RESEARCH ARTICLE

INVESTIGATIONS OF HELLENISTIC MORTAR FROM UMM QAIS (GADARA), JORDAN

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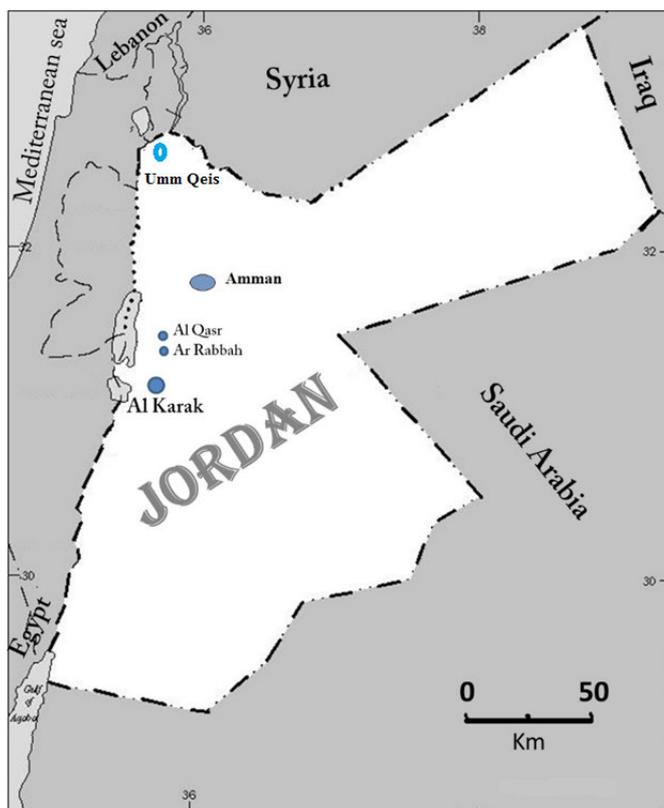
Figure 1. Location map of the study area.

ABSTRACT. *Ancient mortars have been widely studied, in connection with both diagnosis and applications required for restoration. This study is primarily based on analyses of different mortar samples from a Hellenistic temple. The study was done by means of a polarizing microscope equipped for observations in transmitted and reflected light; and X-ray powder diffraction (XRD). Scanning Electron Microscopy/Energy Dispersive X-Ray Spectroscopy (SEM-EDS) were also used to confirm and supplement the microscopic data, and wet chemical analyses were performed on the acid filtrate for soluble oxides of Fe, Al, Ca, Mg, S, Na and K. Chemical and petrographic analysis were used to determine chemical composition and physical properties, which in turn provide an in-depth understanding of the structural behavior and durability characteristics as they relate to the composition. The results show that the plaster used was a lime-based mortar. The cementing binder was a lime (identified by spot test) with fossilized shell, very fine grain size quartz, and some other minerals as the aggregate. The EDS analysis showed the presence of calcium and a small proportion of magnesium; in addition, silicon, aluminum, potassium, and iron were detected. Possibly, the silicate compounds contributed to the hydraulic component. We found strong similarities among mortar samples used in the temple. Physical methods provided useful information on the mineralogical compounds and the surface structures of samples, allowing for the postulation of deterioration mechanisms and overall decay, including the identification and crystalline morphology of reaction products and salts. These results aid in both understanding the technology of historic mortars and planning the restoration of these mortars.*

KEYWORDS. *Mortar analysis; SEM-EDS; Gadara; Lime; Restoration.*

INTRODUCTION

Degradation of building materials in monumental structures is a well-known phenomenon. In antiquity, people seemed to know, to distinguish, and to classify materials according to their durability and suitability. Morgan (1960) states, for instance, that some stones

can easily deteriorate under the effects of freeze/thaw cycles, rain, and salts while others are more resistant, the combined effect of stone inherent properties and environmental conditions. Pliny (2004) gives recipes for how to preserve some materials, such as wall paintings and metallic objects, using organic materials. These are just two examples showing the high value people attach to construction durability.

Lime is one of the oldest chemical industries known to humans; the process of burning lime has been known since the age of Hajji, where the term lime is a general term for all products of limestone. Lime mortar was used by the Greeks, Romans, Arabs, Marors, and Japanese. However, researchers agree that the spread of the lime mortar as a basic mortar began in Greek civilization (Zappia *et al.* 1994) and then moved to neighboring countries, including Egypt. Deterioration of carbonate building stones and mortar may be caused by acid rain, soluble salts, and to SO_2 deposition. The latter has been a major focus of conservation research over the past two decades; many publications devoted to the effect of SO_2 on calcareous stones can be found (e.g., De Santis 1995; Kumar 1998; Hayes and Bluck 1996). Although new energy policies have reduced atmospheric SO_2 to low levels, SO_2 remains a major corrosive agent (Del Monte *et al.* 1996).

