



## EXPERIMENTAL STUDY TO PREPARE A COMPATIBLE MORTAR FOR FILLING THE CRACKS IN ARCHAEOLOGICAL BASALTIC STONES AT ABU SIR, EGYPT

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#### Abstract:

Basalt represents one of the most common stones used in the construction of our archaeological structures. However, basalt is a very dense, compact and hard igneous stone; it is highly deteriorated by physicochemical deterioration factors especially in the open air environments. Cracks are among the dangerous deterioration aspects in basaltic stones. They significantly contribute to accelerate their deterioration rates. Consequently, the cracks in basaltic stones have to be filled with a suitable mortar to stop their harmful impacts. In the present work, four types of organic binder based mortars were prepared and comparatively evaluated to select the best one for filling the cracks occurring in the studied archaeological basaltic stones at Abu Sir area. Examinations and analytical study were performed using transmission electron microscope, polarizing microscope, x-ray diffraction, colourimetric measurement, static water contact angle, compressive strength and scanning electron microscope attached with EDS. The results declared that the mortar named M4 (Acrylirsil + basaltic powder) is the most suitable composite for filling the cracks in the studied basalt.

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### 1. Introduction

Basalt is the most common extrusive igneous rock that widely spreads on the earth's surface. It is occurred by the rapid cooling of lava on or close to the earth's surface. It is mainly composed of calcic plagioclase feldspar and pyroxene minerals with or without olivine. Due to very rapid cooling of basaltic lava on the earth's surface, it usually has aphanitic texture with very small crystals, which are indistinguishable to the naked eye. Also, it may be porphyritic with phenocrysts of augite, olivine, or plagioclase. As a result of the high content of mafic minerals (especially pyroxene) in basalt, it usually has dark grey

to black colour [1-7]. In ancient Egypt, basalt was employed for carving small vessels, sarcophagi and statues from the late pre-dynastic period until the sixth dynasty. During the old kingdom especially in the fourth and fifth dynasties, basalt was largely used for paving the floors of some pyramid funerary temples at Saqqara, Giza and Abu Sir necropolises [8]. Basaltic pavements and walls were used in the mortuary temples of kings Djoser (3<sup>rd</sup> dynasty), Userkaf (5<sup>th</sup> dynasty) and Pepi I (6<sup>th</sup> dynasty) at Saqqara; king Khufu (4<sup>th</sup> dynasty) at Giza; and kings Sahure, Neferrirkare, and Niuserre (5<sup>th</sup> dynasty) at Abu

Sir [9]. In addition, basalt was used in the walls and pavements at the pyramid complex of Pepi I. After the old kingdom, from the first through third intermediate periods, the use of basalt was limited. It was rarely employed for monumental construction purposes, and mainly utilized for carving statues and small vessels. During the late Ptolemaic and Roman periods, it was mainly used for statuary and occasionally for small architectural elements [8-10]. Basalt quarries are widespread at numerous places in Egypt such as Abu Zaabal, Cairo-Suez desert, Abu Rawash, Faiyum, El-Bahansa, Samallut, Gebel El-Teir and Abu Simbel. The quarry of Widan El-Faras on Gebel El-Qatrani in the northern Faiyum desert was the main source of basaltic blocks used for construction purposes in the old kingdom [11-14]. Although basalt represents one of the hardest and most durable stones, it is highly affected by physico-chemical deterioration factors especially in the open air environments [15]. Cracks are among the serious deterioration forms of basaltic stones. They may result from the external mechanical stresses such as overloading, vibrations, mechanical shocks, fault transferring and structural defects. In addition, the physico-chemical weathering processes (*temperature variation, hydration, salt crystallization frost*) are usually accompanied by developing internal stresses inside the stones, which finally lead to the formation of cracks [16-18]. In this paper, the basaltic walls and pavements in the temple of Niuserre at Abu Sir were selected as case study, fig. (1-a). They have suffered from cracks, fig. (1-b) which urgently need to be filled with a suitable mortar in order to prevent their harmful impacts. Rare researches have been conducted to fabricate and estimate the mortars used in filling the cracks of basaltic stones. The main goal of this study is to prepare and evaluate some mortars in order to select the best one for filling the cracks in the selected basaltic

stones. Examinations and analytical study were performed using TEM, PM, XRD, SEM-EDS, colourimetric measurement, static water contact angle and compressive strength.

