



Chemical and physical characterization of mortars and plasters from a Coptic paternoster: new excavation in the temple of Ptolemy XII, Sheikh Hamad, Athribis, Sohag, Egypt

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ABSTRACT

The present study focuses on the compositional characterization of mortars and plasters from a Coptic paternoster excavated in the temple of Ptolemy XII, Sheikh Hamad, Athribis, Sohag, Egypt. A physiochemical study using analytical techniques such as USB microscope, XRPD, FTIR, DTA and SEM-EDX was carried out. This study, on one hand made characterization of the physical properties and chemical composition of joint mortars and different plasters used in this period; on the other hand, it defined in detail the hydraulic mortars or plasters of this period. All analytical methods indicated that the brick joint mortar and whitish plaster layers made of calcite (lime) with the addition of quartz (sand) and sometimes brick gravels. The reddish plaster layers consist of calcite (lime) with quartz (sand) and brick dust which was used as natural pozzolanic additive and that make the plasters more suitable for their use as water tank external plasters. The most important result related to the internal dark grayish plaster layer sample of the big upper tank is the detection of calcite, hedenbergite, quartz and hatrurite which confirms the hydraulicity of the lime plaster.

Keywords: ancient mortar; plaster; Athribis; pozzolanic additives; Coptic paternoster.

INTRODUCTION

The Ptolemaic temple site of Athribis is situated near to the city of Sohag around 200 km north of Luxor on the west bank of Nile Valley (Figure 1a) (Leisen et al., 2008). The name of this city Athribis should not be confused with Athribis in Delta, the modern Benha. Delta town was Hat-her-ab, “the fortress in the midst” of the plain; the southern town is Hat-repyt, “the fortress of Repyt”; a lion-headed goddess, who is scarcely known elsewhere.

Despite of the similarity of Auletes temple plan with the temples of Dendera and Edfu, it differs from any temple yet known because of the Greek influence in its colonnade (Figure 1b, Figure 2).

It is obvious that the source of temple stone is cut from the near cliff (Petrie, 1908). Leisen et al. (2008) studied well the building stones of the temple. They returned

back the building stones of the Repit temple of Athribis to several limestone varieties extracted from the mountain range adjacent the west of the temple site. This mountain range may be attributed to the Drunka Formation of the Early Eocene (shallow marine sediments).

Some of the specialists attributed the destroyed state of the temple to a collapse caused by ancient earthquake. El-Sayed et al. (2008) and Karakhmian et al. (2010) described several destructive earthquakes in Egypt history. Really, we cannot neglect the historical utilization of the temple, as Petrie (1908) mentioned that this temple had undergone several changes such as converting the temple into a church as the Figures of gods in relief on the inside were chopped away, and the whole whitewashed. After that, it was used as a quarry for Deir Amba Shenudeh. Then it became a cattle pen, more quarrying was done,

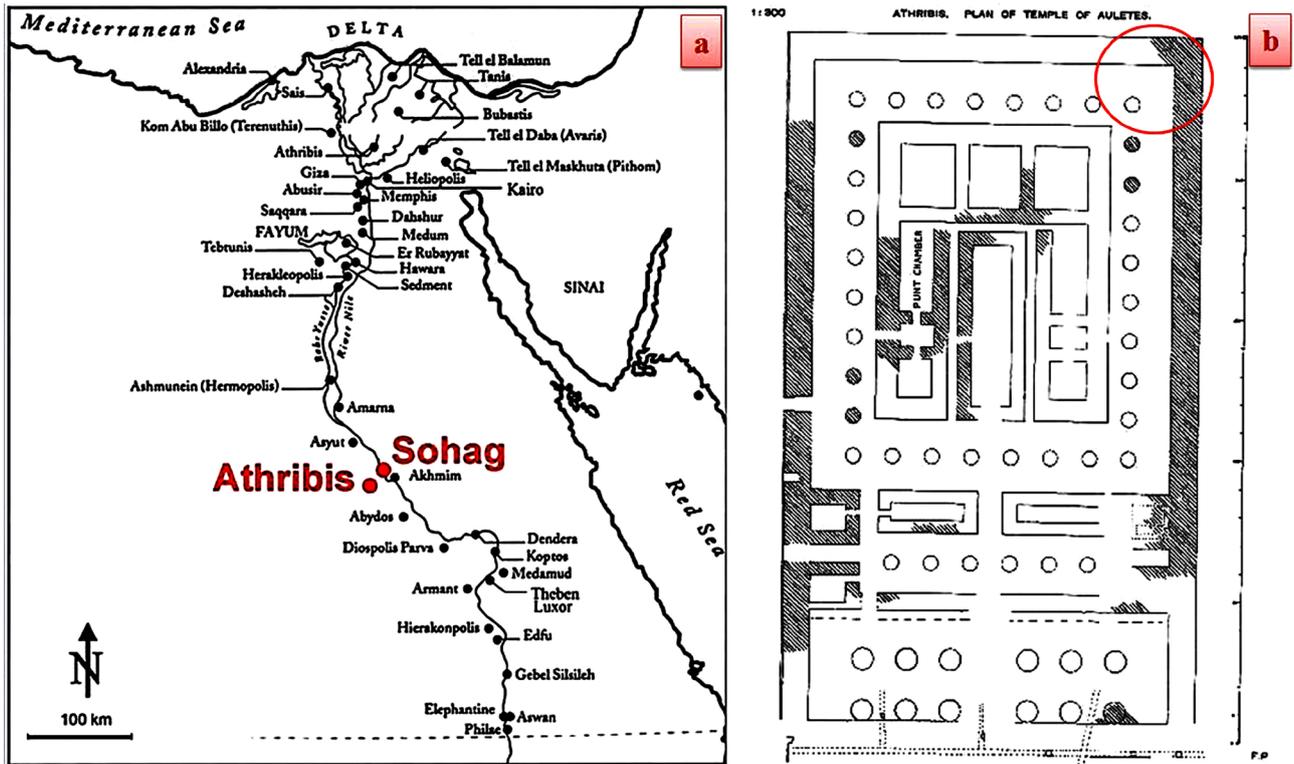


Figure 1. a) Map shows the location of the Athribis temple in Upper Egypt, (Leisen et al., 2008), b) the Petrie plan of the temple of Auletes (Petrie, 1908), the red circle shows the location of Coptic paternoster addition.



Figure 2. a and b show the location of Coptic paternoster addition.

rubbish from the town was heaped in the ruins, and lastly it was hidden by blown sand (Petrie, 1908).

The temple was subjected to three excavations, firstly by Petrie in the beginning of the 20th century, secondly, the excavations made by the Supreme Council of Antiquities of Egypt worked in Athribis (from 1983 until 1996), finally, the excavations made by German-Egyptian project (since 2003) (Leisen et al., 2008).

This research focuses on the physicochemical study of plasters and mortars collected from a new excavated Coptic paternoster through utilizing analytical techniques such as USB microscope, XRPD, FTIR, DTA and SEM-EDX. These techniques will help in identifying the binding materials used in the plasters and mortars. Also, this study will facilitate the identification of the additives used to enhance the hydraulicity of the used mortars and

plasters for this kind of water structures. Finally, the study will help in outlining a proposal for the conservation procedures of the paternoster.

MATERIALS AND METHODS

The sampling procedure for plasters and mortars has been carried out by careful collecting of eight representative samples by a micro scalpel from deteriorated and separated parts from the paternoster (Figure 3). All of the analyzed and investigated samples were collected from areas with less historic and aesthetic values and sometimes the same sample was used for more than one analysis or investigation (Table 1). These samples were studied to identify the chemical composition, deterioration aspects and to discover the used building materials techniques in this period.

Four plaster samples were investigated by handheld USB digital microscope (model PZ01- made by Shenzhen Supereyes Tech) to study the stratigraphy of plaster layers and the additives used for the enhancement of their properties. X-ray diffraction patterns of the mortars and plasters powders were obtained using a diffractometer

type Philips PW 1840, operated at 35 kV, using a Cu K α radiation wavelength of 1.540598 Å. The reference data base used for matching is PDF-4+2015RDB. To confirm the XRPD results, FTIR spectroscopy was used. IR spectra were obtained using a spectrophotometer JASCO FT/IR-4100 typeA. The resolution is: 4 cm⁻¹, Region 4000:499 cm⁻¹ and scanning speed is: auto (2mm/sec). In addition to the previous techniques, differential thermal analysis (DTA) by a Shimadzu DTA 50, using a temperature rate of 10 °C/min, hold temperature=1000 °C, atmosphere: nitrogen with rate flow 30 ml/min was carried out to identify the pozzolanic additives used in the plasters. The microstructure and morphology of mineral constituents in the stuccos were recorded with a scanning electron microscope: Quanta 250 FEG (Field Emission Gun) attached with EDX (Energy Dispersive X-ray) unit, with accelerating voltage 30 kV. The measurements were carried out under low vacuum conditions as samples did not show charging effects. In this way, it was possible to avoid coating the samples with a highly conductive thin film of gold. EDX microanalysis was operated to obtain information on the elemental composition of the samples.