Two processes are generally seen as driving SO_2 reaction with stone. The first is a wet process, consisting of dissolution of the carbonate in acid rain and precipitation of the sulfate in the form of gypsum. The second is a dry process consisting of deposition of SO_2 directly on to the stone surface. Although consistent (to some extent) with aqueous chemistry, the wet process cannot satisfactorily explain the formation of the gypsum crust. First, dissolution implies mass transport (of cations), as long as the solution is not getting saturated with respect to MSO_4 (where M is an alkali earth ion). Second, dissolution and precipitation do not occur simultaneously, since this is a dynamic process and the solid product (gypsum) is more soluble in acid rain than the carbonate itself (Viles 1990). Third, given the low concentration of SO_2 in air, it is unlikely that saturation with respect to gypsum could be attained. Fourth, it is well known that gypsum forms in the sheltered parts of the stone rather than in the parts exposed to the rain. Thus, it is believed that the wet process mainly contributes to weathering of the stone and mortar, which is still a devastating effect. In our case study, the studied temple is subject to numerous pressures, both man-made and natural; some can be pre-
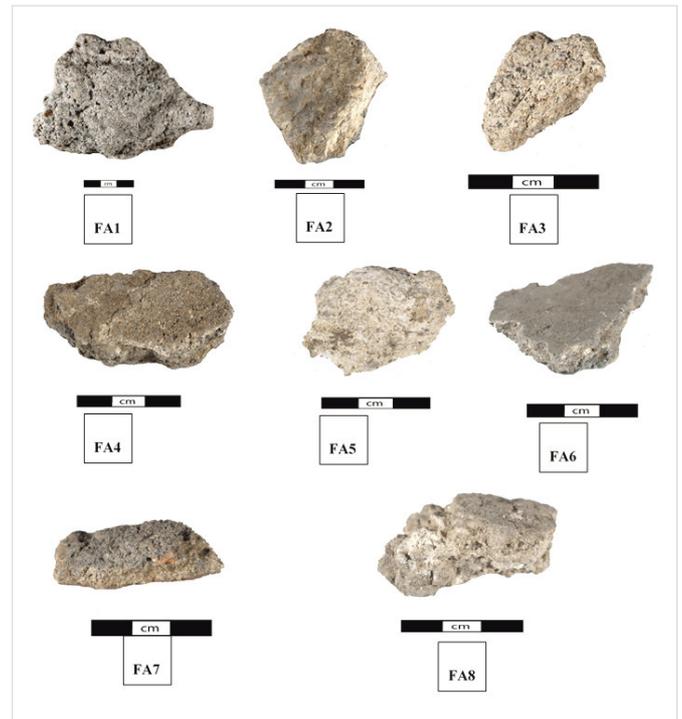
Figure 2. Studied mortar samples.

vented, while others are unavoidable. Natural disasters, such as earthquakes cannot be prevented. The potentially catastrophic effect of natural disasters on cultural property can only be forecast through risk management exercises, and steps taken to minimize or at least manage any damaging effects (Rosa *et al.* 2008). Nonetheless, virtually all material cultural heritage must be considered vulnerable to severe natural disaster and to phenomena associated with climate change.

The presence of moisture and the humidity level in different regions are important factors contributing to the deterioration of building materials. In fact, without the presence of moisture or high humidity level, there is no chemical reaction between the components of construction and soluble salt. Without sufficient moisture, soluble salts cannot be ionized, transported through materials and recrystallize, nor can the soluble compounds of construction materials be taken into dissolution. Also, moisture is an essential factor in several deleterious mechanisms that ultimately cause deterioration through the generation of internal stress (Amoroso and Fassina 1983).

Decreasing temperature or increasing water vapor in the air or in the environment surrounding of the archaeological building will increase moisture in the building materials. The advection process is the primary mechanism by which moisture is absorbed through capillary pores and distributed throughout a material.

Figure 3. Location of mortar samples.

This process can be accelerated through cracks at the surface or through moisture rising from the ground or foundation (Seinfeld 1985). The wicking process is a secondary mechanism, which facilitates the absorption of moisture (rain, steam, drops of dew) from the exposed outer surfaces into and throughout the wall or building. It should be noted that at this site, the drying process starts from the inner matrix to the outer surface (inside out). This is due to ongoing cycles of the mass transport of ions from within the material to the evaporation front at the outer surface. This movement of soluble salts becomes saturated near the outer surface and precipitates or crystallizes, which plugs the internal capillary pores, thereby creating attenuation and internal stress. In addition, concentrations of soluble salt may be extremely caustic or acidic in nature and can deteriorate the outer surface of the material (Genkinger *et al.* 2007).

In this study, we analyzed different mortar samples using a polarizing microscope equipped for observations in transmitted and reflected light. XRD and SEM-EDS were used to confirm and supplement the microscopic data. Wet chemical analyses were performed on the acid filtrate for soluble oxides of Fe, Al, Ca, Mg, S, Na, and K.

Finally, chemical and petrographic analysis were used to determine chemical composition. These physical properties provide a very good understanding of the mechanical behavior and durability and relate these to material composition.

