CARACTERISTICILE TEHNOLOGICE ALE TENCUIELILOR APLICATE ÎN MONUMENTELE DIN SECOLUL al IV-lea î.Hr, DIN GRECIA
TECHNOLOGICAL CHARACTERISTICS OF COATINGS APPLIED IN MONUMENTS OF THE 4TH C BC IN GREECE

MARIA STEFANIDOU*, VASILIKI PACHTA
[1]Lab. of Building Materials, Dept. of Civil Engineering, Aristotle University of Thessaloniki, PO Box:482, 54124, Thessaloniki, Greece

Coatings, such as plasters and renders have been continuously used for dwellings' protection even from the Neolithic period. Diachronically, they present similar technological characteristics forming unwritten normes of manufacture. This paper focuses on the analysis and characterization of coatings from four monuments of Greece, dated through the 4th C BC. The physico-mechanical and chemical properties of samples were determined, leading to the characterization of their consistency and structure. Their stratigraphy showed that they consisted of two to four well compacted layers, the width of which decreased from the inner to the surface layer. The main binding system used was hydrated lime in combination with natural pozzolan, while the aggregates gradation as well the binder/aggregates ratio varied according to the coating's stratigraphy.

Based on the results, it can be concluded that even from the 4th C BC the functional role of renders and plasters was clear and their technology definite. A carefully selected binding system was used, while additional parameters were taken into account in order to achieve the optimum performance and efficasy of the final material.

Keywords: *Historic mortars, Renders, Plasters, Masonry, Stratigraphy, Technology*

1. Introduction

Mortars started to be used as protective coatings of dwellings, during the Neolithic period (8th millenium BC) and since then they constantly contributed to the longevity and durability of constructions [1-5]. In Greece the first clay-based renders are dated during the 7th millenium BC in the wooden huts of the Neolithic settlement of Sesklo [6].

Diachronically, their manufacture was based on the available raw materials, while their application technology was aiming at a resistant final product, the outer layer of which was often finished with grinding, polishing or decoration [3, 7-11].

Pre-historic and historic coatings were usually applied in successive (two to four) layers, characterized by good compaction and coherence, the width of each being decreased to the outer surface [1,2, 5, 11,12].

Neolithic renders and plasters were usually in clay-based, which was often mixed with organic (grasses, roots, straw, reeds) and inorganic (sand, gravel) additives, in order to improve the final properties of the mixtures (volume stability, cracking elimination) [1,2, 13]. According to literature [1, 14-15], hydrated lime started to be used in Greek renders, during the Minoan Period (1700 – 1450 BC). In the Archaic (610-490 BC) and Classic (490 – 323 BC) period, the megalithic domes of temples were usually covered with a thin layer of lime-based render for protective and aesthetic purposes [16], while archaic cisterns were rendered with durable hydraulic mortar layers based on hydrated lime and natural pozzolan [17,18]. Hellenistic (323-31 BC) renders and plasters were also based in hydrated lime according however to limited Researchers [19, 20].

The study of their technological evolution reveals a great source of information regarding the type and proportions of raw materials used (binders, aggregates, additives, admixtures), their application technology, as well as their resistance to ageing and environmental factors [12, 15, 21-24].

This paper focuses on the analysis and characterization of historic renders and plasters taken from four monuments of Greece, dated through the 4th C BC. The aim of the study was to identify their consistency and structure (type and proportion of raw materials), as well as to evaluate their technological characteristics.

The study of renders and plasters dated during the specific century (4th C BC), covers two historic periods in Greece (Classic and Hellenistic), which are characterized by a high level of civilization closely related to monumental constructions. It provides significant research data regarding the technological characteristics of mortars used for specific applications of that period, which were aiming in the durability of structures and resistance to environmental factors.

