

# Consolidation of Egyptian Faience Using PARALOID B-72: The Influence of Production Techniques on the Depth of Consolidation

## ABSTRACT

*Ancient Egyptian faience was glazed using three techniques that play an important role in its structural fragility. To improve mechanical stability, consolidation treatment using a resin may be required. Because each glazing method influences an object's microstructure, the effectiveness of consolidation treatment is expected to differ among objects produced using the different glazing techniques. Polished samples from replicas were compared with those from ancient objects using scanning electron microscopy-energy dispersive X-ray spectroscopy. Research was undertaken into the penetration depth and distribution of PARALOID B-72 applied to representative faience replicas. After consolidation, the distribution of the PARALOID B-72 in the replicas was assessed using three-dimensional images created using neutron tomography. Penetration depth was considerably greater in application- and cementation-glazed replicas than in efflorescence-glazed replicas. Differences in consolidant penetration depth reflect differences in porosity, which is considered to be influenced by interparticle glass formation in the core material.*

## KEYWORDS

Egyptian faience · Consolidation · PARALOID B-72 · Neutron tomography

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## INTRODUCTION

Egyptian faience was first produced in Egypt or Mesopotamia and is one of the earliest glazed materials (Matin and Matin 2012, 763). Its production in Egypt developed in pre-dynastic times, ca. 5500-3050 BCE, and continued until the Late Period, 713-332 BCE (Nicholson 1993, 18). The material consists of a silica-rich core, which is covered by a glaze layer. Researchers have proposed three different techniques for the ancient production of faience: one interior method, efflorescence glazing, and two exterior methods,

cementation and application glazing (Figure 1) (Smith 1996, 846).

In the collection of the National Museum of Antiquities (RMO) in Leiden, the Netherlands, a number of faience *shabtis*, funerary figurines meant to carry out the tasks the deceased would be asked to perform in the afterlife, displayed damages such as chipping, crumbling, and even disintegration (Figure 2). These observations raised questions about the vulnerability of faience and its conservation.

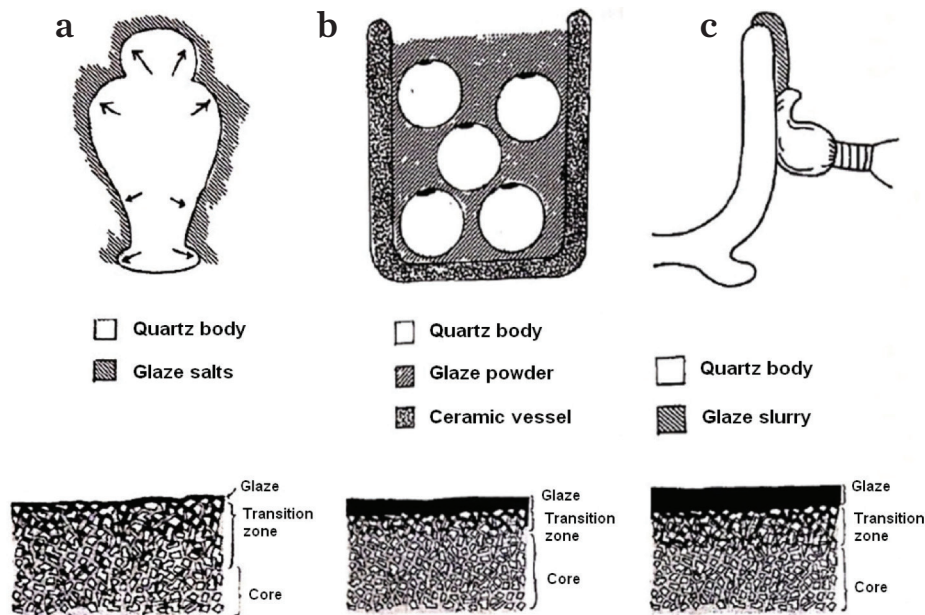


Figure 1. Diagrams of the three faience glazing methods, a) efflorescence, b) cementation, and c) application and resulting cross sections (below) · Copyright of Smith 1996, 846

Faience *shabtis* were produced from the Middle Kingdom, 2040-1782 BCE, until the Ptolemaic period (Schneider 1977, 235). The 11 *shabtis* that were examined for this study were made between the 20<sup>th</sup> and 30<sup>th</sup> Dynasties, 1187-343 BCE. Most are of unknown provenance, but four figurines from the Third Intermediate period, 1070-712 BCE, were found at Deir el-Bahri in Thebes. While *shabtis* from the 18<sup>th</sup> and 19<sup>th</sup> Dynasties are claimed to have the hardest core material, those from the New Kingdom and the Third Intermediate Period are described as having a very friable body, with thick glaze layers (Schneider 1977, 235).