HISTORICAL BACKGROUND AND SAMPLING STRATEGY

Gadara, a Decapolis city, is situated in Northwest Jordan, 110 km north of Amman and ca. 30 km north of Irbid (Figure 1). Today, the site of Gadara is divided into three main areas: the archaeological site (Gadara), the traditional village (Umm Qais), and the modern town of Umm Qais. The great ruins, the environments and the geographical setting of the site, overlooking Wadi Al-Arab, the Jordan Valley, the sea of *Tiberias* and the Golan highlands, make the site one of the most historical and valuable tourist attractions in Jordan. The oldest archaeological evidence at the site, some pottery
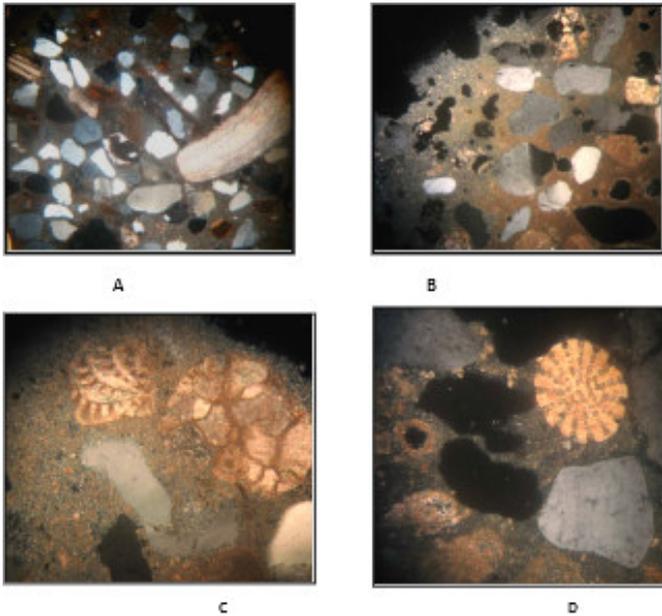
Figure 4. Lime mortar samples FA 1, 4, 6 and 8 show well-sorted quartz and shell fragments (A); quartz with micrite (B); shell fragments, interclast, quartz with micrite (C); and foraminifera, quartz and calcite with micrite (D).

The focus of this research is the newly discovered Hellenistic Temple; this was built of limestone and basalt in the Ionic style, belonging to the Di-style temple type (Shiyab 2017). Eight mortar samples were collected from temple walls (Figure 2). The samples were taken within each wall to represent the different elevations.

An important aspect of sampling strategy, significant for determining the chemical composition of mortar, is to have a fresh sample to prevent contamination by mineral accumulation and alteration. In this study, the samples were taken from the open face of the exterior masonry where a facing stone was missing (Figure 3).

sherds recovered from the southern side of the Acropolis, extends back to the second half of the third century BC.
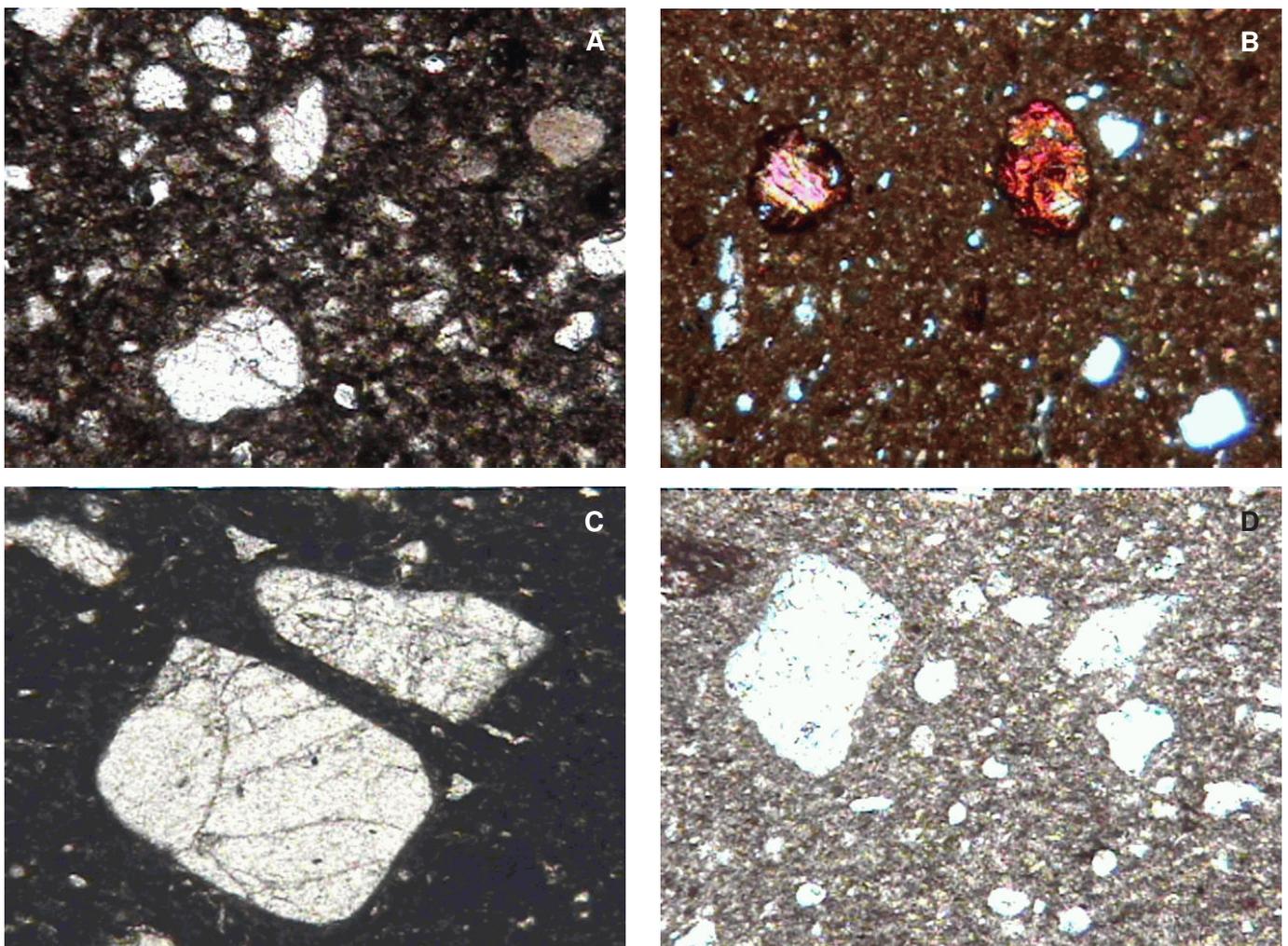
Figure 5. Lime mortar samples FA 2, 3, 5 and 7 (A-D); which contain fine to medium aggregates of quartz, plagioclase, and calcite. The fine-grained to medium-grained aggregates of quartz and plagioclase are embedded in the carbonate matrix.
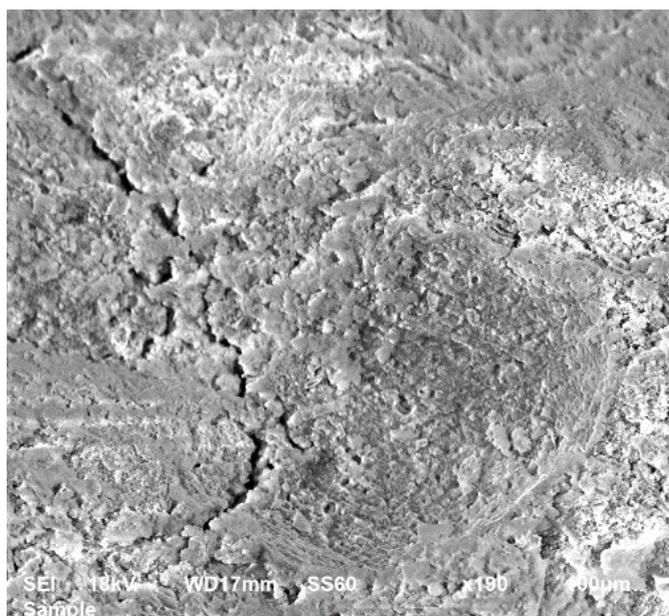
Figure 6. SEM image showing cracks present
on the surface of the mortar.