* Autor corespondent/Corresponding author,
E-mail: stefan@civil.auth.gr

2. Materials and Methods

The monuments from where the samples were taken, are situated in Northern Greece (Fig.1) and concern the following:

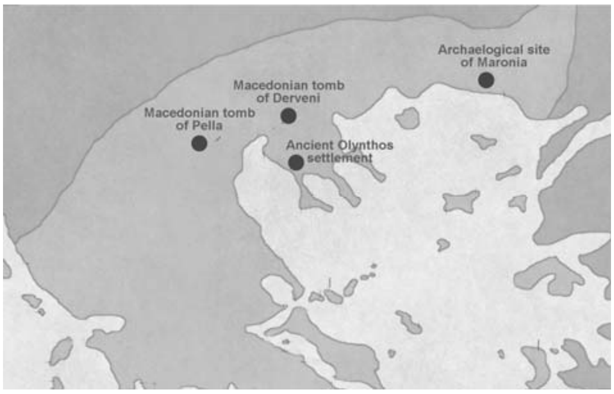


Fig. 1 - Map of Northern Greece depicting the monuments' location.

- Ancient Olynthos settlement (beginning of the 4^{th} C BC): the ancient city of Olynthos is situated in Chalkidiki peninsula and was inhabited from the Neolithic period (5^{th} millennium BC). During the 4^{th} C BC it became one of the most important settlements of N. Greece, covering an area of 600x300m^2, according to the Hippodamius planning system [25]. The analyzed render samples (two samples were analyzed) were taken from the external surface of a rubble masonry, built with unshaped stones and structural mortars based on clay and lime [24].
- Dionysus Sanctuary, archaeological site of Maronia (4^{th} C BC): The ancient city of Maronia was established during the 7^{th} C BC and was gradually transformed to an important politico-economic center [26]. The archaeological site concerns various Hellenistic and Roman monuments, among which it is the sanctuary of Dionysus. The analyzed plaster samples (two samples were analyzed) were taken from a rubble masonry, built with unshaped stones.
- Macedonian tomb of Derveni (end of 4^{th} C BC): The Macedonian tomb is located in the Eastern part of the necropolis of Derveni. It is a monumental construction, consisting of two chambers, the external façade of which was according to the Ionic rhythm [27]. The analyzed plaster samples (two samples were analyzed) were taken from the ashlar masonry of the external façade, built with large stone domes and thin mortar joints.
- Macedonian tomb C' of Pella (end of 4^{th} C BC): The Macedonian tomb C' of Pella is situated in the main road of Thessaloniki – Edessa. It consists of two chambers the external façade of which was according to the Ionic rhythm. The analyzed plaster samples (two samples were analyzed) were taken from the ashlar masonry of the external façade, built with large stone domes and thin mortar joints.

All samples were analyzed, in order to determine their physico-mechanical and chemical properties [10, 28,29].

Insitu and macroscopic observation assisted in identifying the samples' stratigraphy (number- width of layers), as well their preservation state. Microstructure observation was performed with stereoscope (Leica Wild M10) and polarized microscope (Leitz Laborlux12 POL S) assisted by image analysis (ProgRes), as well as Scanning Electron Microscopy (JEOL840A JSM). Aggregates granulometry was tested by hand grounding and sieving, according to EN1015-1:1998. Porosity and apparent specific gravity were performed according to RILEM CPC 11.3, while compressive strength was tested only in samples of adequate width (approximately 2cm), by shaping cubic samples [28,29]. In one sample taken from the Olynthos settlement, drilling resistance technique was applied (DRMS Sint Technology), in order to record the profile of the successive layers strength behavior. Finally, wet chemical analysis assisted by Atomic Absorption was performed in the fine fraction of each sample (<75μm).

Additional comparative observations were made, regarding their structure and constituents, such as binding-system, aggregates' granulometry and type, binder/aggregate (B/A) ratio, colour and texture of each plaster sample. The B/A ratio was estimated by correlating the results of the stereoscopic observation and the granulometry of each sample.

3. Results and Discussion

The results regarding the stratigraphy, as well as the physico-mechanical and chemical properties of the samples are presented in Tables 1,2 and Figure 2.