Figure 3. a, b and c shows the samples locations of the Coptic paternoster.

Table 1. Description of the analyzed and investigated samples collected from the Coptic paternoster in the temple of Ptolemy XII, Athribis, Sohag, Egypt.

Sample no.	Investigation and analysis methods	Sample description (all samples locations were shown in Figure (3))
(3)	XRD, SEM-EDX	Joint mortar sample from the upper part of the south wall of the big tank
(4)	XRD	Joint mortar sample from the upper part of the east wall of the big tank
(5)	XRD, FTIR	The first white plaster layer sample from the middle part of the big tank eastern wall
(6)	USB digital microscope, XRD	The first white plaster layer sample from the southern wall of the upper big tank
(7)	USB digital microscope, XRD	The outer plaster layer sample of the southern-western corner of the upper big tank
(8)	XRD, FTIR, SEM-EDX	The second reddish plaster layer sample of the eastern wall middle part of the upper big tank
(9)	USB digital microscope, XRD, FTIR, SEM-EDX	The third reddish plaster layer sample of the eastern wall middle part of the upper big tank
(10)	USB digital microscope, XRD, DTA, SEM-EDX	The internal dark gray plaster layer sample of the big upper tank

RESULTS AND DISCUSSION

Investigation by USB digital microscope

The observations made by USB digital microscope revealed the following results (Figure 4):

- Figure 4 (sample 6) shows some particles of brick in the first layer of the white plaster layer sample from the southern wall of the upper big tank. This addition may be made to give the air lime mortar some hydraulic properties to be suitable for its use as a plaster in water tank. However the big pieces of brick may be used as a filler material for plaster (See XRD analysis results of sample X6).

- Figure 4 (sample 7) shows the outer two plaster layers sample of the southern-western corner of the upper big tank. As can be seen in the image; the inner layer of plaster is rich in brick powder which was added to increase mortar hydraulicity.

- Figure 4 (sample 9) shows the reddish color of the third reddish plaster layer sample of the eastern wall

middle parts of the upper big tank, this color is related to the addition of brick powder and its fragments. The main purpose of this addition is to make the tank suitable for use as water tank by giving some hydraulic properties to the used mortar.

- Figure 4 (sample 10) shows the cross-section of the plaster layers of the internal face of the big upper tank, which consists of three layers and can be characterized by their color; (1) the whitish internal layer, (2) the reddish layer because of the addition of brick powder and (3) the dark-grayish layer which its color may be related to the addition of pozzolanic material to the lime.

X-ray powder diffraction (XRPD)

XRPD patterns results of the analyzed plasters and mortars samples are resumed in Table 2. They indicate the following results of their semi-quantitative analysis of their mineralogical components:

- XRD analysis of joint mortar samples (X3a, X3b, X4a

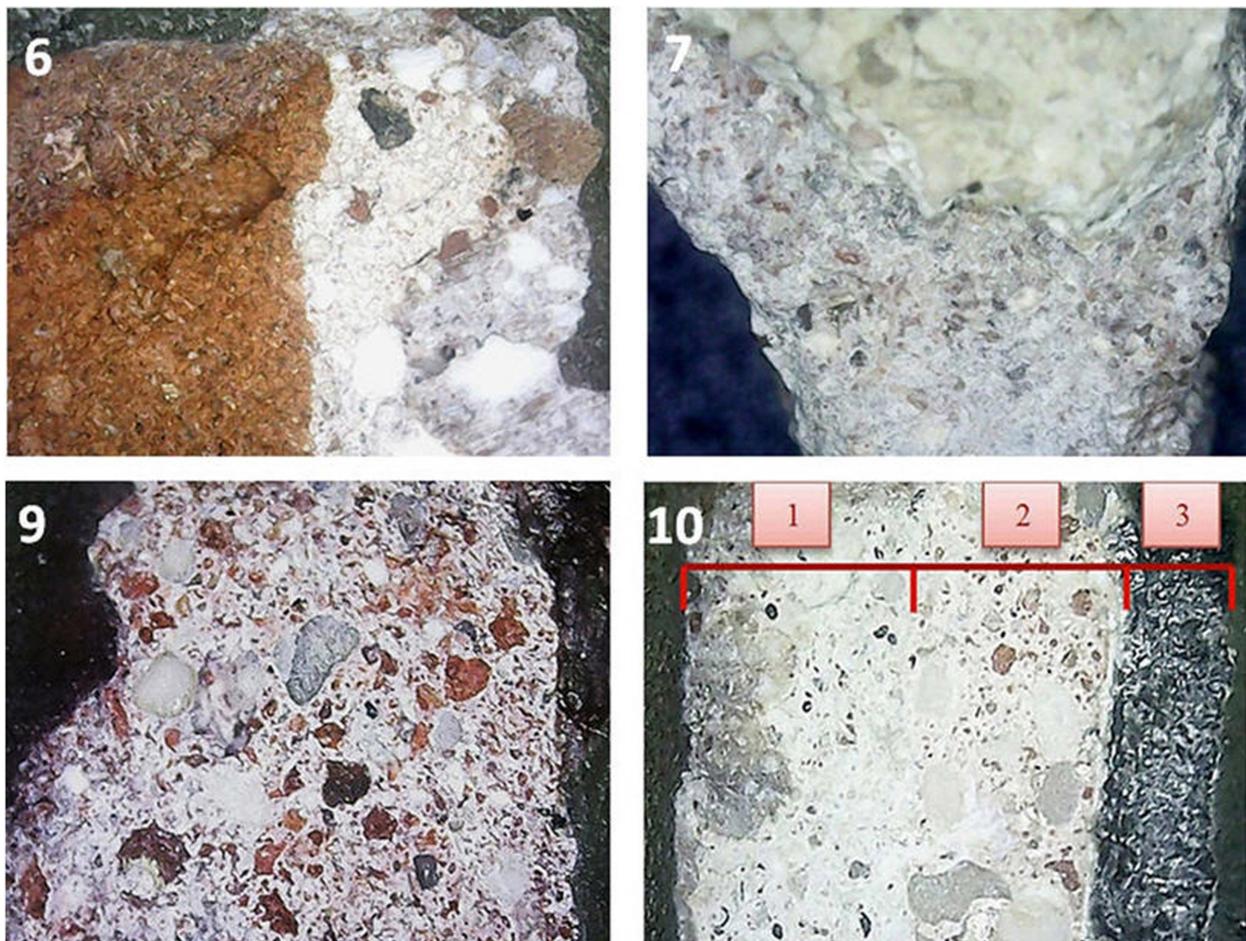


Figure 4. USB digital microscope results of some samples collected from the Coptic paternoster in the temple of Ptolemy XII, Athribis, Sohag, Egypt.

and X4b) revealed that calcite is the main component and the binding material of these mortars samples. As well as quartz as minor constituent with the exception of its high percentage in sample X3b which may be related to poor mixing of mortar or to deterioration affecting the binding materials through dissolution and leaving sand (Kamel et al., 2015a). The presence of anhydrite traces in joint mortar sample X3b can be explained as an impurity related to the limestone used as a source for lime (Kamel, 1986) or as gypsum impurity which it was transformed to anhydrite because of the firing temperature of lime i.e., gypsum can be transformed to insoluble anhydrite in temperatures lower than which were used for lime production (Kamel et al., 2015b; Charola and Centeno, 2002).

Detection of portlandite may be related to the traces that have not been carbonated because of the relative shortage of carbon dioxide in the inner parts of joint mortar i.e., carbon dioxide is essential for carbonation of lime (Elert et al., 2002) (Figs. 5 and 6).

• XRD analysis of plaster samples (X5a, X5b, X6, X7a, X7b, X8a, X8b and X9b) resembles the results of the joint mortars, with the appearance of hematite as traces in samples (X6, X7b, X8a and X9b). Hematite is a deliberate additive to lime mortar where ground bricks have been used in mortar mixes since ancient times. Although the chemistry of the pozzolanic reaction may not completely have been understood before the 19th century, experience had proven that the addition of powdered bricks to lime mortars can improve the hydraulic properties (Rogers, 2011). These materials are called as Homra in Arabic

countries.

Heating of the raw materials used in the manufacture of bricks results in the appearance of pozzolanic amorphous substances like aluminosilicates (such as metakaolin). When these materials are subjected to heating at temperature roughly between 450 and 800 °C, these amorphous substances react with lime to produce calcium silicate hydrate and/or calcium aluminate hydrate at the brick-lime interface and at the pores of the bricks.

Formation of the interlocking crystals of these products improves the strength of the mortars and plasters which, in turn, justifies their use in the construction of many historic buildings since ancient times (Böke et al., 2006) (Figs. 7 to 11).