Mortar was removed by hammer and chisel in pieces averaging several centimeters in diameter.

ANALYTICAL METHODS

Analysis was performed using a polarizing microscope equipped for observations in transmitted and reflected light; XRD and SEM-EDS were used to confirm and supplement the microscopic data. Wet chemical analyses were performed on the acid filtrate for soluble oxides of Fe, Al, Ca, Mg, S, Na, and K. Chemical and petrographic analysis were used in the determinations of chemical composition. Micro-chemical analysis used water-soluble salt to identify sulfate, chloride, carbonate, nitrite and nitrate as well as calcium alumina silicate.

The chemicals and reagents used for the water-soluble salt were:

– Hydrochloric acid (10%) and barium chloride (10% solution, 5 g in 50 ml deionised water), to measure sulphates; a white precipitate indicated the presence of a sulphate.

– Nitric acid (10%) and solution of silver nitrate (1%, 0.5 g in 50 ml deionised water), to measure chloride. One drop of nitric acid was followed with one of silver nitrate. A white precipitate indicated the presence of a chloride.

– Acetic acid and Griess-Ilosvay's reagent in equal amounts with a pinch of zinc powder added, to measure nitrates. The solution turns pink when nitrates are present. The limit of detection is about 5 ppm. However, in this research, we used a nitrates/nitrites paper test (Quantofix for semi quantitative determination of nitrate and nitrite (10–500 mg/l NO_3^-, 1–80 mg/l NO_2^-). For this test, we dipped a test stick briefly in the test solution (pH 1–9) and, after 60 seconds, compared the test paper with a colour scale. When nitrates are present, the outer test paper turns red. The second reaction zone on the strip shows the nitrite concentration.

Results and Discussion

Preliminary examination, conducted both by the naked eye and with a microscope (10X), indicated that mortar samples varied in their rate of deterioration as well as in the nature (mechanism) of deterioration. It seems likely that the weathering crusts found on sample FA 5 and FA 8 are an extreme form and cover the entire sample. The surfaces of these objects are covered by a thick blackened enamel-like weathering surface, which in some areas has flaked away to reveal an iridescent layer beneath (a variegated coloration of the surface). The corroded surfaces had become very soft, powdery, and easy to destroy. Some of the invaluable mortar fragments with deteriorated crusts have flaked away or separated from the mortar surface.

