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CHARACTERIZATION OF QARH'S WALL PLASTERS, AL-ULLA, SAUDI ARABIA. A CASE STUDY

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Abstract

During five years (2008 to 2012) of field work in the restoration and conservation of the Qarh's monuments at Al-Ulla northwest Saudi Arabia, many kinds of wall plasters were studied, investigated and analyzed by the following methods: field observations,(visual examination), Optical Microscope (OM), Polarized Microscope (PM), Scanning Electron Microscope (SEM), equipped with (EDS), and X-Ray Diffraction (XRD).Those methods of investigation and analysis helped us determine the Qarh's wall plasters. Four kinds of plasters were identified; white gypsum plaster, white gypsum and lime plaster, gypsum plaster over an insulating layer and lime plaster over an insulating layer. Their main components are gypsum, lime, sand and an additional material for improving plasters properties. Those results helped us to make a correct diagnosis, which is the first step in any conservation strategy.

Keywords: *Qarh site; wall plaster; investigations; diagnosis.*

Introduction

Qarh is an archaeological site located between Al-Ulla in the north and Matran in the south [1], 38° 6’ E longitude and 26° 30’ N latitude in the Medina area. It is just a few kilometers northwest of the town of Mokerra [2]. Today Qarh is known as "Al-Mabbiayat" for the local people of Al-Ulla. Qarh occupies an area of 640 thousands of square meters, which is empty land, surrounded by the ruins of a zigzag wall [3]. Al-Ulla is known as the village valley, or the city of the village valley [4]. Qarh was an important marketing center before Islam (sixth century AC), and it flourished in the Islamic period. By the tenth century AC, it was the second important city in Hijaz, after Mecca. Qarh's name disappeared with the beginning of the thirteenth century AC [5]. The excavation made by the Saudi commission of antiquities [6, 7], and the excavation made by the King Saud University at the Qarh site [8] discovered many mudbrick buildings that contained various architectural details, such as: burned brick columns, irrigation canals, burned brick grounds, plastered walls, stone pillars, a zigzag wall surrounding the city (Fig. 1a. and b), and many archaeological artifacts of stone, pottery, glass, wood and metal. Those discoveries conveyed to Qarh archaeological site its importance in Arabian Peninsula.

The Qarh site was exposed to different destructive factors, such as: torrents, rain, wind, storms, etc. [9], so the wall was in danger of collapsing and the plasters in danger of decaying.

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This paper aims to study the current state of the wall plasters used in the Qarh archaeological site, to identify the different kinds of wall plaster components, to study the wall plaster morphology and to identify the significant properties of those wall plasters.

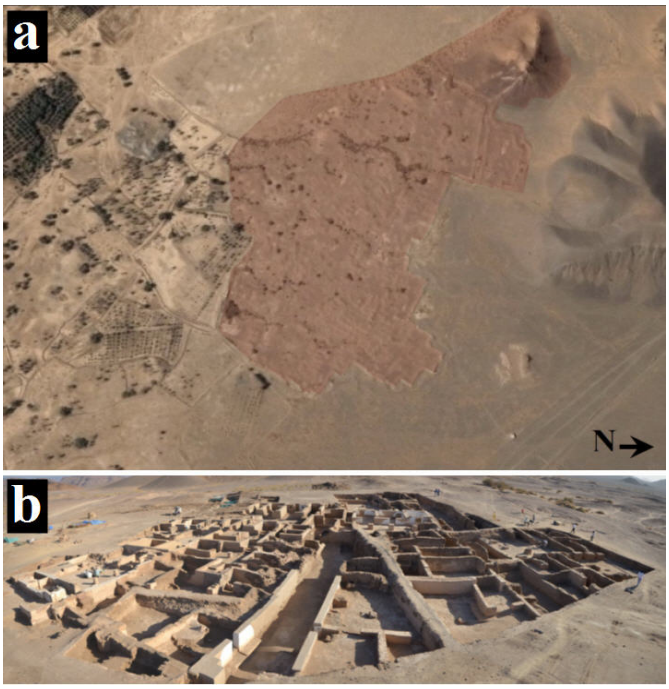

Fig. 1. Qarh archaeological site: a - air view, b - the excavated area.

Materials and Methods

This paper focuses on field observations (a descriptive study – documentation of visible damages) of the wall plasters layers that were discovered by the scientific excavation performed by The College of Tourism & Archaeology - King Saud University. To achieve those objectives, Qarh's plaster samples were collected and analyzed by the following methods: Optical Microscopy (OM - with a Smart-Eye USB Digital Microscope at various magnifications, from a maximum of 170x fixed magnification), in order to characterize the morphological features [10, 11], superficial shape and the grain size of the wall plasters, Polarized Microscopy (PM – with a MT 9000 Series MEJI Co. LTD Japan) used to determine the mineralogical composition and the grain features, by using many cross section and thin section samples [12, 13].

The Scanning Electron Microscope (SEM) JEOL/EO, JSM-6380 device, equipped with an EDS detector operating up to an accelerating voltage of 25kV and a working distance of 9mm was used to investigate the morphology of the deteriorated surfaces [14] of the wall plasters and to detect the distribution of the chemical elements on the plasters layers.

