

“Conservation of Architectural Heritage, CAH” 23-27 November 2015, Luxor

Alternative lime based grouts used in re-pointing of deteriorated ancient mortars and their structural effects on composite unreinforced masonry walls of Mekaad Radwan, ottoman Cairo, Egypt (Case study)

Mona F. Ali^a, Abubakr Moussa^{a,b,*}, Mahmoud Abdelhafez^a

^a*Department of Conservation, Faculty of Archaeology, Cairo University, 12611 Orman, Giza, Egypt.*

^b*Department of Tourism and Antiquities, Jazan University, 114 Jazan, KSA.*

Abstract

Mortars and plasters of Mekaad Radwan are badly affected by decay hazards, therefore; they were studied by means of XRD and polarized microscope to detect their chemical composition, physical and petrographic properties, in addition to identify their characteristics. The analyses revealed gypsum mixed with lime as dominant component with some aggregates of sand particles, sodium chloride was detected in all the studied samples. Standardized alternative mortars were prepared, exposed to artificial aging and tested in order to select the most suitable type to replace the old mortars and plasters at Mekaad Radwan.

© 2016 The Authors. Published by Elsevier Ltd. This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>).

Peer-review under responsibility of IEREK, International experts for Research Enrichment and Knowledge Exchange

Keywords: Mekaad Radwan; building materials; XRD; polarized microscope; alternative mortars; lime based grouts; re-pointing

1. Introduction

Mekaad Radwan is situated in the neighborhood of Bab Ziweila in the historical Cairo (fig. 1a, b). It was built in the 17th century (1650 AD). The Mekaad Radwan monumental building consists of three storeys: the ground floor, the opened floor (the Mekaad room), and the housing top floor (the roof). The building is made of timber and masonry load bearing walls. The floors and ceilings are supported by timber beams, whereas the window and door openings are strengthened by timber or stone lintels. The facades of the building are composed of non-plastered walls made of

* Corresponding author. Tel.: Tel.: +2-010-9666-4411.

E-mail address: dr_abubakr@msn.com

large well-edged parallelepiped and/or cubed limestone blocks with regular horizontal and rare vertical mortar joints. The internal mason load bearing walls are mostly thick and plastered by lime-gypsum mortars. The building is affected by severe deterioration phenomena and patterns of damage which occurred during the time. These deterioration and damages are mainly due to foundation problems, subsoil water and also to the earthquake that affected the whole of Greater Cairo in October 1992.

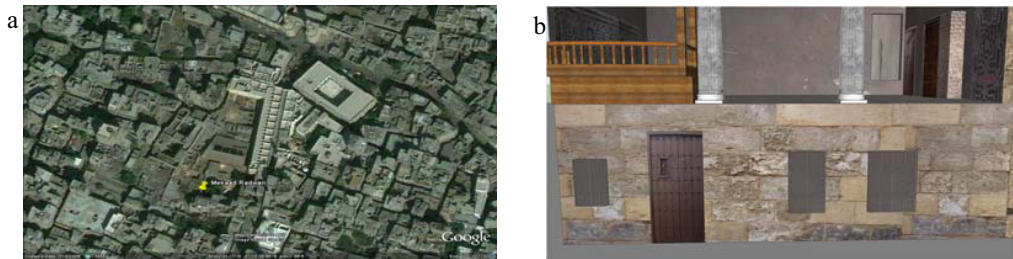


Fig. 1 (a) site of the case study; (b) front façade of the case study.

The plasters and mortars of the Mekaad walls were subjected to loss and cracking due to internal and external reasons. The internal reasons are concerning with petrographic characteristic of the building stone and bricks as well as the type and composition of the mortars and plasters used. The external reasons may be relate to the construction deficiency, exterior salt invasion or differential settlement and subsidence as well as the earthquake affects.

Physical and mechanical characterizations are important requirements for the development of compatible mortars. However, for ancient mortars they are sometimes impossible to evaluate due to the difficulty to do rigorous determinations with irregular, friable specimens, cutting standard-sized test samples or sampling from an ancient wall of high aesthetic value. The tests that are most often performed are compressive resistance, water absorption and porosity (Candeias, A.E., Nogueira, P., & Mirão, J., et al., 2004).

2. Materials and Methodology

Several samples were collected from ancient mortars and plasters as well as from recent plasters that were used during the last restoration upon either limestone blocks or the red bricks (table, 1). However, some physical characteristics are impossible to evaluate due to the difficulty to do rigorous determination with irregular, friable specimens cutting standard sized test sample that could be collected from very soft ancient mortar.

Table 1. The specific locations of the collected mortar and plaster samples.

