية ودفنية أثرت على الفخار. ولذلك، يلعب أثر الفخار المزجج دورًا فعالًا في تشكيل خصائص القطعة الأثرية. وقد تم اختيار مناهج تحليل العناصر، مثل فلورة الأشعة السينية (XRF) والمجهر الإلكتروني الماسح (SEM-EDX)، لاستكشاف التنوع المنهجي في هذه الدراسة. بالإضافة إلى ذلك، استُخدمت تحاليل تركيبية باستخدام حيود الأشعة السينية (XRD) ومطيافية تحويل فورييه الكلي المخفف بالأشعة تحت الحمراء (ATR-FTIR) لفحص التركيب البلوري للباتينا على الفخار المزجج وبنيتها الجزيئية. أشارت النتائج إلى أن القطع الأثرية تحتاج إلى ترميم للحفاظ على المادة الأصلية.

Notes

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