3.1 Olynthos settlement

In the case of Olynthos settlement, the two samples were taken from the render which was covering externally the rubble masonry of two houses [24]. The first sample was taken from a house that was at the main street of the settlement and was near to a drainning channel, while the second one was sampled from a house of a side road. The characteristics of both samples were similar (Table 1, 2). They consisted of two very well compacted layers, of the same width (internal: 5-6cm, external:1mm), the internal layer of each was of pale brown color. Their difference was that in the first sample the external layer was red and in the second it was white (Table 1).

Table 1

Technological characteristics of samples

Monument / Date	Type	Stratigraphy / layers' width	Color
Olynthos settlement / beginning of 4th C BC	render	2 layers Internal: 5-6cm External: 0.1cm	Internal: pale brown External: red
		2 layers Internal: 5-6cm External: 0.1cm	Internal: pale brown External: white
Dionysus Sanctuary, archaeological site of Maronia / 4th C BC	plaster	4 layers Internal: 1.5cm Intermediate: 1.5cm External: 0.5cm Surface: 0.1cm	Internal: pinkish, Intermediate : whitish External: white Surface: white
Macedonian tomb in Derveni / end of 4th C BC	plaster	2 layers Internal: 0.6cm External: 0.2cm	Internal: pinkish External: white
Macedonian tomb C' of Pella / end of 4th C BC	plaster	3 layers Internal: 1.0cm Intermediate: 0.5cm External: 0.1cm	Internal: brown External: white Surface: white

Table 2

Physico-mechanical and chemical properties of samples

Monument	Layer	Binder type L: Hydrated lime P: Pozzolan	Total soluble oxides (% w/w)		B/A ratio	Aggregates type / gradation (mm)	Compr strength (MPa)	Po-ro-sity (%)	Ap. specific gravity (gr/cm^3)
			CaO	Fe_2O_3+ Al_2O_3+ SiO_2					
Olynthos settlement	Internal	L+P	40	31	1/2.5	Siliceous / 0-8	4.8	22.06	1.870
	External	L	45	20	1/1	Carbonate / 0-1	*	*	*
	Internal	L+P	39.2	32.3	1/2.5	Siliceous / 0-8	*	21.5	1.872
	External	L	46.5	19.1	1/1	Carbonate / 0-1	*	*	*
Dionysus Sanctuary	Internal	L+P	30.5	45.7	1/2	Siliceous origin / 0-4 Crushed brick / 0-4	1.21	24.91	1.860
	Intermediate	L+P	40.3	36.6	1/1.5	Siliceous origin / 0-2.5	1.08	21.49	1.803
	External	L+P	38.5	38.9	1/1	Siliceous origin / 0-2.5	-*	16.35	1.914
Macedonian tomb of Derveni	Internal	L+P	35.5	47.2	1/2	Siliceous origin / 0-2.5	-*	15	1.747
	External	L	-	-	-	No aggregates	-*	-*	-*
Macedonian tomb C' of Pella	Internal	L+P	24.8	59.8	1/2	Siliceous origin / 0-4 Crushed brick / 0-2	-*	14.47	1.750
	Intermediate	L+P	21.6	63.9	1/1.5	Siliceous origin / 0-2 Crushed brick / 0-2	-*	-*	-*
	External	L	49	16.8	-	No aggregates	-*	-*	-*

* physical or/and mechanical properties were not measured since there were no sound samples

The contact zone between the two layers was 10μm thick and extremely firm in both cases (Fig.3). The internal layer in both samples was rich in hydrated lime, while as it is concluded by the high content in soluble SiO_2+Al_2O_3+Fe_2O_3, a reactive soil (probably of local origin) acting as pozzolanic material seems to have been used (Table 2). It consisted of a high proportion of rounded aggregates mainly of siliceous origin, with strong paste-aggregate interfaces (Fig.3). The B/A ratio, as recorded by the combination of stereoscopic analysis and granulometry, was approximately 1:2.5. The porosity was around 22% and the apparent specific gravity 1.87. These hydraulic mortar layers were dense in structure and it seems that they were purposely manufactured to resist moisture.