Type	Symbol	Specific location
Mortar samples	MBM	Binding mortar between red bricks
	LSM	Binding mortar between limestone blocks
	ZLSP	Binding mortar between zigzag limestone blocks
	MME	Collating mortar of the mosaics
Plaster samples	MBP	Plaster upon the red brick
	LSP	Plaster upon the limestone blocks

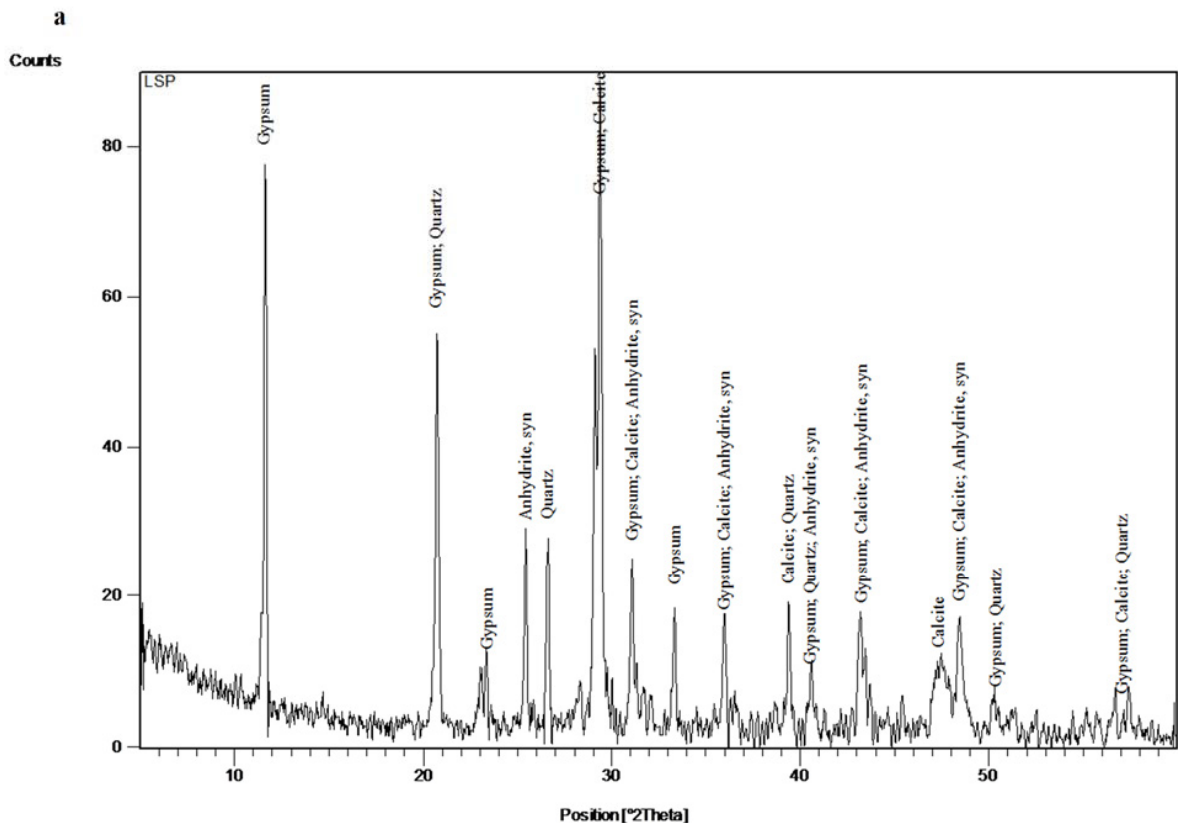
Preliminary morphological observation of the raw surface and polished thin section of the samples were carried out using Zies optical light microscopy. Thin sections of the samples were examined using polarized transmitted light microscopy model Nikon opti photo x23 equipped with photo camera S23, under 10x and 20x magnification in plane-polarized light. X-ray diffraction (XRD) was performed on powdered samples of the core and glaze materials in addition to the mortar used to adhere the tiles into the walls, using a Philips (PW1840) diffractometer with Ni-filtered