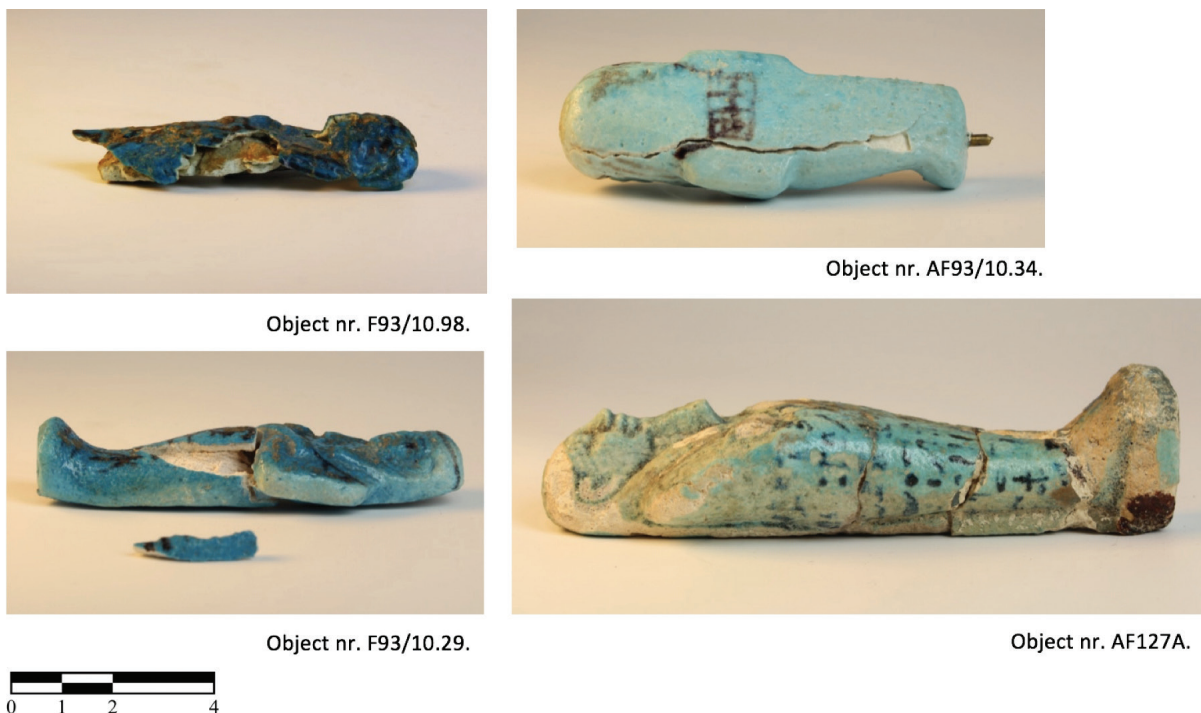
A few researchers have investigated the production methods of faience *shabtis*. Kaczmarczyk and Hedges (1983, 128) observed impressions of stand marks on the glaze that are generally associated with application or efflorescence glazing. Tite, Shortland, and Angelini (2008, 91) posited that *shabtis* are likely to have been application glazed, based on these objects' generally large size. Liang et al. (2012, 3688) suggested that a 21<sup>st</sup>-dynasty *shabti* might have been glazed by efflorescence and convincingly argued that a Late Period *shabti* was application glazed.

Application glazing involves shaping an object, usually in a mould, from a paste of ground sand or

stone mixed with water, possibly with the addition of an organic binder such as gum arabic (Nicholson 1998, 51). Subsequently, a glaze slurry was applied to the object using a brush or by immersion. The slurry would have been made by mixing ground sand or stone with an alkali such as natron or the ash of halophytic plants, and often with a copper-containing mineral, producing a green or blue glaze upon firing.<sup>i</sup> Calcium in the glazes might have been present as a component of the sand, or added intentionally as ground limestone (Nicholson 1998, 50).

The other exterior method, cementation glazing, involves immersing the dried shape in a glazing powder. The silica content of the powder must have been considerably lower than for application glazing, in order to avoid complete vitrification and allow for the alkali and copper to form a successful glaze at the surface of the immersed shape during firing (Matin and Matin 2012, 775).