The external layer of both samples consisted of lime as binder and crushed carbonate aggregates of various sizes (maximum 700-800μm) (Fig.3). The porosity, as recorded by microstructure observation, was low, due to pores of 60μm diameter. The red color of the first sample, as shown by SEM analysis (Fig. 4), was due to the presence of Fe rich components and was probably serving aesthetic reasons.

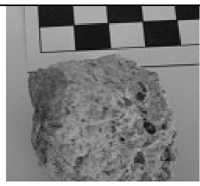
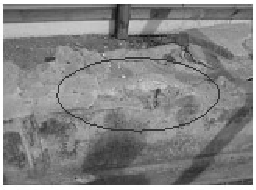
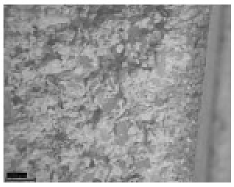
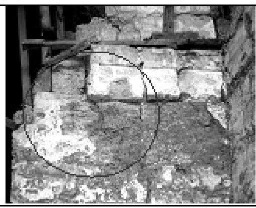
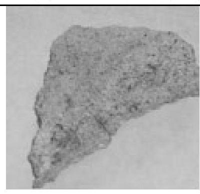
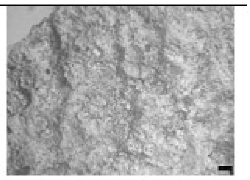
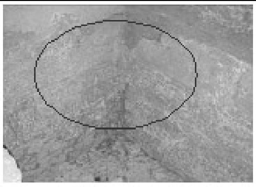
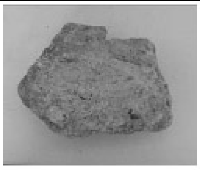
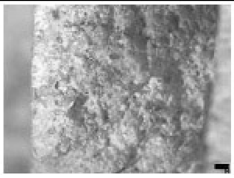
Monument / Date	Sampling	Macroscopic photo	Microscopic photo
Olynthos settlement / beginning of 4th C BC			
Dionysus Sanctuary, archaeological site of Maronia / 4th C BC			
Macedonian tomb of Derveni / end of 4th C BC			
Macedonian tomb C' of Pella / end of 4th C BC			

Fig. 2 - Sampling, macroscopic and microscopic figures of samples

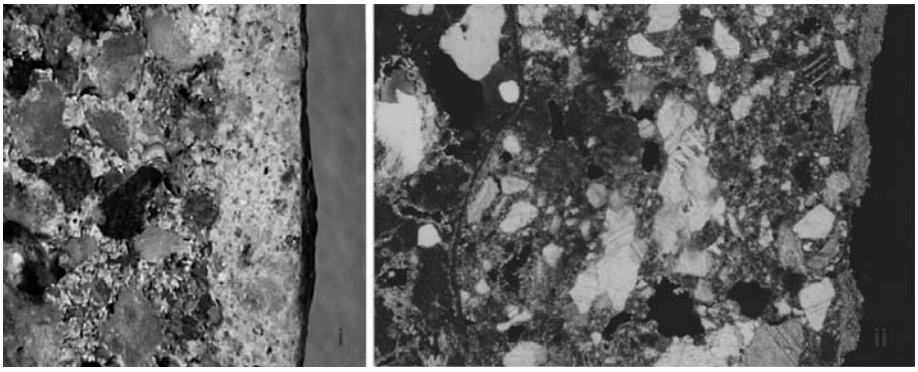
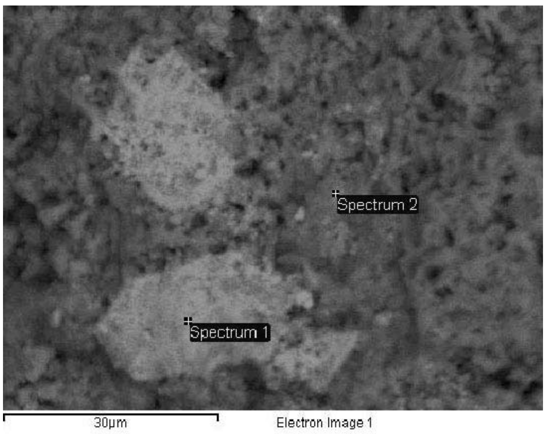


Fig. 3 - Stratigraphy of the sample with the white external layer from Olynthos settlement and contact zones between the layers.



