

Pottery from Halat Aobeer and Hazem Al-Jasrah, Qatar[☆]M.A. Al Maadeed^a, N.J. Al Thani^{a,*}, M. Sadeq^b, R. Sobott^c^aCenter for Advanced Materials, Qatar University, P.O Box: 2713, Doha, Qatar^bHumanities Department, History Program, Qatar University, P.O Box: 2713, Doha, Qatar^cInstitute of Mineralogy, Crystallography, and Material Science, Leipzig University, Scharnhorststr. 20, D-04275 Leipzig, Germany

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Abstract

In this paper the results of investigations on six archaeological ceramic sherds of the most common Islamic earthenware from the site of Halat Aobeer and Hazam Al-Jasrah are presented. The data was obtained by using a multi-analytical approach, comprising chemical and mineralogical analyses. External features were investigated by optical and scanning electron microscopy (OM, SEM) while the chemical composition of the pottery surfaces was analysed by X-ray fluorescence spectroscopy (XRF) in order to obtain information about the production technologies employed in the past. The results showed not unexpectedly that the sherds were different in shape, style, and composition and the diversity of ceramics can be regarded as evidence for economically prosperous communities.

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Keywords: Pottery; Qatar; Materials characterisation; XRF; SEM

1. Introduction

Geologically Qatar is primarily composed of lower Eocene limestone and gypsum rocks overlain by Miocene marls and limestone in the South and South West. Generally, Qatar has three characteristic forms of landscape: the limestone plateau, the sand desert, and the coastal region [1]. Due to its location, Qatar has attracted seasonal settlements particularly along the coastal line since the Stone Age. Successive survey and excavation projects have been conducted in Qatar since the mid twentieth century.

The first was by a Danish archaeological expedition in 1956 identifying over a hundred Stone Age sites, different in character representing small encampments, seasonal settlements, and flint working sites [1]. They are dated to a period extending from 48,000 to 5000 BC. Many of these sites such as Al-Khor in the North East of Qatar, Bir Zekrit, and Ras Abaruk are marked in Holger Kapel's "Atlas of the Stone Age cultures of Qatar" [2]. At these locations flint tools such as scrapers, axes, and arrow heads of the Neolithic type were found [2]. Other sites furnished painted ceramics and vessels indicating Qatar's connexion with the

Al-Ubaid civilisation which flourished in Mesopotamia during the period of the 5th–4th millennium BC.

The latest pre-Islamic archaeological remains on the Qatar peninsula are dated to the Sassanid period (224–651 AD). Qatar was ruled by al-Munther Ibn Sawi, one of the Muntherid kings (also called Lakhmid Kingdom referring to their origin from Banu Lakhm tribes) who were allies to the Sassanid dynasty and lived in the present day Southern Iraq with al-Hirah as the capital in 266 AD.

Islam arrived in Qatar in 629 AD at the time of the Prophet Mohammed. The archaeological site of Murwab is the only major settlement discovered so far in Qatar representing the early Islamic period, particularly the Abbasid period (750–1258 AD) [1]. It consists of approximately 260 houses, two mosques, and a fortified palace. Another site showing Early Islamic pottery alongside with pre-Islamic artefacts in burials is Halat Aobeer and Hazem Al-Jasrah (Al-Madina al-Fadha'yya) which is excavated by the Qatari Department of Antiquities since 2009.

Chemical and mineralogical analyses of ceramic artefacts are apt means to furnish information about the provenance and manufacturing technology. This includes all production steps from the procurement of the raw materials to the finishing of the object by various decoration techniques and the firing process. One of the best non-destructive analytical tools for the characterisation of pottery sherd surfaces is the X-ray

[☆]Hazem Al-Jasrah is nowadays called Al-Madina al-Fadha'yya.

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fluorescence spectroscopy (XRF) and scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM-EDX), which have been widely used for analysing archaeological ceramics [3–9].

The current paper aims to examine earthenware recovered from the site of Halat Aobeer and Hazem Al-Jasrah (Al-Madina al-Fadha'yya) in order to determine (a) whether the pottery was produced locally or imported from the surrounding regions, and (b) to develop a clay reference of the most frequent earthenware found at this site based on OM, SEM, and XRF conducted at Qatar University.

2. Archaeological background

2.1. The archaeological site

The site is located north of Sumaisima (latitude: $25^{\circ}37'40.94''$ N, longitude: $51^{\circ}30'29.15''$ E), approximately 39 km north of Doha (linear distance) on the coast between the towns of Al-Khawr (also spelled as Al-Khor) in the North and Sumaisima in the South (Fig. 1). The site has some significance and lies on an elevated level overlooking the sea (fishing, pearl fishing, sea trade, communication and transportation) and also close to the

