LIME MORTAR AND PLASTER FROM THE HOUSE XVII-XVIII, UMM EL-JIMAL, JORDAN: ARCHAEOMETRIC ANALYSIS

Khaled AL-BASHAIREH
Department of Archaeology, Yarmouk University, Irbid, Jordan
Email: khaledsm@email.arizona.edu

ABSTRACT

This research studies cement materials of mortar and plaster mostly of Byzantine age from the House XVII-XVIII located in the southeast of Umm el-Jimal archaeological site (east Jordan). A set of physical, chemical, mineralogical and petrographic analyses were performed to characterize the materials aiming at defining their production technology adopted by the Umm el-Jimal’s ancient craftsmen. The results show that Umm el-Jimal building technique relied on the manufacture of lime-based cement materials of hydraulic properties acquired by the addition of natural scoria and recycled pottery fragments. The high amount of charcoal added to a group of three samples colored them grey; while the use of straws, bones and considerable amount of scoria and ceramic in the rest of samples did not affect their lime white-beige color. The grain size distribution of the two groups does not show substantial difference compared to the binder: aggregate ratios. The addition of fibers, scoria and ceramics most likely produced porous lightweight white cement material.

KEYWORDS

Production technology, Lime-Mortar, Scoria, Recycled Pottery, Umm el-Jimal, Jordan
INTRODUCTION

Ancient structures are major sources of archaeological and historical information about ancient architecture and building materials such as stones, bricks and cement materials. Archaeological documentation and analyses of building materials are essential to identify their types, reconstruct their production technology and conserve the structures. Cement materials of mortar and plaster were used for the construction of archaeological structures since ancient times. They have been used for different applications: foundations, stucco, binding and coating masonry blocks and filling the gaps between stones, etc; therefore, their production recipes varied according to their function and/or application.

Plasters and mortars are composed of a binder and aggregates of organic and inorganic origin. Lime and gypsum were widely used as binders, while hydraulic (pozzolana, crushed pottery and bricks, etc.) and non-hydraulic (sand, limestone, marble, etc.) aggregates were used as inorganic aggregates. Organic materials including charcoal, seeds, straws, egg whites were also added. The production of the lime plaster and mortar begins by burning calcareous raw materials, usually hard white limestone, at ca. 900°C to produce the quicklime (CaO) when carbon dioxide is driven off from the limestone. The addition of water to the quicklime forms slaked lime Ca(OH)$_2$, which is mixed with aggregates before its application to the structure under construction or restoration. In the last step, the slaked lime sets by absorbing the atmospheric CO$_2$ and reforming a calcium carbonate matrix that adheres the aggregate together. The different choices to perform the final product at these production steps lead up to different production technologies, which influence the physical and mechanical properties of these materials.

Archaeological scientists mainly study cement materials to determine their production technologies, uses and chronology while conservators study them to prepare compatible materials for repair and restoration. The mortar and plaster, which were widely used in different archaeological sites in Jordan, have been scientifically studied and characterized. For instance, Al-Qaisiyah (2002) and Rezkallah (2006) examined mortars and mortar-like materials from different structures at Khirbet Edh-Dharih; while, the mortars studied by Shaer (1997), Al-Aseer (2000) and Bonazza (2013) were from some Petra complexes.

1 White, Greek and Roman Technology, 1984
2 Davey, History of building materials, 1961
3 Stefanidou and Papayianni, The Role of Aggregates on the Structure, 2005
4 Wright, Ancient building technology, 2005.
5 Al-Bashaireh and Hodgins, Lime mortar and plaster: a radiocarbon dating, 2012
6 Adam, Roman Building, 1992.
7 Folk and Valastro, Successful technique for dating of lime mortar, 1976.
8 Lindroos et al., Mortar dating using AMS $^{14}$C, 2007.
9 Heinemeier et al., Successful AMS $^{14}$C dating, 2010.
10 Al-Bashaireh, Chronology and technological production styles, 2008.
11 Al-Bashaireh, Chronology and technological production styles, 2008.
12 Murakami, Characterization of lime carbonates, 2013.
13 Casadio et al., Evaluation of binder/aggregate, 2005.
14 Al-Bashaireh, Dating of Nabatean and Islamic Structures, 2013.
15 Al-Bashaireh, age determinations of mosaic mortar layers, 2015.
16 Al-Qaisiyah, Conservation and Restoration of Nabatean Temple, 2002.
17 Rezkallah, Study of Mortars and Mortar-like Materials from Kirbet Edh-Dharih, 2006.
18 Shaer, The Nabatean Mortars in Petra Area, 1997.
19 Al-Aseer, Chemical analysis of the Nabatean Water Dam Mortar at Petra, 2000.
20 Bonazza, Characterization of hydraulic mortars from archaeological complexes in Petra, 2013.
structures. Yaseen et al. (2013)\textsuperscript{21} characterized Roman mortars from Jarash by using different archaeometric techniques. In addition, Al-Bashaireh and Hodgins (2011, 2012)\textsuperscript{25} characterized plaster and mortar samples from Petra and Udruh and radiocarbon dated their lime and/or organic inclusions. Al-Shreideh et al. (2018)\textsuperscript{23} presented a technological study of the production technology and provenance of raw materials used in the production of mortar and plaster samples from two of Gadara structures.