The efflorescence method involves shaping the object from a paste consisting of ground sand or stone, small amounts of alkali, and a copper mineral. The alkali migrates to the surface upon drying and fuses with silica and copper to create a glaze during firing. Because alkali is still present in the body of the object during firing, partial



**Figure 2.** Egyptian faience shabtis from the National Museum of Antiquities, Leiden, displaying degradation phenomena: Third Intermediate Period, L 8.3 cm. F93/10.98; Third Intermediate Period, 21<sup>st</sup> Dynasty, L 10.2 cm × W 3.7 cm. F93/10.34; Third Intermediate Period, 21<sup>st</sup> Dynasty, L 10.2 cm × W 3.7 cm. F93/10.29; Late Period, 30<sup>th</sup> Dynasty, L 15.2 cm × W 5.2 cm. AF127a · Courtesy of the National Museum of Antiquities, Leiden

vitrification occurs in the core material as well. For all three methods, a firing temperature between 800-1000 °C is assumed (Nicholson and Peltenburg 2000, 191).

Although faience objects may appear stable, this is not always the case. Differences in the microstructure as a result of the production process are expected to influence mechanical vulnerability. Faience core material can be very fragile and vulnerable to physical damage, to the extent where the object cannot be safely handled. In this paper, the authors first investigated what might have caused the damages observed on the Leiden *shabtis*, in relation to how the objects were produced. The *shabtis* were tested for the presence of soluble salts. Visual inspection and microscopic and microchemical analyses were performed on samples taken from eight *shabtis* to determine their production methods.

As structural stability will often need to be improved during the conservation of faience,

consolidation treatment may be required, involving impregnation with a binding material. Consolidation as a general topic has been explored extensively in conservation literature, but the consolidation of faience has received little scholarly attention. Although Smith (1996) and Davison (2006) recommend the use of PARALOID B-72, B-67, and B-99, this approach has not been methodically researched. Therefore, we also studied the behavior of PARALOID B-72, a copolymer of ethyl methacrylate and methyl acrylate, inside the faience. Each glazing method results in different structural characteristics, likely influencing the depth and distribution of the consolidant. Known for improving the internal cohesive strength of a number of porous materials (Podany et al. 2001, 15), PARALOID B-72 is the most commonly used consolidant for faience.

Mapping the distribution of synthetic polymers such as PARALOID B-72 is problematic. Previously employed methods have had serious limitations and could not fully monitor or measure the

depth of consolidation in a conservation context. Techniques like optical microscopy, fluorescent labelling, and Fourier transform infrared spectroscopy (FTIR) have been employed with moderate or little success (Dröber 2006; Louwers 2003; Hamers 2005). The exceptional opportunity arose at the Reactor Institute at Delft University of Technology (TU Delft) to take part in a pilot study involving neutron tomography, which was used to map the penetration of PARALOID B-72 applied to faience. This method has been used successfully to evaluate the consolidation of tin-glazed tiles (Prudêncio et al. 2012, 964-969).

## EXPERIMENTAL

The archives of the RMO were searched for records of old restoration treatments on the 11 *shabtis* in the museum's collection that showed degradation phenomena. Small samples were collected and put in 10 mL distilled water, which was then tested for the presence of sulphates, chlorides, and nitrates by dipping in QUANTOFIX test strips. The solution was assayed after one, two, and seven days, until the water volume had evaporated to 5 mL.

To determine the production method, the *shabtis* were carefully observed in visible light and under magnification using a stereomicroscope. Samples were taken from eight *shabtis* and polished as cross sections. These were examined using a Zeiss Axioplan Imaging 2 optical microscope and with scanning electron microscopy-energy dispersive X-ray spectroscopy (SEM-EDX) using a JEOL 5910LV scanning electron microscope operating in low vacuum and a Thermo Scientific System Six EDX detector.

### Production of replicas

Based on preliminary tests of several recipes reported in the literature, one recipe was selected for each production method (Table 1).<sup>ii</sup> These resulted in replicas that approached the chemical composition and the silica particle size of the core material of the *shabtis* from the RMO. The representativeness of the replicas was assessed by visual inspection and with SEM-EDX analysis of cross sections.

Deionized water was added to the core material ingredients of each of the recipes to create a workable paste. The paste was pressed into one-sided rectangular earthenware molds of 4 cm × 1 cm × 1 cm, removed to drying stands, and left to dry in an oven for 48 hours at 40 °C. These same stands were also used during firing. Some stands were made based on impressions observed on faience *shabtis* from the RMO, while others were modelled after supports found at Memphis (Nicholson 1993, 170).