The X-ray diffraction method (XRD) was performed with an Ultima IV, multipurpose X-ray diffraction system, equipped with a copper anticathode. The measuring conditions were set as follows: Cu target, 40kV accelerating voltage, 40mA current, the scanning range of 2θ was from 4 to 70° and the scanning speed was 2°/min. It was used to identify the chemical compositions [15, 16] of the wall plasters at Qarh.

Results and Discussions

Field observations

Many plaster layers (Fig. 2) were found at the Qarh archaeological site. Those plasters used to cover the lower parts and the interior room walls of the units from our field observations; some reports mentioned the following:

- Wall plaster thickness ranges between 1.5cm and 2.5cm in most areas, and up to 3.8cm in some areas. The variety of the wall plasters thickness resulted from the unevenness of the mud-brick surfaces under those plaster layers. On the other hand, this caused the interior surface of the plaster to be uneven.
- The visual appearance of wall plasters seems homogenous.
- In most cases, the wall plaster was found to be connected with the ground plaster, especially in the living rooms.
- Wall plaster was used to plaster architectural elements, such as columns and pillars built of burnt brick.
- The wall plaster consists mostly of more than one layer, but in a few cases it consists of a single layer. The multiple layers of the wall plaster can be observed on the uneven mud brick wall, so the builder added more than one layer to get a flat surface.
- An insulating layer (gray to black in color) was observed between the wall plaster and the mud brick wall. It was observed only in the places where water was used, such as bathrooms, kitchens, water basins and water tanks, which may indicate relationship between this gray layer and waterproofing the mud brick wall.
- The fragility and weakness of the upper parts of wall plaster resulted from the weakness of the wall ruins in the Qarh archaeological site.
- Crystallized salts, superficial salts and subsurface salts deteriorated the wall plaster in various degrees from one area to another.

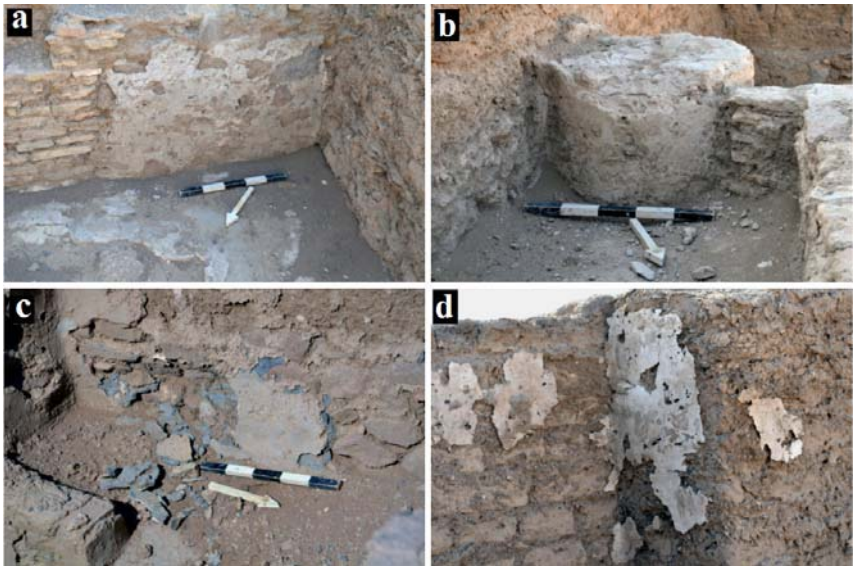

Fig. 2. Different kinds of wall plaster at the Qarh archaeological site.

- Separation of the wall plaster layers, partially or completely, caused by the crystallization of the subsurface, in addition to the effect of other destructive factors.
- The difference of the wall plasters hardness resulted from the difference of the primary and secondary components in the wall plaster layers at the Qarh archaeological site.
- Wall plasters do not contain any paint pigments.

Studied samples

To analyze the different kinds of wall plasters, many samples were collected, representing the different kinds of wall plasters, as shown in Table 1 and Figure 3.

Table 1. Description of the studied samples and their location.

No.	Description	Cross Section's Description	Thickness (cm)	Hardness	Reaction with HCl	place	Photo
MP.1 (a)	MP. 1 Wall plaster consists of two layers; exterior layer MP. 1 (A) having a smooth surface with salt crystals, and interior layer MP. 1 (B) Having a gray color	White color with sand grains and rocks fragments in various colors and sizes.	1.2	It is hard, but can be crumbled by hand	–	Sq. 21 A.	
MP.1 (b)		It has gray color with white clusters	0.8	It is hard, but can be crumbled by hand	-	Sq. 21 A	
MP.2	White plaster plastering the walls and a column	Wall plaster consisting of two layers; exterior layer of fine grains, and interior layer of coarse grains	3.2	fragile	-	Sq. 20 C	
MP.9 (A)	MP. 9 Wall plaster consists of two layers, exterior layer MP. 9 (A) having a smooth surface, and interior layer having a gray color MP. 9 (B)	White color with sand grains, rocks and pottery fragments in various colors and sizes	1.9	It is hard, but can be crumbled by hand	+++	Sq. 23 B	
MP.9 (B)		Gray to blackish color with white clusters	1.8	It is hard, but can be crumbled by hand	+++	Sq. 23 B	
MP. 10	Grayish wall plaster with a smooth surface	Gray to blackish color with white clusters	1.8	It is hard, but can be crumbled by hand	+++	Sq. 23 C	

(-) no reaction to HCl (+++) strong reaction to HCl

Fig. 3. The places where the wall plasters samples were taken from.

