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Etruscan render mortars from *Domus dei Dolia* (Vetulonia, Italy)

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ABSTRACT: The ruins and artifacts of *Domus dei Dolia* testify to its tragic end and the hasty scape of its wealthy owners in the 1st century BC. The excavating campaigns that started at 2009 have revealed several materials of personal and constructive use that allow an integrated archaeometric study on the way of life of Etruscan owners and excellence of construction. This work intends to contribute to the characterization of mortars and construction materials within a broader multidisciplinary study. The data obtained in renders show a careful selection of materials and applied techniques whereas the provenance of the raw materials is in agreement with a distance until 8 km.

1 INTRODUCTION

1.1 *Archaeological contextualization and motivation*

The *Domus dei Dolia* is located in the Hellenistic quarter of the old town of Vetulonia, now Poggio Renzetti within Tuscany region in Italy. Destroyed as a result of a fire, the ruins of the *Domus dei Dolia* remained hidden until 2009, the year of the beginning of the archaeological work. Through the classification of the archaeological materials recovered, the *domus*, and the whole city, was probably destroyed around the 1st century BC (Agricoli *et al.*, 2016). Destruction was due to the reprisals made by Lucio Cornelio Silla after the victory over Gaio Mario in the bitter dispute that saw the Etrurian cities take party in favor of the latter during the Roman civil war. The different materials used and the artifacts found reveal the richness of the building and its inhabitants (Agricoli *et al.*, 2016).

Through the compositional and textural characterization of render mortar samples, this work intends to unravel the production techniques of the Etruscan people in an urban civil house. No less important is to address the choices and decisions in the search for raw materials through probable/potential exploitation sites.

1.2 *Sampling and methodology*

Eight render mortar samples were collected (placed at disposal by Museum "I. Falchi" in Vetulonia – Town Hall of Castiglione della Pescaia and by Superintendence Archaeological of the Tuscany), for their compositional and textural characterization. The samples of render mortars come from the same division of the house – the *triclinium* – and present mural painting. Most of the samples exhibit a clear stratigraphy (Fig. 1): a chromatic layer over a white/grey mortar render (*intonaco*), which in turn rests on a beige/yellowish mortar (*arricio*). Within the upper layer (*intonaco*) it is possible to detect a discontinuity (Fig. 1) probably related to two application moments.

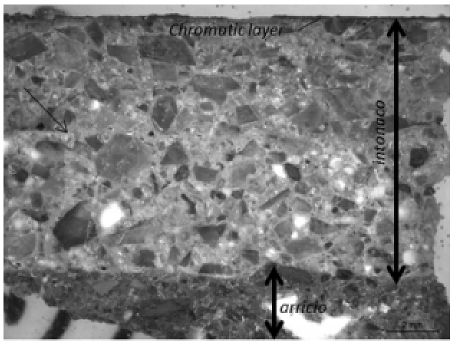

Figure 1. Stereo-microphotograph of a cross section of a representative sample of a render mortar (arrow highlights a discontinuity within *intonaco*).

The data acquisition consisted of set of multi analytical techniques composed by X-ray diffraction (XRD), thermogravimetric analysis (TGA), scanning electron microscopy coupled to X-ray dispersive energy detector (SEM-EDS) and thin-section optical microscopy. Powdered global fractions were obtained for each layer.

2 RESULTS AND DISCUSSION

2.1 *Optical microscopy*

The mortars were thoroughly examined in the laboratory using a stereo-zoom microscope. Optical microscopy observations, photographic documentation as well point counting were obtained by a bright field microscope Leica DM2500P equipped with a Leica DFC 290HD camera associated with PELCON automatic point counter apparatus.

Petrography (Fig. 2) enables to confirm preliminary observations with stereo-zoom-microscope (Fig. 1) as well highlight some textural features. The aggregates of the upper layer are crystals of calcite with very angular shapes, evidencing twins and cleavage, immerged on a microcrystalline continuous binder matrix. Some primary porous in the binder show a calcitic recrystallization in the rims. On these layers it was made point counting in order to obtain the binder:aggregates volumetric ratio. It was counted around 1000 points for each sample and which indicate approximately a proportion 1:1.