Spectrum	Na	Mg	Al	Si	K	Ca	Fe
Spectrum 1	0.25	1.34	0.78	0.07	0.89	2.43	87.14
Spectrum 2	0.06	0.63	0.07	0.33	0.76	84.97	15.43

Fig. 4 - SEM image of the grains in the red layer and analysis of the components

In the second sample, the layer was calcitic and was probably for protective purposes (Fig. 3).

The compressive strength of the internal layer of the first sample was relatively high, 4.5-4.8MPa, indicating the dense structure of the samples, as well as its good state of preservation. In an effort to verify the strength of the sample, a drilling resistance technique was applied in a depth of 20mm. Softer and harder zones were recorded, probably due to the components met during the drilling procedure, but generally a strong structure was recorded (Fig. 5).

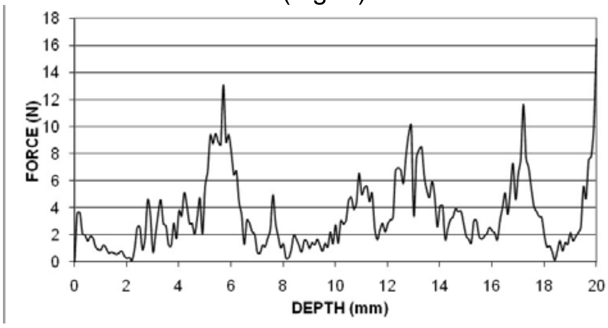


Fig. 5 - Drilling resistance of the Olynthos render at the depth of 20mm.

3.2 Dionysus sanctuary

The analyzed plaster samples were attached to a rubble masonry. They consisted of four well compacted layers, the width of which was reduced from internal (1.5cm) to external (0.5cm) (Table 1). The surface layer (1mm width) was covered with a mural imitating orthomarmarosis (no analysis results have been provided for this layer due to the few remnants) .

The binding system of the three main layers consisted of hydrated lime and natural pozzolan (confirmed by the high content in $SiO_2 + Al_2O_3 + Fe_2O_3$) (Table 2). The B/A ratio was increased to the external layer and rated from 1/2 internally to 1/1 externally. Aggregates gradation was also reduced to the façade and rated from 0-4mm (internal) to 0-2.5mm (external). The aggregates were siliceous, of natural origin and pale color,