• XRD analysis of the internal dark gray plaster layer sample (X10) of the big upper tank of the paternoster proved that it consists of calcite, hedenbergite, quartz and hatrurite. The detection of hedenbergite and hatrurite confirms the hydraulicity of the mortar. As Hedenburgite may be used as a pozzolanic slag additive (Piatak et al., 2015; Dariz and Schmid, 2017), or may be related to the addition of brick powder which is recognized as pozzolanic material in ancient Rome where it was used as a hydraulic additive to lime mortars. (Kepperta et al., 2017). The appearance of hatrurite may be attributed to the reaction of calcium hydroxide with quartz (Labiadh et al., 2009). Other researchers attributed the presence of hatrurite to the addition of marble powder (Buyuksagisa et al., 2017) (Figure 12).

Table 2. The approximate XRD analysis results of plaster, mortar and pottery water pipes of paternoster.

Sample/ component	Ca	Qu	Gy	An	He	Port	Hat	Hed
Sample(3)X3a	+++	++	-	-	-	-	-	-
Sample(3)X3b	+++	+++	-	+	-	+	-	-
Sample(4)X4a	+++	++	-	-	-	-	-	-
Sample(4)X4b	+++	++	-	-	-	-	-	-
Sample(5)X5a	+++	++	-	+	-	-	-	-
Sample(5)X5b	+++	++	-	-	-	-	-	-
Sample(6)X6	+++	+++	-	-	+	-	-	-
Sample(7)X7a	+++	+++	-	-	-	-	-	-
Sample(7)X7b	+++	+++	-	-	+	-	-	-
Sample(8)X8a	+++	++	-	-	+	-	-	-
Sample(9)X9b	+++	+++	-	-	+	-	-	-
Sample(10)X10	+++	++	-	-	-	-	+	+++

Ca=calcite; Qu=Quartz; Gy=Gypsum; An=Anhydrite; He= Hematite; Por=Portlandite; Hat=Hatrurite; Hed=Hedenbergite; - = not determined; + = traces; ++ = minor constituent; +++ = major constituent.

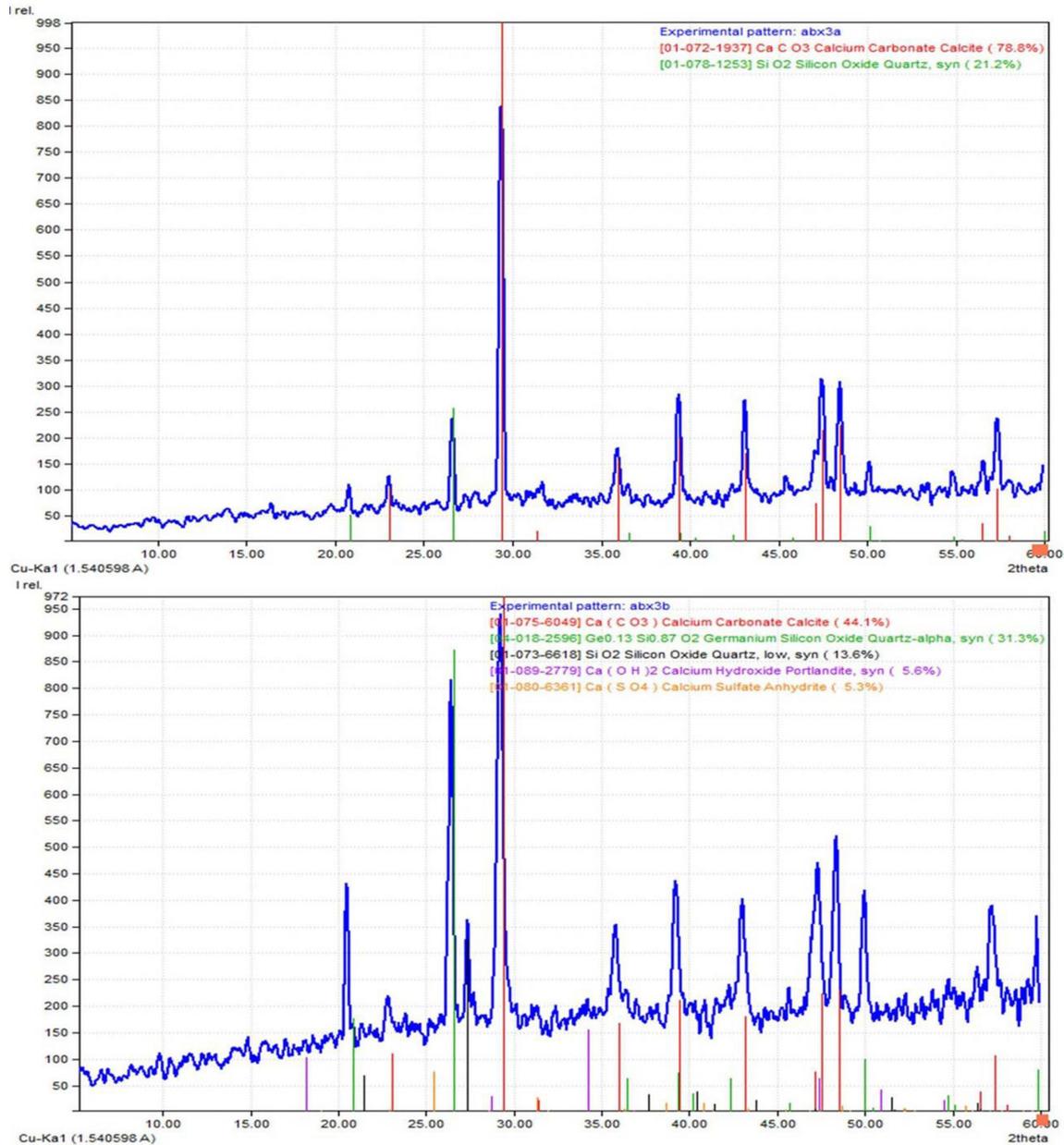


Figure 5. X-ray diffraction patterns of the joint mortar sample from the paternoster upper parts of the south wall of the big tank proved that it consists essentially of calcite, quartz and traces of portlandite and anhydrite.

Fourier transform infrared spectroscopy (FTIR)

Despite of the fact that FTIR is usually used to analyze organic compounds, this technique is also useful for characterizing inorganic substances. A qualitative analysis from the characteristic frequencies provides an information to identify chemical constituents in a compound. In addition, a correlation of FTIR and XRD may be very helpful (Galván-Ruiz et al., 2009). With the aid of (Domenech et al., 1997; Galva-Ruiz et al., 2009; Falchi et al., 2013) FTIR results of the three selected

plaster samples were as follows: calcium carbonate was identified in the three samples (9, 8 and 5) through the C=O stretching band of CO_3^{2-} group of calcium carbonate at about 1795 cm^{-1} and 1440 cm^{-1} plus the O-C-O bending bands of carbonate group at about 875 cm^{-1} and 710 cm^{-1} (Mahmoud et al., 2011). FTIR spectra of samples 8 and 9 (Fig. 13 IR9 and IR8) show an intense broad band at $1000\text{--}1100\text{ cm}^{-1}$ corresponding to C-S-H vibrations of hydraulic compounds. These hydraulic compounds may be related to the interaction between crushed-brick or

