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Analysis of mortars applied to the remains surrounding the Monastery of Apa Shenoute, Sohag, Egypt

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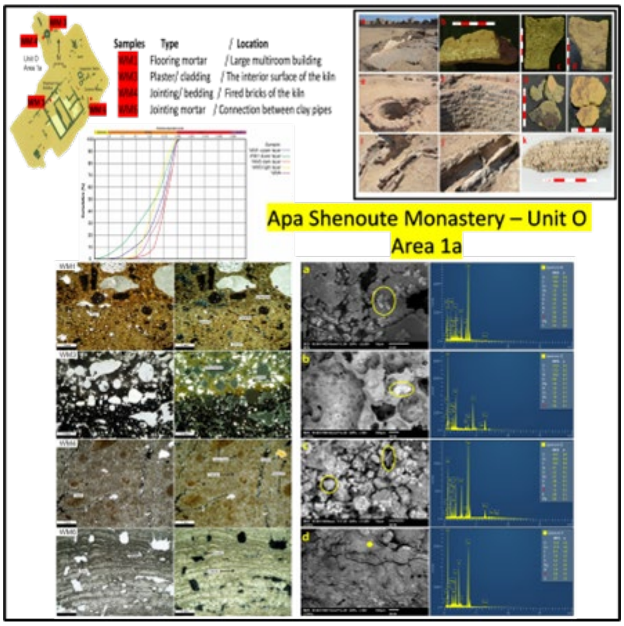
HIGHLIGHTS

- Characterizing of ancient mortars at risk of extinction, employed at Apa Shenoute Monastery, including those used for flooring, bedding, cladding, and jointing.
- Documenting the composition and technologies of these mortars provides insights into their current state of preservation and informs future restoration efforts.
- All analyzed mortars utilize calcitic lime as a binder, combined-with different aggregates such as sand, limestone fragments, and brick dust.
- Cladding and jointing mortars exhibit a range of colors (orange, grey-olive, black) and diverse binder-to-aggregate ratios, varying from 1:1 to 2.3:1 reaching to almost free aggregate mortars used in connecting mortars of clay pipes.
- The collected data is crucial for making informed decisions regarding the selection of appropriate materials and techniques for restoration purposes.

GRAPHICAL ABSTRACT



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ABSTRACT

This paper aims to characterize the different types of mortars used in a specific area most at risk of extinction at Apa Shenoute Monastery, including flooring, bedding, cladding and jointing mortars, in order to document their compositions and technologies. Due to the continuous exposure to erosion that has resulted in their deterioration over time, such a survey enables a proper understanding of their state of preservation, their composition as well as assists in the proper planning of future restoration work.

To achieve this goal, representative samples of different types of applied mortars were subjected to in situ visual inspection, polarizing light microscope, X-ray diffraction, thermal analysis, grain size distribution analysis, and scanning electron microscope equipped with energy dispersive X-ray unit. Obtained results characterize optical, mineralogical, and technical properties of selected mortar samples. The results revealed the use of calcitic lime as a binder in all types of mortars with various Binder/Aggregate ratio, ranging from 1:1 to 2.3:1 with the exception of almost free aggregate mortars used in connecting mortars of clay pipes. Moreover, flooring and plastering mortars are composed of multiple layers. In the case of flooring mortars, it has been observed that fragments of limestone (0.5 to 3.5 mm) are added to the lower layer and fragments of brick dust are added to the upper layer as an artificial pozzolanic material. Different colours of the cladding and the jointing mortar of the kiln, varying between orange, grey-olive and black were attributed to the exposed firing temperature. The information gathered should be useful for specific selection criteria for compatible restoration materials and techniques.

1. Introduction

The study of ancient mortars reveals their fundamental role in construction throughout history. In Egyptian history, mortars made with binders such as mud, gypsum, and lime were extensively used [1]. During Late Antiquity and the Roman/Hellenistic periods, mortars primarily consisted of lime, pozzolans, aggregates (sand and crushed stone), and sometimes organic additives. Research into mortar composition during Late Antiquity and Roman/Hellenistic times reveals significant advancements in construction materials and techniques both in Egypt and beyond. In the Roman era, aggregates such as quartz, feldspar, and ceramic fragments were utilized to provide durability and stability. The addition of pozzolans, volcanic ash, or similar materials enhanced the strength and water resistance of the mortars [2,3].

Organic additives were used to improve workability and adhesion. Technological advancements, particularly within the Roman Empire, led to the creation of hydraulic mortars like opus signinum, which could set underwater [4]. These innovations, along with regional variations and specific construction project requirements, resulted in diverse mortar formulations across the ancient world. Regarding the mixing proportions, Vitruvius describes 1:3 binder/ aggregate ration for structural mortars [5, 6]. In addition, high proportions of brick dust and crushed brick in lime or lime-pozzolan mortars expanded during the Byzantine era (4th–15th century AD) [7, 8, 9]. In the Hellenistic period, lime-based mortars were prevalent, often combined with quartz aggregates [10]. In Late Antiquity, mortars continued to rely on lime and gypsum, with both organic and inorganic additives to increase stability and durability.

In our case study, the Apa Shenoute Monastery region, located approximately 8 kilometres west of the city of Sohag, is recognized as one of the most significant archaeological Coptic sites in Egypt. Locally known as Al-Dayr al-Abyad (The White Monastery), named after the white limestone used in its construction [11, 12, 13]. This area is of great importance due to the high-value church and other structures dating back to the 4th/6th century AD (2nd half of the 5th century) [14]. In addition to the church building, there are several surrounding ruins, which according to recent excavations, are divided into three areas [15,16] as shown in Fig.1a and 1b. This work focuses on Area 1 - Unit O including a large multiroom building, a kiln, and ceramic piping. The ruins have been studied architecturally and archaeologically during the excavations carried out by the Supreme Council of Antiquities (SCA) in 1985 and later by foreign missions [17, 18 , 19] which are still ongoing. However, this region has not been studied from the point of view of characterizing their mortars which represent the dominant element in all types of buildings, used in more than one place and function, including mortar for bedding bricks, cladding, joint mortar between the clay pipes, and flooring mortar. The latter covers large areas and is exposed to extinction due to the continuous exposure to erosion resulting in loss of some parts over time. Also, despite all the efforts of the inspectors to preserve it, some parts are deliberately broken due to the lack of awareness of the people since it is still an open area. Unfortunately, apart from flooring mortar, the whole ruins are subjected to erosion with time due to weathering factors. Since different types of mortars were discovered, they are in need to be technically documented in terms of their composition, optical properties, mineralogical, and chemical composition as a first step in any possible restoration work that may be carried out to prevent further deterioration. In addition, characterization helps in the diagnosis of deterioration causes. Therefore, this effort aims to realize the nature of these mortars and to identify their composition and state of preservation in order to properly support the choices for future restoration work [20]. Considering the historical significance and antiquity of the Apa Shenoute Monastery, this study is crucial for characterizing the old mortars. This work, combined with other efforts in historical, architectural, and structural specializations applied to the church and its annexes, contributes to the proper rehabilitation of the site. This revitalization aims not only to enhance its appeal as a tourist destination that includes religious activities but also to emphasize the architectural and functional characteristics of all annex units outside the archaeological church, particularly those associated with industrial activities [21].