At Umm el-Jimal (figure 1; see De Vries (1994,1998)\textsuperscript{24}), Dunn and Rapp (2004)\textsuperscript{25} presented a fairly detailed characterization of mortar samples from Umm el-Jimal based on a variety of analytical techniques such as X-ray diffraction and microscopic petrography for conservation purposes. The samples were collected during the early excavation seasons (1981, 1984 and 1998) from different structures: the so-called Nabatean Temple, Praetorium, Northwest Church, Village, and others. Al-Bashaireh (2014, 2016, 2017)\textsuperscript{26}\textsuperscript{14}C dated organic materials from the mortar and plaster samples from House XVII-XVIII, the West Church and the Cathedral. Al-Bashaireh (2017)\textsuperscript{27}’s study not only radiocarbon dated the structure, but also characterized the mortar by archaeological techniques, and determined the source of the scoria used in the fallen dome of the West Church.

\begin{figure}
\centering
\includegraphics[width=\textwidth]{location_map.jpg}
\caption{Location map of Umm el-Jimal, Jordan.}
\end{figure}

This study presents the results of an archaeometric characterization of mortar and plaster samples from House XVII-XVIII collected during the excavation and documenting activities of the house. It aims to define the type, recipe and production technology of this

\begin{itemize}
\item \textsuperscript{21} Yaseen et al. Petrography and mineralogy of Roman mortars, 2013.
\item \textsuperscript{22} Al-Bashaireh and Hodgins, AMS 14C dating of organic inclusions of plaster and mortar, 2011; Al-Bashaireh and Hodgins, Lime mortar and plaster: a radiocarbon dating tool, 2012.
\item \textsuperscript{23} Al-Shereideh et al., Characterization of Architectural Mortars from Buildings at Umm Qais.
\item \textsuperscript{24} De Vries, What's in a name, 1994; De Vries, Umm el-Jimal: A Frontier Town, 1998.
\item \textsuperscript{25} Dunn and Rapp, Characterization of mortars and pozzolanic materials, 2004.
\item \textsuperscript{26} Al-Bashaireh, Reconstructing the Chronology of the House XVII–XVIII, 2014; Al-Bashaireh, Use of Lightweight Lime-Mortar, 2106; Al-Bashaireh, Umm el-Jimal Cathedral, Jordan, 2017.
\item \textsuperscript{27} Al-Bashaireh, Umm el-Jimal Cathedral, Jordan, 2017.
\end{itemize}
A group of samples with the purpose of producing compatible materials for the preservation of the house.

**MATERIALS**

**Ten samples** (1-10) were collected from the wall courses and niches of different rooms and yards of the first and second floor, and one sample (sample 11) was collected from the entrance of the reservoir; see figure 2 and table 1 for the location and description of the samples. The samples were intact and undisturbed; therefore, they most probably represent the initial plastering or the last plastering stage of the house before its destruction. The samples were recently dated to the Byzantine period by radiocarbon dating some of their organic inclusions of annual flora and charcoals\(^\text{28}\).

![Figure 2: The plan of House XVII-XVIII illustrating the samples’ locations (after Al-Bashaireh 2014\(^\text{29}\)).](image)

**METHODS**

**The samples** were collected using a chisel and a hammer after the removal of their outermost material in order to eliminate possible stuck contaminants. After the collection and documentation of the samples, they were macroscopically described and then analyzed by several scientific techniques including X-ray diffraction (XRD), petrographic (polarized light microscopy PLM) and scanning electron microscopy (SEM). Sieves were used to determine the grain size distribution of samples' aggregates.