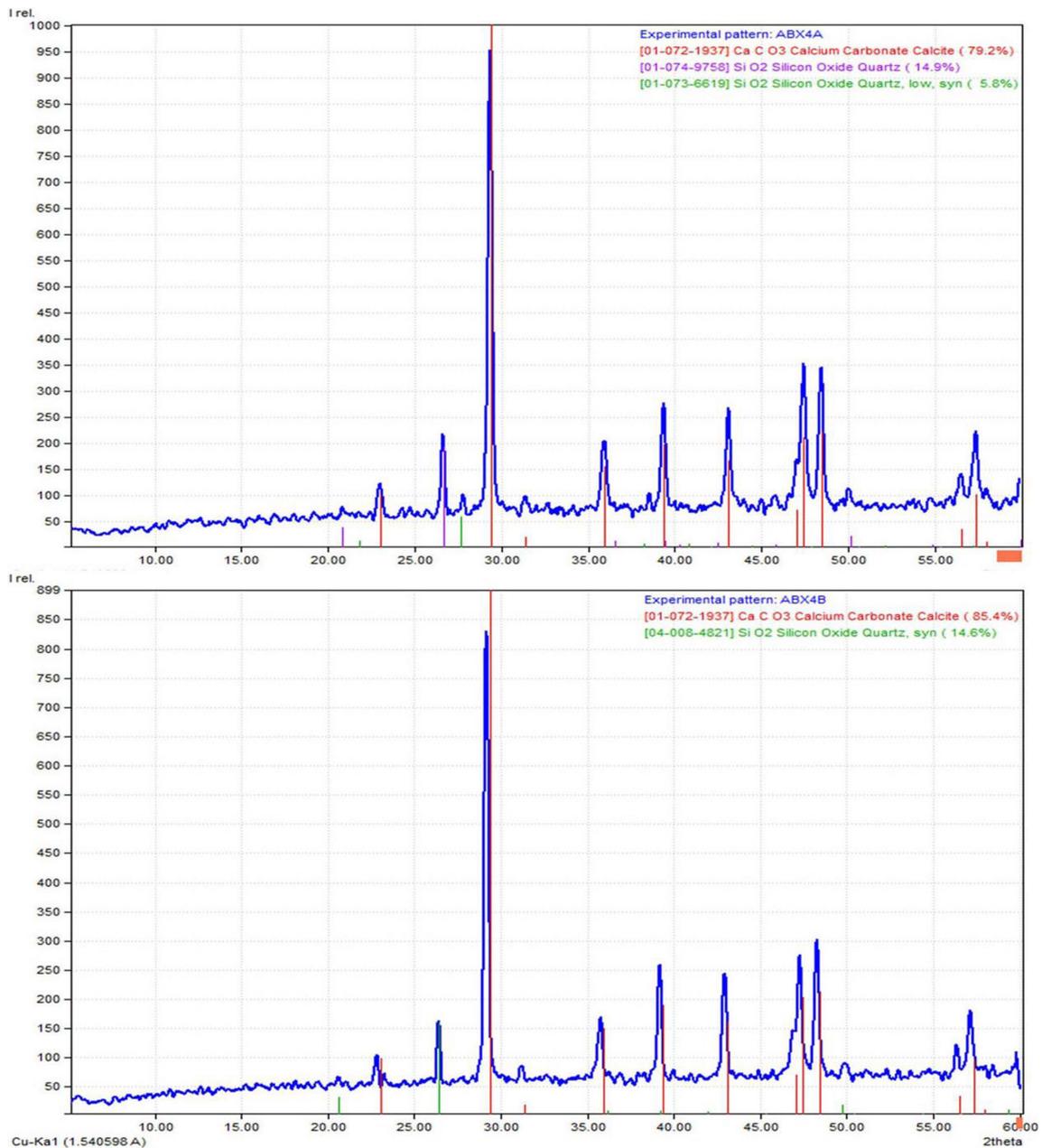


Figure 6. X-ray diffraction patterns of the joint mortar sample from the patenoster upper parts of the east wall of the big tank proved that it consists essentially of calcite and quartz .

pozzolanic aggregate additives and lime. Presence of hydraulic compounds was also confirmed by the results of XRD analysis (Maravelaki-Kalaitzaki et al., 2010; Diekamp et al., 2012).

Quartz was identified by the vibrations at about 780 cm^{-1} and the asymmetric Si-O-Si stretching band at $1000\text{-}1100\text{ cm}^{-1}$ in the three samples (Bosch et al., 2002; Derrick et al., 1999). Presence of quartz may be related to the traces of sand which was detected by XRD analysis (Figure 13).

Differential thermal analysis (DTA)

Differential thermal and thermogravimetric analyses are suitable to establish the characteristics of ancient mortars as it is easy to detect the main components, the nature of the aggregate and other aspects, with a small quantity of sample (Montoya et al., 2004). The (DTA 10) sample presents a DTA curve characterized by two important peaks. The first one was at $123.58\text{ }^{\circ}\text{C}$ which is related to the region of pozzolanic product dehydration. The second peak appeared in the range between $700\text{:}800\text{ }^{\circ}\text{C}$ which is

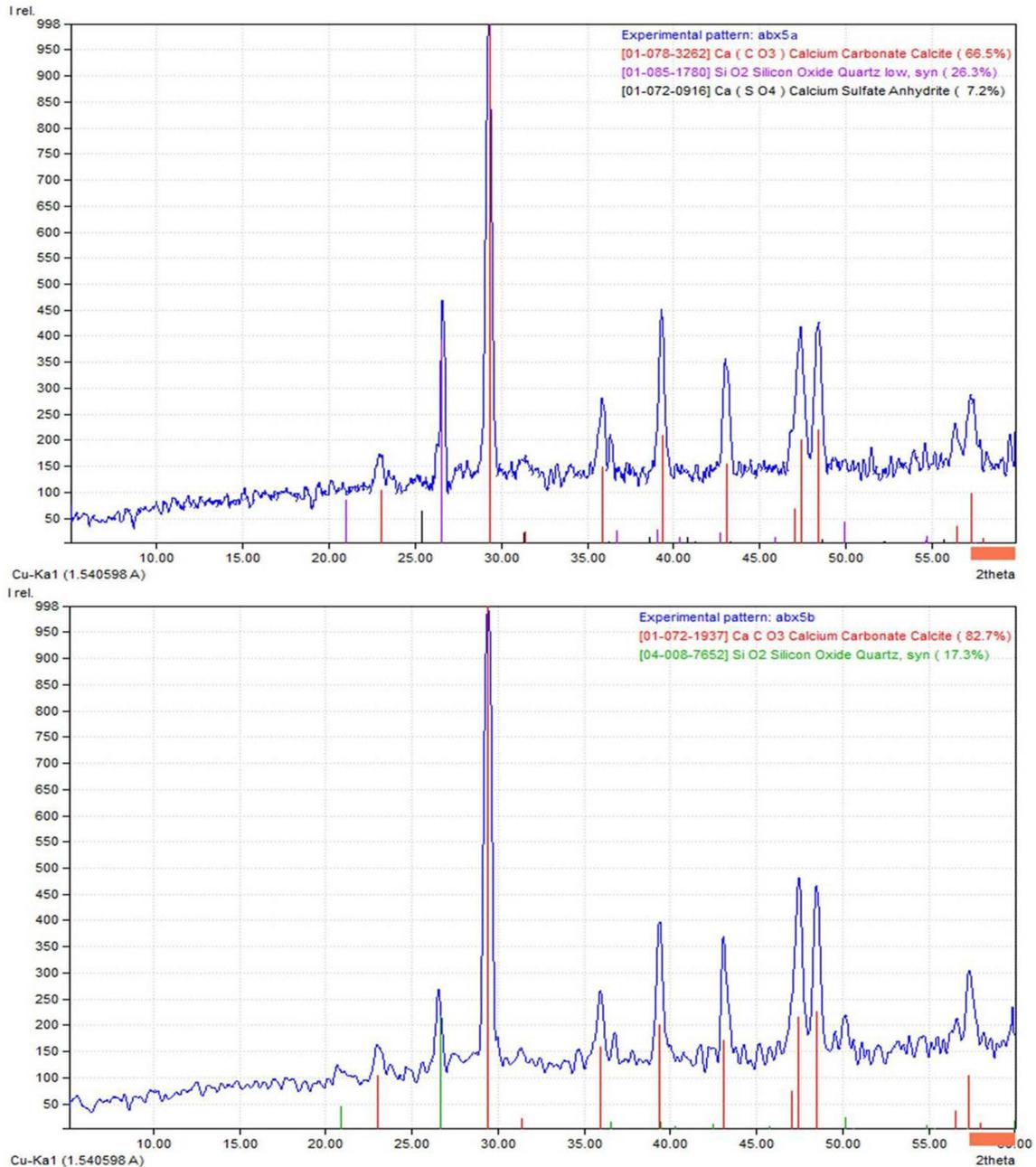


Figure 7. X-Ray diffraction patterns of the first white plaster layer sample from the middle parts of the big tank eastern wall proved that it consists essentially of calcite, quartz and traces of anhydrite.

related to decarbonation of calcite and the weight loss associated has been originated by the $\text{CO}_2(\text{g})$ evolution: $\text{CaCO}_3 \rightarrow \text{CaO} + \text{CO}_2$.

The (DTA 9) sample showed a peak at 686.72°C which may be related to the decomposition of calcite (Figure 14). Montoya et al. (2004), Alvarez et al. (2000) and Gameiro et al. (2014) mentioned that low temperature of the decomposition of calcite may give an evidence for the presence of dolomite traces (Figure 14).