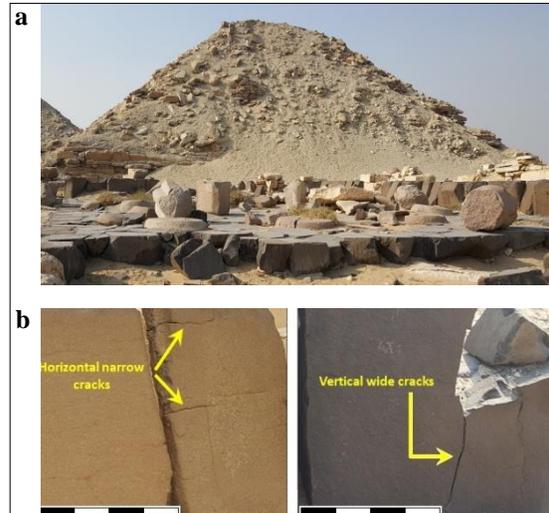


Figure (1) Shows **a.** remains of basaltic walls and pavements in the temple of Niuserre at Abu Sir, **b.** horizontal and vertical cracks affected the basaltic walls

## 2. Materials and Methods

### 2.1. Sampling

Weathered basaltic samples were collected from the fragments spread in the site of Abu Sir, Egypt.

### 2.2. Mortars

Four types of mortars were made in order to select the best of them for filling the cracks in the studied archaeological basalt. The binding materials used in the prepared mortars are acrylic based products:

- (1) Paraloid B72 (*CTS Company, Italy*)
- (2) Eucocolle (*SwissChem Construction Materials, Egypt*)
- (3) Primal AC 33 (*CTS Company, Italy*)
- (4) Acryclirsil (*SwissChem Construction Materials, Egypt*).

The filler material used in the prepared mortars composes of fine powder of basalt which collected as by-product of modern basalt quarrying activities. The size of basaltic powder was determined by JEOL

2100 high resolution transmission electron microscope. TEM micrographs declared that the used basaltic powder consists of tiny particles ranging in diameter from 100 nm to 1000 nm, fig. (2-a). Compositions of the prepared mortars are presented in tab. (1). In order to prepare mortars with good consistency and workability, each organic binder was mixed with basaltic powder in the proportion 0.5:1 by volume. To simulate the procedures of treatment as they happen in the practical field, each mortar was vigorously stirred by a trowel till a homogenous mixture is composed. The mixtures of mortars were then casted in 2 cm<sup>3</sup>, 3 cm<sup>3</sup>, 5 cm<sup>3</sup> moulds, and de-moulded after 7 days and left for 1 month at room T until the complete curing took place, fig. (2-b). Afterwards, the samples of each mortar type were comparatively tested.

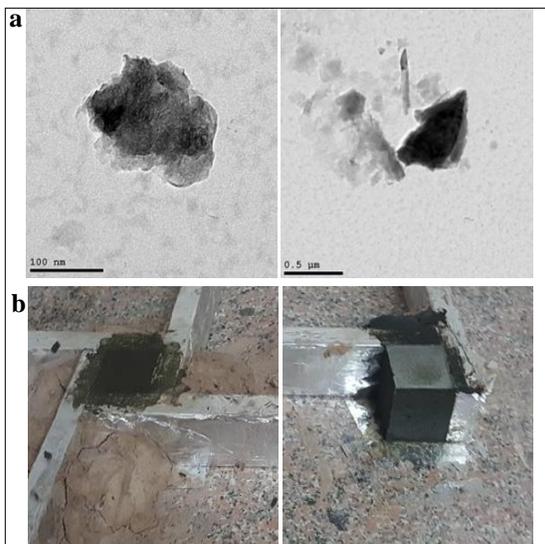


Figure (2) Shows **a**, HR-TEM micrographs of the filler material (*basaltic powder*), **b**, casting and de-moulding stages.

Table (1) Compositions of the prepared mortars.

Symbol	Organic binder	Solvent	Filler
M1	Paraloid B72 (30 %)	Toluene	Basaltic powder
M2	Eucocolle (30 %)	Water	
M3	Primal AC 33 (30 %)		
M4	Acryclirsil (30 %)		

### 2.3. Polarizing microscope

Basaltic samples were investigated under polarizing microscope to study their mineral

constituents and other petrographic properties. Thin sections of weathered basalt were firstly prepared, and then were examined under Nikon eclipse LV100POL polarizing microscope.