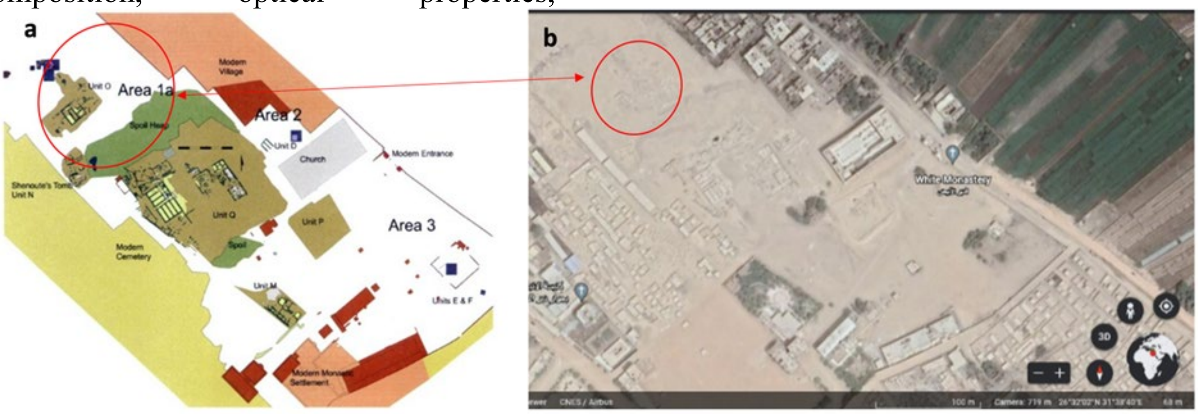

Fig.1. a. The location of Area 1 at Apa Shenoute after Darlene L. Brooks Hedstrom and et.al (2011); b. Satellite view of the whole area.

2. Materials and methods

Given the richness of this region in terms of the utilization of mortars, their diverse applications, and construction phases, comprehensive studies require taking samples to the extent that allows high accuracy of the results statistically. However, given the high value of these built units, taking samples was restricted by authorities. Consequently, sampling was limited to some specific representative samples from the northern part of the Monastery *Area 1- Unit O* extracted by a hammer and chisel [22]. The samples collected encompass a variety of mortars, including flooring mortar, cladding, bedding mortar, and jointing mortars. A summary of the number of samples, their types, and locations is given in Table 1. These samples were taken from a variety of locations, including the floor of a large multi-room building, the kiln, and the spaces between the ceramic pipes, as shown in Fig. 2. Then, the samples were transported to the laboratory in small, sealed plastic bags to prevent contamination. Upon arrival at the laboratory, the samples were prepared for further examination. This preparation included gentle cleaning with a soft brush to remove any potential contaminants.

2.1. Visual inspection and physical properties.

The investigation of the samples began with a macro visual inspection, providing an initial understanding of their physical characteristics to the naked eye [23]. The samples were photographed using a Nikon D5200 digital camera for detailed documentation. They were then examined for colour using the Munsell chart [24,25], state of preservation, visible aggregates, number of layers -if present- along with their thickness were examined using a digital calliper. Porosity was calculated after determination of bulk density, dry density and volume which had been measured by Gas Pycnometery method [26].

2.2. Petrographic analysis.

Thin sections, approximately 30 μm in thickness, were meticulously prepared from each sample. This process involved embedding intact pieces within a low-viscosity epoxy resin under vacuum conditions [27]. A series of overlapping photomicrographs was captured using a Zeiss Axiolab Opton polarizing microscope. The microscope was equipped with a Canon Powershot G2 digital camera, which was coupled to the microscope via an eyepiece adapter. These individual images were then automatically stitched together using Adobe Photoshop S3 software.

2.3. Grain size distribution and acid dissolution.

The composite image of the overlapping photomicrographs was used for subsequent image analysis. Binder/aggregate (B/A) ratios and grain size distributions have been obtained by point counting method and determination of the longest Feret diameter for approximately 200 grains per thin section respectively, using JMicrovision V.1.2.7 software [28,29,30,31]. About 25g of each sample had been digested with a dilute HCl acid to determine the percentage of aggregates after physical separation of limestone fragments acting as a filler [32,33,34].

2.4. XRD analysis.

A few grams of each mortar sample were extracted and prepared for X-ray diffraction analysis [35]. Samples were analysed [36,37,38] using a Bruker D 5000 diffractometer operating at 36 mA, 36 kV, with CuKα radiation. All measurements were performed in the 2θ range from 5° to 75°. Identification of different mineralogical phases including major and minor minerals was done using PANalytical X'pert high score plus software.

2.5. SEM/EDS examination.

For microstructural analysis [39], uncoated and freshly broken pieces of samples, as well as polished thin sections were sputter-coated with carbon have been examined by scanning electron microscope SEM-VEGA LSU TESCAN, which was equipped with an energy dispersive X-ray detector (EDX) (Oxford IncapentaFETX3 detector).

2.6. Thermal analysis.

Differential scanning calorimetry (DSC) and thermo-gravimetric analysis (TG) were carried out for phase identification [40,41] using a Perkin-Elmer STA6000 calorimeter equipped with an Al2O3 sample pan, in the temperature range of 40. °C to 990. °C at a constant heating rate of 15 °C/min, in an N2 atmosphere. PYRIS software was used for data interpretation. Thermographs of the examined samples allowed to calculate the proportions of carbonates according to the mass loss at certain temperatures [42].

Samples	Type	/ Location
WM1	Flooring mortar	/ Large multiroom building
WM3	Plaster/ cladding	/ The interior surface of the kiln
WM4	Jointing/ bedding	/ Fired bricks of the kiln
WM6	Jointing mortar	/ Connection between clay pipes

Fig.2. Shows samples' locations on a plan of area 1a – Unit O drawen by Darlene L. Brooks Hedstrom and et.al (2011) and table with number of samples, their types, and locations.