Scanning electron microscopy (SEM) with energy dispersive X-ray analysis (EDX)

- The results of EDX analysis of joint mortar sample 3 (SEM-EDX 3a and 3b) show a high proportion of Si and O in sample 3a (Figure 15) indicating the occurrence of quartz (sand), while the results of sample 3b, (Figure 15) show a high proportion of Ca, O, C proving the occurrence of calcite as a major component, Si ratio testifies to the occurrence of quartz (sand). Finally, the presence of Fe, Al

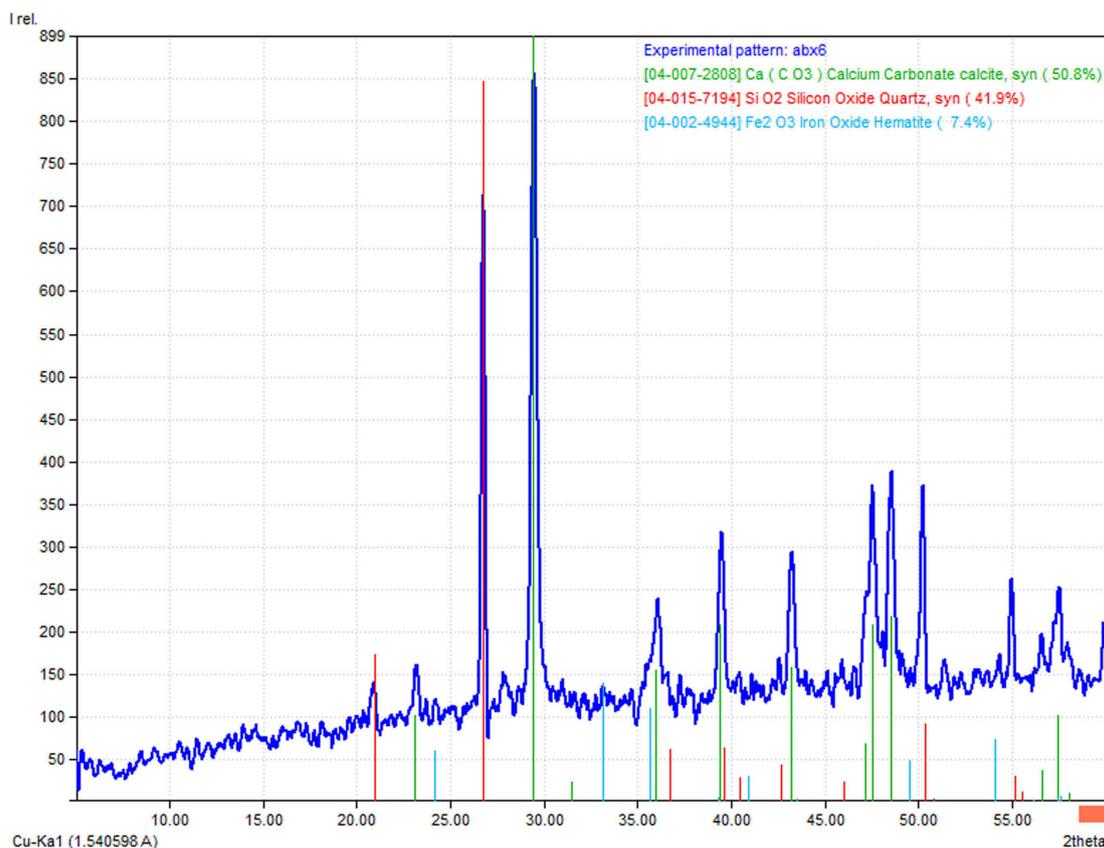


Figure 8. X-ray diffraction patterns of the first white plaster layer sample from the southern wall of the upper big tank proved that it consists essentially of calcite, quartz and traces of hematite which may be related to the addition of brick powder.

and Si proves the presence of traces of pozzolanic materials; that may be related to the addition of ground bricks.

- EDX analysis results of the reddish plaster layer sample 8 (SEM-EDX 8a) (Figure 16) show the presence of (Fe) element which gives an evidence of the occurrence of brick powder which was added to give the plaster its reddish color. This result was confirmed by the detection of hematite by XRD analysis. SEM-EDX 8b (Figure 16) reveals the presence of a high proportions of Ca, O, C and Si indicating that the plaster is a lime mortar which consists of calcite, quartz and the addition of brick powder.

- EDX analysis results of the third reddish plaster layer sample of the eastern wall middle parts of the upper big tank (SEM-EDX 9a and 9b) show the presence of Ca, Si, Al and Fe which make this sample resemble sample 8, but the appearance of S ion may testify to traces of one of the gypsum phases as an impurity (Figure 17).

- EDX results of the three layers sample which was collected from the internal face of the big upper tank show the following results:

- The whitish internal layer sample (SEM-EDX 10 Layer1) shows a high proportion of calcium Ca as the

major ion contained, which refers to the existence of calcite as a binder (Figure 18).

- The reddish layer sample (SEM-EDX 10 Layer 2) shows the existence of (Fe) and (O) which testifies to the occurrence of brick powder as an additive (Figure 18).

- The grayish layer sample (SEM-EDX 10 Layer 3) shows the existence of high proportions of Ca, Si as well as Al and Fe that testifies the occurrence of hedenbergite and hatrurite minerals. These minerals may be related to the addition of pozzolanic materials to the lime to yield a hydraulic mortar compatible with the usage of the water tank (Figure 18). This conclusion was confirmed by the results of XRD and IR analyses.

- The superficial sample of the grayish layer (SEM-EDX 10 x) shows a proportion of S ion which may be testified to traces of one of the gypsum phases as an impurity or related to sulfation of lime mortar (Cultrone et al., 2008) (Figure 18).

CONCLUSION

The chemical-physical study of mortars and plasters samples of the Coptic paternoster allowed obtaining of

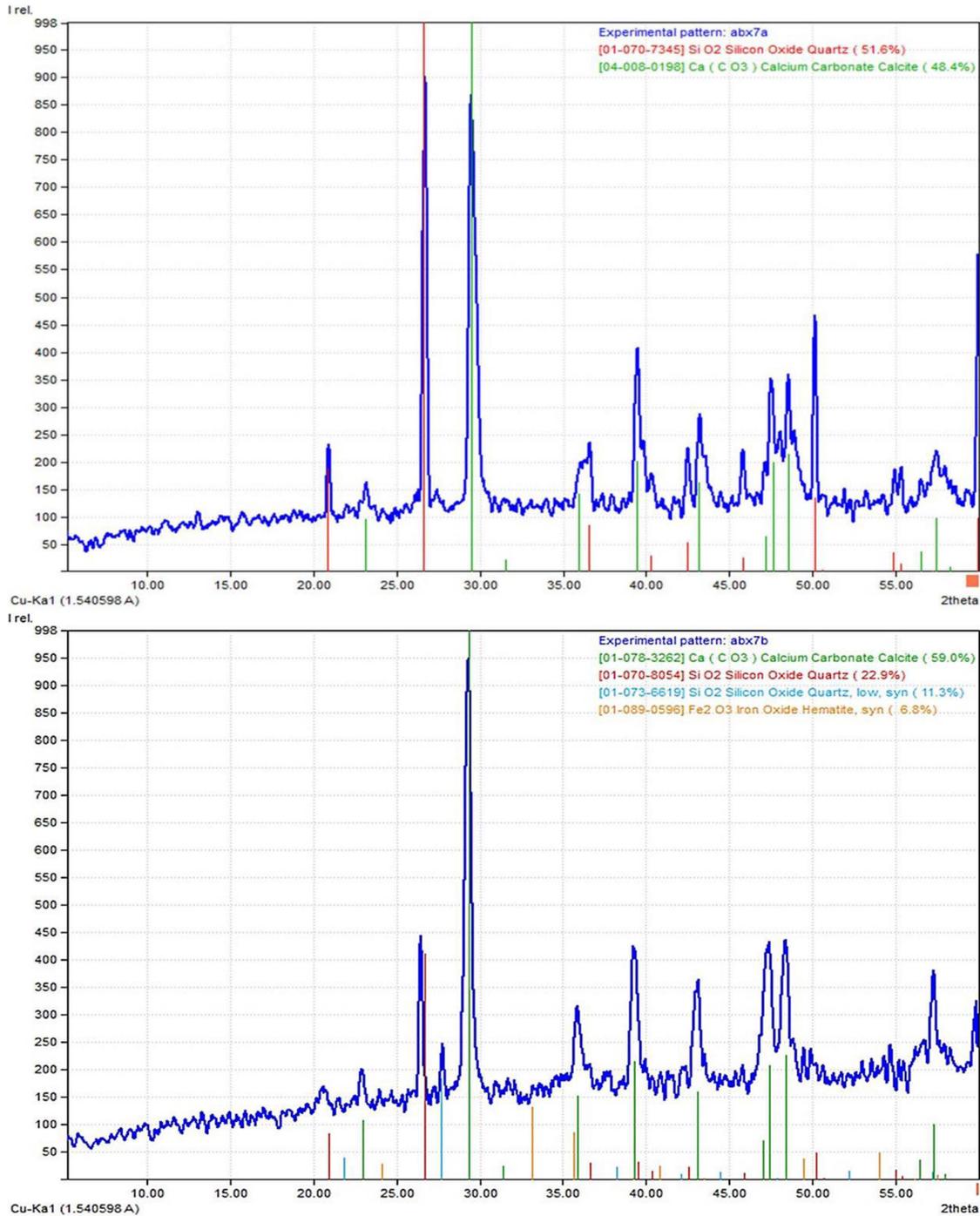


Figure 9. X-ray diffraction patterns of the paternoster outer plaster layer sample of the southern-western corner of the upper big tank proved that it consists essentially of calcite, quartz and traces of hematite which may related to the addition of brick powder.

interesting information about the composition of these building materials and the identification of hydraulic plasters in Coptic period in Egypt.