OM Results

Thin and cross section samples were prepared to be investigated under the optical microscope. The results were as the following:

Sample MP. 1 reveals the presence of salt crystals on the surface. It consists of three layers: the lower layer is remains of mud brick, the intermediate layer has grayish components and the top layer has white components (Fig. 4).

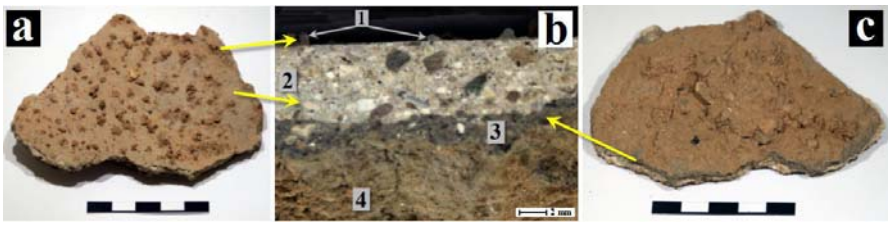

Fig. 4. Sample MP1 (a) salt crystals on the surface, (b) cross section shows: salt crystals (1), white layer (2), grayish layer (3), mud brick remains (4), (c) lower layer of mud brick remains.

By examining the cross and the thin section of the white and gray layers, the following components were revealed: a white layer (the upper layer) consisting of a mixture of white background, sand, stone fragments (SF) pottery fragments (PF), and white clusters (W) that are different in color, shape and size (Fig. 5). We may assume that the cementing material was a white material, while sand, rock and pottery fragments were fillers.

The gray layer (the intermediate layer) consists of a grayish background, burnt ash mixed with sand, and traces of burnt organic materials (BO) (Fig. 5).

The salt crystals on the surface of the white upper plaster layer crystallized in various forms (Fig. 6). They are pure and transparent, which indicates a crystallization in suitable conditions and the existence of an internal source of saline water [15, 16].

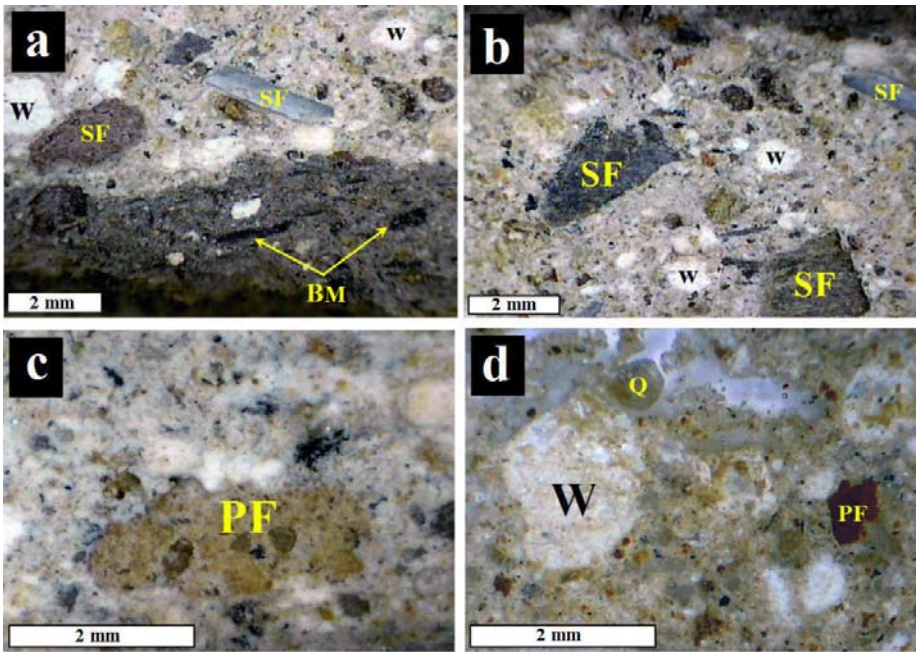

Fig. 5. (a) The white upper layer consisting of white background, stone fragments "SF" and white clusters "W". The intermediate grayish layer showing grayish background containing burnt material remains "BM" and an amount of white clusters. (b), different shapes, sizes and colors of stone fragments. (c) archaeological pottery remains "PF", (d). quartz "Q" and white "W" clusters.

Fig. 6. Various forms of crystalline salts on the sample MP.1a surface.

Sample MP.2 used to plaster the walls and the column at square 20 C. The plaster layers have many cracks causing the separation and falling down of the plaster. Its thickness is up to 3.2cm and it consists of two layers (Fig. 7).