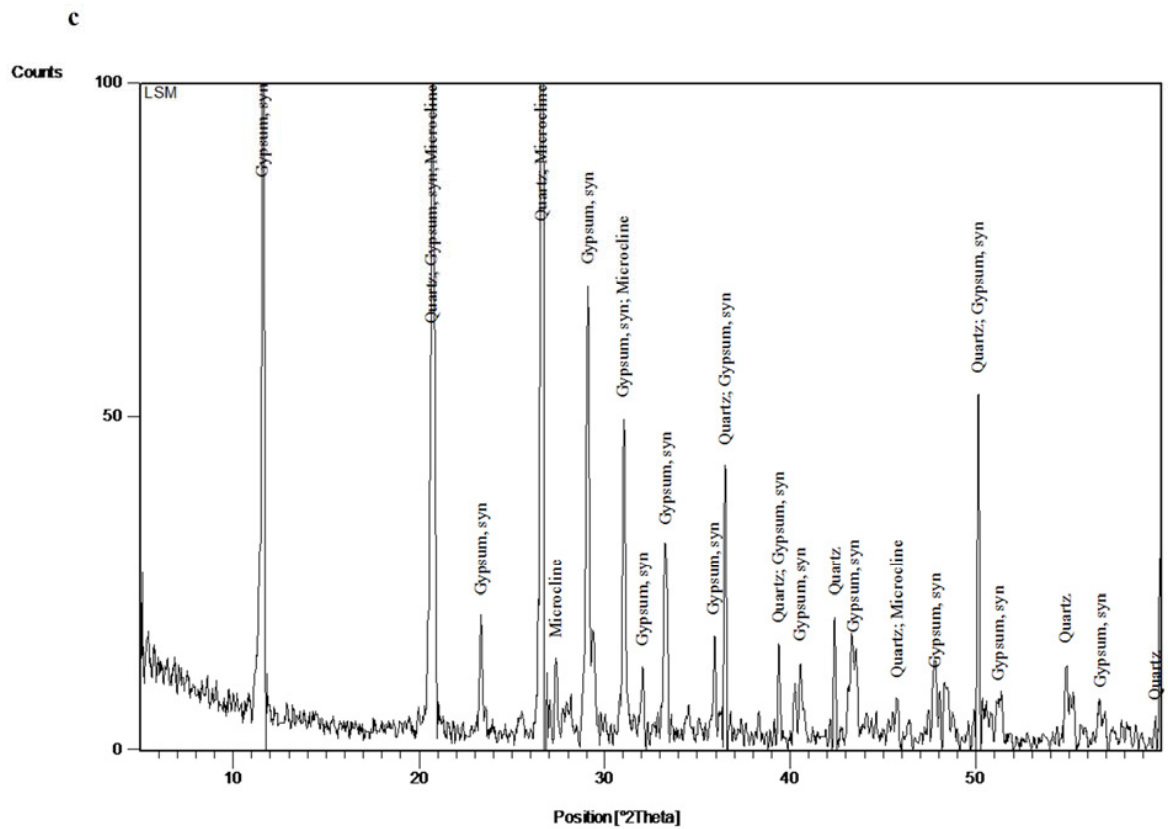
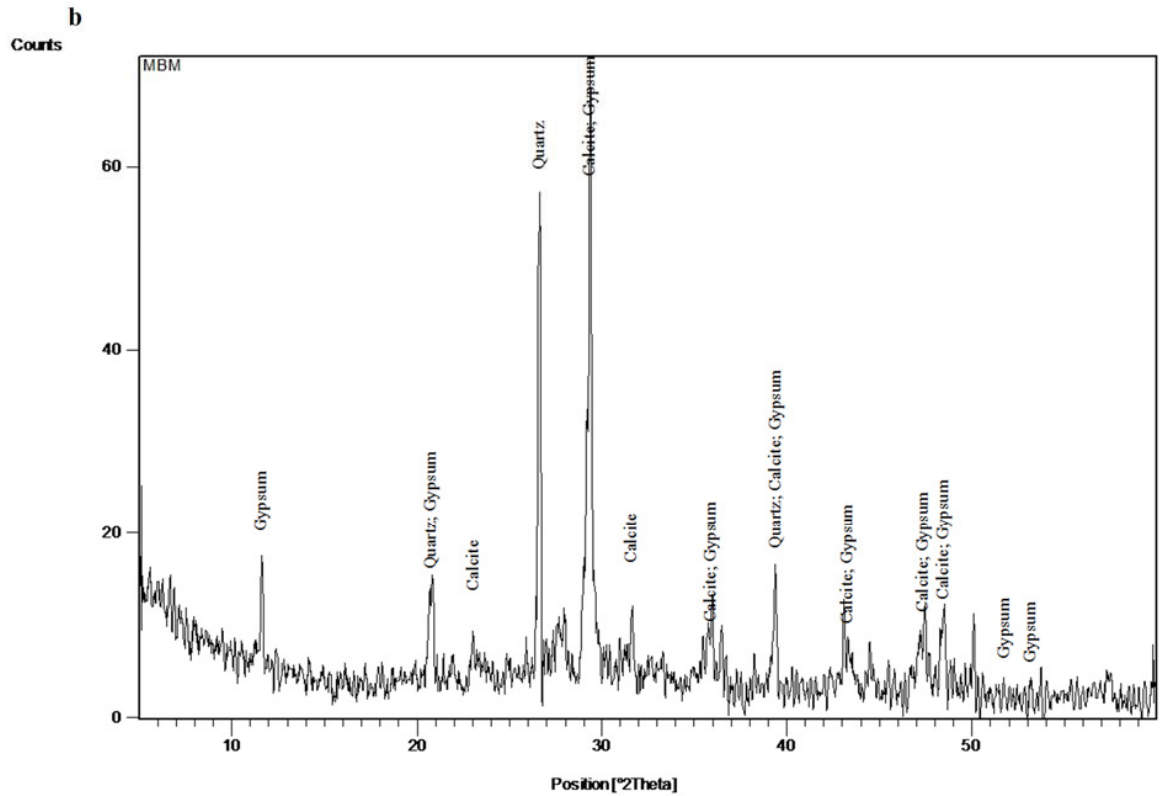
Cu-K α radiation The samples were scanned over the 0-60° 2 θ intervals, at a scanning speed of 1.2° min⁻¹ A° quantitative estimate of the abundance of the mineral phases was derived from the XRD data, using the intensity of certain reflections and external standard mixtures of minerals compared to the (JCPDS standards of 1967); the detection limits of the method were ± 1 w/w %.

3. Results and discussion

The microscopic investigation proved that the plaster sample (LSP) located upon the limestone blocks is similar to the mortar sample that binding the same blocks. Both are composed of lime lumps represented by fine calcite particles with gypsum, few anhydrite crystals, and few fine quartz sand grains. Some additives (very fine fibrous and organic materials) are also observed embedded in the gypsiferous lime matrix. Some gypsum particles seem to be altered to anhydrite crystals.