XRD analysis of powders was used to determine the mineralogical composition of the samples. All the samples were analyzed using a Shimadzu Lab X, XRD 6000 X-ray diffractometer. Powder diffraction patterns were obtained by applying the following conditions: CuK\(\alpha\) radiation (1.5418 Å) with 30 kV, 30 mA energy and a graphite monochromator.

Petrographic analyses identified the samples' content of aggregates and their type, size and distribution. A small block of each sample was hardened by impregnating it under vacuum with a low-viscosity resin\(^\text{30,31}\). Afterwards, a polished side of the block was mounted on to

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\(^\text{28}\) Al-Bashaireh, Reconstructing the Chronology of the House XVII–XVIII, 2014.
\(^\text{29}\) Al-Bashaireh, Reconstructing the Chronology of the House XVII–XVIII, 2014.
\(^\text{30}\) Al-Bashaireh, Chronology and technological production styles, 2008.
\(^\text{31}\) Elsen, Microscopy of historic mortars, 2006.
a microscope slide, then its thickness was reduced by polishing and sawing to about 30µm. The percentages of aggregates and additives were visually estimated on thin sections by comparison with standard charts used by sedimentologists. Thin-section examinations were carried out using a Leitz 7062 model polarizing microscope with an attached digital camera. Thin sections and petrographic analyses were made at the workshop of the Faculty of Archaeology and Anthropology at Yarmouk University.

SEM was used to show the binders' growth and composition and their interaction with the aggregates and organic inclusions. SEM analysis were carried out using an FEI Quanta 200, Netherlands Scanning Electron Microscope equipped with EDS (Energy Dispersive X-ray Microanalyzer) at the SEM laboratory, Department of Earth and Environmental Sciences, Faculty of Science, Yarmouk University, Jordan. Fresh fractures of small pieces of bulk samples were coated with a thin layer of carbon and analyzed under the following conditions: run at an accelerating voltage between 0.3 and 30 kV, with the chamber’s pressure about 50 Pa in a variable-pressure mode.

Grain size analysis was performed by the disaggregation of the aggregates from the binder by the gentle crushing of a known weight of each sample. The material was dry sieved using 63, 125, 250, 500, 1000, 2000 and 4750µm sieves. The fine material that passes through the 63µm sieve is more enriched with binder, while the coarser material retained on the sieve is more enriched in aggregates. The binder : aggregate ratio was calculated and expressed in the form of 1: a/b.

The apparent porosity of the samples was determined by drying them with (around 70°C), evacuating the dried weights Wd for 24 hours in a vacuum chamber, then introducing and covering the samples with water in the vacuum chamber for 24 hours. The samples were weighed while suspended in water (Ww) and weighed in air (Wa). The apparent volume porosity (%) was calculated by applying the formula of Grimshaw (1971) and Rice (1987), Apparent porosity = (Wa-Wd/Wa-Ww)×100.

RESULTS AND DISCUSSION

The samples that have been preserved for hundreds of years are coherent, but not tough enough to resist crumbling when broken. Macroscopically, the samples can be divided into two groups according to their color; the white (1,2,3,4,5,7,9,10) and grey (6,8,11) groups (figure 3). The white samples are used to infill the spaces and gaps between the basalt masonry stones, while the grey samples are used as a bedding layer between the masonry stones, a coat and rendering for the walls and niches at the second floor and the cistern. The abundance of charcoal in the grey samples of the second group caused their grey color, while its small amount and the use of fine ground white straws of annual plants, and bones did not affect the lime’s original white to beige color of the samples of the first group. All samples of each group appear to be similar in texture; only the grain size and quantity of the aggregates allow a kind of differentiation. The texture of the samples is characterized by embedded aggregates composed of volcanic materials (basalt and scoria), recycled ceramics and mortar, and calcareous sand and gravel in a fine-grained carbonate matrix. Sand calcareous grains varied in shape from spherical to subspherical because of their weathering history. The subsphericity of the sand grains, most likely, indicate that they were not crushed before their addition to the mixture; while, the angularity and sub-angularity of the rest of aggregates indicate that they were crushed. Few scoria particles

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32 Grimshaw, The Chemistry and Physics of Clays, 1971.
33 Price, Pottery Analysis, 2015.
could be seen by the naked eye, but it is possible to observe smaller ones under the microscope (figure 3: sample 7, and see below figure 6A).