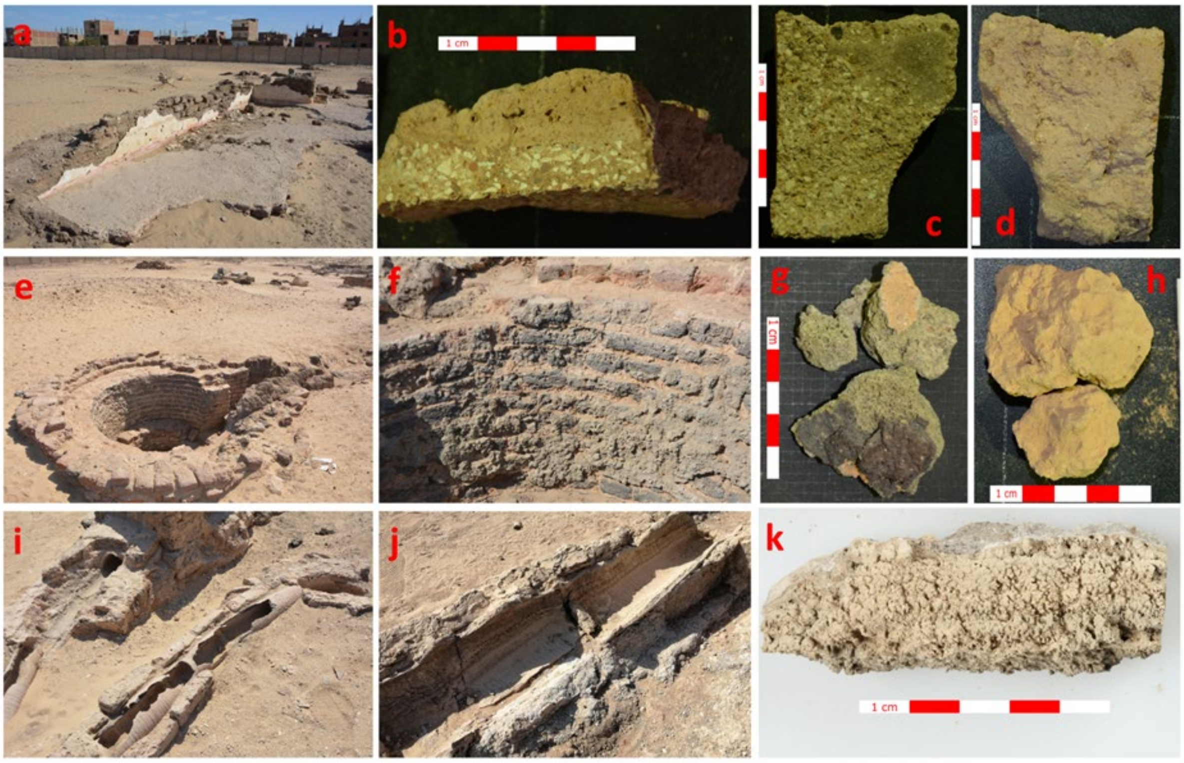

Fig.3. a. Sample WM1 represents flooring mortar from the large multiroom building; b. Composed of two layers, c. The lower layer; d. The upper layer; e. Sample locations of WM3 and WM4 from the kiln; f. plaster/ cladding (WM3) and bedding mortar (WM4); g. Black and olive coloured layers of cladding mortar; h. Orange bedding mortar; i. Damaged ceramic pipes; j. Sampling location of WM6 Jointing mortars between clay pipes; k. Jointing mortar sample WM6.

3. Results and discussion

3.1. Visual inspection and physical properties.

The results of the naked eye investigation in situ and measurements in laboratory are summarized in table 1. The results of simple visual examination of the samples varied in terms of properties including colour, thickness, cohesion and composition. The examination showed a difference in the degree of cohesion and porosity of the samples. Samples used as flooring mortars are characterized by good cohesion in the upper and lower layers, reaching the very hard sample No. WM6 of connecting mortars between clay pipes. It was also found that sample WM3 was characterized by a more porous surface structure than the other samples in both its dark and light layers. As for the materials used in the composition of the different mortar samples, it was noted that there were some materials that distinguished some types from others, such as the presence of ceramic fragments in sample No.WM1a,which gave it a distinctive pale red colour, while to the presence of limestone pebbles in the lower layer of flooring mortar sample WM1b was noted. As for the thickness of the samples, the thickness of the samples varied, with averages reaching a maximum of 2.4 cm in the mortar sample used to connect the clay pipes, passing through the flooring mortar samples, in which the average thickness ranged from 1.3-1.5, reaching the joint mortar and plaster mortar samples WM4 and WM3 to 1 cm or less respectively.

Table 1. Visual inspection and in-situ investigation.

Samples	Colour (Munsell Chart)	Average. Thickness (cm)	Brick fragments	Limestone fragments.	Notes
WM1 a	10 R 7/3 Pale red	1.3	+++	-	Coherent
WM1 b	2.5Y 8/1 White	1.3	+	+++	Coherent
WM3	Dark 7.5Y 3/1 Black Olive Light 7.5Y 5/2 Grayish Olive	0.5 0.9	- -	- -	Porous Porous
WM4	10YR 8/4 Orange	1.1	-	-	Dense
WM6	7.5YR 7/1 Light brownish	2.4	-	-	Hard

(+++) very abundant; (++) abundant; (+) rare; (-) none.

3.2. Petrographic analysis.

Petrographic examination using a polarizing microscope confirmed that sample WM1 flooring mortar is composed of two layers, where calcite is the binder with sparse lime lumps in both layers, while the aggregate is composed mainly of brick powder and limestone fragments in the upper layer and lower layer respectively. Quartz was observed as an accessory constituent in both layers, while limestones occur sporadically in the lower layer. Sample WM3 is composed of aphanitic groundmass, intergrown with numerous acicular microscopically unidentified crystals. The aggregate is dominated by: quartz, heavily cracked, feldspars are subordinate, while accessory constituents are amphibole, opaque minerals, ceramic (brick?) fragments coming from adjacent substrate. Sample WM4 also shows calcitic lime as a binder with small sized lime-lumps. The aggregate is composed mainly of quartz with accessory constituents like opaque minerals. Sample WM6 revealed also calcite as a binder while the aggregate is composed of quartz (very uncommon) as well as opaque minerals as an accessory constituent.