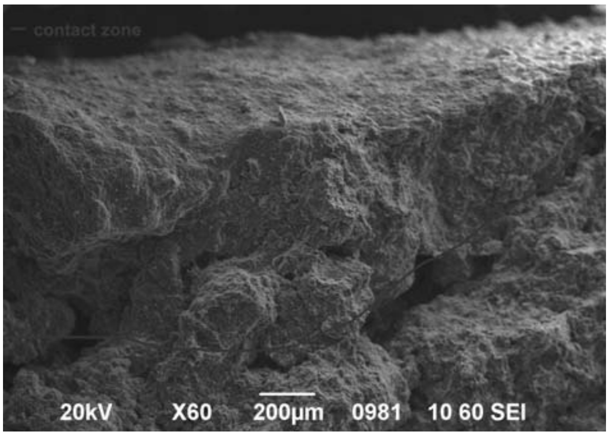


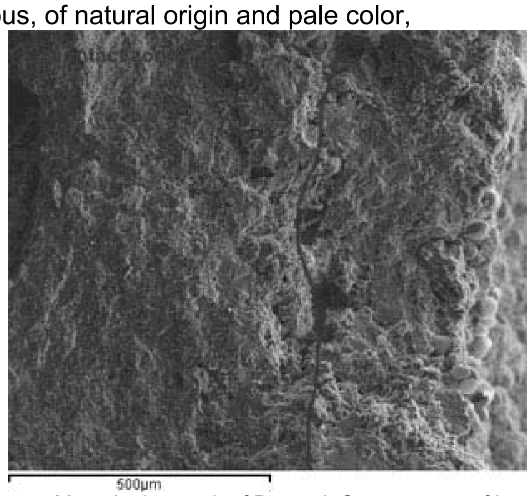
Fig. 6 - Contact zone of the two layers of the Olynthos render

while in the internal layer a short proportion of fine crushed brick was identified, resulting in its pinkish hue. Porosity was reduced to the external surface, as a result of the aggregates gradation decrease and the B/A ratio increase, while apparent specifc gravity tended to be increased. Compressive strength seemed to be decreased to the outer layer. The cohesion between the intermediate and the external layer is shown in Figure 6.

3.3 Macedonian tomb of Derveni

In the case of the Macedonian tomb of Derveni the samples were taken from the plaster which was covering an ashlar masonry made with large stone domes and thin mortar joints. The cohesion of the plasters to the masonry was loose and was related to the bad conservation state of the monument. The samples consisted of two well-compacted layers, with a decrease of width from the internal (0.6cm) to the external (0.2cm).

The internal layer concerned a lime-pozzolan mortar (confirmed by the high content in $SiO_2 + Al_2O_3 + Fe_2O_3$) (Table 2, Fig. 7), with aggregates of siliceous origin of 0-2.5mm granulometry. The porosity was around 15% with a respective apparent specific gravity of 1.747.



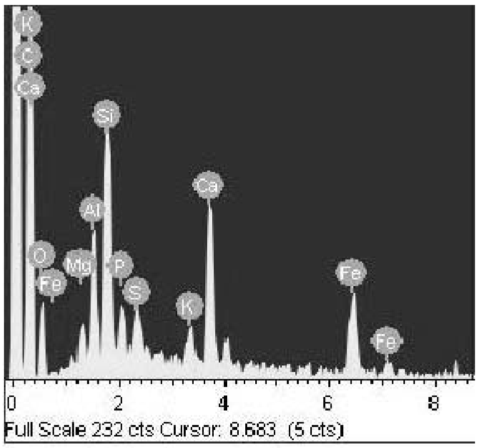


Fig. 7 - Macedonian tomb of Derveni. Contact zone of layers and composition of the internal layer by SEM-EDS.