Figure 3: Samples 2 and 7 of white plaster show plant straws, brown scoria and large basalt filler, sample 11 of grey mortar shows lime lumps and charcoal. It is notable that grain angularity and grading yield a higher strength to the mortar or plaster than the grain roundness. Therefore, all the samples have moderate strength because they contain graded grains of sharp edges well interlocked with the binder and other grains of rounded shapes less packed with the binder (figure 3). In addition, the straws, bones (see petrographic analysis below) and charcoals increased the strength of the plasters and enhanced their adherence. SEM analyses confirmed that lime binder has moderate bonds with rounded calcitic grains (figure 4a), while it has stronger bond and more compact contacts with pozzolanic and angular aggregates (figure 4b).

Figure 4: SEM images showing left: weak contacts between rounded grains and the binder, middle: stronger contact with scoria, and right good adheres of lime binder to organic straws.

Similarly, large basalt grains and ceramic fragments were seen by the naked eye in some samples (figure 3), as well as under the microscope (figure 6d). It is likely that these large size fragments were inserted in the mortar to fill the large size joints between the basalt stones in order to reduce the amount of needed slaked lime. XRD analysis shows the presence of calcite and quartz and the absence of gypsum in all the samples. This result indicates clearly that the samples are lime-based and shows that Umm el-Jimal craftsmen during the Byzantine period produced mortars and plasters based on lime technology rather than gypsum-based mortar despite the fuel shortage. XRD patterns of samples 2 and 9 show feldspar and pyroxene groups, the patterns of samples 1, 5, 6, 7, 8 show very short peaks of the two groups, while the patterns of the rest of the samples do not show these peaks clearly (figure 5). It is clear that the appearance of feldspar and pyroxene minerals reflects the presence of basalt and/or scoria in the analyzed powders.

34 Lanas and Alvarez-Galindo, Masonry repair lime-based mortars, 2003.
Table 1 summarizes the samples’ contents, their color and percentages, and their binder/ aggregates ratios

| S. | Context                                                                 | Description                                                      | Binder % | Lst. | Q. | Bas. | Tuff | Cer. | Or. | Mor. | lump | A/B ratio | Sieves 1/b/a | Petrography B:A |
|----|------------------------------------------------------------------------|------------------------------------------------------------------|----------|------|----|------|------|------|------|------|-------|-------------|----------------|---------------|
| 1  | House 17, room 21, the south face of the north wall, 5th row, to the right of the window, directly beneath the roofing corbels; | White, brownish aggregates, straws                              | 29       | 38   | 1  | 5    | 5    | 10   | 3    | 4    | 5     | 1:3.31      | 1:3            |
| 2  | House 17, room 24, the back wall of the second manger of the western wall | White, brownish aggregates, some pebbles, straws                 | 28       | 32   | 1  | 8    | 5    | 10   | 12   | 2    | 2     | 1:3.17      | 1:3            |
| 3  | House 17, room 27, 6th row of stone of the north wall, on the left side, one stone beneath the corbels, two stones to the right of the west wall. | White color, brownish aggregates, straws                         | 38       | 20   | 1  | 8    | 4    | 15   | 4    | 2    | 8     | 1:2.81      | 1:2            |
| 4  | House 17, room 25, on the left side of the doorway leading into the room. | White, brownish aggregates, straws                              | 28       | 25   | 1  | 10   | 5    | 15   | 8    | 2    | 6     | 1:3.15      | 1:3            |
| 5  | House 17, the north face of the north wall of house 17, 6th row, 2 stones to the left doorway. | White, brownish aggregates, straws                              | 39       | 10   | 1  | 8    | 5    | 15   | 8    | 4    | 10    | 1:2.91      | 1:2            |
| 6  | House 18, room 8, 2nd floor, the inner layer of plaster of the niche of south wall. | Grey, some pebbles, charcoal                                     | 21       | 22   | 1  | 5    | 3    | 10   | 22   | 8    | 8     | 1:4.22      | 1:4            |
| 7  | House 18, room 8, 2nd floor, the outer layer of plaster of the niche of south wall. | White, brownish aggregates, some pebbles, straws                 | 28       | 15   | 1  | 15   | 5    | 10   | 6    | 5    | 5     | 1:2.90      | 1:2            |
| 8  | House 18, room 7, 2nd floor: the inner layer of plaster of south wall, southeast corner above the roof. | Grey, brownish aggregates, some pottery fragments, charcoal      | 22       | 15   | 1  | 8    | 5    | 15   | 20   | 8    | 6     | 1:4.14      | 1:4            |
| 9  | House 18, room 7, 2nd floor: the outer layer of plaster of south wall, southeast corner above the roof. | White, brownish aggregates, straws                              | 26       | 17   | 1  | 12   | 3    | 20   | 7    | 6    | 8     | 1:3.25      | 1:3            |
| 10 | House 17, yard of 1st floor, above the staircase, from the east part of north face of the north wall, 6th row above floor and two stones from the doorway. | White, brownish aggregates, straws                              | 27       | 22   | 1  | 15   | 5    | 20   | 2    | 3    | 5     | 1:2.97      | 1:3            |
| 11 | House 18-room 6, 1st floor: from west face of east wall, in the basement, covering the arches of the cistern. | Grey, some pebbles, charcoal                                     | 19       | 16   | 1  | 12   | 5    | 15   | 20   | 6    | 6     | 1:4.45      | 1:4            |
Petrographic analysis was used to better identify the components of the samples and their distribution. Analysis of the white samples revealed the presence of a fairly compact microcrystalline carbonate matrix enclosing mostly reddish ceramic fragments (figure 6 a, f). On contrast, the grey samples have a microcrystalline matrix enclosing different kinds of aggregates including black charcoals (figure 6 c, e). Additionally, small size particles are embedded in the binder around the aggregate grains.