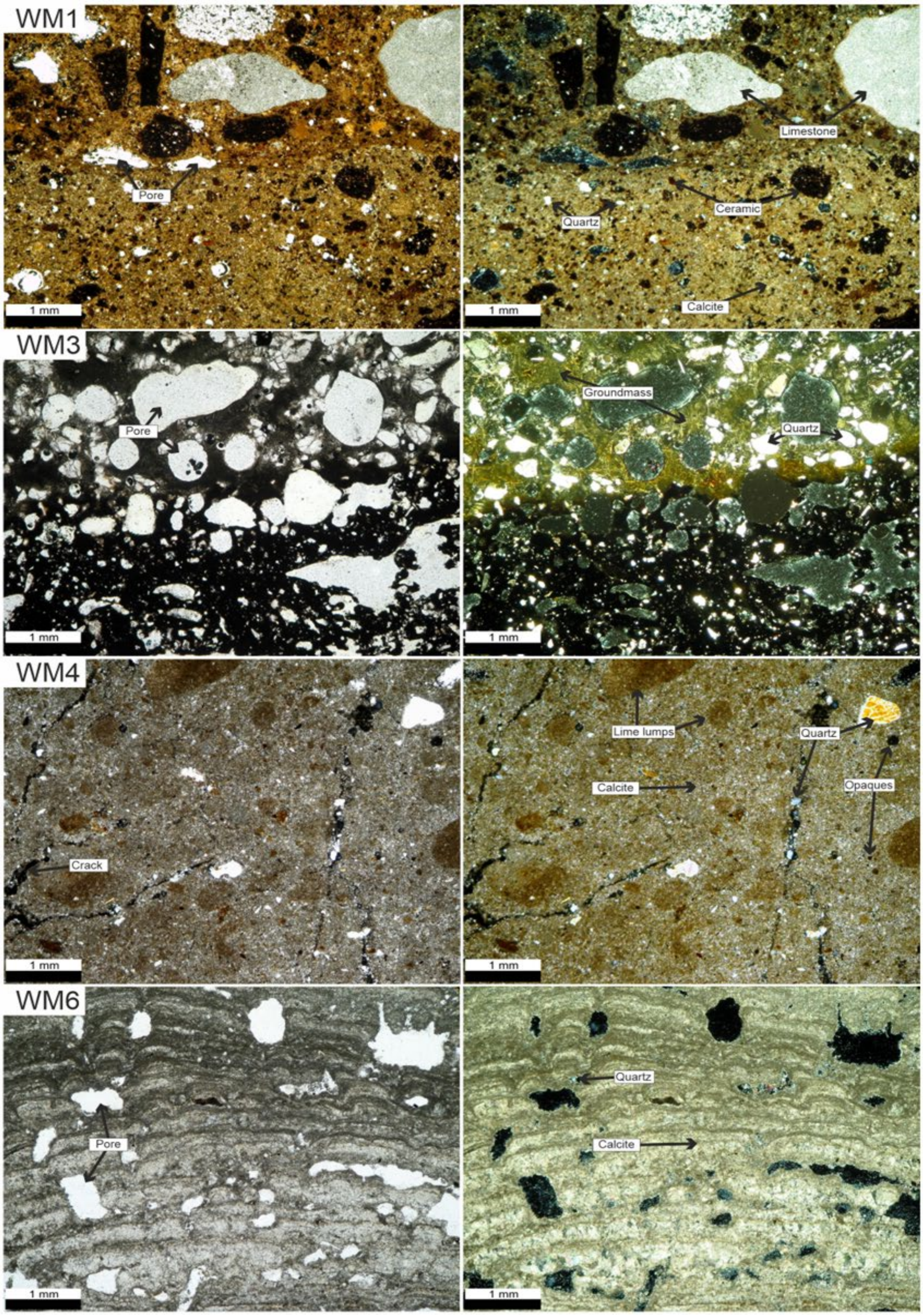

Fig.4. Microphotographs of samples WM1, WM3, WM4 and WM6 of different mortars taken in plane-polarized (PL) (left column) & under cross polarized light (CN) (right column).

3.3. Grain size distribution and acid dissolution.

Sample WM1a (upper layer of flooring mortar) and **Sample WM1b** (the lower layer) have the aggregate classified as slightly gravelly sand, polymodal poorly sorted. Sample WM1a is composed of (very fine sand 46.3 % + fine sand 30.1% medium sand 12.3% coarse sand 8.3% + very coarse sand 2.5%, while Sample WM1b is composed of 4.2 % gravel and fine to very fine sand poorly sorted (very fine sand 32.8 % + fine sand 26% medium sand 15.7+ coarse sand 13.7% + very coarse sand 7.6%). **Sample WM3 dark layer and Sample WM3 light layer** has aggregate classified as a sandy bimodal moderately well sorted and moderately sorted sand respectively. Sample WM3 dark is composed of (very fine sand 63.5% + fine sand 31.1% + medium sand 3.4% coarse sand 2.0%) very fine sand, while Sample WM3 light is composed of (very fine sand 26% + fine sand 45.5% + medium sand 24.9 % coarse sand 3.7%). **Sample WM4** has aggregate classified as a polymodal moderately sorted sand (very fine sand 54.7% + fine sand 28% + medium sand 16 % coarse sand 1.3%) fine sand moderately sorted.

Sample WM6 was not subjected to the grain size distribution analysis due to the small number of grains, as previously stated.

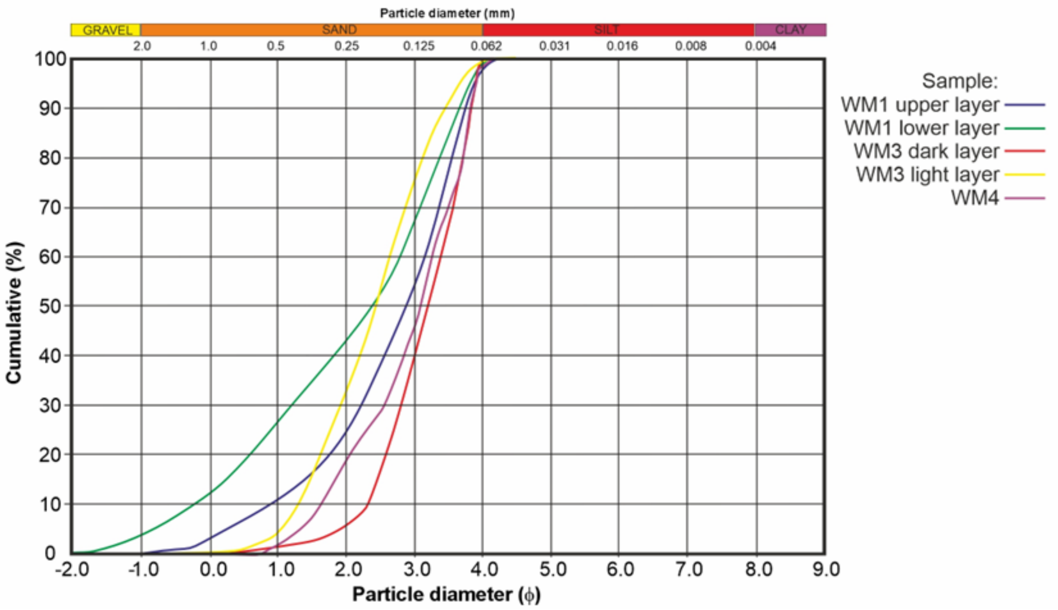

Fig.5. Cumulative grain-size distribution curves of samples WM1a (upper layer of flooring mortar), WM1b (the lower layer), WM3 (dark layer) of cladding mortar from the Kiln, WM3 (light layer) and WM4 jointing mortar from the kiln respectively.