### 2.4. X-ray diffraction analysis

X-ray diffraction analysis was utilized to identify the mineralogical composition of the studied basaltic samples. This analysis was performed using PAN analytical x-ray diffractometer X'Pert PRO working with secondary monochromator, Cu-radiation ( $\lambda = 1.542\text{\AA}$ ) at 45 K.V., and scanning speed  $0.04^\circ/\text{sec}$ . The diffraction peaks between  $2\theta = 2^\circ$  and  $60^\circ$ , corresponding spacing ( $d$ ,  $\text{\AA}$ ) and relative intensities ( $I/I^\circ$ ) were obtained. The diffraction charts and relative intensities are obtained and compared with ICDD files.

### 2.5. Scanning electron microscope attached with EDS

Quanta FEG 250 SEM was used to study the morphological features of archaeological basalt and prepared mortars. Also, EDS unit connected to this microscope was employed to determine the elemental composition of archaeological basalt. This examination was performed on non-coated samples with the conditions: a 10.1 mm working distance and in-lens detector with an excitation voltage of 20 kV.

### 2.6. Aesthetical properties

The preservation of general appearance of the treated stones represents an important criterion in the field of restoration and conservation. For this purpose, the colour homogeneity between archaeological basalt samples and prepared mortars was determined by colourimetric analysis. Optimatch 3100 spectrometer was utilized to measure the chromatic alterations between the basaltic samples and prepared mortars. The test was performed based on the  $L^*$ ,  $a^*$  and  $b^*$  coordinates of the CIELAB colour space [19].



Table (2) Approx. percentages of the minerals detected in the studied basalt.

Minerals	Approx. percentages
Labradorite	55
Augite	41
Montmorillonite	04

### 3.3. Microstructure features and elemental composition of basalt

Scanning electron microscope equipped with EDS spectrometer was conducted to investigate the microstructure features of the studied basalt. SEM micrographs of basaltic sample, fig. (5) illustrate that it is substantially affected by physicochemical and mechanical deterioration aspects. The sample suffers from surficial deformation, micro-cracking, flaking and accumulation of weathering products. Moreover, EDS analysis of the same basaltic sample reveals the presence of O, Si, Al, Fe, Ca, Mg, Na, K and Ti, tab. (3).

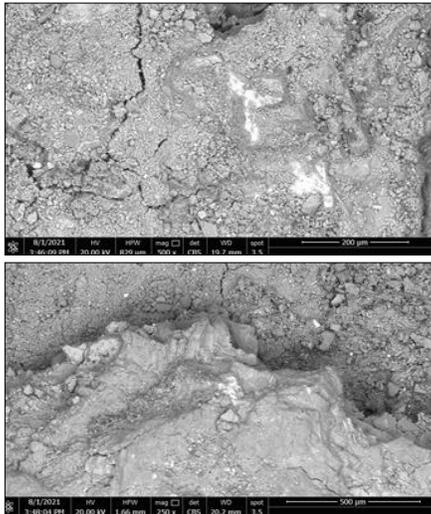


Figure (5) Shows SEM micrographs of the studied basalt surficial deformation, micro cracks, flakes and weathering products.

Table (3) Elemental composition of the studied basalt.

Elements	Concentration (wt %)
O	43.10
Na	01.13
Mg	02.62
Al	09.90
Si	23.30
K	01.82
Ca	08.40
Ti	00.62
Fe	09.11
<b>Total</b>	<b>100 %</b>

### 3.4. Aesthetical properties

The preservation of general appearance of the treated stones represents an important criterion in the field of restoration and conservation. For this purpose, the aesthetical compatibility between the archaeological basalt and prepared mortars was determined by colourimetric measurements. The total colour change ( $\Delta E$ ) was calculated using the following equation:  $\Delta E^* = \sqrt{[(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]}$ . Where  $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$  are the differences in the,  $L^*$ ,  $a^*$  and  $b^*$  coordinates of archaeological basalt and prepared mortars. The data obtained from this test are reported in tab. (4).

Table (4) Chromatic alterations between the prepared mortars and archaeological basalt.

Mortar	$\Delta E^*$
M1	8.2
M2	6.4
M3	3.8
M4	4.6

### 3.5. Hydrophobicity

Hydrophobic properties of the prepared mortars were assessed by measuring their contact angles. Table (5) and fig. (6) clarify the results of static water contact angle measurements.