The XRD results (fig. 2a) support the composition of the plaster sample (LSP). The XRD result of mortars indicated that there are three types; the first type (fig. 2b) mainly consists of calcite (68%) and gypsum (7%) with quartz (25%); the second type (fig. 2c) was used as binding material for the building limestone blocks contains gypsum (24%), calcite (32%) with excess quartz (35%) in addition to some feldspar grains (9%). Such feldspar (mainly k-feldspar) associated with a part of quartz may be derived from granitic rock powder which possibly was added as additives to ancient mortar, and the third type (fig. 2d) consists of gypsum (72-76%), little calcite (11-12%) and traces of quartz (5-6%).





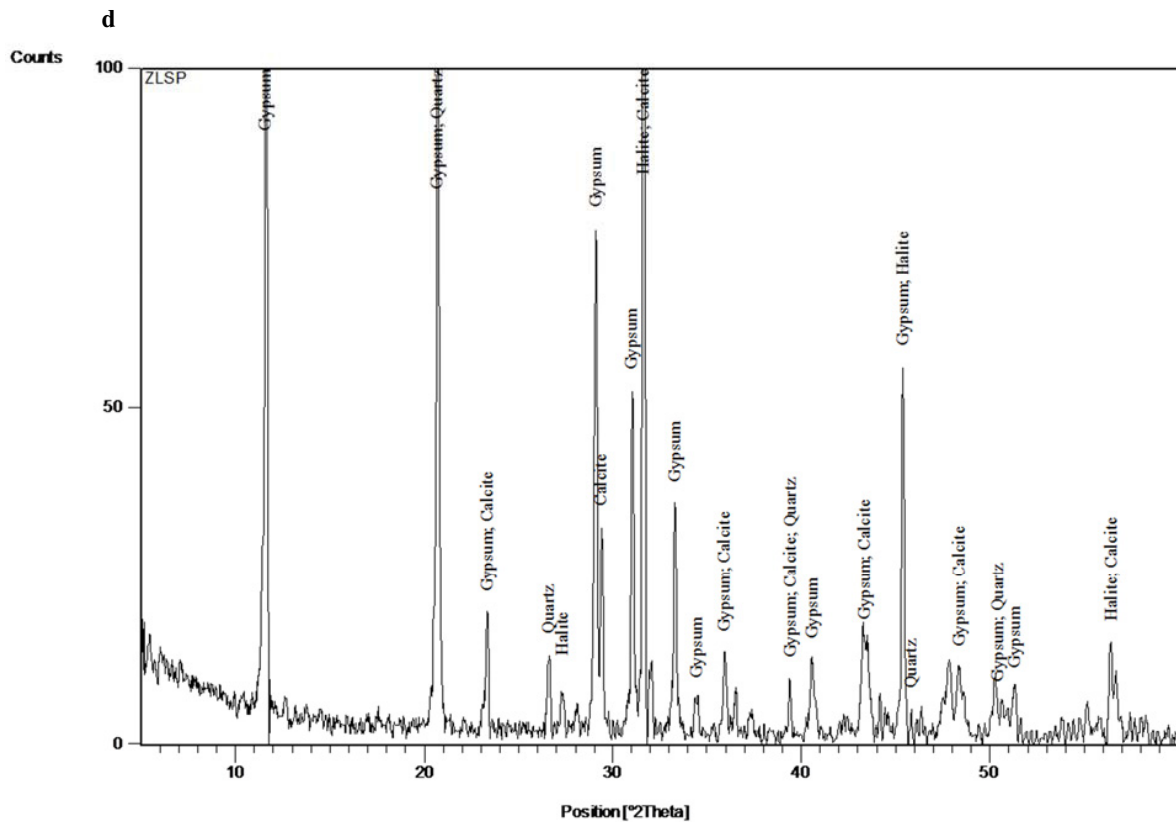
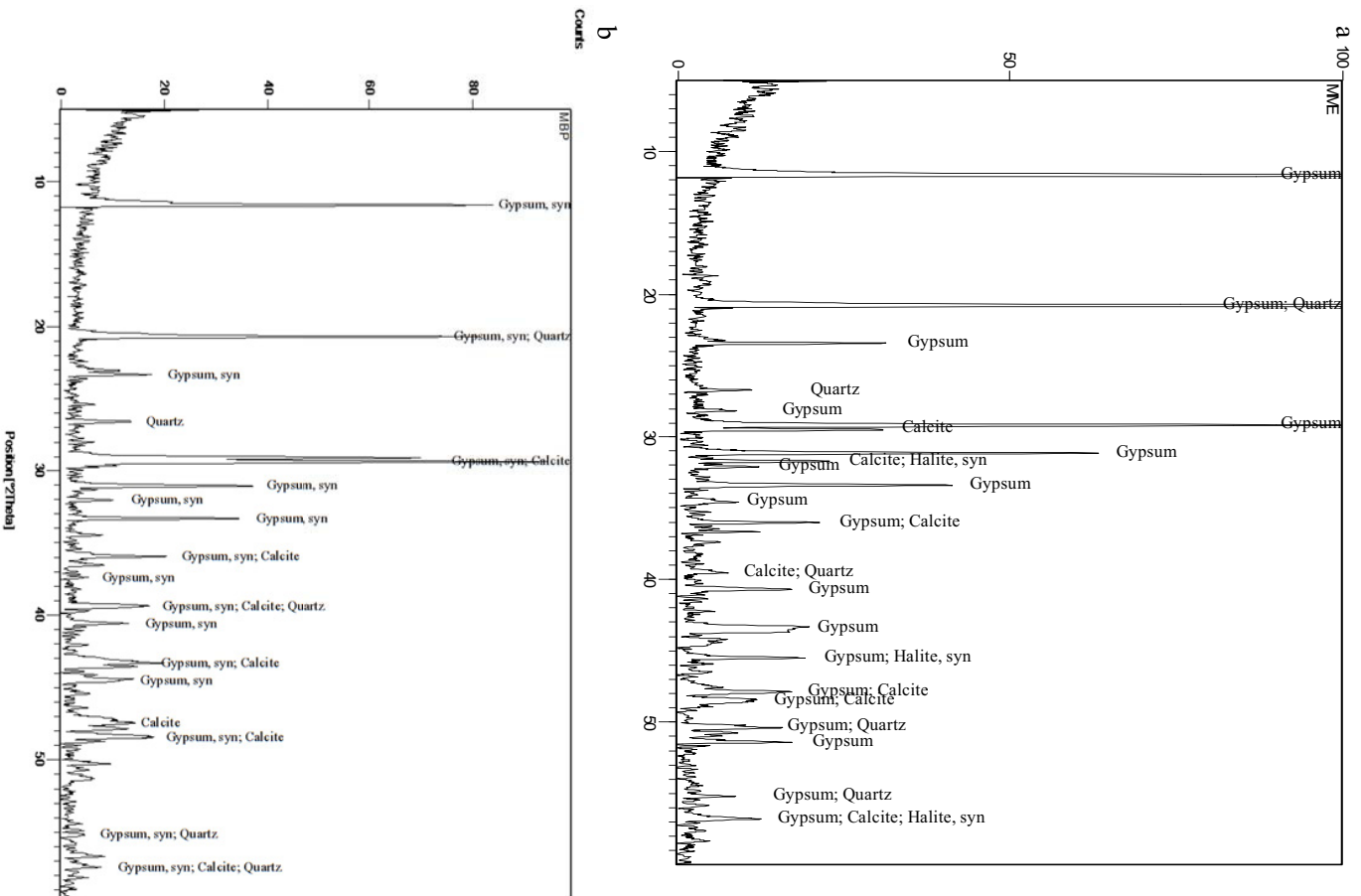