In particular, we detected the prevalent use of lime mortar and plasters and the use of homra (brick powder)

as a pozzolanic additive to create hydraulic mortars in the Egyptian Coptic period, especially in the external surfaces of paternoster walls. In the internal surfaces of water tanks, we detected the occurrence of hedenbergite and hatrurite minerals which may be related to the addition of

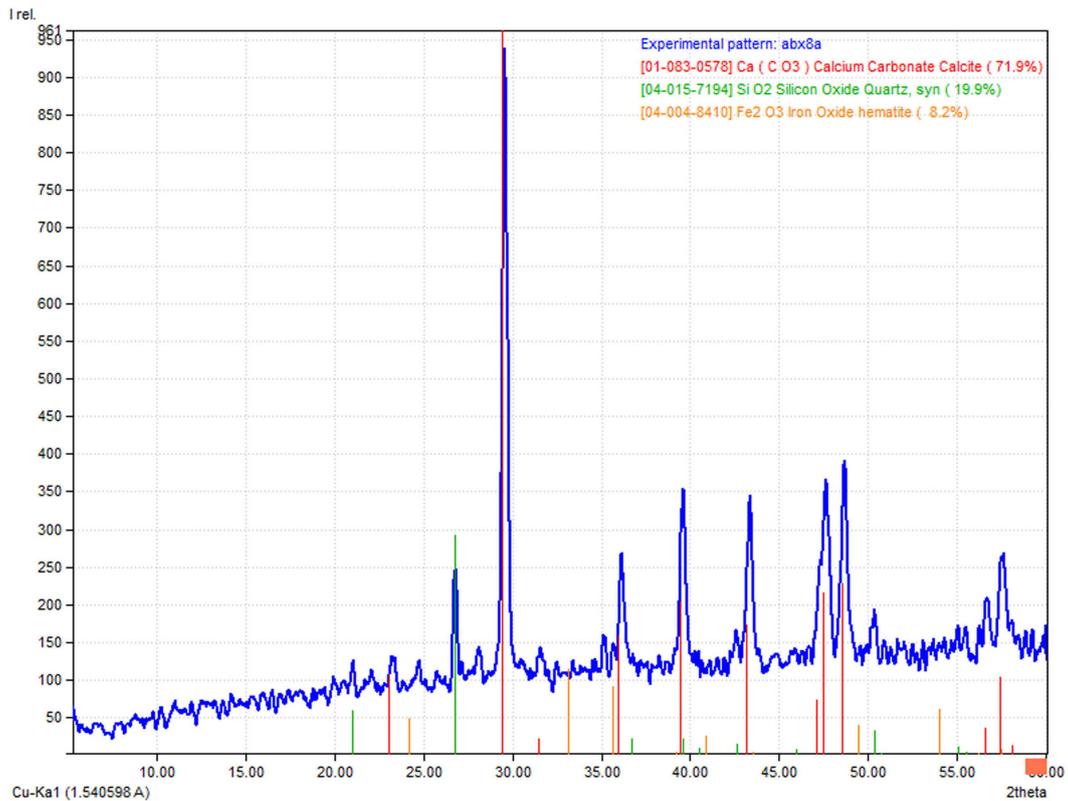


Figure 10. X-ray diffraction pattern of the second reddish plaster layer sample of the eastern wall middle parts of the upper big tank proved that it consists essentially of calcite ,quartz and hematite which may related to the addition of brick powder which gave the plaster its reddish color.

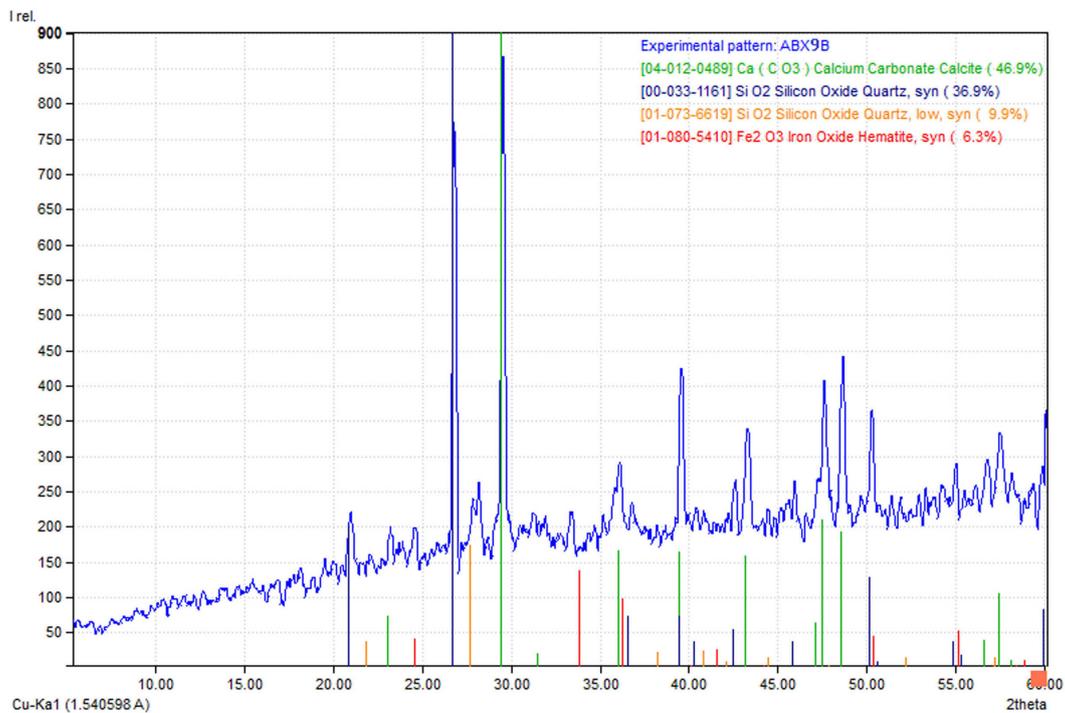


Figure 11. X-ray diffraction pattern of the third reddish plaster layer sample of the eastern wall middle parts of the upper big tank proved that it consists essentially of calcite, quartz and hematite.

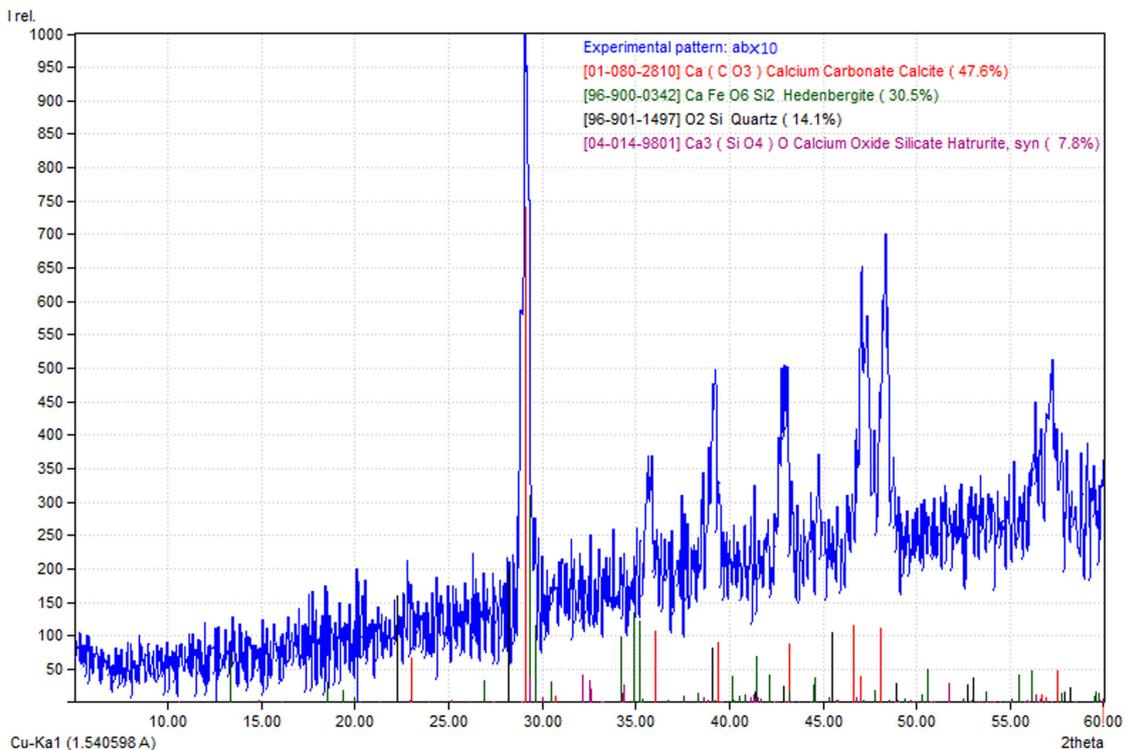


Figure 12. X-ray diffraction pattern of the internal dark gray plaster layer sample of the big upper tank of the paternoster proved that it consists of calcite, hedenbergite, quartz and hatrurite.