PETROGRAPHIC ANALYSIS

Optical microscopic observations (FA 1–8, as shown in Figure 4) indicate all the mortars are comprised mainly of calcite with some accessory minerals such as quartz and plagioclase. Examination of the mortar samples, showed that the mixture of the mortars was mainly quartz sand with lime as a binder and brick, fine grain size ironstone, quartzite, shell fragments in different kinds, shapes and colors, and charcoal. In addition, organic material (straw) was found in the mixture of some samples. The mortars were very hard and dry and micro cracks were visible on the p surface with rough structure. The thicknesses of the mortar were between 5–10 cm with some up to 15 cm. Not all the fragments were the same thickness, because they came from different locations within the building.

Thin sections of the samples that were part of the same chemical group show similarity among the minerals included (Figure 5). Quartz predominates. Char-
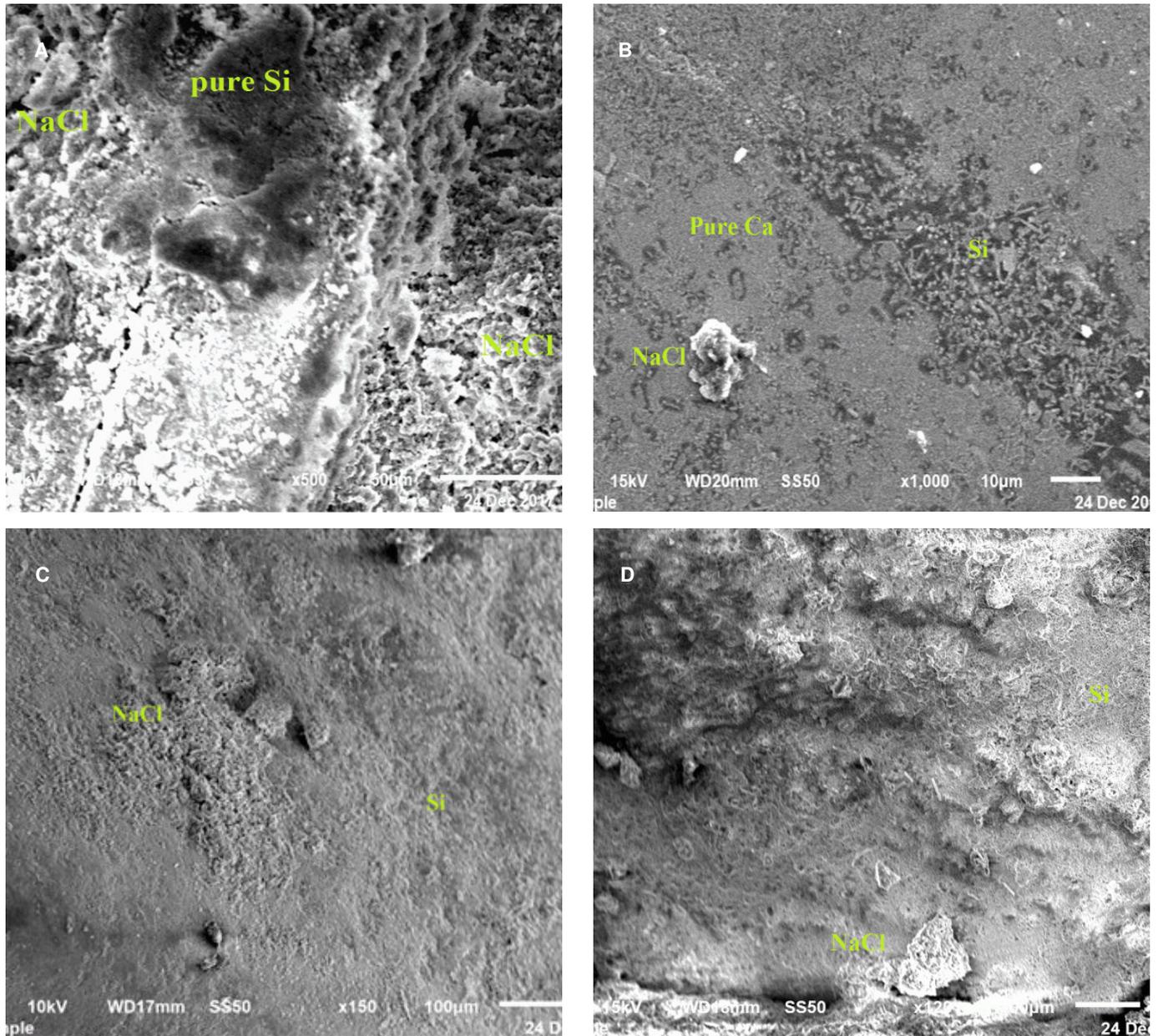
Figure 7. Surface structure of mortar samples FA 1, 4, 6 and 8. A shows the cracks present on the surface; B shows analysis area 1 (yellow arrow) with Ca, Si, S, Al, Mg and Fe present; C shows area 2, containing Ca, S, Si, A, Mg, Na and Fe; and D shows analysis area 3, containing elements Ca, Si, S, Al, Mg, Fe and Na.

coal, brick fragments, and lumps of lime putty or other binder materials, as well as pozzolanic additives, are easily distinguished. The examination of the polished sections suggests that some samples have the same mixture as well as the same stratigraphy. In particular, crushed brick lime mortars consist of fine to medium aggregates of quartz, plagioclase, and calcite; the fine-grained to medium-grained aggregates of quartz and plagioclase are embedded in the carbonate matrix. The mortar mainly consisted of quartz sand, quartzite, lumps of lime/chalk and brick in sample FA 7. The mortar can thus be classified into two groups. The first

mortar group contains lime with very fine grain sizes, but the mixture is predominantly quartz sand. The composition of the second group consists of quartz sand, quartzite and shell fragments as well as brick in addition to ironstone. Charcoal and some shell fragments were visible as well.