Fig. 2. (a,b,c,d) XRD diffraction patterns of the analyzed mortar samples.

The weathered gypsum mortar usually includes 8-10% salt (sodium chloride) which is called in the X-ray chart "synthetic halite". This salt is not considered as one of the components of the mortar, but it was formed from salinization weathering of mortar. That salt has a negative influence on the quality of the mortar. Therefore it is unlikely an additive (fig. 3a). The plaster samples are lime-gypsum type. The X-ray analysis of the plaster samples (MBP) and (LSP) shows that they are composed of (45-51%) calcite (lime), (39-44%) gypsum and traces of quartz (5-8%), (fig. 3b). Some ancient mortar or plaster, such as sample (LSP) occasionally contains few anhydrite crystals (8%) that may be formed by dehydration of the original gypsum at higher temperature.

Particularly the proportion of gypsum and sand additives of the mortars is another significant factor controlling the level of cracking and losses. Generally in preparation of mortar or plaster for restoration processes, the addition of gypsum improves the strength of lime – various pozzolan mixes, but the gypsum content should remain limited because the ettringite formation causes swelling and disintegration of the material (Toumbakari, 2002).



Sometimes, it is recommended to use lime/sand mortar, like that of sample (RBM) because it is more appropriate for repair of older masonry with an existing lime mortar, and where exposure to climate loads is low. It is important to avoid the presence of soluble salts, or salt crystals in the prepared mortar for restoration and therefore avoiding the appearance of efflorescence and subflorescence. These properties can be relatively modified by altering the process of production of the mortar, the type of aggregate, the aggregate/binder and water/binder ratio...,etc. (Palomo, A., Blanco-Varela, M.T., & Martinez-Ramirez, S., et al., 2004).

Two destructive expansive reactions are usually taking place in the presence of gypsum leading to the formation of ettringite and thaumasite. The latter can be formed in historic mortars and plasters by the reaction of gypsum and calcium aluminum hydrates in a moist condition (Böke and Akkurt 2003). The formation of ettringite or thaumasite leads to the complete destruction of the material (Van Balen, K., Toumbakari, E.E., Blanco-Varela, M.T., et al. 1999). Fissuring and cracking phenomena are widely observed in the case study. The possibility of ettringite formation in historic mortars or plasters due to air pollution effect was previously discussed as showed in the reactions (Sabbioni, C., Zappia, G., & Riontino, C., et al. 2001, Sabbioni, C., Bonazza, A., & Zappia, G., 2002):