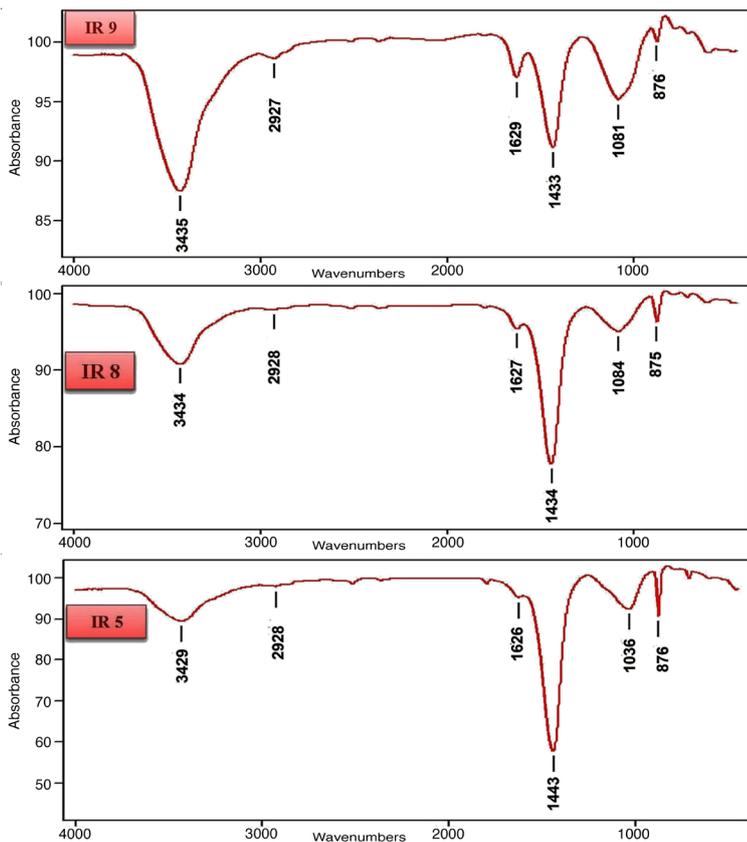


Figure 13. (IR9, IR8 and IR5) Infrared spectra of three plaster samples from the Coptic paternoster confirmed the results of XRD analyzes.

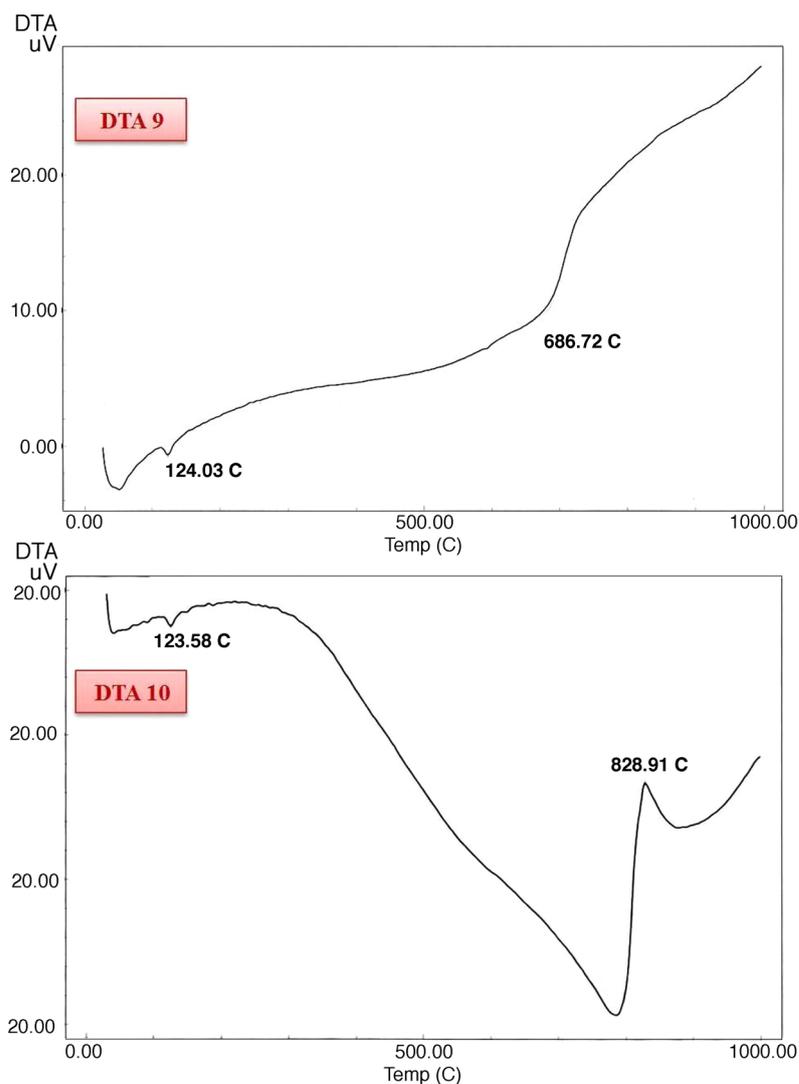


Figure 14. DTA curves of samples 9 and 10, their results are compatible with XRD and IR results.

other pozzolanic materials.

All previous results were proved by USB microscope XRPD, FTIR, DTA and SEM-EDX; since the results of XRPD analysis revealed that calcite is the main component of the brick joint mortar and the whitish plaster, as well as quartz (lime mortar) and sometimes brick gravels were used. The reddish plaster layers consist of calcite (lime) with quartz (sand) and brick dust which was used as a pozzolanic additive to give the plaster some hydraulic properties through the reaction of lime with brick dust by time to yield calcium silicates that makes the plasters more suitable for their use as water tank external plasters.

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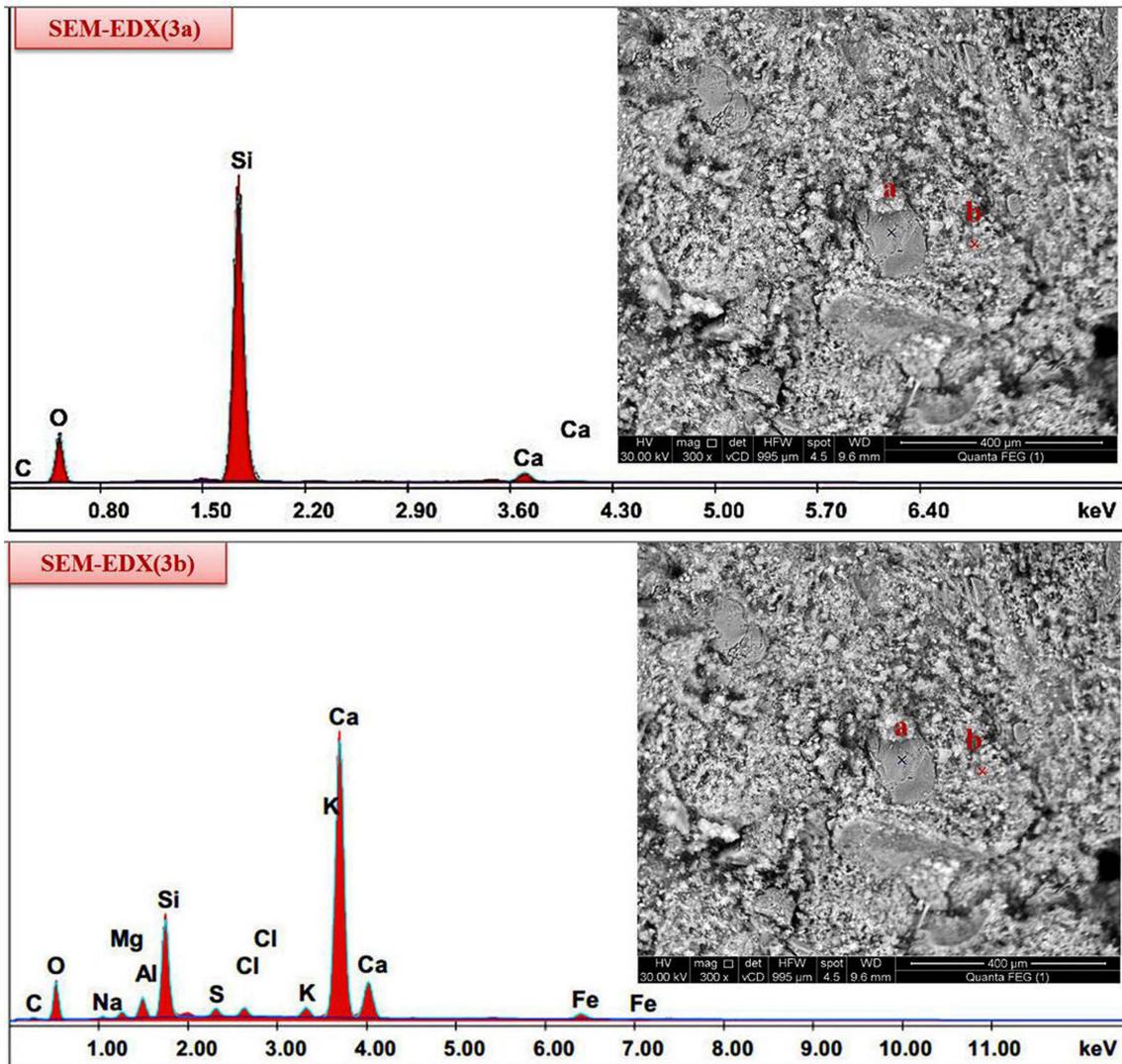


Figure 15. (3a and 3b) EDX spectra of joint mortar sample from the upper parts of the south wall of the big tank as the appearance of Si, Ca, O, C, Fe and Al testifies the occurrence of quartz, calcite and traces of pozzolanic materials.