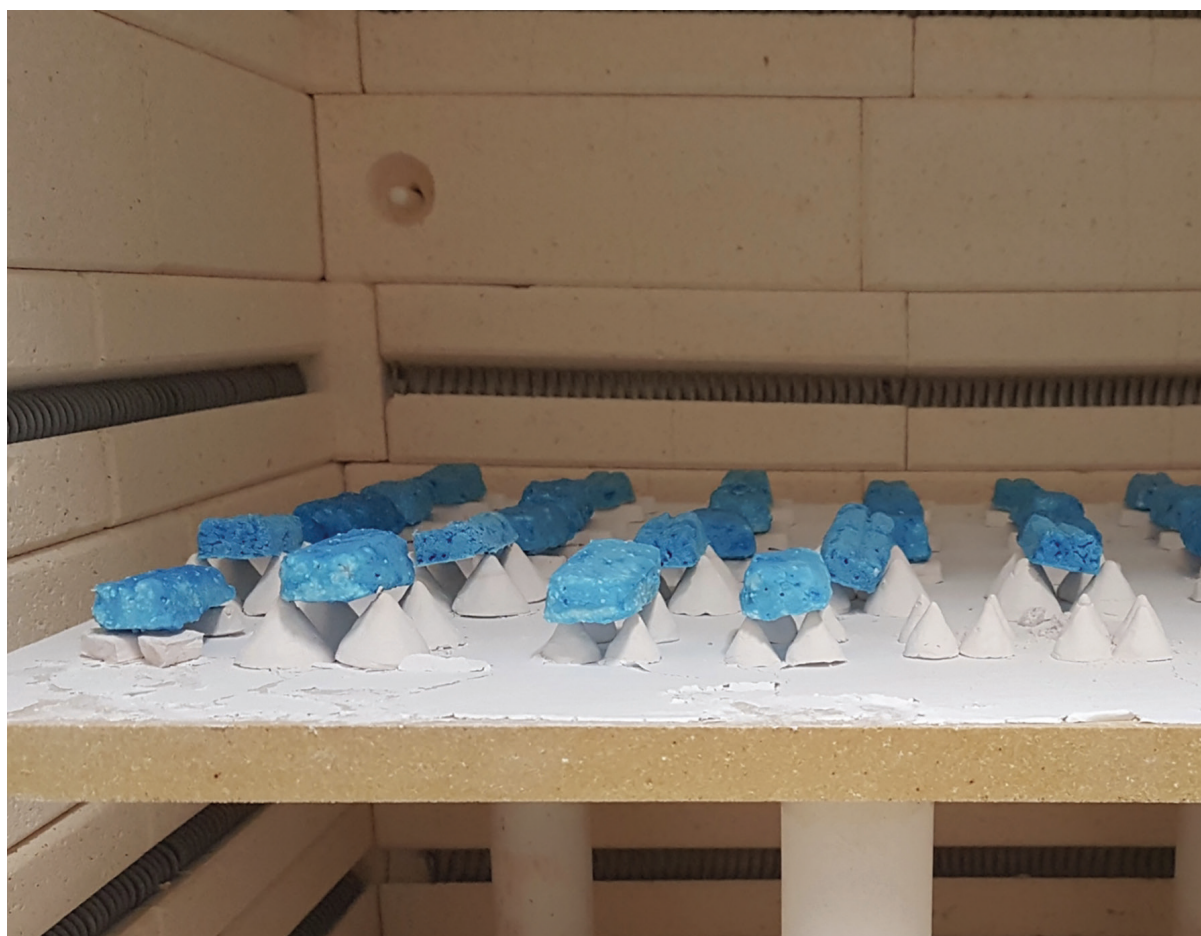
For application glazing, the dry ingredients were again mixed with deionized water (Table 1). Several layers were applied with a brush to the dried cores. The glazed cores were left to dry under the same conditions for another 48 hours. Once dry, the forms used for cementation glazing were placed in an unglazed porcelain bowl and were sandwiched between two layers of glazing powder. This bowl was then placed in a large lidded saggar in the kiln.<sup>iii</sup> The efflorescence- and application-glazed replicas were fired on stands (Figure 3). For all three glazing methods, the replicas were fired in an electric kiln, increasing the temperature by 100 °C per hour to 980 °C. Soaking time was 30 minutes. Cooling down took approximately 12 hours.