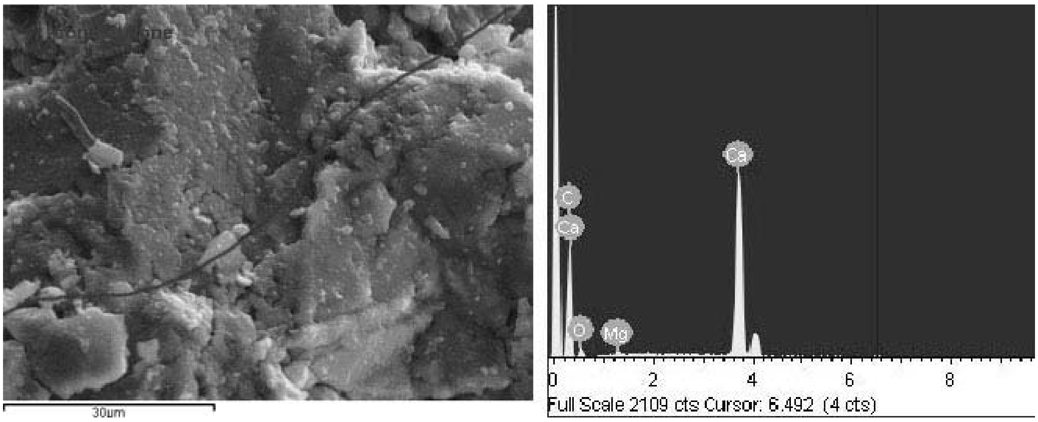


Fig. 8 - Macedonian tomb of Derveni. External layer of calcite nature by SEM-EDS

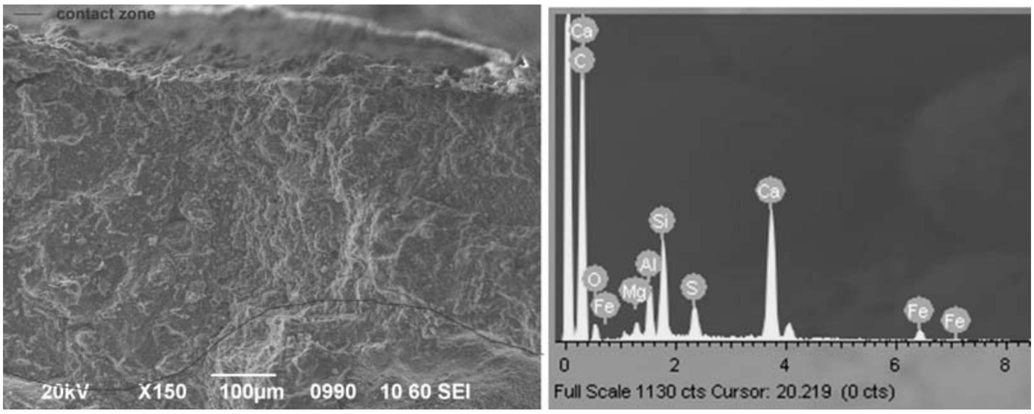


Fig. 9 - Macedonian tomb C' of Pella. Contact zone of the two layers and composition of the internal zone by SEM-EDS.

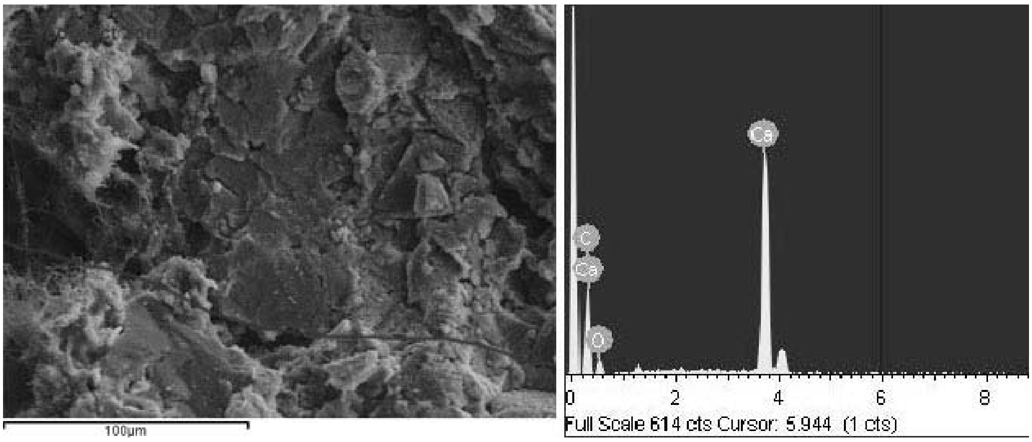


Fig. 10 - Macedonian tomb C' of Pella. SEM-EDS of the external calcite layer.

According to the SEM analysis of the external layer (Fig. 8), hydrated lime was the only binding agent detected, while no aggregates were observed. This layer was mainly decorative, for smoothening the final surface.

3.4 Macedonian tomb C' of Pella

In Macedonian tomb C' of Pella the samples were also taken from a plaster covering an ashlar masonry made with large stone domes and thin mortar joints. It consisted of three well-compacted layers, with a decrease of width from the internal (1cm) to the external (1mm) (Table 1).