The binder has a homogeneous texture of densely packed lime material with micro-pores. Some areas of the binder have lime lumps of more compacted material (6b,c). The lime lumps are present in different sizes and shapes, but mainly sub-rounded (figure 3: sample 11). Their presence offers pieces of information about the lime production technology, where they most likely indicate imperfect slaking of the quicklime in contact with the aggregate and insufficient mixing of the aggregates with the slaked lime\textsuperscript{35,36}.

\textsuperscript{35} Bruni et al., White lumps, 1997.
\textsuperscript{36} Elsen, Microscopy of historic mortars, 2006.
Petrographic analysis confirmed the use of different kinds of aggregates including rock fragments (limestone, basalt, scoria), isolated grains of river sand, reused ceramic and mortar, organic bones, plant fibers and charcoal (figure 6 a, b, c, d; and table 1). Micritic limestone vary in shape from rounded to angular, but mostly are sub-rounded (figure 6 a), while basalt grains are mostly angular (figure 6 e). Some of the basalt aggregates appear in dimensions that reach centimeter sizes (figure 6 f). The valley sand aggregates are rounded to subrounded (figure 6 a). Most scoria grains are small and rounded (figure 6 a). Ceramic and mortar particles are mostly elongated, but rounded particles also exist (figure 6 b). Charcoals, bones and fibers appear elongated (figure 6 c).

Polished sections and petrographic analysis showed the reaction rims at the boundaries between the slaked lime and the hydraulically active scoria and ceramic fragments, which are considered pozzolanic materials (figure 6). Umm el-Jimal craftsmen used the most common recipe at that time to induce hydraulic properties by adding artificial pozzolanic and crushed ceramics (cocciopesto) and natural scoria, an available and abundant pozzolanic material in Umm el-Jimal vicinity. It is known that these materials increase the mortar’s hydraulicity, when in contact with highly reactive lime, depending on their firing temperature, mineralogy and content during their amorphous phase and surface area. Ceramic fragments are the most reactive component to the slaked lime, which increases the cohesion of the aggregates and improves the mortar's mechanical properties and resistance to environmental factors. The use of volcanic material in lime mortar are widely documented.

The occurrence of organic charcoals (figure 6) is probably a remnant of the burning process of the fuel; however, it is argued that it can also be added in some cases as a pigment to darken the mortar and/or to contribute to the hydraulicity of the mortar. It is likely that the large content of charcoal in the grey samples support the second hypothesis.