SEM and EDS Results

For detailed characterization of surface structure, SEM-EDS analysis was carried out on different fresh samples as well on polished cross-sections. We examined sur-

Figure 8. SEM image showing the presence of the white layer on mortar surface.

face structures and identified the elements present. All samples and sections were coated with carbon before entering the SEM. Different samples show the same surface characteristics and structures, which gives insight into the deterioration mechanisms and decay that has affected the mortar, as well as allowing the identification of various known salt crystallization formations. Cracks seen due to mechanical stress were visible on some samples, but there were also cracks on the surface (as seen in Figure 6). The surface cracks may be caused by expansion related to certain physico-chemical reactions. Microanalysis showed variation in the microstructure of the surface deterioration in all samples (Figure 7).

SEM analysis identified the following elements: calcium (Ca), silicon (Si) and sulphur (S), aluminium (Al), magnesium (Mg), and minor amounts of iron (Fe). Chloride (Cl) and sodium (Na) were found in most samples as well. The highest amount of Na and Cl was found in samples FA 5, 6 and 8. These samples in particular show a complex compound structure, as well as a more severely deteriorated surface.

The presence of calcium (Ca) and sulphur (S) on some samples reflects the transformation of calcium carbonate (mortar or limestone) into a calcium sulphate, a process which results in deterioration and decay. However, another important factor to consider is that the surfaces of the mortar were uneven.
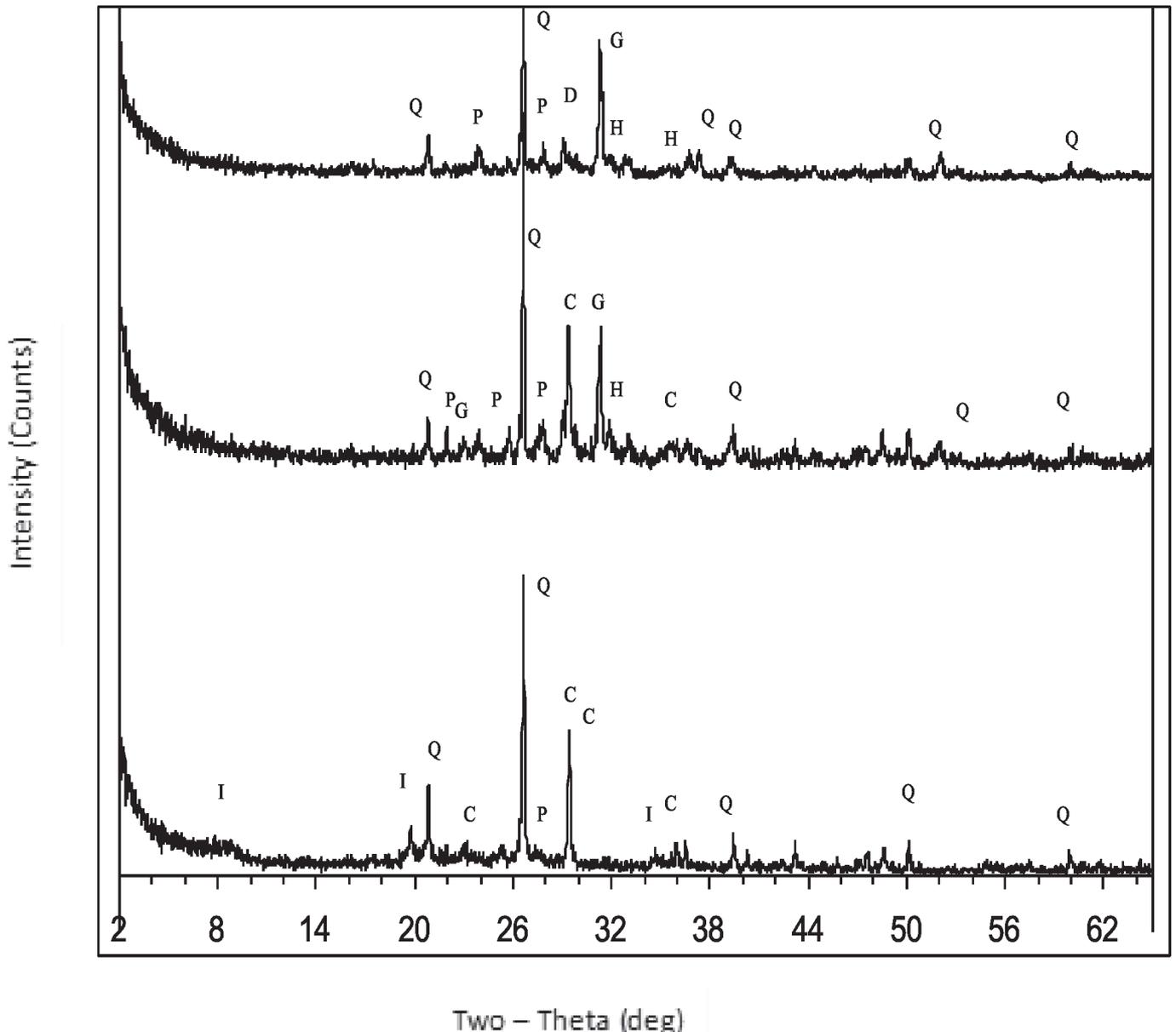
Figure 9. Representative XRD diagrams of mortar samples. The indicated mineral phase are illite (I), quartz (Q), plagioclase (P), K-feldspar (F), calcite (C), sulfur (G) and hematite (H).

