

**Archaeometric Investigation of Nabataean Mortars from Petra
“PRODOMEA Project: Project on High Compatibility Technologies and Systems
for Conservation and Documentation of Masonry Works in Archaeological Sites of
Mediterranean Area”**

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Abstract

Scientific Analysis of mortar samples from different sites in Petra were conducted using X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD) and Fourier Transform Infra Red Spectroscopy (FTIR), in addition to Optical Microscope (OM) analysis. Results were coincident and reflected the nature of Petra mortars through their chemical composition and mineralogical identification. Further experimental work is suggested to improve these mortars.

Introduction

Sites Description

The Petra Garden and Pool Complex is located in the centre of Petra, overlooking the colonnaded street. A preliminary survey and excavation of the site started in 1998, revealing an elaborate hydraulic system associated with a swimming pool and island pavilion [Bedal 1999, 2000, 2001, 2002].

Excavations at Petra Great Temple started in 1993 and have continued since then. It is located in the centre of Petra, occupying an area of 7560m², which includes the monumental entrance (propylaeum), the sacred area (lower temenos) and the upper temenos comprising the temple itself [Joukowsky, 1998].

Qasr el-Bint (No. 403) is located at the western end of the temenos gate, at the end of the colonnaded street. It is considered as the only freestanding structure that withstood the earthquakes of 363 and 747 AD. The first to describe the structure was Kohl (1910) followed by Wright (1961), while excavations in and around the site began in the 1960s. the plan of this temple is square-shaped, built of massive sandstone blocks, sometimes reaching ca. 2m. Parr (1967-8: 17) dates the temple to the time of King Obodas III (30-9 BC), while McKenzie (1990: 34-35) gives it a terminous ante quem of the first century AD.

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EXPERIMENTAL ANALYSIS

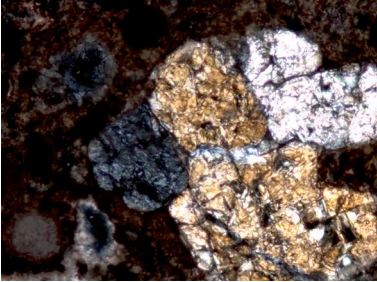

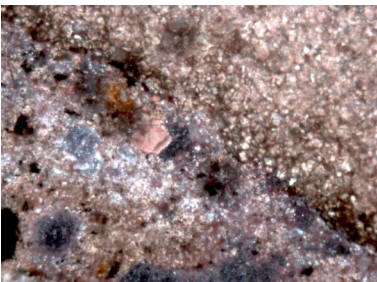

Eight samples were collected by the Hashemite University team members and Table (1) below represents a short description and provenance of each sample under study.

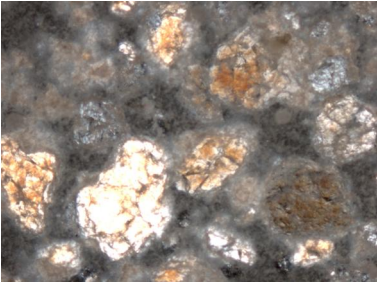
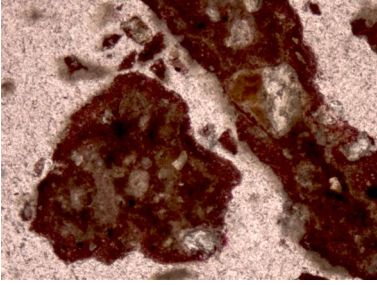
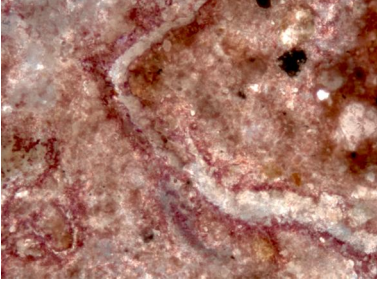

Table (1): Provenance and Description of samples under study

Sample ID	Provenance	Description
PTR 5	Petra Garden and Pool Complex	South-west of site, plaster on the west wall
PTR 6	Petra Garden and Pool Complex	South-west of site, mortar on the floor
PTR 7	Petra Garden and Pool Complex	Mortar mixed with stone, structure south of “pavilion”
PTR 8	Petra Garden and Pool Complex	Plaster covering structure south of “pavilion”
PTR 12	Petra Garden and Pool Complex	Plaster at corner of north-west walls
PTR 14-A	Petra Great Temple	Channel at its end (basin or cistern?), outer layer
PTR 14-B	Petra Great Temple	Channel at its end (basin or cistern?), inner layer
PTR 16	Qasr el-Bint (No. 403)	Jointing mortar, from interior of north wall