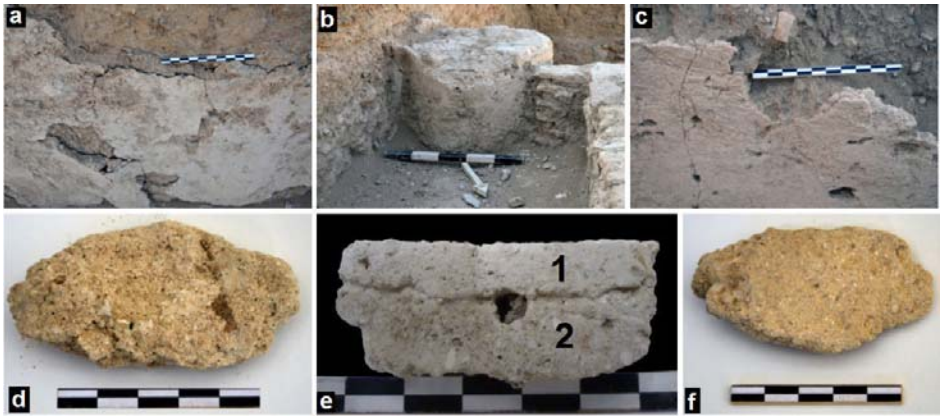

Fig. 7. Images of the cracked plaster layers(a, b and c), the lower surface of the sample MP.2. (d), cross section of sample MP.2 showing the two layers (e) and the upper surface of the sample MP.2 (f)

The detected results of the optical microscope analysis for sample MP.2 showed that it consists of a mixture of white background, an amount of fine sand with rounded edges "Q", a small amount of pottery fragments 'PF", straw "S", and white clusters "M" (Fig. 8).

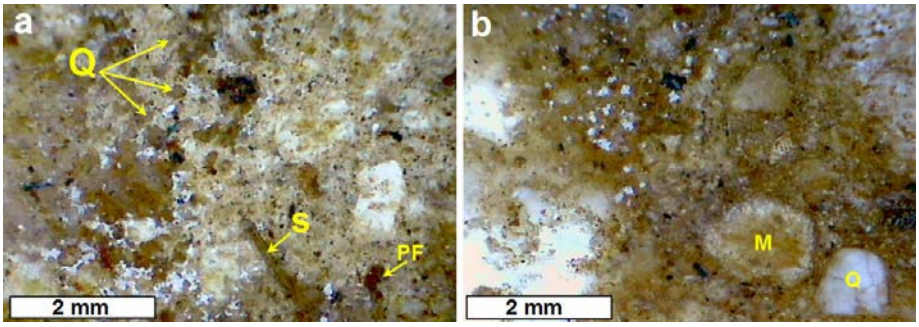

Fig. 8. MP.2 sample consisting of a mixture of white background, a large amount of fine sand with rounded edges "Q", a small amount of pottery fragments "PF", straw "S", and white clusters "M".

Sample MP.9 taken from the plaster was used on the walls and the floor in the bathroom at Sq. 23 B. The grayish layer was used as a contact layer between the mud brick wall and the outer, white plaster layer. The foundations of the bathroom were built from sandstone, which was more resistant to moisture than mud-brick (Fig. 9).

Fig. 9. MP.2 sample showing the two layers (the white layer and the grayish layer): a and c - cross section, b - the area where MP.9 sample was taken from.

By examining the cross sections and the thin sections of sample MP.9 under the optical microscope observations we detected that sample MP.9 consists of two layers: the first layer MP.9a is a mixture of white background, fine sand with sharp edges "Q", stone fragments "SF", yellow and white clusters, as well as re-used parts of old mortar (OM), as mentioned by Miriello and others [18]. It may indicate that the components of sample MP.9 were of a white cementing material and fillersof sand, stones fragments and remains of old mortar (Fig. 10). The second layer, gray in color MP.9b consists of a grayish cementing background that contains remains of burnt material "BM" (Fig. 10g, h and i).

Sample MP 10 was taken from the plaster used on the mud brick wall and a burnt brick pillar at Sq. 23 C. Field observations revealed that the plaster was stable on the burnt brick pillar but completely destroyed on the mud brick wall. The results of the optical microscope analysis show that sample MP.10 consists of grayish cementing background, containing remains of burnt material "BM". This sample is different from the other samples, as it has only one gray layer used on a mud brick wall and a firebrick pillar (Fig. 11).

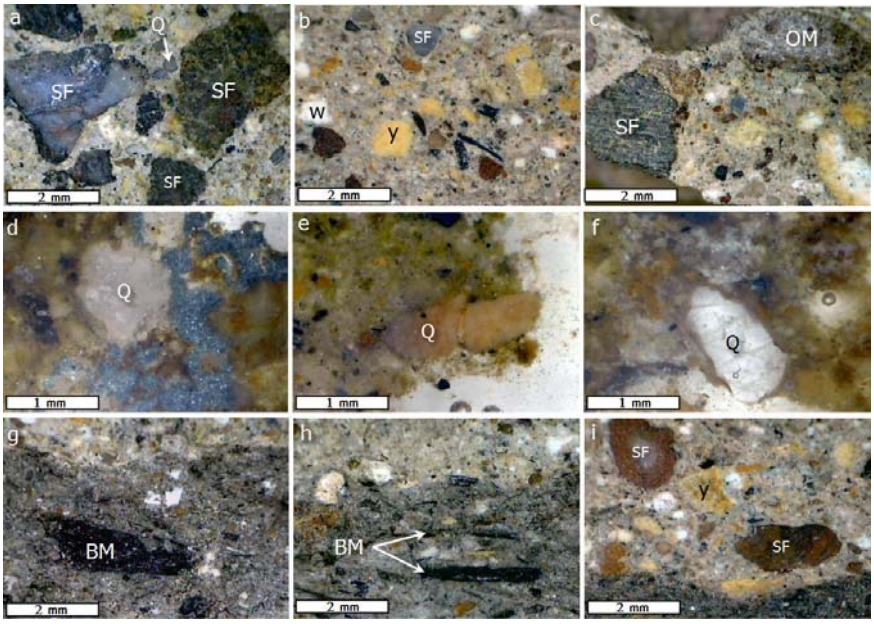

Fig. 10. Images of sample MP.9a (a, b, c, d, e, f and i) and MP.9b (g, h and i).

