



Characterization of multi-layered wall paintings from a Roman *domus* in Santa Maria Capua Vetere

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ABSTRACT

This paper regards the preliminary characterization of wall paintings sampled in a Roman *domus* located in the city of Santa Maria Capua Vetere. Despite its history and archaeological remains, there is a lack of scientific studies on Roman materials and techniques adopted in this site. Samples were taken from different walls of the *villa*. Pigments, binders, and mortars were analyzed by optical and electronic microscopies, vibrational spectroscopies including Fourier transform infrared and micro-Raman imaging, and mass-spectrometry coupled to gas and liquid chromatography. Altogether, the multi-layers preparation, the rich pigments palette (both natural and synthetic ones) and the variety of the organic ligands used (terpenic resins or animal glue) support the hypothesis of a very wealthy owner. The absence of calcite in the pictorial layer and the presence of organic binders clearly indicates an *a secco* technique.

Section: RESEARCH PAPER

Keywords: Pigments; binders; mortars; proteomics; Raman imaging; measurement

Citation: M. Alberico, M. Rossi, L. Birolo, G. Ntasi, P. Maddalena, M. Salvatore, A. Tomeo, A. Vergara, Characterization of multi-layered wall paintings from a Roman *domus* in Santa Maria Capua Vetere, Acta IMEKO, vol. 13 (2024) no. 3, pp. 1-6. DOI: [10.21014/actaimeko.v13i3.1794](https://doi.org/10.21014/actaimeko.v13i3.1794)

Section Editor: Fabio Leccese, Università Degli Studi Roma Tre, Rome, Italy

Received February 27, 2024; **In final form** March 12, 2024; **Published** September 2024

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Funding: This work was financially supported by PNRR PE5 CHANGES (PE00000020).

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1. INTRODUCTION

In the Roman age, paintings were mostly executed on wall plaster, even though other supports were common as wood, ivory and others. The colours used to obtain wall paintings were typically composed of mineral pigments with calcite and/or organic binders. Depending on the type of binder, multiple preparative techniques of wall paintings were classified and first described in Vitruvius's manual *De Architectura* and in Pliny's *Naturalis Historia* [1]. In a *fresco* painting technique (meaning on fresh plaster) the colour was spread directly on wet plaster. Alternatively, other colours were often added *a secco* (on dry plaster), resulting in mixing the mineral powder with organic binders (mostly wheat paste, egg, resins, and emulsified beeswax) to easily spread them. Historical mortars are composed of

hydraulic or aerial binding material, aggregates in crystalline or amorphous form and additives (passive or active) which react with the binding material. Among the elements that compose historical mortars, additives easily can undergo modifications due to hardening and ageing processes [2]. Among the additives that can be used there are minerals. In Roman times it was popular the minerals addition as pozzolan or ceramic crushed fragments, used as latent hydraulic because they combine with lime to give other hydration products useful for cement stability [3].

In this work we present a characterization of wall paintings found in a Roman *domus*, located in the actual city of Santa Maria Capua Vetere corresponding to the ancient Capua [4]. In Section 2 the archaeological site will be discussed. The characterization of the murals in terms of colours and/or organic binders, examined in Section 4, can reveal important aspects

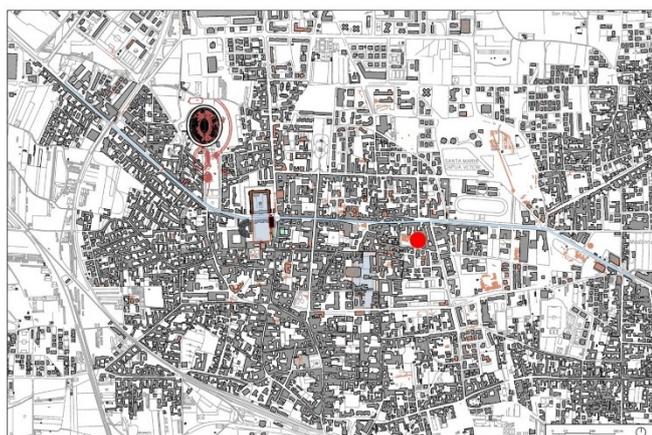


Figure 1. Topographical framework in the urban context of ancient Capua [4].