The two main layers (internal and intermediate) were of dense structure. The internal one was based in hydrated lime and pozzolan, as has been confirmed by the chemical (Table 2) and SEM analysis (Fig.9) that showed a high content in SiO_2 + Al_2O_3 + Fe_2O_3. The aggregates were of siliceous origin and their granulometry was decreased from of 0-4mm (internallly) to 0-2.5mm (externallly). A low proportion of crushed bricks (0-2mm) was identified in both layers. The porosity was 14.5% with a respective apparent specific gravity of 1.750.

The external layer of the sample (1mm width) was polished, without aggregates and was based in hydrated lime (Fig. 10). This layer was mainly decorative, for smoothening the final surface.

3.5 General remarks

According to the results, during the 4th C BC, renders and plasters were applied on rubble or ashlar masonries, in order both to protect the structure and improve their appearance. Their application followed succesive layers of reduced width to the outer surface. In the case of rubble masonry (Olynthos settlement and Dionysus sanctuary), thick coverings were applied, while in the case of ashlar masonry (Macedonian tombs of Derveni and Pella), the coverings were fine. The compaction of all layers was high, as has been proved by their dense microstructure and the low porosity values of the tested samples.

The parameters related to the longevity of the analyzed coatings (layers' compaction and adhesion, compressive strength) implies the knowledge of ancient masons in manufacturing and applying mortars. The application of a multi-layer mortar and the way the layers were succeeded seems to have followed some rules. From internal to external, the layers were more compact, reducing the porosity and blocking the moisture to enter in the masonry. The compact transition zones between the layers confirm the material's stability.

Regarding their binding system, the combination of fine crystalline hydrated lime and natural pozzolan was used in the internal layers of all cases, leading to the manufacture of durable hydraulic mortars, resistant to humidity. Taking into account the location and functional role of samples (Olynthos: vicinity to drainning system, Dionysus sanctuary: mural substratum, Macedonian tombs: intense humidity), it is clearly pointed out that the need for waterproof materials emmerged the deliberate use of natural pozzolan. The use of natural pozzolan in lime based mortars has been also identified in other Hellenistic mortars of specific applications, such as the substratum of floor mosaics in Pella and Vergina [30-31], which were also manufactured according to the need of resistance to humidity.

In all analyzed samples, a very thin (1mm), calcitic external layer was used, which either had no aggregates or a short proportion of fine aggregates (0-1mm) . This layer was either polished (Macedonian tombs, Olynthos) or decorated with mural (Dionysus sanctuary). Its functional role seems to be both protective and aesthetic.

These results allow the conclusion that hydrated lime in combination with natural pozzolan have been systematically used in the internal layers of renders and plasters, from the beginning of the 4th C BC, while externally they were covered with a fine calcitic layer.

4. Conclusions

The analysis of the structural, physico-mechanical and chemical properties of historic renders and plasters dated during the 4th C BC, proved their high quality, as well as the systematic application of specific technological principles for their manufacture. Their functional role (protection of the structure from humidity, aesthetic improvement of facades) implied their consistency and structure, forming unwritten normes of manufacture.

Synoptically, the main characteristics of the analyzed samples were:

- Stratigraphy of two to four well compacted layers of total width ranging from 1 to 6cm. Thick renders and plasters were used in rubble masonry, while thin plasters, were applied in ashlar masonry.
- Decrease of layers' width, aggregates gradation and porosity to the tested external layer
- Increase of the B/A ratio to the external layer
- Use of hydrated lime and natural pozzolan, in the internal layers, forming hydraulic mixed type binding systems resistant to humidity.
- Thin external layers of 1mm width, based on hydrated lime, used for protective and aesthetic reasons.

Taking into account the aforementioned principles, it seems that from the 4th C BC, ancient masons were fully aware of raw materials' and final products' properties and they were capable of producing durable, resistant to moisture mortars by exploiting the available materials and techniques to the maximum.

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