Analysis of mortar samples for precise identification of mineralogical composition was done using different analytical techniques for comparison and assurance. Optical Microscopy (OM) of thin sections was the first method of analysis applied to mortar samples, Table (2) describes the petrographic analysis of each mortar sample and in Figures (1-2) selected microscopic images were chosen to represent the samples. Fourier Transform Infra Red Spectroscopy (FTIR) was used as well for the analysis, a representative sample spectra is shown in Figure (3) and Table (3). X-Ray Fluorescence (XRF) was performed where the most abundant metals present in the samples were represented as metal oxides. This can be seen clearly in Charts (1-3). The last available method used for analysis was X-Ray Diffraction (XRD) but this was done just for four samples, Figure (4) represents one spectra and Table (4) summarizes the analysis results.

Table (2): OM Analysis of Petra Mortar samples

Sample ID	Description
<p>PTR 5</p> 	<ul style="list-style-type: none"> • Rock fragment of sandy micrite in bedded in micrite • Sandstone • Oolitic micrite • Cryptocrystalline Silica • Micrite
<p>PTR 6</p> 	<ul style="list-style-type: none"> • Rock fragment of marly micrite 50% • Highly abundant of remain wood • Iron oxide • Rock fragment of Ferruginous Calcareous Sandstone (Micrite) in cement Iron oxide and Sprite • Large sandstone • Heavy metal (Zircon) • Clay (Ceramic)
<p>PTR 7</p> 	<ul style="list-style-type: none"> • Cryptocrystalline silica • Grain of Quartz, highly fracture • Calcite < 1% • Organic matter
<p>PTR 8</p> 	<ul style="list-style-type: none"> • Rock fragment of ferruginous limestone ,contain grain of Quartz (fracture Angular rounded) ,fine sand, moderately sorting with cement iron oxides • Marly micrite • Marly to sand micrite • Grain of Quartz • Rock fragment of dolomite • Remain of Ash. In cement marl and like the tuff shape

<p>PTR 12</p> 	<ul style="list-style-type: none"> • Sandy micritic limestone • Cracked quartz crystals (c.a. 65%)
<p>PTR 14-A</p> 	<ul style="list-style-type: none"> • Marly micrite • Cryptocrystalline silica • Micrite Rounded belts of micrite <5% • Containing fine grain of Quartz, sandy size • Remain of fragment wood • Also contain rock fragment of sandy micrite containing veins of iron oxides • Rock fragment of dolomite show rhombs shape of dolomite • Rock fragment of Marl • Contain micro sprite
<p>PTR 14-B</p> 	<ul style="list-style-type: none"> • Marl con not reacts with alizarin Red S (ARS) • Micrite • Marly micrite • Remains of wood (Ash) • Fine grain of quartz • Rare of dolomite • Silica (Chalcedony)
<p>PTR 16</p> 	<ul style="list-style-type: none"> • Rock fragment of marl containing highly abundant of marl & remain of wood • Iron oxide 10% • Small grain of Quartz • Rounded belts • Containing evaporate (Gypsum & Anhydrite)

Figures (1-2): Optical Microscope Analysis of Petra Mortar Samples

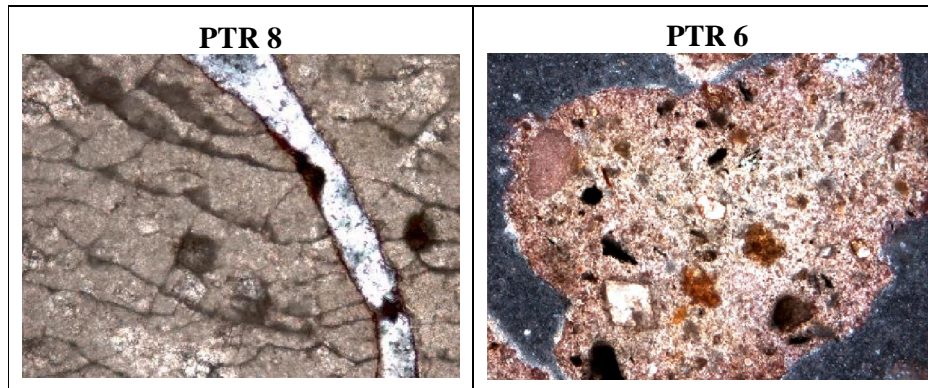


Table (3): FTIR Analysis of Petra mortar sample with ID (PTR 05)