In the lower layer is noticeable the variety of materials: minerals such as quartz and feldspar, but also sedimentary rocks such as chert, sandstone and clay most of them with rounded shapes. Some lime lumps were observed. In the same way as it was observed in the *intonaco* the texture of the binder is continuous and microcrystalline.

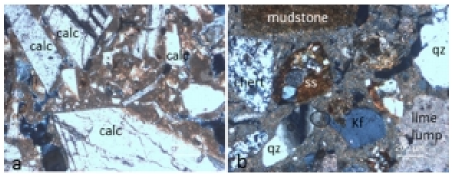

Figure 2. Photomicrographs with transmitted light under crossed nicols of *intonaco* upper layer (a) and lower layer *arricio* (b). calc – calcite, qz – quartz, Kf – K feldspar, ss – sandstone.

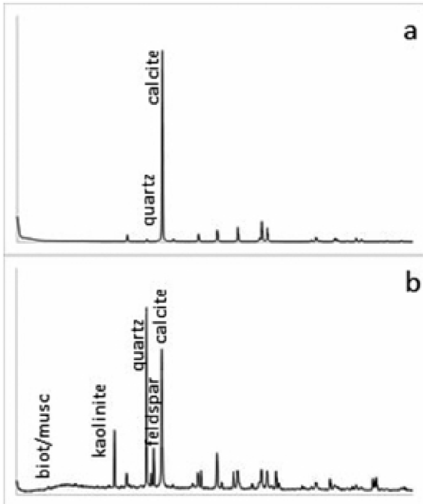

Figure 3. Diffractograms from (a) *intonaco* upper layer (b) *arricio* lower layer.

2.2 *XRD*

In order to confirm the mineralogical phases of the mortars, X-Ray diffraction was performed using a BRUKER AXS D8 Discovery diffractometer with a Cu Kα radiation source at 40 kV and 40 mA and the Lynexe 1D detector. The diffractograms were obtained in the interval 3–74° 2θ, step of 0.05°, time per step of 1s. The identification of the phases was performed with the Bruker EVA software package (Version 3.0) using the PDF-ICDD Powder Diffraction Database (International Centre for Diffraction Data).

The information obtained by XRD reinforces the differences of the two layers. The upper layer with a simple composition; mainly calcite associated to tiny amounts of quartz (Fig. 3a) and the lower layer diffractograms highlights the complexity of geological material as aggregates (Fig. 3b). These are dominated by quartz (present as single grains, chert and sandstone), feldspar (present as single grains and sandstone), micas (present in some sandstones and pelitic sediments and kaolinite.

2.3 *TGA*

The mortar's binder composition and CO_2 content were assessed by thermogravimetry (TGA) performed on a Netzsch STA 449F3 Jupiter analyser, under nitrogen atmosphere, with heating rate of 10°C/min, from room temperature to 1000°C. TGA measures the weight (mass) change of a sample as a function of temperature and so allows to estimate the amount of phases that are decomposed by heat such as $CaCO_3$ (calcite).

On the analyzed global fractions of mortars the observed drop of mass weight between 500°C and 900°C is attributed to decomposition of calcite (e.g. Földvári, 2011) by liberation of CO_2. The major mass loss is observed on the upper layer of samples and is between 39.9% and 42.7% which indicates a range of 91 to 97% of calcite. That is almost 100% of the layer is composed by calcite (binder and aggregate). On the other hand the drop of the mass for the lower layers is respectively 8.6% and 10.9% which indicates 20 to 25% of calcite that is a proportion binder:aggregates of 1:4 and 1:3.