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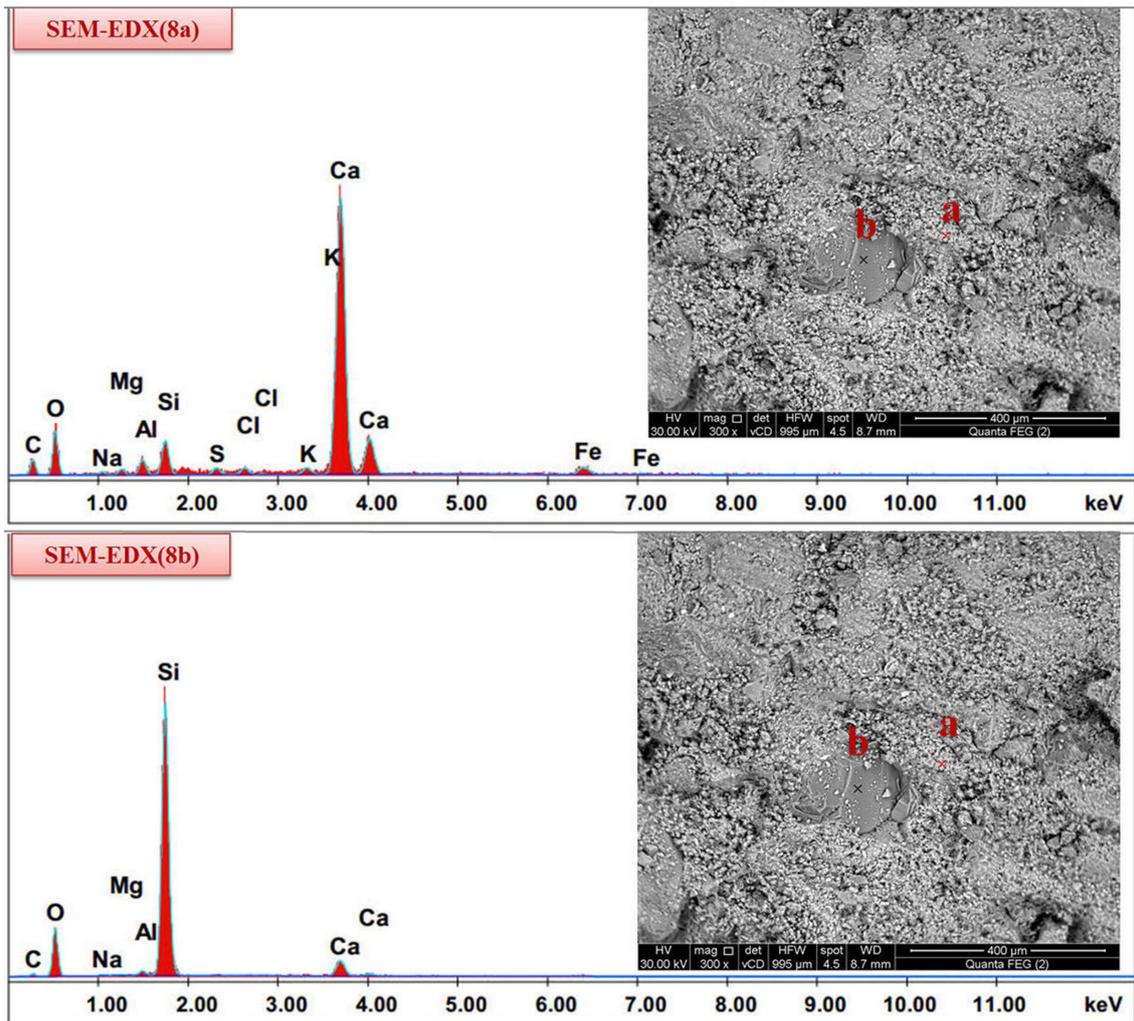


Figure 16. (8a and 8b) EDX spectra of the second reddish plaster layer sample of the eastern wall middle parts of the upper big tank as the appearance of Fe, Ca, O, C and Si confirms that the plaster is a lime mortar consisting of calcite, quartz and brick powder.

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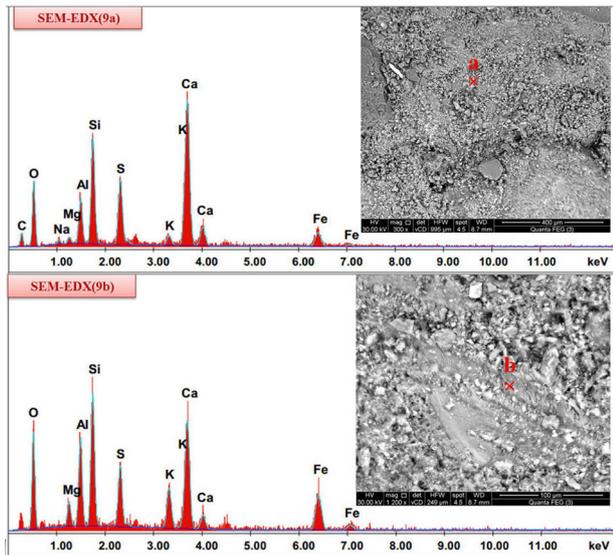


Figure 17. (9a and 9b) EDX spectra of the third reddish plaster layer sample of the eastern wall middle parts of the upper big tank show the presence of Ca, Si, Al and Fe which make this sample resemble sample 8, but the appearance of S may testify the occurrence of traces of one of the gypsum phases as an impurity.

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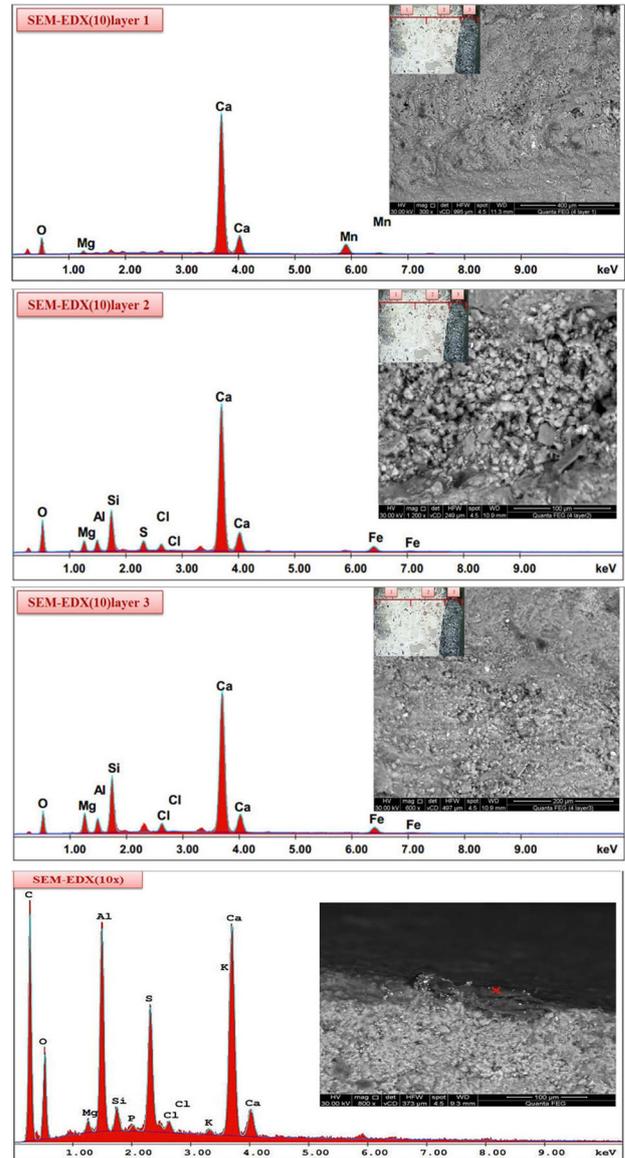


Figure 18. EDX spectra of the three layers sample collected from the internal face of the big upper tank which confirming that the whitish internal layer 1 is a lime mortar while the reddish layer 2 is a lime mortar mixed with brick powder and the elemental composition of the grayish layer 3 testifies to the occurrence of pozzolanic material but the appearance of S in the superficial sample of the grayish layer (SEM-EDX 10x) testifies the occurrence of traces of one of the gypsum phases as an impurity or related to sulfation of lime mortar.

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