wavenumber (cm ⁻¹)		Mode [#]
Experimental	Published	
420.00	K	Si-O ^B
468.67	K	Si-O ^B
536.62	K	Si-O-Al
711.23	K	Si-O-Al ^S
791.86	Q	Si-O ^S , perp. to optical axis
873.84		
917.21	K,M	Al-OH ^S
1032.75	K,M,Chry	Si-O-Si ^S (very intense)
1091.32	M	Si-O ^S
1163.02		
1428.83	Cal,D	very intense
1623.90	H ₂ O(in Mt)	H-O-H
2863.16	OM	Organic Matter
2929.73	OM	Organic Matter
2986.06	OM	Organic Matter
3426.79	H ₂ O,Chl	H-O-H(wide)
3618.87	K,Mt	Al-OH(inner)
3651.78	Mt	Al-OH
3694.73	K	Al-OH

According to published kaolinite and Quartz spectra analysis [1]

^ S: Stretching Vibrational Mode

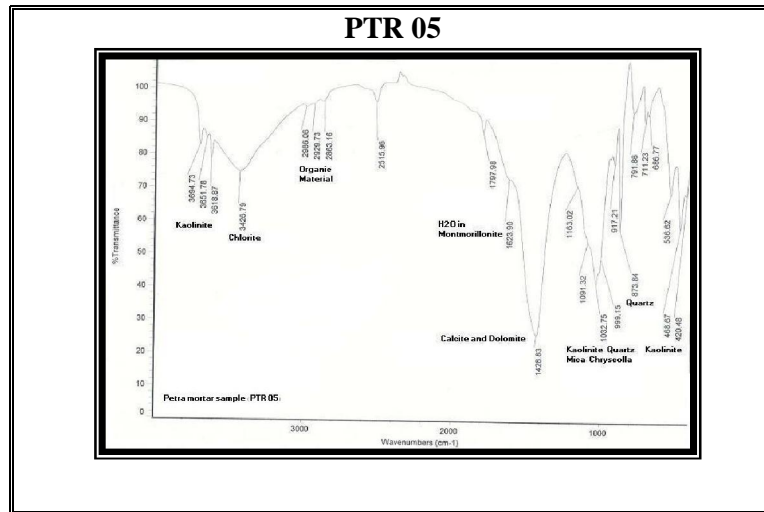
^^ B: Bending Vibrational Mode

A: Attapulgite
S: Sepiolite
Cal: Calcite
Chl: Chlorite
Chry: Chrysocolla
D: Dolomite
K: Kaolinite
M: Mica
Mt: Montmorillonite
Q: Quartz
OM: Organic Matter

References

- [1] Atlas of Infrared Spectroscopy of Clay Minerals and Their Admixtures, by H.W. van der Marel and H. Beutelspacher.
- [2] Geology, Mineralogy, Geochemistry and Industrial Applications of the Clay Deposits from Wadi Musa-Petra Area, South Jordan. by B.Y. Abu Dayyeh. M.Sc. Thesis, University of Jordan. 1993. Page 79.

Figures (3): Selected FTIR Spectra of Petra mortar samples



According to FTIR analysis, all mortar samples contain large amount of Calcite and Dolomite, Kaolinite and Montmorillonite were also found in different ratios. Quartz and micrite were found in all samples in appreciable amounts. This was also proved through the analysis of same samples by XRD and XRF as shown below.