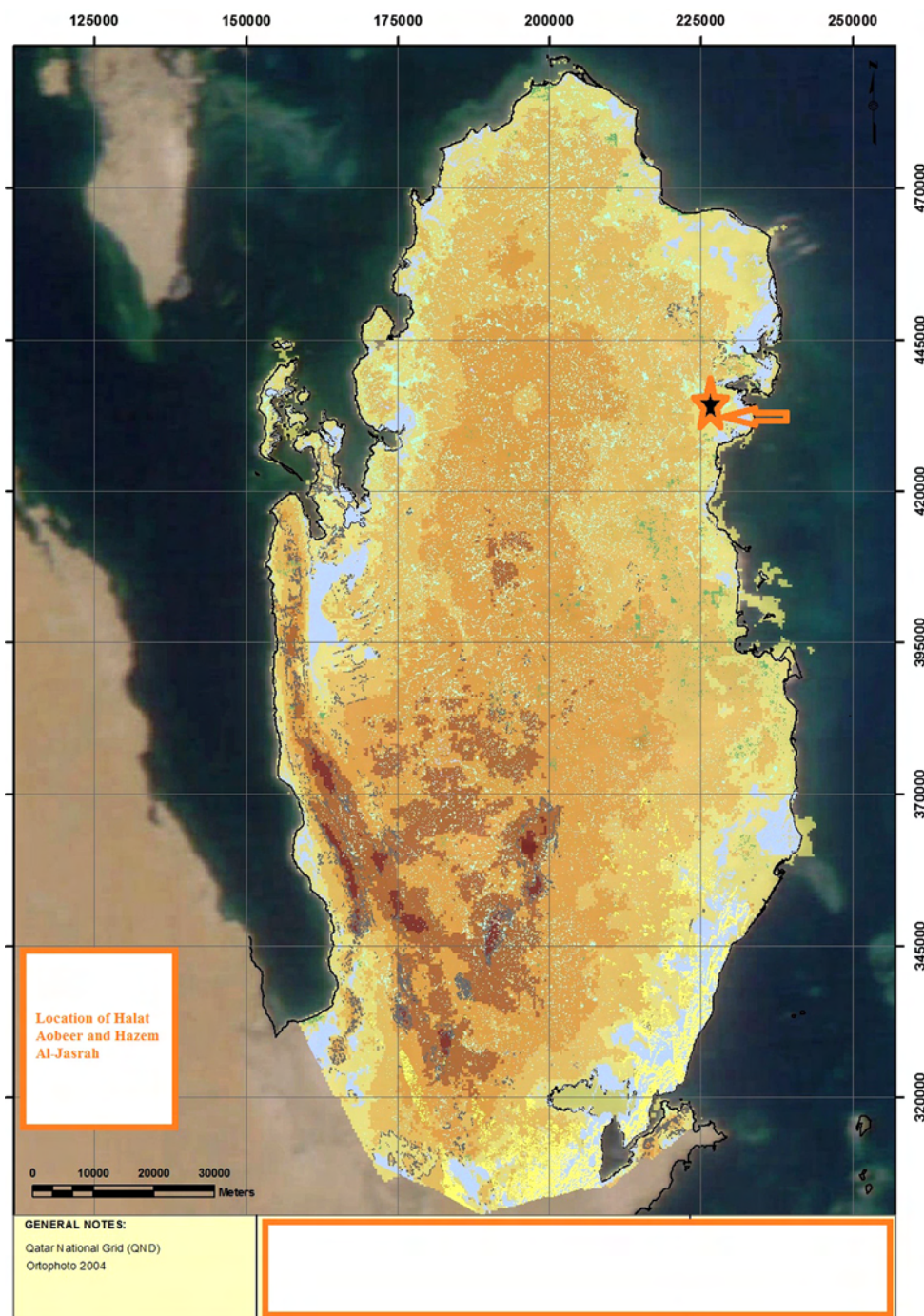

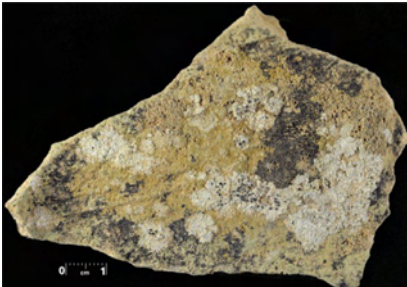
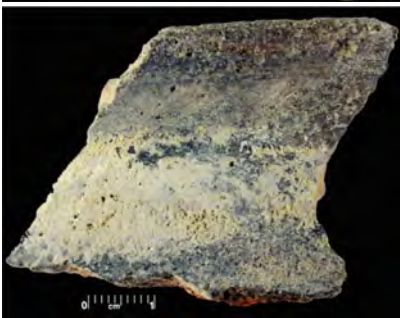


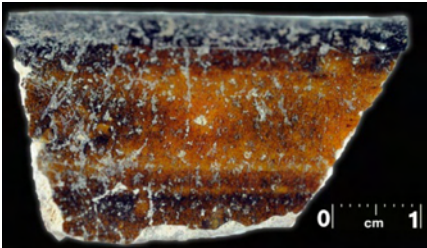


Fig. 1. Map of Qatar showing the location of Halat Aobeer and Hazem Al-Jasrah.