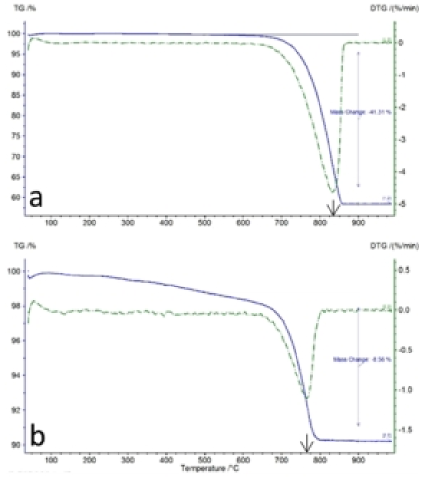

Figure 4. Representative thermograms from: (a) upper layer (*intonaco*) and (b) lower layer (*arricio*).

It is noteworthy to mention the higher decomposition temperatures (~825°C) of primary calcite (metamorphic) within upper layers (Fig. 4a) when compared to recrystallized calcite (−775°C) from the lime/binder as in the lower layer (Fig. 4b).

2.4 *SEM-EDS*

Scanning electron microscopy coupled with energy dispersive X-ray spectrometry (SEM-EDS) was carried out using a Hitachi S-3700N variable pressure scanning electron microscope coupled with a Bruker XFlash 5010 SDD Detector. The observations were done under a pressure of 40 mPa and the accelerating voltage used was 20 kV.

SEM-EDS analysis allowed to assess the composition of some aggregates as well as to estimate the composition of the binder. At *intonaco* layers the EDS spectrum suggests that the binder is a calcitic aerial lime (Fig. 5a–b). On the other hand the presence of Si and Al in the binder of *arricio* (Fig. 5c–d) is in agreement with the presence of thin phyllosilicates in the binder matrix.

2.5 *Discussion of results*

The integration of the various techniques indicates that render mortars consist of a preparatory layer (*intonaco*) with calcitic aggregates displaying very angular contours which suggest the use of in situ brittled recrystallized limestone/marble. Quartz aggregates are rare and very small in size. The binder is an aerial calcite lime. Due to the presence of calcitic aggregates in this layer, the determination of the binder:aggregate ratio was made by point counting on thin section and corresponds to a volume proportion of 1:1.

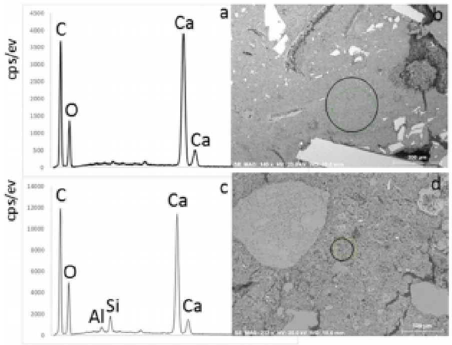

Figure 5. Analyze of binders: (a) EDS spectrum from the binder in the intonaco and (b) BSE image of intonaco binder, (c) EDS spectrum from the binder in the arricio; (d) BSE image of arricio binder.

The underlying beige/yellowish mortar is clearly contrasting from the compositional and textural point of view. The aggregates are mainly silicates prevailing the quartz over the feldspars and lithics (sandstones, slates, cherts), being still observed lime lumps. The shape of the aggregates is rounded to well-rounded suggesting a significant transport from geological source, probably corresponding to a river sand. Although with thin phylosilicates the binder is identical to the *intonaco* that is an aerial lime of calcitic composition. The TGA enable a binder: aggregates ratio of 1: 4 to 1:3 for the *arricio* layers.

According to the available geological map, the river sands are compatible with a distance of less than 5 km, but the raw material for the crushed *intonaco* aggregates (marble) and limestone required for lime are 8 km apart.

3 CONCLUSIONS

For the studied samples the results show a strong coherence between them which is in agreement with the fact that they belong to the same division. *Arricio* layer applied with a proportion of 1:3 or 1:4 with a calcitic aerial lime and river sands as aggregates. *Intonaco* layer applied in two moments with a proportion of 1:1 with aggregates of crushed marble.

The specificity of the materials used in each layer reveals a careful preparation of the materials according to the nobility of *triclinium* and the wealth of its owners.

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