Figures (4): XRD Spectra of selected mortar samples

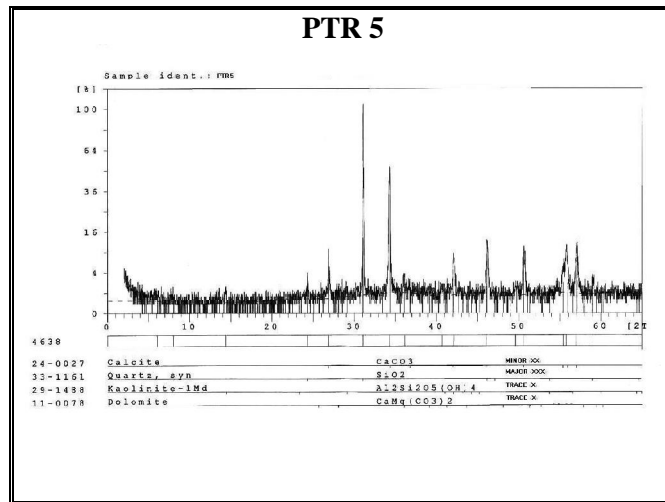
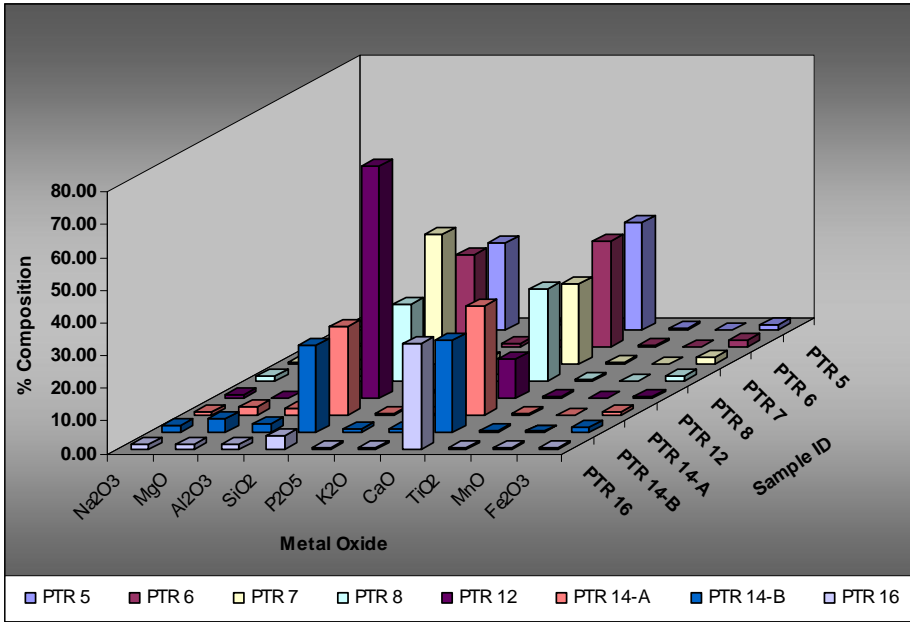
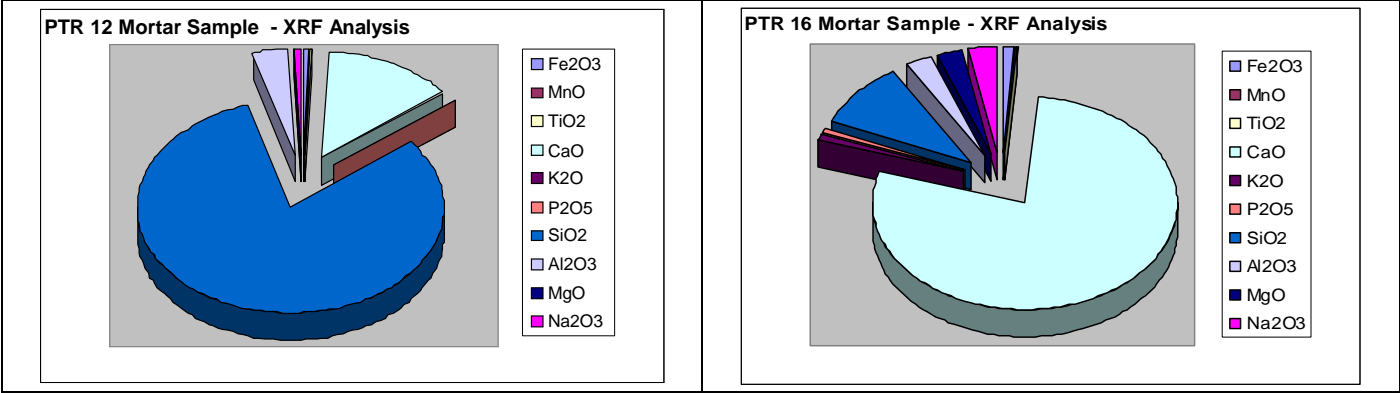