- $3(\text{CaSO}_4 \cdot 2\text{H}_2\text{O}) + 3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O} + 20\text{H}_2\text{O} \rightarrow 3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$ (Ettringite)
- $\text{CaSO}_4 \cdot 2\text{H}_2\text{O} + \text{CaCO}_3 + \text{CaSiO}_3 \cdot \text{H}_2\text{O} + 12\text{H}_2\text{O} \rightarrow \text{CaSiO}_3 \cdot \text{CaSO}_4 \cdot \text{CaCO}_3 \cdot 15\text{H}_2\text{O}$ (Thaumasite)

Charola and Centeno state that the dehydration of gypsum to hemihydrate does not occur on monuments (Charola and Centeno, 2002). This does not preclude that gypsum, particularly in cryptocrystalline forms, may partially dehydrate and eventually rehydrate giving rise to further contraction–expansion cycles (Charola, 2003). This hypothesis is supported by the detection of both anhydrite and gypsum in the plaster layer of the Mekaad Radwan.

Anhydrite was detected abreast with gypsum in the rendering layer. It is our position that the material used was anhydrite and the effect of the slight humidity started the anhydrite to absorb water molecules and return partly to gypsum in an uncompleted hydration process. This change is usually followed by an increase in the volume of anhydrite. During the transformation of anhydrite to gypsum, a volume expansion of up to 61% is possible, this develops hydration pressure which depends on the temperature and humidity of the air (Moussa, A., Kantiranis, N., & Voudouris, K., et al., 2009).

4. Experimental work

From the historical perspective, repointing should be carried out using the same material as originally used. From a practical point of view, a masonry wall should be pointed with a carefully chosen lime mortar, which is just softer and more porous than the material with which the wall is constructed. Generally, the best aggregate for lime mortar is a well graded sand mix comprising angular particles ranging in size from large to small (from 6 millimeters in diameter to dust) in a suitable color (Moussa, 2007).

In order to prepare the most appropriate mortar for re-pointing purposes of the deteriorated mortars at Mekaad Radwan, many standard cylinders of different types of mortars were prepared; and given alphabetic symbols table (2).

Table 2. Composition of the studied alternative mortars.

Symbol	Composition
A	1 part calcium carbonate + 1 part sand + 2 parts palm
B	½ part calcium carbonate + 1 part sand + 1½ part palm + 1 part white cement
C	1 part lime + ¼ part Sand + 1 part ash mortar “qusrmil” + ¼ part white cement
D	1 part lime + ¼ part sand + 1 part qusrmil
E	5 parts lime + 1 part sand
F	3 parts lime + 1 part sand + 1 part qusrmil + 2% primal AC 33
G	3 parts lime + 1 part sand + 1 part qusrmil + 1 part white cement
H	3 parts lime + 1 part sand + 3 parts qusrmil

I	3 parts lime + 1 part sand + 3 parts qusrmil + 2% Primal AC 33
J	3 parts lime + 1 part sand + 3 parts qusrmil + 1 part white cement

Prepared mortars were left to dry in room temperature, they were optically investigated in order to register their color, then the mortars were weighed and measured before they were exposed to artificial ageing to test their resistance to the environmental factors which Mekaad Radwan is facing indeed, table (3).

Table 3. Alternative mortars after drying.

Code of mortar	Weight before drying	Weight after drying	Color before drying	Color after drying	Dimension before drying (cm)	Dimension after drying (cm)
A	485g	470g	Off white	Off white	13.6 x 5.5	13.6 x 5.5
B	445g	430g	Off white	Off white	13.6 x 5.5	13.4 x 5.4
C	340g	330g	Light grey	Light grey	13.3 x 5.5	13.1 x 5.4
D	340g	330g	Light grey	Light grey	13.2 x 5.5	13.0 x 5.4
E	340g	330g	Dark off white	Dark off white	12.1 x 5.5	12.1 x 5.5
F	335g	330g	Dark grey	Dark grey	13.2 x 5.5	13.0 x 5.3
G	420g	405g	Light grey	Light grey	13.7 x 5.5	13.7 x 5.5
H	360g	355g	Dark grey	Dark grey	13.8 x 5.5	13.6 x 5.4
I	330g	325g	Dark grey	Dark grey	13.2 x 5.5	13.0 x 5.3
J	420g	405g	Dark off white	Dark off white	14 x 5.5	14 x 5.5

4.1 Investigation

No significant changes in color or weight were registered after the ageing cycles, table (4).

Table 4. Alternative mortars after accelerated ageing.

Code of mortar	Density before ageing	Density after ageing	Color before drying	Color after drying	Color After ageing	Water absorption (%)	Apparent porosity (%)
A	1.45	2.02	Off white	Off white	Light off white	22.9	33.2
B	1.40	1.77	Off white	Off white	Light off white	26.8	37.5
C	1.10	1.38	Light grey	Light grey	Light grey	47.0	51.7
D	1.11	1.28	Light grey	Light grey	Light grey	xxxxxx	xxxxxx
E	1.14	1.50	Dark off white	Dark off white	Light off white	40.4	46.1
F	1.15	1.38	Dark grey	Dark grey	Dark grey	40.9	47.0
G	1.24	1.69	Light grey	Light grey	Light grey	34.1	42.3
H	1.14	1.48	Dark grey	Dark grey	Dark grey	41.7	47.5
I	1.13	1.38	Dark grey	Dark grey	Dark grey	45.5	51.4
J	1.21	1.63	Grey	Grey	Light grey	37.9	45.9

Mortars were exposed to impregnation tests to value their abilities to resist the transportation of moisture through them, the results are shown in table (5), figure (4).