The results of acid attack and point counting to determine the binder/ aggregate ratios as well as approximate porosity of all samples are summarized in Table 2. They revealed the use of calcitic lime as a binder in all types of mortars with various binder/aggregate ratio, ranging from 1:1 to 2.3:1 with the exception of almost free aggregate mortars used in the connecting mortars of ceramic pipes.

Table 2. Summarizes the approximate percentages of binder and aggregates in all samples.

Samples	Carbonates (Binder) %	Rock fragments %	Brick fragments %	Approx. Sand %	Total aggregates %	Approx. Porosity %	B/A ratio
WM1a	63.6	-	{30.4	6}	36.4	12	≈ 1.5:1
WM1b	51	{26.8	19.8	2.4}	49	31	≈ 1:1
WM3 Dark	48.3	-	3.1	42.3	45.4	35.8	≈ 1:1
WM3 Light	48.0	-	-	42.2	42.2	34	≈ 1:1
WM4	70.4	-	-	29.6	29.6	24.3	≈2.3:1
	97.8	-	-	2.2	2.2	39.1	almost pure lime

3.4. XRD analysis.

X-ray diffraction charts of bulk samples are shown in Fig.6. They revealed that sample WM1a is composed of the main constituents; *Calcite, Quartz, Halite, Magnetite, Gypsum* with accessories including Hematite, *Albite, Calcian Anorthite.* Sample WM1b is dominated with *Calcite*, *Quartz*, *Halite* with accessories including *Vaterite*, *Sylvite*, *Hematite*, *Anorthite.* Sample WM3 is composed of *Diopside, Aragonite, Quartz, Analcime, Halite, Whitlockite, Clinohypersthene, Wollastonite*. Sample WM4 is composed of *Calcite, Quartz, Halite, Dolomite, Sylvite, Cristobalite, Muscovite, Phillipsite-Ca.* Sample WM 6 *Quartz, Calcite-Magnesium, Anorthite and Gypsum.*

Table 3. Summarizes results of the XRD analysis of Apa Shenoute Monastery samples.

Sample	Locations/function of mortars	Composition
WM1a	Upper layer of flooring mortar	Calcite, Quartz, Halite, Magnetite, Gypsum Hematite, Albite, Calcian Anorthite
WM1b	Lower layer of flooring mortar	Calcite, Quartz, Halite, Vaterite, Sylvite, Hematite, Anorthite
WM3	Cladding mortar from the Kiln	Diopside, Aragonite, Quartz, Analcime, Halite, Whitlockite, Clinohypersthene, Wollastonite-
WM4	Jointing mortar from the Kiln	Calcite, Quartz, Halite, Dolomite, Sylvite, Cristobalite, Muscovite, Phillipsite-Ca
WM6	Connecting mortar between clay pipes	Quartz, Calcite Magnesium, Anorthite, Gypsum

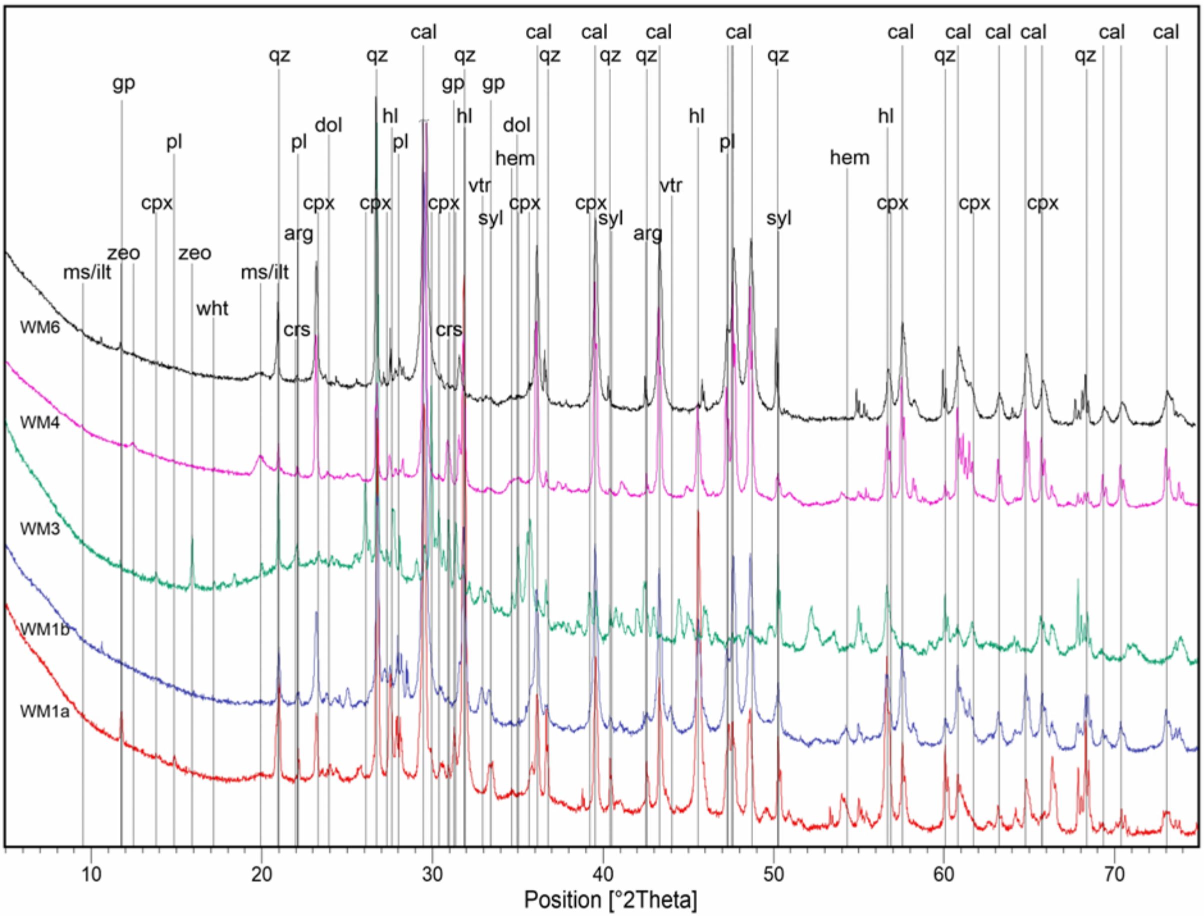

Fig.6. XRD patterns of mortar samples. Mineral abbreviations after Warr (2021) [43]: cal-calcite, qz-quartz, gp-gypsum, hl-halite, dol-dolomite, pl-plagioclase, hem-hematite, vtr-vaterite, cpx-clinopyroxene, syl-sylvite, arg-aragonite, zeo-zeolite, ms/ilt-muscovite/illite, wht-whitlockite, crs-crystobalite.