Carbonated/precipitated layers partially covered the surface in some instances; therefore, certain elements were found in one area of the surface, but not in another area of the same specimen. Several analyses were carried out on each sample to obtain a general idea on the elements present (Haynes *et al.* 2010).

It was interesting to observe in sample FA 6 particles of K-feldspar, gypsum and Na-feldspar; in addition some samples contained Ti, which is characteristic of clay. The primary elemental constituents present were calcium (Ca), silicon (Si), aluminium (Al), magnesium (Mg), sodium (Na) and chloride (Cl), with minor elemental constituents of sulphur (S) and iron (Fe). The presence of salts in all samples may be related to the atmospheric conditions and pollution or as a result of the decomposition of calcium carbonate, sodium sulphate, potassium sulphates, calcium and magnesium. The lime mortars of are made of dolomitic lime. This compound may react with sulphate ions in the water. SEM also helped to identify the cause of surface deterioration, such as micro cracking and the presence of various crystalline reaction products (Figure 8).

XRD Analyses

To assess the validity of mortar classification and to obtain a better understanding of mortar chemistry, samples were characterized by XRD. Observation of the

Table 1. Results of the calcium alumina silicate test.

Samples No.	Notably Present	Present	Absent
FA 1			○
FA 2		●●	
FA 3			○
FA 4	●●●		
FA 5			
FA 6		●●	
FA 7		●●	
FA 8		●●	

Table 2. Results of the sulfate and chloride tests.

Samples No.	Type of Sample	Sulphates (SO$_4$)	Chlorides (Cl)
FA 1	Lime Mortar	-	+ wp
FA 2		-	++ wp
FA 3		-	++ wp
FA 4		+	++ wp
FA 5		-	++ wp
FA 6		-	++ wp
FA 7		+	++ wp
FA 8		+	++ wp

++ = Notably Present; WP = White Precipitates; − = Absent; +− = Traces; + = Present

diffraction patterns for samples reveals important similarities (Figure 9). The XRD patterns indicate calcite as the main component of the matrix, with the presence of quartz, plagioclase, feldspars, and muscovite as accessory minerals. The XRD analysis of soluble silicates contained in the binder from a significant number of samples reveals hydraulic cement compounds such as calcium silicate hydrate (C–S–H). The presence of C–S–H in mortars can be attributed to the reaction of lime with SiO$_2$ and Al$_2$O$_3$ in the presence of water; SiO$_2$ and Al$_2$O$_3$ may be contained in the natural pozzolan, in the finely ground bricks and tiles, as well as in the lime used. In a large number of mortars, pozzolanic initiators such as pyroxenes and feldspathoids were found, suggesting the purposeful inclusion of pozzolanic admixtures (i.e., Robert *et al.* 2008).

Micro-Chemical Analysis

Micro-chemical tests were necessary to distinguish the materials before carrying out full analyses. All samples were dissolved in acid (3% hydrochloric acid, HCl) to ascertain the presence of lime mortar. The solution in some samples was yellowish in colour indicating the

Table 3. Results of the nitrate and nitrite tests.

Samples No.	Type of Sample	Nitrates (NO₃) mg	Nitrites (NO₂) mg
FA 1		25	0.5
FA 2		**100**	1
FA 3		25	-
FA 4	Lime Mortar	50	-
FA 5		20	0.5
FA 6		25	1
FA 7		25	0.2.5
FA 8		50	5

Table 4. Results of the water soluble salt tests.

Sample No.	Type of Sample	Original Weight/g	Dry weight after analysis NaCl, CaSO₄, NO₂ & NO₃	% Residue	% Dissolved
FA 1		2	1.974	98.7	1.3
FA 2		1.948	1.889	**96.97**	**3.03**
FA 3	Lime Mortar		1.951	**97.55**	**2.45**
FA 4			1.943	**97.15**	**2.85**
FA 5			1.941	**97.05**	**2.95**
FA 6		2	1.885	**94.25**	**5.75**
FA 7			1.917	**95.85**	**4.15**
FA 8			1.967	98.35	1.65

presence of iron; this was confirmed by a spot test using potassium ferrocyanide (2%). Sample residue showed very white fine grain sizes with brick in some samples. One of the mortars did not dissolve completely in acid, suggesting that it may contain gypsum (Nehdi *et al.* 2005). For the identification of carbonates, micro-chemical tests were carried out using hydrochloric acid (HCl). However, another possibility for the presence of gypsum is ionic conversions with the underground water, which may transform calcium carbonate into calcium sulphate under certain condition.