### Consolidation with PARALOID B-72

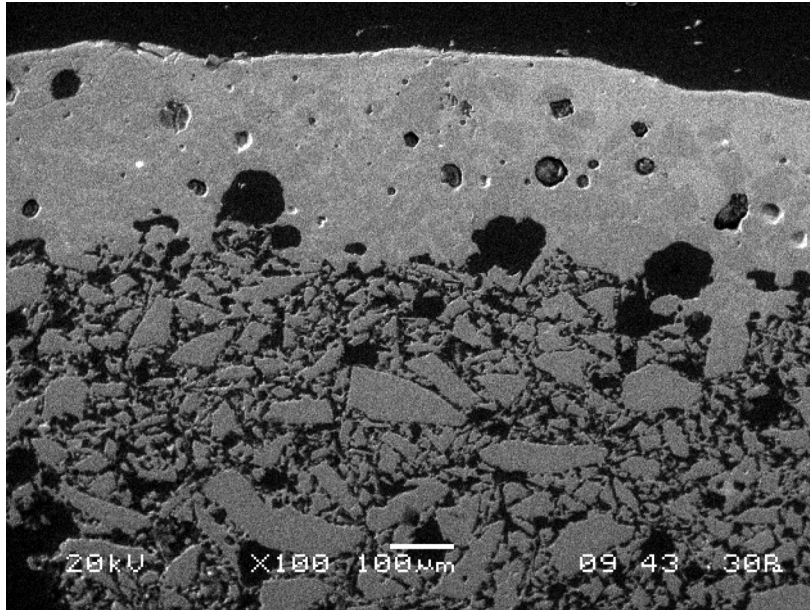
Using a diamond-tipped micro drill, 1 cm deep holes were drilled into the top of the faience test pieces to simulate a damaged area. These holes were deep enough to provide access to the core material. Then, 0.5 mL of PARALOID B-72 in solution was introduced into each hole using a pipette. Six test pieces of each glazing method were consolidated with different solutions: 3, 5, and 10 percent (w/v) in acetone and 3, 5, and 10 percent (w/v) in 1:1 acetone:ethanol. The 10 percent concentration is often used for consolidation of porous silica-based materials, while the 5 percent and 3 percent solutions were chosen to evaluate the influence of concentration on penetration depth. The addition of ethanol was intended to slow the rate of solvent evaporation, allowing more time for the resin to penetrate. The test pieces were placed in an upright position during and after consolidation and were left untouched for 12 hours.

GLAZING METHOD	SOURCE	SILICA GRAIN SIZE (µm) BODY	SILICA GRAIN SIZE (µm) GLAZE	SILICA CONTENT BODY (wt.%)	SILICA CONTENT GLAZE (wt.%)	ALKALI (wt.%)			METAL OXIDES (wt.%)		LIME (wt.%)		FIRING TEMPERATURE (°C)
						Na <sub>2</sub> CO <sub>3</sub>	NaCl	K <sub>2</sub> CO <sub>3</sub>	Cu <sub>3</sub> (CO <sub>3</sub> ) <sub>2</sub> (OH) <sub>2</sub>	CuO	CaCO <sub>3</sub>	Ca(OH) <sub>2</sub>	
<b>Efflorescence</b>	Tite, Manti, and Shortland (2007)	<63/250 (80:20)	NA	90	NA	6				2.3	1.7		980
<b>Cementation</b>	Matin and Matin (2012)	<63/250 (80:20)	<63	100	24	5	4	3		3		61	980
<b>Application</b>	Not based on sources from the literature	<63/250 (80:20)	<63	100	76	15		5	3		1		980

*Table 1. Recipes used to produce the faience replicas. The ingredients are based on descriptions of replica production by Tite, Manti, and Shortland (2007) and Matin and Matin (2012).*



*Figure 3. Efflorescence- and application-glazed replicas after firing*



*Figure 4. SEM image of a shabti believed to have been application glazed. Third Intermediate Period, 21<sup>st</sup> Dynasty, L 4.9 cm. National Museum of Antiquities, Leiden, RBK14272 · Courtesy of the National Museum of Antiquities, Leiden*

### Neutron tomography

Neutron tomography is used to visualize the interior of bulk objects, similar to computed tomography with X-ray radiation. By performing tomography with neutrons, thick and heavy objects can be penetrated non-invasively, yet the technique is particularly sensitive to light elements including hydrogen, lithium, and boron. Hydrogen-rich materials like PARALOID B-72 are less transparent to neutrons than the constituents of faience, which results in contrasting greys in a neutron image. As compared to X-rays, the particular sensitivity of neutrons for both light and heavy elements originates from the fact that neutrons interact with the core of atoms, while X-rays interact with the electrons of atoms. Neutron tomography therefore interacts strongly with hydrogen and can highlight different types of materials as compared to other types of analysis.

Neutron tomography is based on a set of (2D) transmission images of the object, created by placing the object between the camera and the impinging neutron beam. By rotating the object, images are taken from different angles (Paul Scherrer Institut 2019). From these images, a full virtual three-dimensional (3D) image, of the sample can be produced (De Beer 2015, 916).