3. 5. SEM / EDS examination

The results obtained from scanning electron microscope (SEM) examination, coupled with spot microanalysis using an energy-dispersive X-ray (EDX) detector, are presented in Fig.7. The key observations regarding the microstructure of the samples can be summarized as follows:

a) Main constituents: SEM-EDS analyses confirm previous observations that the binder in the samples is calcium carbonate. The filler components are quartz, limestone grains and also grains consisting of aluminosilicates – due to ceramic fragments.

b) NaCl Particles: NaCl particles were detected on the surface of most samples, particularly in sample WM1a (the upper reddish layer) of the flooring mortar and sample WM4 of the jointing mortar. b) Sulfur presence and fine cracks: Sample WM6, which represents the connecting mortar between ceramic pipes, exhibited a relatively high presence of sulfur. This indicates the presence of secondary gypsum. Additionally, fine cracks were observed on the smooth surface in this sample. c) Distinctive Cladding Mortar Structure: Sample WM3, corresponding to the cladding mortar, displayed a unique feature in its structure represented in spherical voids. These findings contribute to our understanding of the composition and characteristics of the examined mortars.

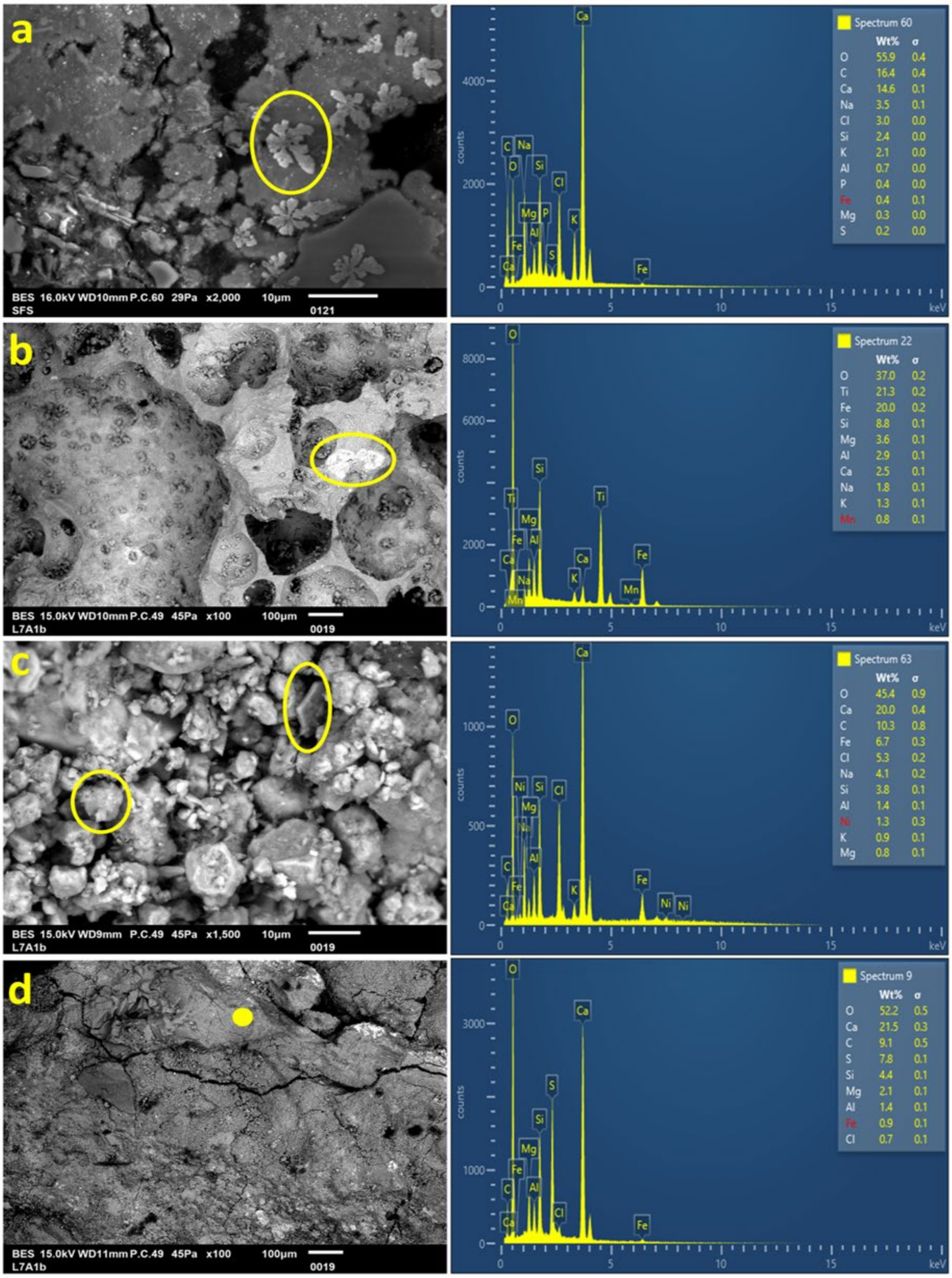

Fig.7. SEM-BSE Microphotographs with EDX spectrums results a. Sample WM1 upper reddish layer of flooring mortar; b. Sample WM3 plaster (microstructure of spherical shapes of voids); c. Sample WM4 of jointing mortar; d; Sample WM6 of connecting mortar between ceramic pipes.

3. 6. Thermal analysis.