We also conducted a spot test for calcium alumina silicates. The presence of aluminum in hydraulic mortars and cements can be verified by adding a diluted alkali to the dissolving samples solution. Any dissolved aluminum will precipitate as gelatinous white material (Morgan 2005:52). The results of this test can be seen in Table 1. The presence of aluminum silicate may indicate the use of hydraulic lime. The sand/aggregate used with the mixture may have contained aluminum silicate as well.

Soluble salt analysis is an essential aspect of diagnosing ancient materials, particularly wall painting and mortar. Such materials usually contain salts from different sources (Teutonico 1988). It is possible to obtain key information on the presence of salts using

micro-chemical tests, which can then be confirmed by physical methods. In addition, the alkaline or acidic nature of a soluble salt is one of the most important mechanisms responsible for weathering decay. Soluble salt damage has been known since antiquity and is still an unsolved conservation problem. It affects not only the building materials, but also causes serious deterioration problem to the rendering and plaster layers (Rosa *et al.* 2007). The analysis was carried out to identify the most important of salt types, these are sulfates, chlorides nitrites and nitrates.

Analysis of Soluble Salts

Soluble salt analyses were done according to Teutonico (1988). Samples were dried at 85 °C and weighed before and after analyzing the carbonates to calculate the percentage of the residue as well as the dissolving amount. Water was added to the samples to analyze for carbonates, using 10% of hydrochloric acid, then samples were washed until pH 7 and dried at the same temperature. Soluble salts results can be seen in Tables 2, 3 and 4.

The most significant salts found were chlorides and nitrates. It is interesting to note that the percentages of dissolved solute varied from 0.9% to 7%; the most significant was FA 3, at 30% whereas FA 1, 2 and 4 remained below 3% and samples FA 5, FA 6 and FA 7 remained below 2%. The presence of chloride reflects the ongoing weathering process, likely rain water or groundwater as it was noted that the walls were moist. Table 3 shows the results of the nitrate and nitrite analysis. Sample FA 2 has the highest amount of nitrates, at 100 mg. Samples FA 3 and 8 contained 50 mg. The lowest amount of nitrates were found in samples FA 1, 4, 5, 6 and 7, which contained 20–25 mg.

Samples FA 4, 7 and 8 showed the presence of both sulfate and chloride. These samples were taken from a location close to the floor, where additional salts it would be expected. In areas of groundwater or moisture impact, three zones of deterioration (zone A, zone B, and zone C) were identified. There is less deterioration in the upper zone A. The mortar and stones of zone B showed significantly more disintegration, crumbling, and scaling. The salts in zone B are mainly sulfates (sodium sulfates, magnesium sulfates, calcium sulfate and potassium nitrate and sodium carbonate). Less deterioration was found in lower zone C, although it is situated in a lower temperature and elevated humidity zone where chlorides and nitrates are concen-

trated. This zone also accumulates salts under normal climatic conditions, which results in advection of moisture due to the strongly hygroscopic nature of salts. However, humidity is one of the important factors for initiating chemical and physical deterioration, as well as biological decay (Kunlin *et al.* 2007). Elements of the materials used in a previous conservation process may have also supplied salts. In addition, cement injected with alkali, silicates and silicones, etc., was used as a consolidant. The reaction between these different materials will lead to increased deterioration and decay. Another possibility is that the materials used in the preparation of mortars and plasters may have contained a small quantity of sulphate impurity. It is known, for example, that minerals can react with ions in water, leading to the presence of sulphate (Tsui *et al.* 2003). It is possible that some of these samples contained trace amounts of sulphate because some of the sand was washed before being used in the mixture.

CONCLUSION

SEM-EDS and XRD are critical analytical tools for the identification of material constituents and reaction products. This study found that the studied mortars were composed of a mixture of lime (identified by spot test) containing fossil shell with very fine grain size quartz and some other minerals. It is possible to conclude that the main binder of the mortars is a calcitic lime manufactured from seashells.

Sulfation is one of the main deterioration process which damages lime mortar in urban environments. The wet and dry deposition of atmospheric sulphur compounds and their subsequent reactions with calcium carbonates are the main cause of the formation of gypsum on the external surface. Soluble salts are another contributor of serious deterioration of mortar. The deterioration and decay caused by weathering (temperature, humidity, water soluble salt, etc.) is varied, and can have an exacerbating effect on other deterioration factors. However, humidity is the main condition for all the chemical and biological decay, as well as physical damage.

Mortar damage is likely related to chemical deterioration from the mass transport of ions towards an evaporation front, creating supersaturated concentrations (caustic or acidic) via advection; or, a physical (expansion and contraction) deterioration such as freeze/thaw or wet/dry; or, a combination of these (physico-chemi-

cal) mechanisms. The investigations of the microstructure and the application of the mortars in this research may help to understand the properties of ancient mortar formulas elsewhere, as well as contribute to future conservation and restoration efforts.

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