Table 5. Impregnation tests of the studied alternative mortars.

Time of impregnation	Sample Weight (g)								
	A	B	C	E	F	G	H	I	J
0	497.3	452.5	350.7	344.8	347.2	429.8	378.1	347	424.5
1sec.	507.6	465.2	367.9	363.3	359	442.6	400	357	441

1min.	552.4	512.1	480.7	454.8	404.9	520.1	480	383	545
5min.	572	565.3	507.5	476.8	456.8	568.8	527.3	439	574.4
15min.	601	566.3	507.8	477.6	481.2	569.5	527.6	483	575.1
30min.	607.2	567.5	509.4	478.5	483	569.7	528.6	496	575.9
1h.	607.3	567.5	509.8	478.6	483	570	528.8	497	576.3
2h.	608.1	569.6	512.5	479.5	484.1	571.4	530.4	498	577.7
4h.	608.6	570	513.1	480	484.5	572.2	530.9	499	578.1
12h.	608.9	570.3	513.6	480.4	484.7	572.7	531.1	499	579.3
24h.	609.4	572.6	514.1	482	485.5	573.3	532.9	501	581.8
48h.	611.3	574.1	515.8	484.1	489.4	576.5	536.1	505	585.4
7days	611.3	574.2	515.8	484.2	489.4	576.5	536.3	505	585.4

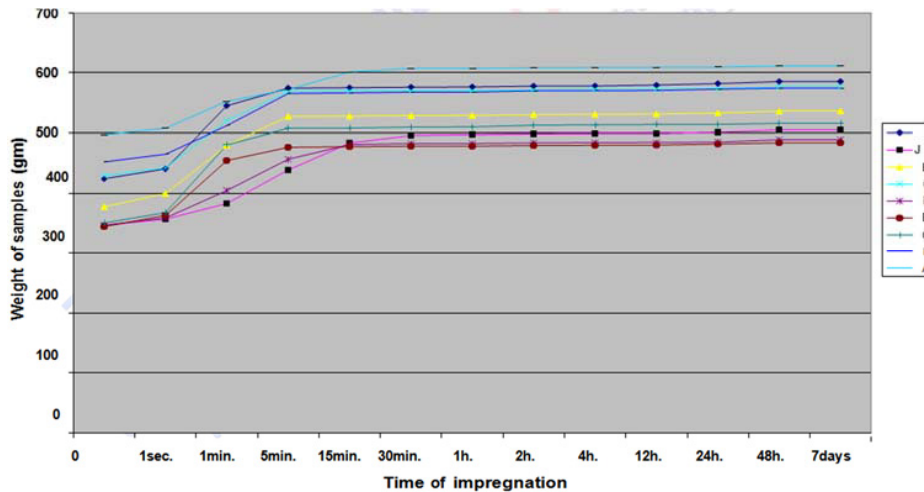


Fig. 4. Diagram shows the impregnation tests of the studied alternative mortars.

Compressive strength of the studied alternative mortars was examined before and after accelerated ageing cycles, the results are shown in table (6).

Table 6. Compressive strength tests of the studied alternative mortars.

Code of mortar	Compressive strength before ageing (kg/mm ²)	Compressive strength after ageing (kg/mm ²)
A	0.240	0.188
B	0.103	----
C	0.060	----
D	Xxxxx	Xxxxx
E	0.058	----
F	0.111	0.086
G	0.145	0.124
H	0.057	0.040
I	0.133	0.102
J	0.086	0.055

5. Results and discussion

It is important to address first that the duration of immersing the samples in water or salt solutions (sodium sulfate salt) was 24 hours, the duration of exposure to temperature was 24 hours at 150° C, and was repeated for 7 cycles, palm was added to mortars to improve their physical properties, while qusrmil is a mortar equals hydraulic lime mortars; this qusrmil was invented by the Muslim architects to compensate the lack of volcanic ash and pozzolanic additives in Islamic countries. Qusrmil composes of the ash results or by-produced from ovens, kilns, and firing waste matter, etc. which works as a cementing material in this mortar, it is mixed with sand and sometimes lime in proper proportion, the sulfate content should not exceed 0.5 %, this mortar was very common in historical Islamic architecture in Egypt, specially the brickwork masonry.

The compositions and methods of preparation of all mortars have been developed through extensive laboratory investigations to develop the particular combination of properties each bonding mortar should possess. Among the factors included are workability, plasticity, water retention, drying and firing shrinkages, chemical composition, and refractoriness, bonding strength, vetrification and resistance to chemical attack (BNZ Materials 2006).

Lime mortars usually consist of one part of lime to 2 to 3 parts of sand by volume; non-hydraulic lime usually contains a significantly higher content of lime than the same volume of hydrated lime. This should be taken into account if the lime content in the mortar is based on volume proportions. Lime/sand mortars are more appropriate for repair of older masonry with an existing lime mortar, and where exposure to climate loads is low (Maurenbrecher, 2004).