First Imaging Station Holland (FISH) is a new thermal neutron imaging station at the Hoger Onderwijs Reactor (HOR) at TU Delft and was described in detail by Zhou et al. (2018, 369-373). An effective spatial resolution of 150 µm with an optical pixel/voxel size of 51 µm for the images was chosen for this work. For each sample, 500 projections and several dark and open beam images were taken for the tomography measurements. The exposure time for each image was 160 seconds to achieve a broad dynamic range in the resulting 16-bit images that were stored in tiff format. The open source image processing programs ImageJ and Tomviz were used for 3D rendering and visualization.

Each replica was marked with the drill to make them identifiable in the neutron tomograms. The replicas were then stacked together, wrapped in aluminum foil, and placed in a soda can that was the maximum size to fit inside the canister of the rotation stage. Four tomographic measurements were taken, each lasting approximately 24 hours. After one week, the radioactivity of the samples had declined enough for the replicas to be taken out of the building.

HISTORIC SHABTI OBJECT NUMBER	COMPOSITION-BODY (wt.%)							COMPOSITION-GLAZE (wt.%)							Max. grain size (µm)	Glaze layer thickness (µm)	Interparticle glass in the core	Observed glazing method
	SiO <sub>2</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	CuO	CaO	Al <sub>2</sub> O <sub>3</sub>	Cl	SiO <sub>2</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	CuO	CaO	Al <sub>2</sub> O <sub>3</sub>	Cl				
F93/10.29	95.1	1.1	0.5	0.5	0.2	1.6	1	90	5.2	2.6	1.8	0.3	1	0.5	250	400	No	Application
F93/10.34	97.8	0.3	0.1	0.3	0.2	1	0.5	97.4	0.5	0.4	0.2	0.5	0.5	0.7	150	400 - 600	No	Application
RBK14272	95.3	1.3	0.3	0.1	0.5	1.8	0.7	79.1	14.6	0.6	1.9	1.5	0.7	1.5	250	333	No	Application
F93/10.97	97.1	1	0.1	0.6	0.2	0.8	0.2	94.3	2.4	0.8	1.3	0.6	0.4	0.2	150	160 - 400	No	Application
AF127a	95.3	1.3	0.3	0	1.5	1.3	0.4	70.4	16	1.3	2	4.1	2.1	2	350	220 - 300	No	Exterior
BA276	95.2	1.5	0.3	0	0.6	1	0.5	73.5	13.3	1	7.2	2.2	0.8	1.5	300	250 - 400	No	Exterior
HIIM19	98.3	0.6	0	0	0.4	0	0.7	98.9	1.3	0.3	1.5	0.3	0	0.4	250	130	No	Exterior
AF42B	98.4	0.8	0	0	0.3	0	0.4	95.1	1.5	0.3	1.3	0.6	0.6	0.3	200	500 - 700	No	Exterior
<b>REPLICAS</b>																		
Efflorescence	95.1	3.3	0	2.4	1.3	0.8	0	92.1	3.3	0	2.5	1.2	0.7	0	< 250	200-500	Yes	Efflorescence
Cementation	98.1	0.6	0	0	0.8	0.5	0	78.8	8	2.6	4.4	5.4	0.1	1	< 250	100-300	No	Cementation
Application	92.1	3.2	1.6	2	0.9	0.6	0	91.1	2.8	0.9	3.5	0.7	0.7	0	< 250	1000	No	Application

**Table 2.** SEM-EDX results of cross sections of faience shabti and replicas embedded in epoxy resin. The concentrations of the oxides are normalized to 100% excluding the carbon which derives from the embedding medium.

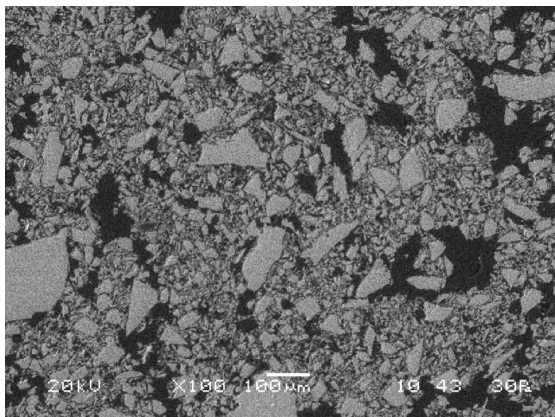
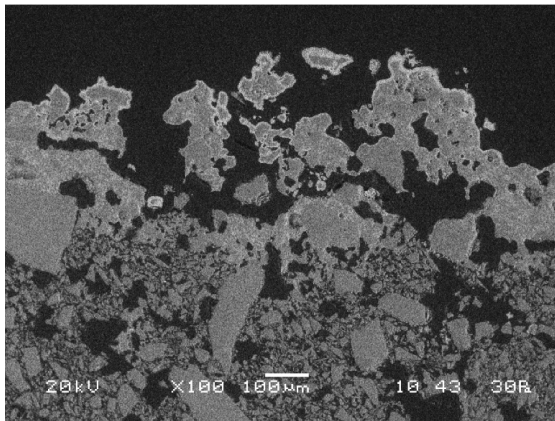
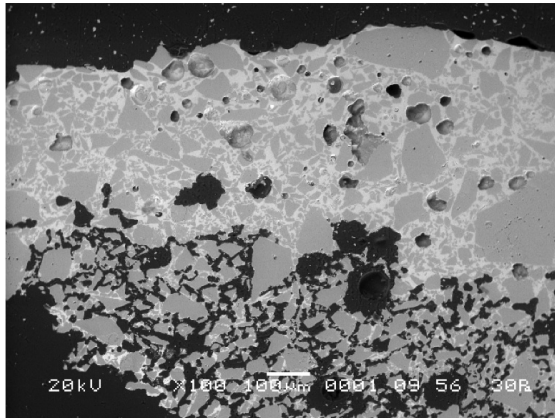
## RESULTS AND DISCUSSION