On the other hand, the frequent occurrence of organic straws and bones in the white samples most likely indicate a common addition of these materials to enhance their durability and their resistance to deformation. They adhere very well to the lime binder to produce significant improvement to the mechanical and physical properties of the mortar. SEM analyses showed a good compaction between the straw fibers and the growing lime micro grains (figure 4 c) which, apparently improved the cracking resistance of samples. Another advantage of the addition of natural fibers is to decrease the unit weight of the plaster which brings their application more comfortable. However, it should be noticed that deteriorated fibers produce pores that cause mechanical weakness of the plaster.

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37 Al-Bashaireh, use of Lightweight Lime-Mortar, 2016.
38 Walker and Pavaia, Physical properties and reactivity of pozzolans, 2011.
39 Baronio et al., Microscopy study of Byzantine mortars, 1999.
40 Walker and Pavia, Physical properties and reactivity of pozzolans, 2011.
41 Tekin and Kurogöl, Physicochemical and pozzolanic properties of the bricks, 2011.
42 Moropoulou et al., technology of historic mortars, 2000.
43 Belfiore et al., Archaeometric Study of Pozzolanic Aggregate, 2015.
44 Fichera et al., Limestone Provenance, 2015.
45 Izzo et al., The art of building in the Roman period, 2016.
46 Elsen, Meroscopical study of ancient mortars, 2004.
47 Leslie and Hughes, Binder microstructure in lime mortars, 2002.
48 Stefanioud et al., Evaluation of inclusions in mortars, 2012.
The grain size distribution of the aggregates and the binder: aggregate ratios estimated by petrographic analyses and sieve analysis of the samples are presented in figure 7 and table 1. The grey samples show more aggregates of different sizes embedded in the matrix than those of the white ones. The grey and white samples present averages of 23.2 and 19.0% (respectively) of binding material in the < 63 mm range, while both two groups have almost the same distribution of the larger grains except the 0.063-0.125mm and 0.250-1.0mm grain size ranges. Few grains are larger than 4.75mm in two samples (7 and 8), but it is worth noting that some larger grains were not sieved but seen by the naked eye. This grain size distribution allows the estimation of the binder: aggregate ratios per weight percentages of the samples (white samples from 1:2.8 to 1:3.3; and the grey samples from 1:4.14 to 1:4:45).

Figure 7: Grain size distribution of mortar and plaster samples from the house 17-18. The porosity of the samples is given in table 1. It is most likely that the porosity resulted from the micro-pores of the binder, the interstices between the binder and aggregates, the pores of the plant fibers and ceramics and the holes of the scoria (figure 4b,6a). It is noticeable that the white samples are more porous than the gray ones which might be explained by their content of the degraded porous fibers, scoria and ceramics. In addition, it is likely that the fibers and scoria were selected to manufacture plasters of thermal insulation properties and lightweight material\textsuperscript{49,50}.

**CONCLUSIONS**

This paper presents an archaeometric analyses of plaster and mortar samples that have resisted erosion and weathering and/or other factors such as earthquakes and human activities; therefore, they still contribute structural stability and artistry to the building. The Umm el-Jimal craftsmen used lime-based cement materials in building their Byzantine and later structures and monuments. The studied samples mainly dated to the Byzantine period were characterized by their white color and content of hard calcareous sand. The samples

\textsuperscript{49} Bouaker et al., natural straw fibers as aggregates, 2014.
\textsuperscript{50} Al-Bashaireh, Use of Lightweight Lime-Mortar, 2016.
have hydraulic properties through the addition of pozzolanic materials mainly ceramics and volcanic scoria. The addition of charcoal appears to be deliberate for the bedding grey mortars. This feature is widely used, not only in the House XVII-XVIII, but also in the churches and water cisterns of the city. It is clear that the used recipes reflect the availability of raw materials in the vicinity of the site. These recipes were mainly based on the use of the basalt which covers the whole area and scoria outcropping southeast Umm el-Jimal, and the recycling of ceramics. In addition, the recipes included the use of recycled mortar and crushed limestone fragments, most likely those are remnants of the dressing process of raw limestone. The results are concord with previous studies on the mortar of other constructions of the Umm el-Jimal. The results likely indicate that Umm el-Jimal craftsmen of the Byzantine period inherited the same lime-cement technology from their ancestors of the Roman period.
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