Table 1
Characteristics of analysed samples.

| Sample no. | Photograph | Object type | Colour and surface treatment | Remarks |
|------------|---|----------------------------------|---|--|
| B4-1 |  | Fragment of service vessel | Cream-coloured sherd with intersecting incised lines topped by two incised horizontal lines | Shoulder fragment from just below the rim |
| B4-2 |  | Fragment of service vessel | Buff-coloured sherd with two parallel incised lines | Shoulder fragment from just below the rim |
| B4-3 |  | Pot fragment | Red-coloured sherd with plain brownish surface | Rim fragment representing 6.5% of the rim with a radius of 10.5 cm |
| B4-4 |  | Handle fragment of a storage jar | Cream-coloured sherd with incised geometric decoration (dots bordered by two double lines) | Handle attached to the shoulder in a vertical position |
| C1 |  | Fragment of a jar body wall | Black-coloured sherd with vertical thumb decoration | Fragment of the upper part of a jar shoulder |
| C5 |  | Fragment of service vessel | White-coloured sherd with reddish glaze on both sides | Rim fragment representing 5% of the rim with a radius of 13 cm |

ancient main road leading from the present day Doha to the northern coastal archaeological sites north and south of the present city of Al-Khawr.

2.2. Investigated objects

In an archaeological survey conducted by the Qatari Department of Antiquities many graves in a circular shape associated with faint remains of building blocks were identified within the upland and lowland of the site. A set of six pottery sherds representing the commonly observed earthenware at the site of Halat Aobeer and Hazem Al-Jasrah (Al-Madina al-Fadha'yya) was selected for the analysis in the present work.¹

The sherds are parts of common and glazed wares and include two rim fragments (C5, B4-3) and four body fragments (B4-1, B4-2, B4-4, C1). External features of the sherds such as colour, glaze, and decorations were used to classify them. The characteristics features of the investigated objects are shown and briefly described in Table 1.

3. Analytical methods

3.1. Optical microscopy

The pottery sherds of Islamic earthenware were documented with a NIKON D3X digital camera and analysed by polarised light microscopy using a ZEISS 3D stereo microscope equipped with an AxioCam HRc camera.

3.2. Scanning electron microscopy

For the study of the surface and structural properties of the sherds a FEI Quanta 200 environmental scanning electron microscope (ESEM) with EDAX was used. The microscope offers operation in either a high- or low-vacuum (HV, LV) mode in the range of 10.7–130.6 Pa. The acceleration voltage ranges from 0.2 to 30 kV and the maximum resolution in the HV mode is 3 nm and 4 nm in the LV mode. Qualitative and quantitative element mapping and line analyses were made with the EDAX Genesis 7000 EDS system. Samples were investigated in the LV mode using a gaseous secondary electron and backscattered electron detector. The sample surfaces were levelled, cleaned and dried, but additional conducting coatings were not applied.

3.3. X-ray fluorescence spectroscopy (XRF)

The JSX-3201 M Element Analyzer was used for the non-destructive investigation of sample surfaces. At least five to seven different positions on the inner and outer surface of each sample were analysed. The element concentrations were recalculated to oxide concentrations. Only in cases where no glaze, slip or deposits due to the burial in the ground were present on the surface it can be assumed that the chemical

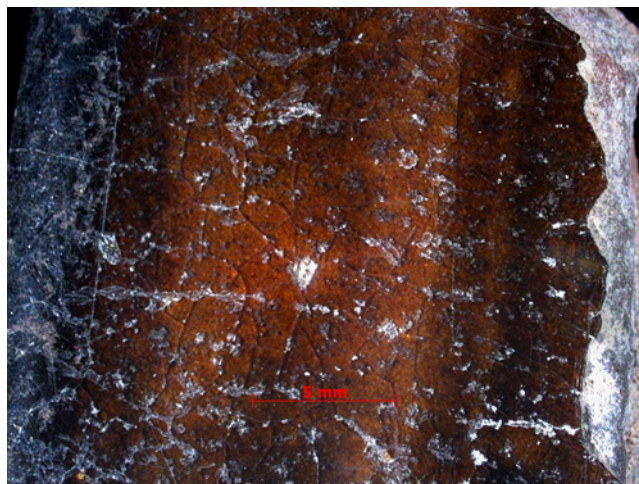


Fig. 2. Glazed surface of sample C5. (For interpretation of the references to colour in this figure, the reader is referred to the web version of this article.)

composition of the sherd was analysed. However, a large scattering of the element concentrations for the set of data points on either of the two surfaces indicated that the sherds were stained during burial in the ground. The sometimes unusual high sulphate concentrations suggest that the stains consist at least partly of gypsum. Whenever possible, it was tried by statistical evaluation of the data to approximate the chemical composition of a sherd as closely as possible.

4. Results and discussion

4.1. Glazed earthenware (sample C5)

Just one fragment of the selected set (C5) is monochrome as seen in Fig. 2, and covered with brown reddish lead-glaze which is usually produced by lead and silicon dioxide.