Fig. 11. The stable plaster layer on the burnt pillar, and the remains of plaster on the mud brick wall (a) and the composition of the sample MP.10 (b, c, d)

Polarized Microscope Results

From the results of the polarized microscope investigation, a difference between the two layers MP.1a and MP.1b was detected. Sample MP.1a consists of different shapes and sizes of quartz "Q" and stone fragments "SF" in a gypsum background. Sample MP.1b consists of different shapes and sizes of quartz (sand) "Q" in a background of clay, iron oxides and traces of burnt organic materials (Fig. 12). These results comply with the results of other investigations, carried out on similar samples [19, 20].

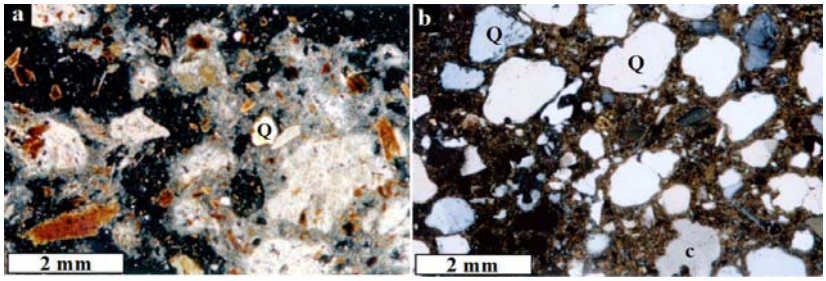

Fig. 12. Microphotographs of Qarh plaster under crossed polars (a) and Quartz in sample MP.1a and in sample MP.1b (b)

The PM result of Sample MP.2 has revealed that it consists of a mixture of a gypsum background with various phases of gypsum nodule, a little amount of quartz (sand) and charcoal fragments. This composition, where the filler was not enough, explains why this sample is weaker than the other samples (Fig. 13).

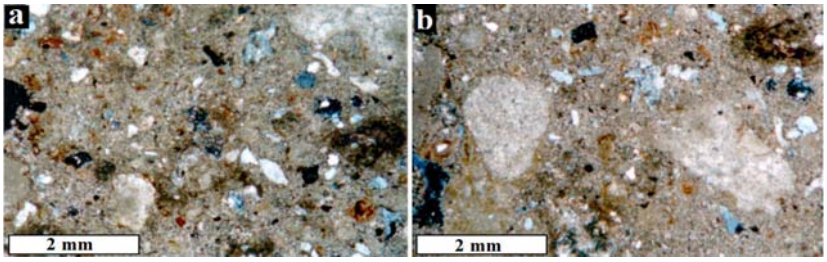

Fig. 13. Microphotographs of sample MP.2 showing the mixture of gypsum, sand and charcoal fragments under crossed polars.

Sample MP.9 (including MP.9a) representing the white upper layer and MP.9b representing the gray lower layer. The Polarized Microscope results show that MP.9a consists of quartz (Q) in different shapes and sizes in a calcareous background, lime lumps and stone fragments. Sample MP.9b consists of a mixture of calcite and quartz as well as traces of burnt organic materials (Fig. 14).

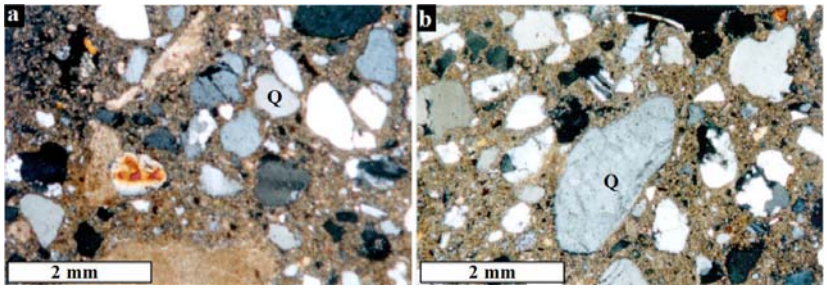

Fig. 14. Microphotographs of white upper layer in sample MP.9a (a), the gray lower layer in sample MP.9b (b) under crossed polars.

We may deduce that the white matter (gypsum) was replaced by calcite (lime). The lime plaster was used to coat the ancient bathrooms that usually used water more than the other units in the ancient homes, as lime plaster is more resistant to water than gypsum plaster [21]. Thus, we assume that the builders knew that information and therefore replaced gypsum plaster with lime plaster. In addition to moisture isolation that layer consists of gray ash, lime and sand.

The polarized microscope investigation of sample MP.10 revealed that it consists of a mixture of gray background of manual kilns ash, gypsum, calcite and quartz, as filler. This sample was different from the other samples, as it is gray color and consists of a mixture of calcite, gypsum and quartz, used for plastering the firebrick pillar (Fig. 15).