Based on the experimental work results; it has been concluded that mortar (A) is the most proper mortar for conservation works in Mekaad Radwan, it consists of (1 sand +2 palm + 1 calcium carbonate), it has shown a high resistance to mechanical tests before and after aging, followed by the mortar (G), which consists of (3 lime + 1 sand + 1 qusrmil +1 white cement).

6. Conclusion

The Petrographic investigation shows a general textural similarity between the ancient and the recent mortar and plaster samples. Both consist of lime lumps (fine calcite particles) with gypsum and anhydrite crystals and few fine quartz sand grains. Moreover, based on the XRD results, three types of mortars are recognized. The first type is lime mortar, composed of 68% calcite, 25% quartz and 7% gypsum. The second type is lime-gypsum mortar, which is frequently used as binding material for the masonry limestone blocks. This consists of 32% calcite, 24% gypsum and 35% quartz. The third type is gypsum mortar composed of 72 – 76% gypsum with 11 – 12% calcite and 5 – 6% quartz. This type is usually used as collating or binding the decoration and mosaics. The XRD analysis identifies the plaster samples as a lime-gypsum type; composed of 45 – 51% calcite (lime), 39 – 44% gypsum and 5 – 8% quartz. Based on the experimental work results; it has been concluded that mortar (A) is the most proper mortar for conservation works in Mekaad Radwan, it consists of (1 sand +2 palm + 1 calcium carbonate), it has shown a high resistance to mechanical tests before and after accelerated aging, followed by the mortar (G), which consists of (3 lime + 1 sand + 1 qusrmil +1 white cement).

7. References

- Candeias, A.E., Nogueira, P., Mirão, J., Silva, A.S., Veiga, R., Casal, M.G., Ribeiro, I., Seruya, A.I. (2004). Characterization of Ancient Mortars: Present Methodology and Future Perspectives, Workshop on Chemistry in the Conservation of Cultural Heritage: Present and Future Perspective, Chairmen of the European Research Councils' Chemistry Committees.
- JCPDS (1967). Joint committee on powder diffraction standards, index to the powder diffraction file, American society for testing and materials, Pennsylvania.
- Toumbakari, E.E. (2002). Lime-Pozzolan-Cement Grouts and their Structural Effects on Composite Masonry Walls, Ph.D., Thesis, Katholieke Universiteit Leuven, Faculteit Toegepaste wetenschappen, Departement Burgerlijke Bouwkunde, Laboratorium Reyntjens, Heverlee, Belgium.
- Palomo, A., Blanco-Varela, M.T., Martinez-Ramirez, S., Puertas, F., and Fortes, C. (2004). Historic Mortars: Characterization and Durability, New Tendencies for Research, Madrid, Spain.
- Böke, H., Akkurt, S. (2003). Ettringite Formation in Historic Bath Brick–Lime Plasters, *Cement and Concrete Research* (33), 1457-1464.

- Van Balen, K., Toumbakari, E.E., Blanco-Varela, M.T., Aguilera, J., Puertas, F., Palomo, A., Sabbioni, C., Riontino, C., Zappia, G. (1999). Environmental Deterioration of Ancient and Modern Hydraulic Mortars, European Commission Protection and Conservation of European Cultural Heritage (EDAMM) Research Report XX.
- Sabbioni, C., Zappia, G., Riontino, C., Blanco-Varela, M.T., Aguilera, J., Puertas, F., Balen, K.V., Toumbakari, E.E. (2001). Atmospheric Deterioration of Ancient and Modern Hydraulic Mortars, *Atmos. Environ* (35), 539-548.
- Sabbioni, C., Bonazza, A., Zappia, G. (2002). Damage on Hydraulic Mortars: the Venice Arsenal, *Journal of Cultural Heritage* (3), 83-88.
- Charola, A.E., Centeno, S.A. (2002). Analysis of Gypsum-Containing Lime Mortars: Possible Errors Due to the Use of Different Drying Conditions, *JAIC* (41), 269-278.
- Charola, A.E. (2003). Salt Deterioration: Open Questions, *Mauersalze und Architekturoberflächen* (Salts in Walls and Architectural Surfaces), (Leitner, H., Laue, S., Siedel, H., Eds), Hochschule fur Bildende Kunste, Dresden, 10-24.
- Moussa, A., Kantiranis, N., Voudouris, K., Stratis, J., Ali, M., and Christaras, B. (2009). Impact of Soluble Salts on the Deterioration of Pharaonic and Coptic Wall Paintings in Al Qurna, Egypt: Mineralogy and Chemistry, *Archaeometry* (51:2), 292-308.
- Moussa, A., Assessing the Decay Agents of Wall Paintings in Al Qurna and Wadi El Natrun Regions-Egypt, Ph.D., thesis, Aristotle University of Thessaloniki, Greece, 2007.
- BNZ Materials, Inc., Refractory Bonding Mortars, USA, 2006.
- Maurenbrecher, A.H.P. (2004). Mortars for Repair of Traditional Masonry, *ASCE Practice Periodical on Structural Design and Construction*, Ottawa, Canada, 62-65.