Table (5) Average values of static water contact angles of the prepared mortars.

Mortar	Water Contact Angle
M1	70°
M2	55°
M3	72°
M4	95°

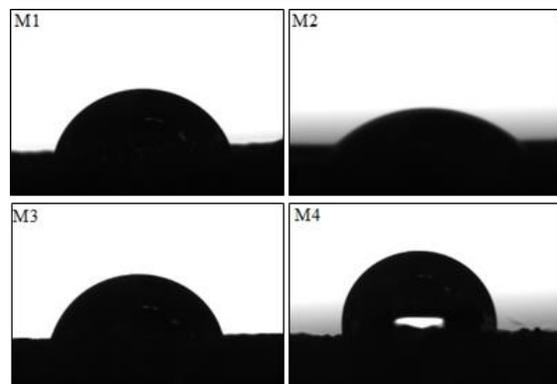


Figure (6) Static contact angle photographs of the prepared mortars.

### 3.6. Mechanical properties

Mechanical properties of the used mortars were comparatively studied by means of compressive strength test. The average values of compressive strength are listed in tab. (6).

Table (6) Average values of compressive strengths of the prepared mortars.

Mortar	Compressive strength (kg/cm <sup>2</sup> )
M1	69
M2	80
M3	72
M4	60

### 3.7. Mortars morphological aspects

The microscopic textures and morphologies of the studied mortars were investigated by scanning electron microscope. SEM micrographs, fig. (7) declare that the tested mortars have homogenous microstructure features. Although M1 and M4 samples show similar structure, M4 has a rough microstructure surface. M2 and M3 samples are characterized by their condensed microstructure but M2 is more compact and has low amount of porosity.

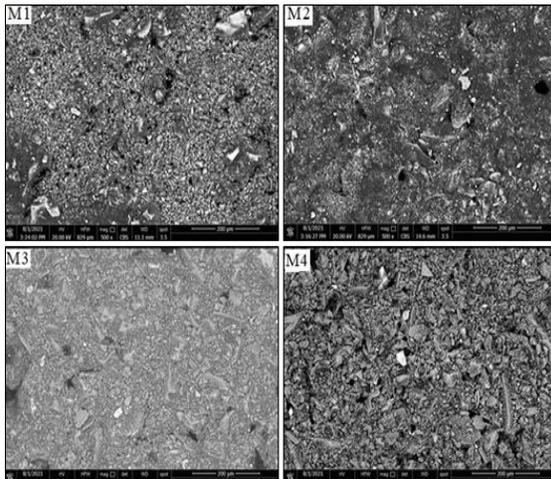


Figure (7) SEM photomicrographs of the prepared mortars.

## 4. Discussion

The field observations of the basaltic walls and pavements in the temple of Niuserre declared that they are affected by many det-

eriation aspects such as discoloration, granular disintegration, cracking, flaking. The petrographic investigation of the studied basalt elucidated its porphyritic texture. It is principally comprised of labradorite and augite associated with minor amounts of iron oxides, and opaque minerals. In addition, it was observed that the studied basalt is highly affected by mineralogical alteration. Labradorite is converted into sericite, epidote and clay minerals while augite is converted into iron oxides, chlorite and clay minerals. X-ray diffraction analysis of the studied basalt emphasized that it is mainly composed of labradorite and augite as primary minerals in addition to montmorillonite as weathering product. SEM graphs of basaltic sample declared that it is highly affected by physicochemical and mechanical weathering features. The sample suffers from superficial deformation, microcracking, flaking and weathering products accumulation. Based on the results of field observations and analytical methods, it was clear that the studied basalt has been subjected to aggressive weathering. Although basalt is a very dense, compact and hard igneous stone, it is rapidly damaged by physicochemical weathering factors, which attack its primary minerals such as labradorite and augite and alter them into secondary minerals such as clay minerals [22]. Moisture is the most effective physicochemical weathering factor that plays the major role in the mineralogical conversion of labradorite and augite into clay minerals [23,24]. The clay minerals formation in the surficial layers of basalt causes a drastic change in their physicochemical properties. For instance, hydration of clay minerals after absorbing moisture is accompanied by their swelling. The pressures resulted from swelling of clay minerals result in granular disintegration, cracking and flaking of basalt [25-27]. Moreover, daily and seasonal thermal fluctuations con-