This manufacturing process is commonly applied in the territories of the Abbasid dynasty (750–1258 AD) extending from Khurasan in the east to North Africa in the west. Gulf states, including Qatar, were part of it. Assemblages of well dated similar Abbasid glazed earthenware have been found at the site of Murwab northwest of Qatar, approximately 80 km northwest of Doha. At this site two complete white slipped Abbasid amphorae with turquoise glaze (copper oxide) have been found standing against the courtyard wall 308 of a house in the northern part of the site ([10], Fig. 15). Three other complete white slipped vessels with turquoise lead-glaze are also displayed at the museum of Islamic Art in Doha (a jar PO. 215.2003, and an ewer PO. 709.2007): All of them originated from Iraq and are well dated to the Abbasid period. The glaze on the two sides of sherd C5 has a different thickness and is 0.2 mm on the inside and 0.1 mm on the outside as shown in Figs. 3 and 4.

On the polished side of the sample (Fig. 5) three data points (9, 10, 11), formed a consistent data set which is interpreted as being representative for the chemical composition of the glaze as is shown in Table 2.

As can be seen in Table 3 it is a high lead glaze. High lead glazes of a brown colour and similar composition of Ayyubid/

¹Preliminary report of "The first season of the rescue excavations at Al-Madina al-Fadha'yya north of Sumaisima (March/April/May 2010)".

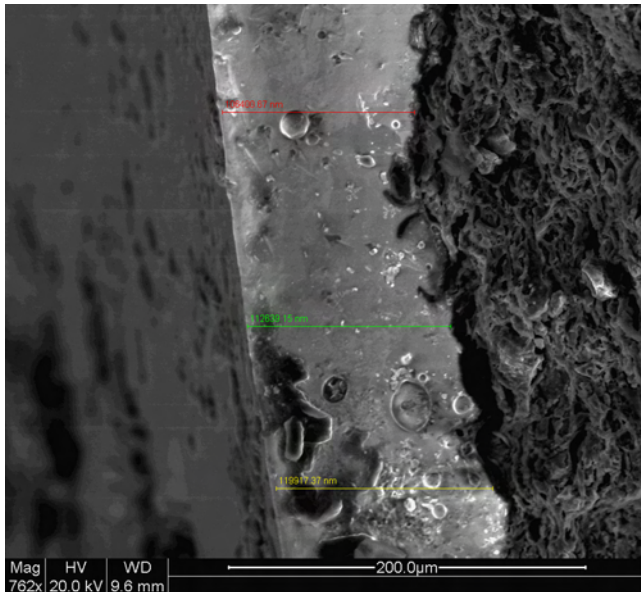


Fig. 3. SEM micrograph of glaze layer of sherd C5 with conspicuous gas bubbles indicating fusion of the glaze.



Fig. 4. Cross section of sherd C5 with measurement of thickness of sherd and glaze.



Fig. 5. Sample C5.

Table 2

Element Analysis data- XRF for C5.

| Element | Si | Ca | Fe | Pb | S | Al | Mg | K | Ti |
|---------|-------|-------|-------|-------|-------|------|------|------|------|
| Spot 1 | 10.15 | 45.47 | 8.04 | 2.70 | 22.58 | 3.40 | 4.00 | 1.73 | 0.76 |
| Spot 2 | 27.93 | 23.03 | 14.06 | 1.31 | 12.48 | 7.97 | 8.02 | 3.33 | 1.21 |
| Spot 3 | 2.13 | 60.53 | 3.05 | 0.436 | 29.35 | 0.74 | 1.83 | 0.66 | 0.22 |
| Spot 4 | 29.14 | 0.140 | 5.49 | 53.19 | 1.03 | 2.32 | 3.56 | 4.83 | nd |
| Spot 5 | 26.61 | 4.41 | 5.73 | 55.80 | 1.59 | 1.94 | 3.23 | nd | 0.20 |
| Spot 6 | 24.49 | 13.44 | 5.51 | 49.16 | nd | 2.15 | 3.25 | 1.37 | 0.31 |
| Spot 7 | 10.69 | 38.27 | 3.74 | 22.93 | 17.88 | 1.57 | 2.48 | 1.43 | 0.23 |
| Spot 8 | 26.43 | 2.99 | 4.44 | 58.60 | nd | 3.01 | 2.30 | 1.76 | 0.25 |
| Spot 9 | 26.09 | 2.82 | 5.75 | 62.34 | nd | nd | nd | 2.44 | 0.56 |
| Spot 10 | 26.52 | 3.66 | 4.91 | 62.79 | nd | nd | nd | 1.65 | nd |
| Spot 11 | 26.14 | 3.72 | 5.76 | 64.39 | nd | nd | nd | nd | nd |
| Spot 12 | 27.73 | 2.89 | 5.07 | 55.15 | nd | 3.75 | 2.96 | 1.94 | 0.23 |

Table 3

Composition of high lead glaze of sample C5.

| K ₂ O | CaO | MnO | PbO | Fe ₂ O ₃ | SiO ₂ | TiO ₂ | Σ |
|------------------|-----|-----|------|--------------------------------|------------------|------------------|-------|
| 1.2 | 3.4 | 0.4 | 48.9 | 5.6 | 40.4 | 0.7 | 100.6 |