Table (4): Representative Results for XRD Analysis of Selected Petra Mortar Samples

Sample ID	Quartz (SiO ₂)	Gypsum (CaSO ₄ .2H ₂ O)	Calcite (Ca(CO ₃) ₂)	Illite, Kaolinite (Al ₂ Si ₂ O ₅ (OH) ₄)	Halite (NaCl)	Dolomite (CaMg(CO ₃) ₂)
PTR 5	XXX		XX	X		X
PTR 6	XX		XXX			
PTR 7	XX		XXX			

PTR 14 (A+B)		X	XXX	X	XX	
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Charts (1-3): Selected XRF Analysis of Petra Mortar Samples



Results and Discussion

The results of the analyses performed on the samples from selected archaeological sites in Petra (great Temple, Petra Garden and Pool Complex and Qasr el-Bint (No. 403)) were concordant with analysis results performed by our partner Dr. Christina Sabioni and with previous analysis done by Miss May Shaer, member in HU group for PRODOMEA project. All lead to the same conclusion that mortar samples from Petra are Hydraulic Mortars, although aggregate nature varied from one structure type from were the sample is to another.

The northern-west part of the Garden and Pool Complex in Petra represented by sample PTR 12, which is a plaster at corner of the north-west wall was depicted to environmental conditions that were rainy and affected by northern winds, in addition the summer season was shiny enough to accumulate salts by evaporation of humidity stored in the wall. Other samples from the same complex, such as PTR 5 and PTR 6 show appreciable content of calcite and dolomite with the presence of quartz to a less extent. Some organic matter (wood) were recognized in sample PTR 6.

Other samples from the same complex but representing structure of the south of pavilion (PTR 7, PTR 8) were mainly consisting of siliceous minerals with the presence of iron oxide (hematite). The presence of organic matter (ash) was recognized in these samples. The presence of hematite gives the red color of some mortars and this value varies from monument to another, but it has no effect on the strength or durability and workability of the mortar.

Analysis of plaster samples collected from the pipeline system of the Great Temple at Petra (outer and inner layers of the channel) revealed that both samples are hydraulic mortars. But the inner layer sample (PTR 14B) showed by optical microscope analysis a coarser aggregate than the outer layer sample. Both samples consisted of siliceous and carbonate minerals and their aggregate appeared as inhomogeneous and poorly-sorted type. The presence of calcite assures the possibility of carbonation of the lime involved as binder in the mortar preparation process. Clay minerals were also found in these samples, mainly as Kaolinite and Illite with different ratios that are never constant.

Gypsum and Halite were recognized in Great Temple samples whereas never found in the others. So this leads us to the possibility that this mortar is a gypsum-based mortar, keeping in mind that this kind of mortars is less resistant to dissolution in water. So, since these samples are from a channel then this could never be correct. So, what explains the presence of gypsum? This could be due to the lackness of timber necessary for fuel, and since gypsum requires lower burning temperature and hence less fuel it was used in the plaster mix, and because calcium sulphate is water soluble then for a long time very little amount is expected to be detected [Shaer, M.]. The other opinion concerns surrounding environmental conditions within the last decade. That is sulpher could be transferred to the archaeological site of Petra from different sources surrounding the area, such as nearby Bedouin activities, nearest cities and industrial plants which are all of them

accumulates with time sulphur concentration in atmospheric air in this area. Then under rainy conditions, this sulphur can be dissolved in rain water forming what is called "Acidic Rain" even it is very low concentrated but it will have an accumulative effect with time. As well, hydrogen sulphide could be formed in air attacking the monuments and stones converting the lime into gypsum with longer contact time and under high temperatures like those found in Petra climate. I believe if longitudinal sampling in-depth within the same channel will either prove this assumption or deny it!!

One more note can be said here, that is the considerable amounts of Illite clay mineral and traces of Kaolinite mineral found in almost all samples revealed that a marly limestone was used as raw material in the preparation of the mortars. And this natural composition can explain the hydraulic property identified in all samples from different sites of Petra concerned in this study as mentioned earlier.

Acknowledgment

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References

1. Shaer, May. **"The Ancient Nabataean Mortars of Petra, Jordan"**. Petra Stone Preservation Project (GTZ), Jordan.