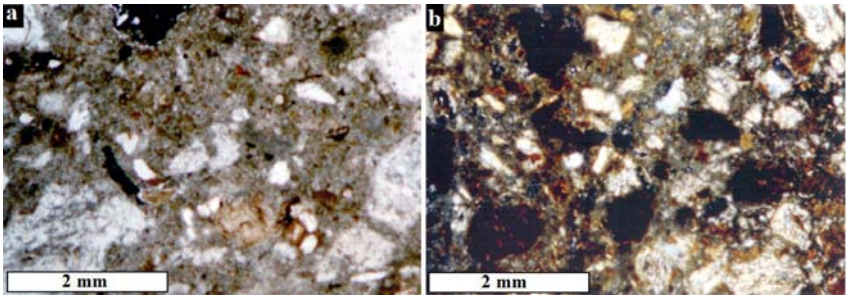

Fig. 15. Microphotographs of sample MP.10 under crossed polars.

SEM results

Results of the scanning electron microscope investigations showed that the surface of sample MP.1a and b was fragmented and damaged (Fig. 16a and b). Sample MP.2 shows halite crystals in a background of gypsum (Fig. 16c), sample MP.9a and b shows the deterioration of the surface that contains halite crystals, sand and lime (Fig. 16d and e) and sample MP.10 shows the deformation of calcite and bassanite grains (Fig. 16f).

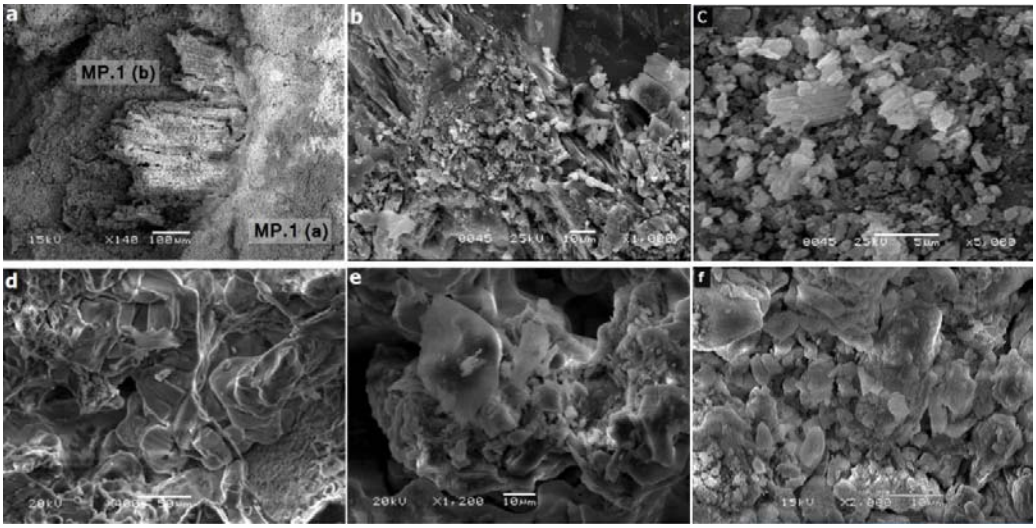

Fig. 16. SEM images: **a** - the difference between the texture of sample MP.1a and MP.1b, b - the surface deterioration of the sample MP.1b, c - halite crystals in the background of bassanite and sand, d - the white upper surface of the sample MP. 9a containing halite crystals, sand and lime, e - gray under layer of the sample MP.9 b, f - the damaged of bassanite and calcite components in sample MP.10.

The results of the EDS analysis carried out on the sample MP.9a and b are shown in Table 2 and Fig. 17.

Table 2. The elemental composition (wt.%) of the studied plaster samples.

Sample	*C*	*O*	*Mg*	*Al*	*Si*	*S*	*Cl*	*K*	*Ca*	*Fe*	*Na*
MP.9 (a)	9.54	22.31	1.98	2.59	7.85	7.34	1.74	2.04	44.61	-	-
MP.9 (b)	7.88	19.70	2.16	5.54	19.02	-	1.07	1.97	37.34	3.78	1.45

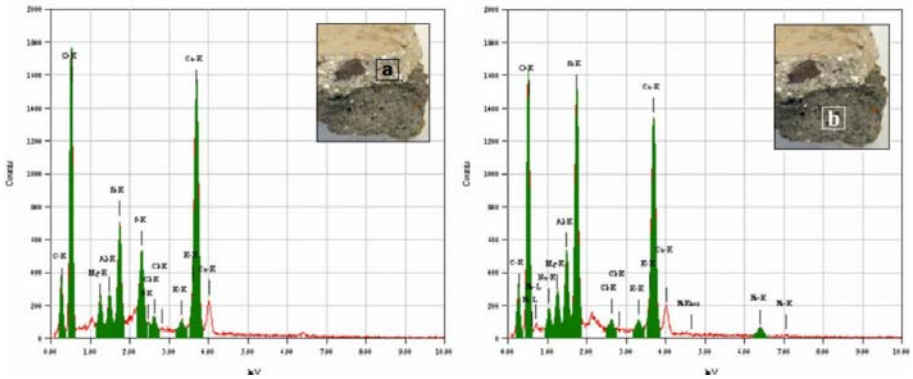

Fig. 17. EDS spectrum of MP. 9 (a and b) samples.