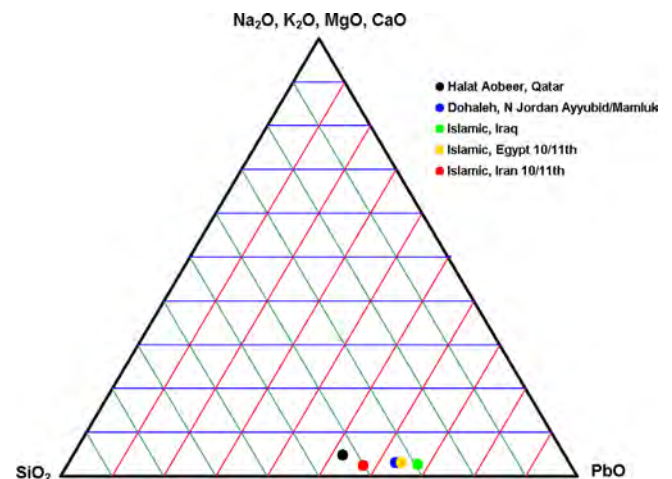


Fig. 6. Ternary diagram showing chemical composition of high lead glazes from the Islamic world. The concentrations are normalised to 100% [11,12].

Mamluk origin were reported by al-Saad [11] from Dohaleh in northern Jordan (Fig. 6). The glazing of pottery was wide-spread in the Islamic world which excelled in this technique of decoration.

The problem of staining on the sample surfaces is evident in Fig. 7 showing the correlation of Cao and SO₃ concentrations for the polished and damaged side of sample C5. While there was no SO₃ detected on the polished side, the SO₃ concentrations on the damaged side range from 2.7 to 42 mass% and are linearly correlated with the Cao concentrations ($r^2=0.95$) suggesting that gypsum was irregularly deposited on the surface.

The brown colour of the glaze is due to a distinct content of Fe(III) oxide which amounts to an average value of 5.6 mass %. Due to the partial contamination of the surface with gypsum it is difficult to make any statements about the chemical composition of the sherd.

4.2. Unglazed, slipped earthenware (samples B4-3, C1)

Clay slip was widely used for decorative and aesthetic purposes and for the reduction of permeability. Firing the clay in an oxidising atmosphere led to the development of a more or less intensive red colour [13].

One of the unglazed earthenware in the set of the six samples is a potsherd with a homogeneous body coated with a red slip. The sherd composition is determined by the chemistry of the clay and the procedure of clay paste preparation [14]. On both sides of the sherd six positions were analysed.

In sample B4-3 the chemical composition (Table 4) of the positions on the outer surface (1–6) scatter (Fig. 8) consider-

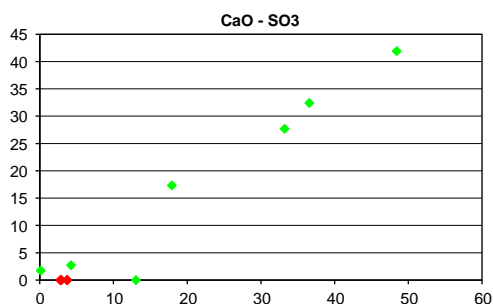


Fig. 7. CaO and SO₃ concentrations on the polished (red marks) and damaged side (green marks) of sample C5. SO₃ concentrations presented as ordinate values; data given in mass %). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Table 4
Element Analysis data- XRF for B4-3.

| Element | Mg | Al | Si | S | K | Ca | Fe | Ti |
|---------|-------|-------|-------|-------|------|-------|-------|------|
| Spot 1 | 6.86 | 4.57 | 13.30 | 16.96 | 1.67 | 49.86 | 5.56 | 0.50 |
| Spot 3 | 10.37 | 13.15 | 26.73 | 2.17 | 4.41 | 16.99 | 23.76 | 1.97 |
| Spot 4 | nd | nd | nd | nd | nd | 24.34 | 75.66 | nd |
| Spot 5 | nd | nd | nd | 24.30 | nd | 65.24 | 10.46 | nd |
| Spot 6 | nd | nd | 6.94 | 25.94 | 1.20 | 61.37 | 2.85 | nd |
| Spot 7 | 6.91 | 9.45 | 22.88 | 3.19 | 3.30 | 35.09 | 16.40 | 1.75 |
| Spot 8 | 4.73 | 18.08 | 32.65 | 3.88 | 6.32 | 7.97 | 23.60 | 2.26 |
| Spot 9 | 4.74 | 15.06 | 29.04 | 3.95 | 5.78 | 11.29 | 26.11 | 2.17 |
| Spot 10 | 4.62 | 16.06 | 32.17 | 3.88 | 6.24 | 12.21 | 21.90 | 2.26 |
| Spot 11 | 4.88 | 16.10 | 31.98 | 2.69 | 5.66 | 9.63 | 26.23 | 2.07 |
| Spot 12 | 4.96 | 16.57 | 30.33 | 2.79 | 5.68 | 9.94 | 26.68 | 2.07 |