tribute to the mechanical deterioration of basalt. This is ascribed to the repeated expansion and contraction of basaltic minerals due to repeated heating and cooling [28]. Cracks are one of the most dangerous deterioration factors of stones. The presence of cracks in studied basaltic stones plays a significant role in their damage mechanisms. They represent weakness points from which the fractures start in the stones. Moreover, they allow water, acidic rain, salts and microorganisms to penetrate inside the stones, which accelerate their deterioration rates [17,29,30]. Freezing-thawing cycles and crystallization of salt in the cracks generate high internal mechanical stresses [31]. Furthermore, penetration of microorganisms through small fissures and cracks causes many physicochemical and mechanical deterioration aspects [32,33]. In order to prevent the harmful effects of cracks, they must be filled with a suitable mortar. In the current paper, four types of organic binder based mortars were prepared and comparatively studied to select the best one for filling the cracks in the studied basaltic stones. Due to the good adhesion properties of acrylic products, they were employed as organic binders in the prepared mortars [34]. Furthermore, the aesthetical compatibility between the mortars and archaeological basalt was achieved by using basaltic powder as fillers in the prepared mortars. The results of colourimetric analysis demonstrated that the mortars of M3 and M4 possess the most compatible appearances with the studied archaeological basalt, as they achieve total colour differences values less than the acceptable limits [35]. These results are agree with the results of visual appraisal, as the mortars M3 and M4 have pale gray colors similar to those of the archaeological basalts, fig. (8). In addition, the mortar M1 has the highest value of  $\Delta E$ , this is can be attributed to its nature as a solvent based polymer whereas

the other three products are water based polymers. The results of water repellency highlighted that the mortar M4 has a hydrophobic character, as it achieved contact angle higher than 90°. The polymers hydrophobic properties significantly depend on their surface energies which are mainly related to their chemical compositions [36]. The mortar M2 achieved the lowest contact angle value which suggests that it has high surface energy [37]. The mortars of M1 and M3 had almost similar values of contact angles due to same chemical compositions of the binding materials (paraloid B72 and primal AC33) used in these mortars [38]. Moreover, the mechanical performances of the prepared mortars were comparatively estimated. The data of clarify that the mortar M2 has the highest compressive strength value. This is due to the high adhesion properties of the product Euccocole resulting from its high surface energy as illustrated by the test of static contact angle. However, M1 and M3 possess similar compositions, M3 achieved higher degree of compressive strength, this is may be due to its condensed microstructure as observed in SEM micrographs.

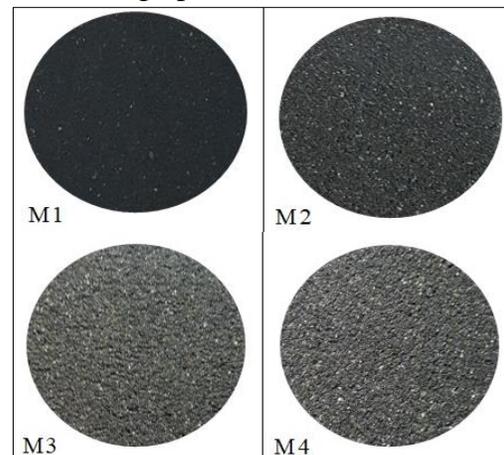


Figure (8) Shows general appearance of the prepared mortars.

## 5. Conclusion

*Basaltic walls and pavements in the temple of Niuserre at Abu Sir have highly suffered from*

many deterioration agents related to the open environment. Cracks are among the aggressive deterioration aspects existed in those basaltic walls and pavements. They are considered to be deterioration aspects and damage factors at the same time. They play substantial roles in accelerating the deterioration rates of basaltic stones. As a consequence, the cracks in basaltic stones have to be treated with a suitable mortar to stop their risks. In the present work, four types of organic binder based mortars were fabricated and comparatively evaluated to select the best one for filling the cracks in the studied basaltic stones. The results of the experimental study elucidated that the mortar of M4 (Acryclirsil + basaltic powder) represents the most optimal mortar for filling the cracks in the studied basalt. It possessed good compatibility with the studied basalt samples with unnoticeable total colour difference. Moreover, it showed good hydrophobic properties by achieving the highest values of water contact angles, so that it has the ability resist water and reduce its harmful impacts. In addition, it produced acceptable value of compressive strength.

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