XRD results

The results of the XRD analysis for the studied samples are shown in Table 3, and Fig. 17(a, b, c, d, e, f) [25, 26].

Table 3. Identified minerals in the plaster samples by XRD analysis.

Samples		Minerals	Formula	Index No.
MP. 1	a	Halite	NaCl	5-0628
		Quartz	SiO_2	5-0490
		Bassanite	$2\ CaSO_4{\cdot}H_2O$	14-453
		Gypsum	$CaSO_4{\cdot}2H_2O$	6-0046
	b	Alleghanyite	$Mn_5Si_2O_8(OH,F)_2$	12- 433
		Quartz	SiO_2	5-0490
		Bassanite	$2\ CaSO_4{\cdot}H_2O$	14-453
		Geothite	α - FeOOH	17-0536
		Anhydrite	$CaSO_4$	6-0226
		Gypsum	$CaSO_4{\cdot}2H_2O$	6-0046
		Halite	NaCl	5-0628
MP. 2		Anhydrite	$CaSO_4$	6-0226
		Halite	NaCl	5-0628
		Quartz	SiO_2	5-0490
MP. 9	a	Calcite	$CaCO_3$	5-0586
		Quartz	SiO_2	5-0490
		Halite	NaCl	5-0628
		Geothite	α - FeOOH	17-0536
	b	Quartz	SiO_2	5-0490
		Calcite	$CaCO_3$	5-0586
		Anhydrite	$CaSO_4$	6-0226
		Halite	NaCl	5-0628
		Gypsum	$CaSO_4{\cdot}2H_2O$	6-0046
MP. 10		Bassanite	$2CaSO_4{\cdot}H_2O$	14-453
		Calcite	$CaCO_3$	5-0586
		Quartz	SiO_2	5-0490
		Anhydrite	$CaSO_4$	6-0226
		Gypsum	$CaSO_4{\cdot}2H_2O$	6-0046
		Hercynite	$FeAl_2O_4$	7- 68

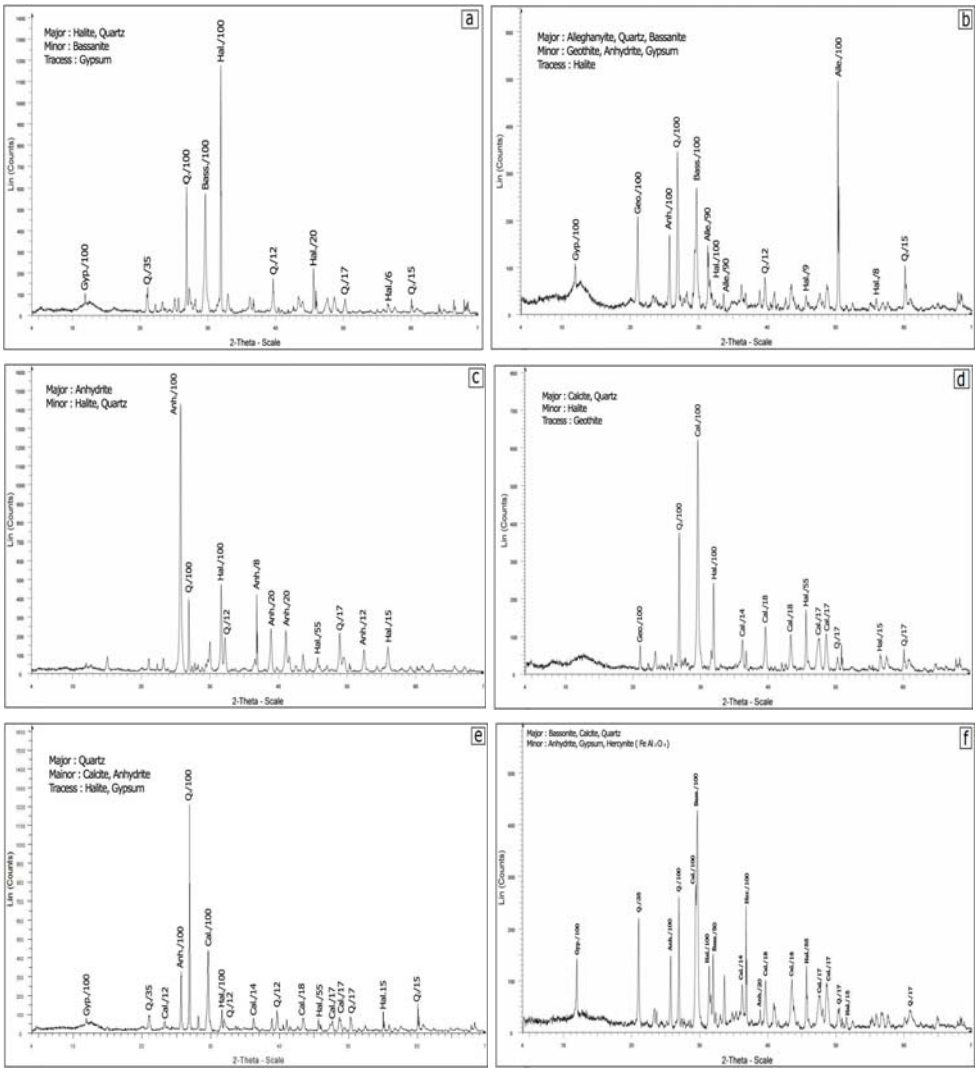

Fig. 17. X-ray spectrums of the identified minerals from the studied plasters samples, Sample MP.1(a and b), Sample MP.2(c), Sample MP.9(d and e), and Sample MP.10(f).