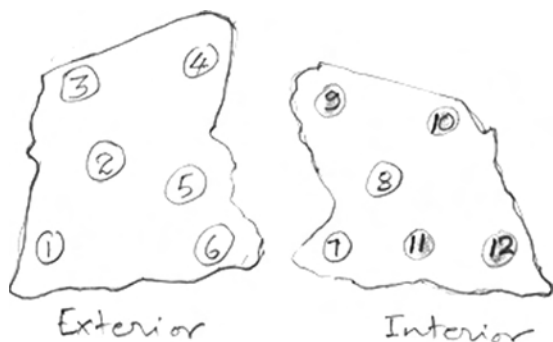


Fig. 8. . Sample B4-3.

ably which is not surprising as the distribution of slip is irregular (Fig. 9). The result for data point 4 shows that iron (III) oxide is a major component of the slip. Data points 5 and 6 are distinguished by high CaO and SO₃ contents. They were most probably caused by a gypsum staining which could have been formed while the sherd was buried in the ground. With the exception of data point 7, the chemical data for the positions 8–12 on the inner surface is much more consistent. Since there was no slip on the inner side of the vessel the calculated average composition for data points 8–12 should reflect the chemistry of the clay paste (Table 5). However, the SO₃ content is uncommonly high and was therefore eliminated as gypsum and the data recalculated and normalised to 100%.

Fig. 10 shows a cross section of the sherd with the thickness of the slip layer determined by optical microscopy to be 0.11 mm. The colour of the slip is determined by high contents of Fe(III) oxide. Apart from SiO₂, Al₂O₃, and Fe₂O₃ as major components, the clay paste contains appreciable amounts of alkali and alkali earth oxides which lower the temperature of sintering and vitrification [15] and favour the formation of a dense sherd fabric (Fig. 11).

Sample C1 has a black surface colour which is probably due to a firing under reducing conditions. Depending on the chemical composition, hercynite, FeAl₂O₄, maghemite, γ-Fe₂O₃, and/or magnetite, Fe₃O₄, are the possible black pigments.



Fig. 9. Exterior surface of sherd B4-3 with marked positions of chemical analysis.

Table 5
Average values of major oxide components of the chemical composition of the inner surface of sherd B4-3 (values given in mass%). SO₃ was eliminated as gypsum and the data recalculated and normalised to 100%.

| K ₂ O | MgO | CaO | Al ₂ O ₃ | Fe ₂ O ₃ | SiO ₂ | TiO ₂ | SO ₃ | Σ |
|------------------|-------------|-------------|--------------------------------|--------------------------------|------------------|------------------|-----------------|---------------|
| 4.06 | 4.50 | 8.11 | 17.55 | 20.22 | 37.91 | 2.05 | 4.87 | 99.77 |
| 4.46 | 4.95 | 5.16 | 19.29 | 22.22 | 41.66 | 2.25 | | 100.00 |

The sherd surface is decorated with horizontal stripes and the observation of the surface under the optical microscope and by SEM reveals further morphological features (Fig. 12).

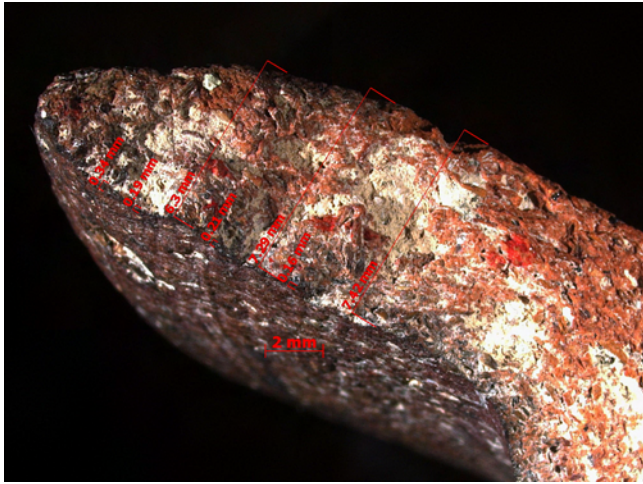


Fig. 10. Macroscopic view of cross section with slip layer.

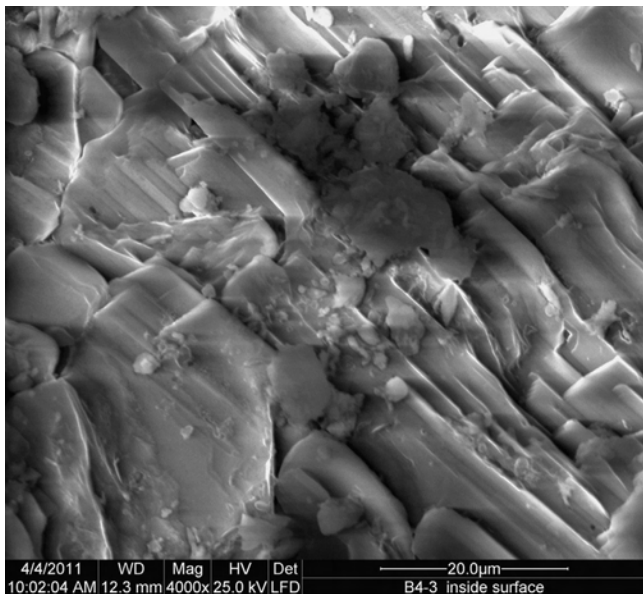


Fig. 11. SEM micrograph of interior sherd fabric, sample B4-3.