Fig.8. showed DSC/TG curves of all analysed samples where sample WM1a and WM1 b curves are characterized with weight loss accompanied with endothermic reactions including loss of unbound water(1),dissociation of gypsum (2), dehydroxylation of clay minerals– illite(4), thermal dissociation of calcite (5 and 6) and double-step endothermic reaction indicating to halite melting and evaporation (7 & 8) with one exothermic reaction indicating to combustion of organic matter at (3). Sample WM1b curve is characterized with wight losses accompanied with endothermic reactions including loss of unbound water (1),dehydroxylation of goethite/lepidocrocite (2),thermal dissociation of calcite of different crystallinity (3,4), as well as halite melting and evaporation (5). Sample WM3 curve showed endothermic reaction including polymorphic transition of quartz low-temperature into high-temperature variety at (1), thermal dissociation of calcite (2) and halite melting and evaporation (3). Sample WM4 curve revealed loss of unbound water (1), and thermal dissociation of dolomite (double step) and calcite at (2,3) respectively with halite melting and evaporation at (4). Sample WM6 curve showed loss of unbound water at (1), dissociation of gypsum at (2) and thermal dissociation of calcite at (3).

The examination and analysis of the representative samples taken from the remains of the built units of Area 1- Unit O at Apa Shenoute Monastery revealed a great deal about the composition of the mortars, the techniques used to apply them and the by-products affecting the structure of those mortars. These samples included flooring, bedding, cladding and jointing mortars between the brick units, as well as the clay pipe units. It is evident from the visual examination by the naked eye and thin section of these samples that multi-layers have been applied to the floors of large multiroom building, as well as cladding in the kiln. This multiplicity of flooring mortars is attributed to the function of those buildings, which in the past could have been used for industrial activities, in which liquids were used in abundance, requiring insulating and resistant properties. In addition, the cladding of the inner wall of the kiln is multi-layered, as evidenced by the clear boundary between the greyish olive and black olive layers due to the petrographic examination of the thin sections, which indicates the possibility of the plaster being applied in two stages.

Petrographic examination by polarizing microscope also revealed the different constituents used as aggregates in the composition of the different layers of flooring mortar, where the older lower layer is characterized by relatively large limestone fragments added with sand, while the younger upper layer is characterized by red brick powder to provide a durable and fluid-resistant coating **[**44**,**45**]**.

In addition to the multi-layers in the flooring mortar and cladding ones, the multi-colours also appeared through the visual examination because of the different components included in the composition as a filler **[**46**]**, such as the reddish colour resulting from the use of red brick powder in the upper layer. This variety of aggregate types affected the colour of the mortars.

According to the Munsell chart, cladding layers of the kiln have the same hue, but with a different value and chroma between

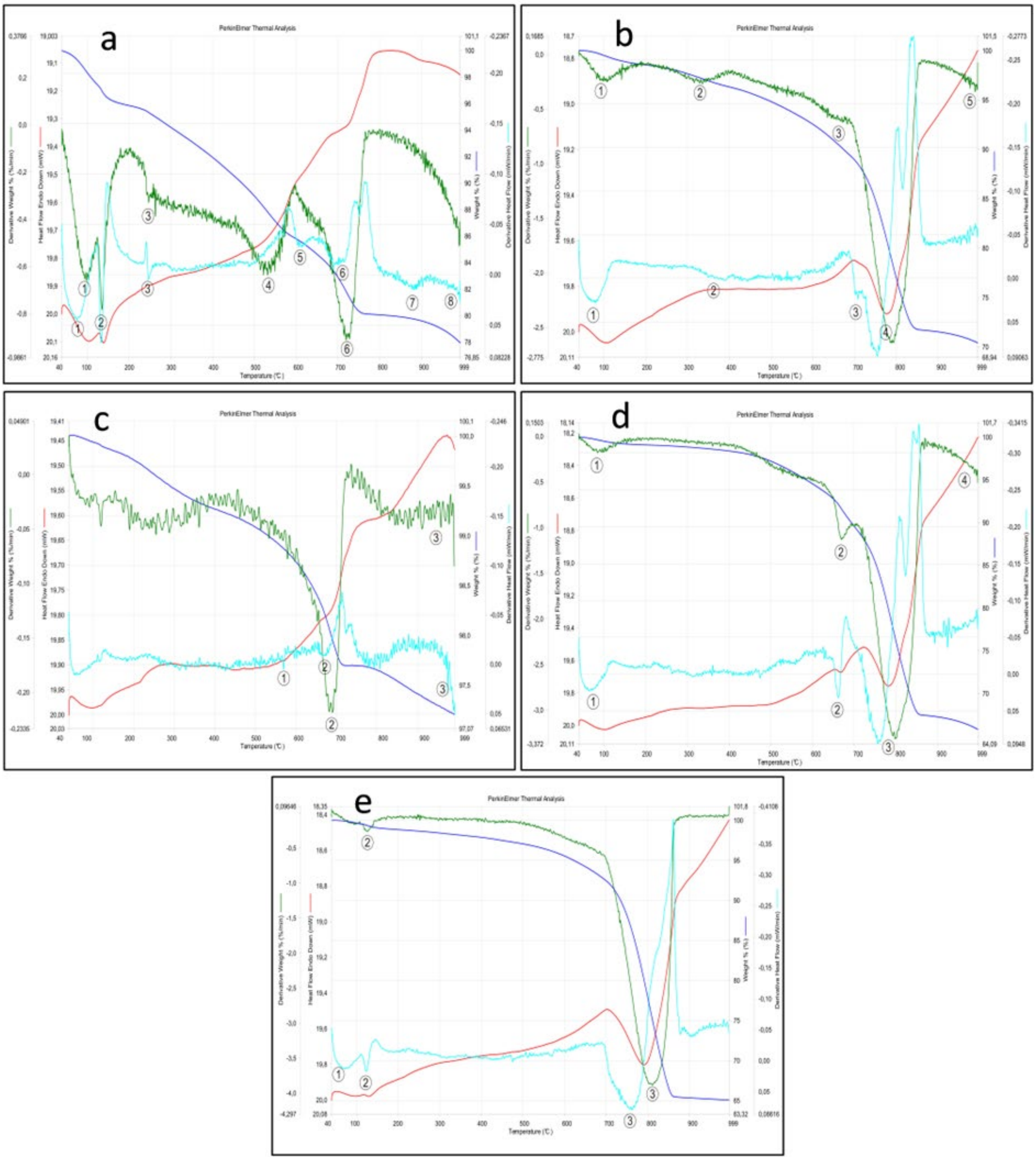

Fig. 8. DSC/TG curves of a. Sample WM1 a (upper layer of flooring mortar); b. Sample WM1 b (lower layer of flooring mortar); c. Sample WM3 (Cladding of the kiln); d. Sample WM 4 (bedding mortar used in wall of the kiln); e. Sample WM 6 (jointing mortar used for connection between ceramic pipes)

the black olive inner layer (**7.5Y 3/1**) and the grey olive outer layer (**7.5Y 5/2**).