The XRD results confirmed the investigation results obtained by optical microscopy, polarized microscopy and SEM-EDS. From those results, we deduced that the main materials used in the different kinds of Qarh wall plasters were:

- Gypsym (Bassanite $2CaSO_4{\cdot}H_2O$, Anhydrite $CaSO_4$)
- Lime (Calcite $CaCO_3$)
- Sand (Quartz SiO_2)
- Qsromel (the ash resulted after burning firewood and other materials in manual kilns), (Alleghanyite $Mn_5Si_2O_8(OH,F)_2$, Hercynite $FeAl_2O_4$)

Halite (NaCl) was detected in samples; MP.1a and b, MP.2 and MP.9a and b, as a salt contamination rather than as a plaster component. Its existence is a result of its transferring from under ground water in the soil through water capillarity.

Gypsum which was detected in samples; MP.1a and b, MP.2 and MP.10, was used as a building material because of its natural properties [22]. It was subjected to a calcification process, crushed to powder and then mixed with water, so it transformed into a paste which hardens by drying. This process involves the chemical reaction of the natural gypsum, that transforms after burning to the dry phase of calcium sulfate (anhydrite), at a temperature 300°C, or to semi-dry phase of the same material (Bassanite or plaster of Paris), at temperatures between 120°C - 160°C [23].

$$CaSO_4 \cdot 2H_2O \xrightarrow{120-160°C} CaSO_4 \cdot ½\ H_2O \text{ (Bassanite)} + 1½\ H_2O$$

$$CaSO_4 \cdot 2H_2O \xrightarrow{300°C} CaSO_4 \text{ (Anhydrite)} + 2H_2O$$

The calcification degree (burning) of the gypsum plays an important role in the mechanical properties of the final product [24]. The partial removal of water molecules from calcium sulphate (gypsum) at 110 °C gradually increases to form bassanite between 120 °C - 160 °C. The last temperature is generally accepted in the traditional gypsum industry. By adding the water that was lost during the calcification process you obtain plaster (in 5-10 minutes) as in the following equation:

$$CaSO_4 \cdot ½\ H_2O \text{ (Bassanite)} + 1½\ H_2O \xrightarrow{Hydration} Ca\ SO_4 \cdot 2H_2O \text{ (Gypsum)}$$

We can get another phase of plaster by adding water, but setting will be very slowly and the resulted plaster (anhydrite) will be less hard than the plaster of bassanite. This explains the weakness of the wall plaster of sample MP.2. Gypsum loses its coherence by burning at a temperature of 600°C. By studying the Qarh wall plasters, we detected two phases of gypsum (Bassanite and Anhydrite), which caused difference in the wall plasters hardness, without ignoring the role of the filling materials in their hardness.

Lime which was detected in sample MP.9a and b is a kind of plaster that takes a long time to harden after drying and turning into a coherent mass. The mechanism of the drying process varies according to the type of lime used; the non-hydraulic lime (air-lime) or the hydraulic lime [25, 26]. The properties of the lime plaster is influenced by additional materials [27] that will affect the solidification process and improvement of its mechanical properties [28], and the most important of these additional materials is Qsromel.

The use of ***Qsromel*** (ash from man-burned materials) was common in the medieval Islamic world as an alternative to the volcanic ash. It was similar to the Pozzolan that consist of silica, alumina, iron oxides, magnesium oxides, calcium oxide, manganese oxide and other raw materials. This explains the presence of Alleghanyite $Mn_5Si_2O_8(HO,F)_2$ and Hercynite $FeAl_2O_4$ in samples MP.1 and MP.9 that were taken from the gray layers. Its color is dark gray, black when it is pure. To assess the degree of purity, we can immerse a little amount in water. If it is pure, it will float and if it is mixed with dust, it will sink. Adding Qsromel to lime plaster or mortar will make it hydraulic (setting in water or moist conditions). This explains the use of this mixture in places where water is used frequently, such as bathrooms, as detected in sample MP.9.

Conclusions

From our analyses and investigations we concluded that the wall plasters layers in the Qarh archaeological site are of four kinds. The first kind is a white plaster consisting of (gypsum + sand) that was represented in sample MP.2. The second kind of wall plaster is a mixture of gypsum, lime, sand, as well as Qsromel, as represented in the sample MP.10. The third kind of wall plaster is gypsum plaster over an insulating layer of Qsromel. This kind consists of two layers complementary to each other: a gray, lower layer consisting of ash, sand and gypsum and an upper white layer consisting of sand and gypsum. This kind of wall plaster was represented by sample MP.1a, b.

The fourth kind of wall plaster in the Qarh archaeological site is lime plaster over an insulating layer of Qsromel. It consists of two layers, complementary to each other: a gray lower layer consisting of ash, sand, lime, a little amount of gypsum and an upper, white layer consisting of sand, lime and a small amount of gypsum. This kind of wall plaster was represented by sample MP.9a, b.

Acknowledgments

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