Bubbles occurring at various locations in the matrix suggest that the clay was not well kneaded. The chemical data for sherd C1 is presented in Table 6.

Comparison of the chemical data in the Tables 5 and 6 reveals that both sherds obviously have a very similar chemical composition which justifies to attribute them to a group.

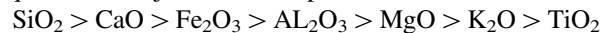
4.3. Decorated, unpainted ware (samples B4-1, B4-2, B4-4)

B4-1 is a sherd from an unpainted bowl. It has a fine-grained, homogeneous, well-fired cream-coloured body decorated on the outer surface by intersecting incised lines followed by two horizontal lines (Fig. 13).

Sherd B4-2 has a brown-coloured surface and shows a well-fired, fine-grained, hard-textured and homogeneous cream white body as shown in Fig. 14.

Sample B4-4 originates from a jar handle and exhibits a whitish well-fired coarse clay body decorated with punches in two vertical rows flanked by two incised double-line bands as shown in Fig. 15.

The samples B4-1, B4-2, and B4-4 have a similar chemical composition (Table 7) which is characterised by the following sequence of major oxide components:



5. Conclusions

The chemical characterisation of archaeological pottery is a very useful tool for studying the technology of production, the provenance of the ware and possibly the raw materials, and the cultural developments and exchanges with surrounding countries.

The present chemical study has revealed that five of the six samples can be attributed to two different groups. Group 1

Table 6

Average values of major oxide components of the chemical composition of the inner surface of sherd C1 (values given in mass %). SO_3 was eliminated as gypsum and the data recalculated and normalised to 100%.

| K_2O | MgO | CaO | Al_2O_3 | Fe_2O_3 | SiO_2 | TiO_2 | SO_3 | Σ |
|----------------------|--------------|--------------|-------------------------|-------------------------|----------------|----------------|---------------|----------|
| 4.13 | 4.35 | 8.77 | 19.22 | 20.49 | 39.62 | 1.98 | 1.05 | 99.61 |
| 4.22 | 4.45 | 8.21 | 19.65 | 20.95 | 40.50 | 2.02 | | 100.00 |

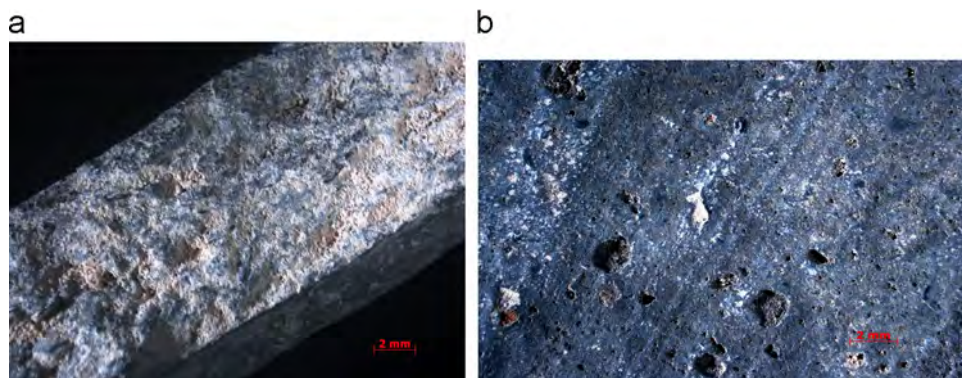


Fig. 12. Cross section (left) and surface of sample C1.

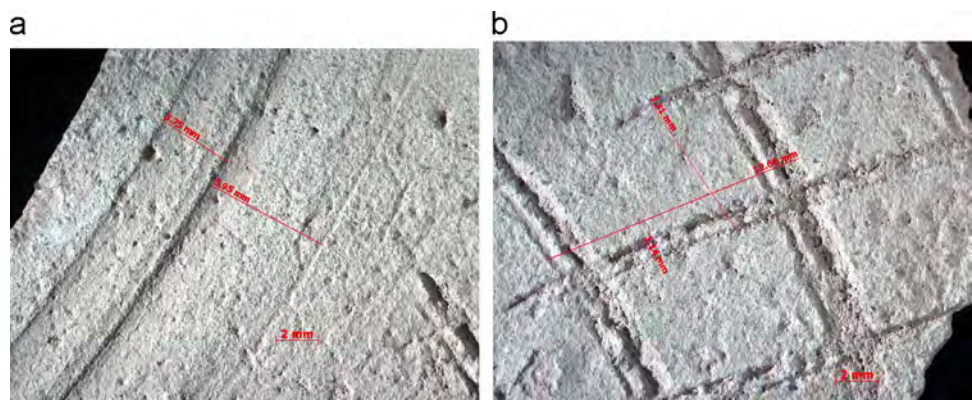


Fig. 13. With incised lines decorated surface of sherd B 4-1. (For interpretation of the references to colour in this figure, the reader is referred to the web version of this article.)