No soluble salts were detected in the sample material taken from the *shabtis* that were studied at the RMO. Visual inspection of the *shabtis* showed marks on the glaze, presumably from stands. On one of the objects, clear drip marks associated with application glazing were observed (De Regt 2017).

As it is difficult to establish the glazing method with the naked eye, Tite et al. (1983; 1986) have suggested microstructural criteria for SEM-EDX analysis. SEM-EDX analysis of the samples of the RMO *shabtis* showed low concentrations of alkali and copper and no signs of interparticle glass in the core (Figure 4 and Table 2). This shows these *shabtis* could not have been produced with efflorescence glazing (Liang et al. 2012, 3687-3688), in which alkali and copper are mixed into the core causing interparticle glass formation (Tite, Manti, and Shortland 2007, 1571). All eight

objects have rather thick glaze layers that range from 130 µm to 600 µm. According to Tite, Manti, and Shortland (2007, 1572), the glaze thicknesses of their cementation-glazed replicas were less than 50-200 µm. The Late Period *shabti* that was argued by Liang et al. (2012, 3688) to have been application glazed shows a glaze layer of 200-800 µm. The relatively thick glaze layers of the *shabtis* from the RMO suggest application glazing as well.

Although the thick glaze layers act as protective coatings, the cores of these objects are inconsistent and powdery. Cracks formed during manufacture expose the vulnerable core material (De Regt, 2017). Interior-glazed faience objects, on the other hand, possess a more cohesive internal structure due to the formation of interparticle glass in the core; however, the glaze layer is thinner and considered to be more fragile (Smith 1996, 846).



**Figure 5.** SEM back-scattered images of replica samples produced by efflorescence (above), cementation (center), and application (below) glazing

## Replicas

As determined with EDX, the component ratios of the replicas approach those of original faience (Table 2). The efflorescence-glazed replicas contain a high amount of interparticle glass in the core. The cementation-glazed replicas show limited interparticle glass towards the surface, suggesting that the glaze layer penetrates into the core material to some degree. Interparticle glass is absent from the application-glazed replicas. The morphology of the replicas adequately reflects that of historical faience objects, as can be seen on SEM back-scattered images of the replica samples, where quartz particles are visible as darker grey areas, glass phases as lighter grey areas, pores in black, and areas comprising heavier elements in white (Figure 5). The efflorescence-glazed replica shows partial vitrification of the core material.

## Distribution and depth of consolidation

The neutron tomograms were studied as individual slices, as well as in 3D composites, using Tomviz software. The brighter color visible in the cross sections is a measure of higher hydrogen density and thus consolidant density in that specific sub-volume (Figure 6). The penetration depth of PARALOID B-72 within the faience microstructure of each test piece was evaluated based on the brightness level intensity distribution. An overview of the maximum penetration depth for each replica is presented in Table 3.

PARALOID B-72 penetrated the faience cores to depths ranging from 0.2-1.4 cm. Penetration depth is often greatest in the exterior-glazed replicas.<sup>iv</sup> The deepest penetration is found in the application-glazed replicas, closely followed by the cementation-glazed replicas. The consolidant penetrated considerably less far into the efflorescence-glazed replicas. The resin is less evenly distributed into the microstructure of the efflorescence-glazed replicas, relating to the vitrification of the core material.