This can be attributed to the exposure to different temperatures, oxidation conditions, and direct/indirect exposure to combustion where the grey olive appeared because of the presence of the diopside mineral **[47]** given by XRD results, while the black olive colour could result from the conditions of indirect combustion **[48]** during the pottery firing process. As for the orange colour of the bedding mortar, it is likely caused by aggregates mixed with lime where the filler has the greatest influence in determining the colour of the mortar **[49,50].** That aggregates which contain sand, or some clay particles have high levels of iron oxide, as confirmed by elemental analysis using EDS. That high percentage of iron in the bedding mortar sample can give the mortar a reddish-orange colour, depending on the iron concentration **[51]**.

The results of b/a ratios revealed diversity, so that the samples have an equal ratio of the B/A 1:1 in the lower layer of flooring mortar and cladding ones, while the upper layer of the flooring mortar represents a ratio of 1.5:1. In the latter, despite the relatively high amount of binder which can lead to lower bulk density **[52]**, the addition of sub-angular and angular brick dust can act as a pozzolan, filling the pores and increasing the density **[53]** enabling good interlocking of the grains and reducing porosity **[54]**. As a final result, this will allow it to act as a lining layer for the purposes of protection against liquids.

The percentage of carbonates reaches its peak in the jointing mortar for ceramic pipes connection to the point where the filler material is almost absent. This could be for providing flexibility and subtility to move with two edges of connection areas between the clay pipes. Furthermore, this plastic paste of almost pure lime mortar is required for smooth and levelled placement of ceramic pipe pieces with horizontal straight line that facilitates the movement of fluids inside and prevents their leakage.

The grain size distribution also showed a diversity due to the different types of aggregates and the function of the mortar itself. Limestone grains with a relatively large grain size of up to 4 mm in the lower layer of the flooring mortar indicate a slightly gravelly texture. Since the surrounding area is rich in limestone, it is an appropriate, economical, and efficient choice for the production and use of the limestone pebbles to provide a strong solid with stable volume of the mortar **[55],** and a durable substrate for the subsequent layers **[56,57]**. However, the predominant feature in other samples is the use of fine to very fine aggregates. The finer grains allow for physically denser and more homogenous mortars. The practice of adding brick fragments, organic proteinaceous materials, and limestone pebbles to lime-based mortars highlights the historical knowledge of material science. Brick fragments and limestone pebbles likely provided enhanced mechanical strength and durability, essential for the upper and lower layers of the mortar respectively. Proteinaceous substances could have contributed to the improved binding properties and flexibility, preventing cracks and enhancing longevity. These practices indicate a sophisticated understanding of how to manipulate materials to achieve specific construction outcomes, demonstrating the advanced engineering capabilities of the time [58].

Some physical properties such as porosity can give an indication of the mortar application technique, where a significant increase in total porosity was noted in the cladding mortar- sample WM3, in its two layers. From a technical point of view, the high porosity can be attributed to the application of the mortar through splashing way. This type of mortar application requires a higher amount of water due to the increase in the water/biner (W/B) ratio, where a higher mixing water content means a higher porosity **[59,60]**. Therefore, the spherical morphology of the pores at the macro and microscopic levels can be clearly seen by the naked eye, P.M. and SEM respectively. This rounded morphology mainly arising from air bubbles **[61]**. The results of X-ray diffraction and thermal analysis confirmed the presence of calcite as a binder (air lime) in all samples. They also proved the presence of some salts, sodium chloride (NaCl), potassium chloride (KCl) and calcium sulphate ($CaSO_4$) through the appearance of the minerals: halite, sylvite, and gypsum as by-products, which was proved by elemental analysis and microstructure using SEM/EDS. The mineral Vaterite that was detected by XRD in the flooring mortar sample, was previously detected as an accessory mineral in the joining mortar on the western façade of the church of the Red Monastery which is located about 3 Km to the north of the Apa Shenoute Monastery **[62]**. The common feature of both mortars is the presence of the protein as an organic additive. This presence can be a further confirmation of the use of organic additives where it was probably formed during the early step of precipitation of the $CaCO_3$ from lime and can be linked to the presence of the organic compounds from the additives used **[63]**.

The thermal analysis confirmed presence of deterioration products such as gypsum and halite salts. Furthermore, it proved the possibility of the presence of organic materials in the floor mortar, confirming the previous result obtained by the wet chemical analysis to detect the protein materials added to the flooring mortar **[64]**. It is a matter under study to define the nature of the protein qualitatively and its percentage quantitatively, because it may be useful to know the technology and materials used in it and its purpose, as well as what is useful for restoration purposes. Finally, all the results obtained from the involved multidisciplinary techniques with their interpretations could be useful for documenting the ancient materials and applied technologies. In addition, it is useful as a first step for designing conservation plan and preventive care of the built units.

4. Conclusion

Based on the results of the investigations presented above, a number of observations have been made: This study focused on the investigation into the mortars employed within the settlements of Area 1-Unit O at the Apa Shenoute Monastery underscores their significance in documenting the construction materials used. Although these mortars applied with same calcitic binder, they exhibit a diverse range of compositions and features. Their binder/aggregate ratio, ranges from 1:1 to 2.3:1 with the exception of almost free aggregate mortars used in connecting mortars of clay pipes. Moreover, flooring and plastering mortars are composed of multiple layers. In the case of flooring mortars, it has been observed that fragments of limestone (0.5 to 3.5 mm) are added to the lower layer and fragments of brick dust are added to the upper layer as an artificial pozzolanic material. Furthermore, our analysis aimed to document and demonstrate the materials and techniques employed in their preparation, particularly in the context of the conservation of these valuable architectural units and extensions. The application of multi-disciplinary examination and analysis methods has been instrumental identifying, and characterizing the components of the mortars used in the construction and finishing of the attached units within the study area. This knowledge contributes to a deeper understanding of these materials, especially considering the exposure of the region to erosive factors due to its open environment. Moreover, our findings shed light on the various deterioration by-products associated with the different mortar types, which were either different or similar in all the samples studied. This understanding enables a more accurate diagnosis, facilitating the development of implementable plans for prevention, maintenance or active intervention including consolidation by either traditional or smart materials, patching and repair mortar to mitigate damage and to protect these architectural units from total loss. Furthermore, the benefits of conducting a multidisciplinary analysis of mortars for conservation purposes lie in obtaining comprehensive information that helps prevent material incompatibility, thereby averting potential significant and irreversible damage [65]. However, further research is needed to explore other areas surrounding the Monastery of Apa Shenoute and to compare the findings with other studies of similar buildings from the same historical period such as Apa Bishoi (the Red Monastery). That will enhance the comprehension of mortar usage and preservation practices.

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