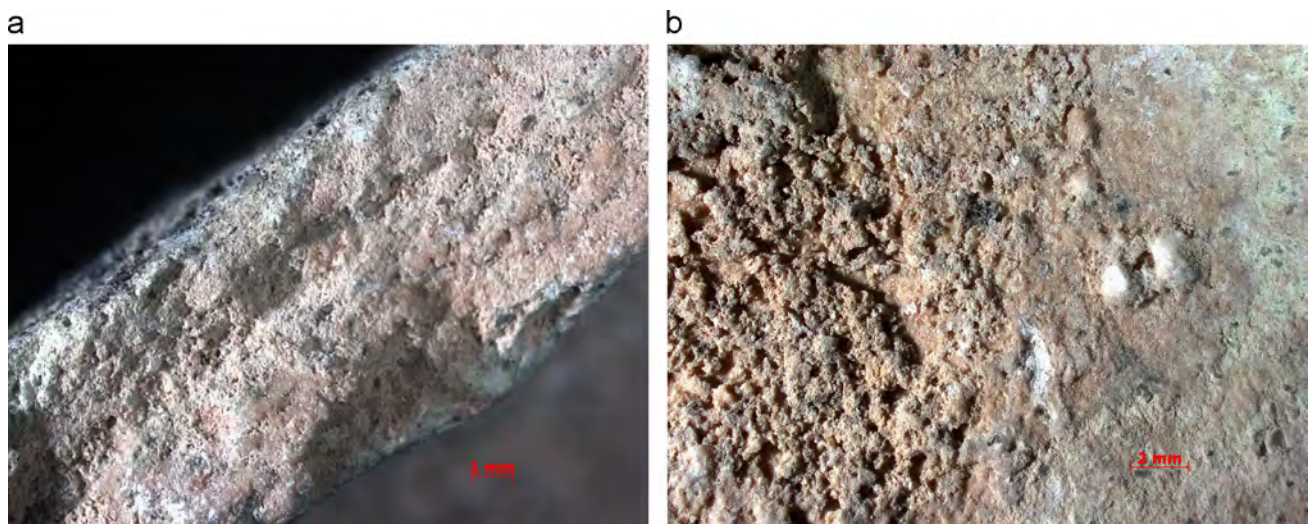


Fig. 14. Cross section (left) and the surface of sherd B 4-2. (For interpretation of the references to colour in this figure, the reader is referred to the web version of this article.)



Fig. 15. Enlarged view of surface of sherd B 4-4. (For interpretation of the references to colour in this figure, the reader is referred to the web version of this article.)

(samples B4-3, C1) is distinguished by high contents of Al_2O_3 and Fe_2O_3 and a comparably low CaO content, while group 2 (B4-1, B4-2, B4-4) is marked by a high CaO content and

Table 7
Average values of major oxide components of the chemical composition of the inner surface of sherd B4-1, the outer surface of sherd B4-2, and the top surface of sherd B4-4 (values given in mass%). SO_3 was eliminated as gypsum and the data recalculated and normalised to 100%.

| Sample | K ₂ O | MgO | CaO | Al ₂ O ₃ | Fe ₂ O ₃ | SiO ₂ | TiO ₂ | SO ₃ | Σ |
|--------|------------------|-------|-------|--------------------------------|--------------------------------|------------------|------------------|-----------------|--------|
| B4-1 | 1.99 | 9.98 | 25.32 | 10.01 | 11.36 | 36.45 | 0.9 | 2.87 | 98.88 |
| | 2.12 | 10.62 | 24.80 | 10.65 | 12.09 | 38.78 | 0.96 | | 100.00 |
| B4-2 | 2.15 | 6.16 | 34.58 | 8.1 | 11.37 | 30.76 | 1.34 | 4.06 | 98.52 |
| | 2.35 | 6.72 | 34.64 | 8.84 | 12.41 | 33.57 | 1.46 | | 100.00 |
| B4-4 | 1.7 | 6.09 | 28.38 | 9.32 | 8.23 | 40.04 | 0.77 | 4.23 | 98.76 |
| | 1.86 | 6.65 | 27.76 | 10.18 | 8.99 | 43.73 | 0.84 | | 100.00 |

comparably low contents of Al_2O_3 and Fe_2O_3 . The analysis of the glaze on sherd C5 showed that it is in the tradition of Islamic high lead glazes. The chemical compositions of sherds deduced from surface analyses require further verification by XRF analysis of bulk samples and non-destructive GADDS X-ray diffraction analysis.

The abundance of different kind of ceramics—glazed and unglazed, decorated and undecorated, fired at oxidising and

reducing conditions—is an obvious indication that at least part of the earthenware was imported from places in the surrounding regions which accordingly means that Halat Aobeer and Hazem Al-Jasrah settlements had strong trade relations with various neighbouring civilisations and cultures. In addition, the different shapes, types and styles of pottery products give evidence for economical prosperity at both sites during the zenith of their development.

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