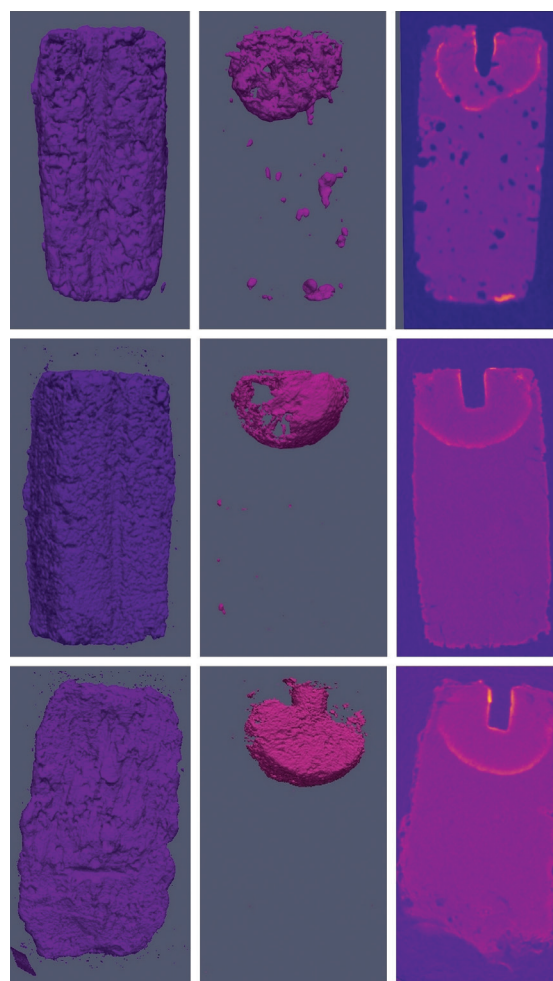


With higher concentrations of PARALOID B-72, the penetration depth was often reduced (Table 3). This reduced ability to penetrate the faience microstructure is expected to be related to the higher viscosity of the solubilized resin. Consolidation solutions including ethanol consistently resulted in deeper penetration of the resin, however the differences were very small.

The noticeable differences in consolidant penetration depth between interior- and exterior-glazed faience reflect differences in porosity, which is influenced by interparticle glass formation in the core material. As exterior glazing produces glass phases only at the surface and in the interaction zone, but not in the core material, the body is of a less compact nature. This is likely to facilitate the transport of the resin towards the core.

## CONCLUSION

This research elucidates penetration depth of PARALOID B-72 as a consolidant for faience objects in relation to the glazing technique used. Neutron tomography is an extremely successful technique for observing both penetration depth and distribution of the PARALOID B-72 in faience. The addition of ethanol to the PARALOID B-72 solution made no statistically significant difference in penetration depth. A considerably less effective penetration of PARALOID B-72 was observed with higher concentrations, presumably due to increased viscosity of the resin solution. Consolidant penetration depth and distribution are also influenced by an object's glazing method. Penetration depth was considerably higher in exterior-glazed replicas, reflecting a difference in porosity arising from differential interparticle glass formation in the core material.



*Figure 6. Neutron tomograms from efflorescence- (above), cementation- (center), and application- (below) glazed replicas showing the outer surfaces (left), consolidant within the faience microstructures (center), and individual slices (right)*

<b>GLAZING METHOD</b>	<b>PERCENT CONCENTRATION (w/v) OF PARALOID B-72</b>	<b>SOLVENT</b>	<b>PENETRATION DEPTH (cm)</b>
Efflorescence	3	Acetone	1.0
Efflorescence	3	Acetone + Ethanol	0.5
Efflorescence	5	Acetone	0.5
Efflorescence	5	Acetone + Ethanol	0.6
Efflorescence	10	Acetone	0.3
Efflorescence	10	Acetone + Ethanol	0.3
Cementation	3	Acetone	0.9
Cementation	3	Acetone + Ethanol	0.8
Cementation	5	Acetone	1.1
Cementation	5	Acetone + Ethanol	0.9
Cementation	10	Acetone	0.6
Cementation	10	Acetone + Ethanol	0.6
Application	3	Acetone	0.9
Application	3	Acetone + Ethanol	1.4
Application	5	Acetone	1.0
Application	5	Acetone + Ethanol	0.9
Application	10	Acetone	0.6
Application	10	Acetone + Ethanol	1.0

*Table 3. Penetration depth of PARALOID B-72 as assessed with neutron tomography*

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## NOTES

<sup>i</sup> In the New Kingdom, antimony-, lead-, and cobalt-containing oxides were used as well (Nicholson 1993, 30-31).

<sup>ii</sup> Preliminary tests were carried out by De Regt in 2018.

<sup>iii</sup> In ancient Egypt, earthenware jars were used (La Delfa, Formisano, and Ciliberto 2008, e114).

<sup>iv</sup> An exception is formed by the 3 percent solution in acetone, as the consolidant penetrated the deepest into the efflorescence-glazed replicas.

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