about its conservation state [1], [5]-[7]. Vibrational spectroscopies, optical and electronic microscopies and mass spectrometric techniques (fully described in Section 3) were herein used to give a chemical picture of the layers composing the wall painting and revealing a large variety of pigments and binders. The data obtained provide information on the components of the samples, including also the mortars (Section 4). Several erratic fragments fallen from collapsed walls were collected by the archaeologist Dr Tomeo during the excavation and classified in terms of stratigraphic units. Among these fragments, we selected the investigated samples (avoiding any further sampling from the site) with a twofold criterion: a) to cover all the colours with different shades used for the paintings and b) to cover different mortar preparations (with both three and four layers). Four fragments of wall paintings were selected on site and analysed (187, 151A, 151B, 151C) with special attention to pigments and binders chemical composition.

2. THE ARCHAEOLOGICAL SITE

During Roman times the city of Santa Maria Capua Vetere lived a very prosperous era, rich enough to be called *Campania felix*. After the domination of the Osci and Etruscans, in 841 the city was almost destroyed by the incursions of the Saracens. Due to its history, the city can count on different archaeological monuments such as the Campano amphitheatre, the arch of Hadrian; however, many archaeological artifacts are still buried.

In 2018 some excavations, in an area located south of the ancient Appian way (Figure 1), have unearthed a multi-level site consisting of three hypogeal nymphaeums; two of them had walls completely frescoed with a type of painting attributable to the so-called IV Pompeian style. The *domus* has a continuity of life from the late republican age to the late antiquity. In plan there are several rooms, a *peristylum* with two ornamental basins and a large area intended for a garden. The plan that can now be read is the result of a progressive increase, as for other *domus* of ancient Capua. Between the late Republican and early Imperial periods, the first large-scale layout of the *domus* can be placed, as documented by the construction of three rooms that develop over a large garden area with a peristyle. In the first half of the third century, an extension was carried out involving the addition of three *nymphaeums* and a large rectangular room with a portico. The three *nymphaeums* of the *domus* present a complexity and a decorative commitment that indicates their exceptional character and is not much compared in other contexts relating to urban residences. This archaeological aspect calls for a scientific

confirmation from our diagnostic study. During the fourth century, several interventions were carried out, including the construction of a porticoed area. Around the middle of the fifth century, probably coinciding with the devastation brought by the Vandals of Genseric in 456 A.D., we witness the plunder, devastation and definitive abandonment of the *domus* [4].

3. MATERIALS AND METHODS

The samples were taken from the archaeologists of Superintendence of fine arts and landscape of province of Caserta and Benevento.

3.1. Optical microscopy

The stereo and petrographic microscopes used are the AXIO ZOOM V16 and AXIO IMAGER A1m by Zeiss powered by the Museum Center of Natural and Physical Sciences of University Federico II. The lighting takes place from a CL 9000 LED CAN cold source for stereomicroscope. The light reflected in the bright field was used for the work carried out. The AxioCam ICc5 camera was used for image acquisition. The software used for image acquisitions are Axiovision SE64 Ref 4.9.1 with Z-Stack, Extended Focus, and Panorama modules.

3.2. Scanning electron microscopy

Quantitative chemical analyses of the major and minor elements were performed by using a scanning electron microscope (SEM) JEOL-JSM 5310, coupled with energy dispersive X-ray spectroscopy (EDS). The setup is equipped with an Oxford Instruments Microanalysis unit, INCA X-act detector, operating at 15 kV primary beam voltage, 50–100 mA filament current, variable spot size, 20 mm WD, and 40 s net acquisition real time. INCA X-act detector uses Energy software with XPP matrix correction scheme and Pulse Pile up correction [8]. Data were processed with INCA software version 4.08 (Oxford Instruments. INCA, the microanalysis suite issue 17a+SP1, Version 4.08. Oxfordshire, UK: Oxford Instr. Anal. Ltd; 2006). Minerals and pure elements were used as standards for the analyses of thin section. For each mineral in the pictorial layers of thin sections, 25 analytical points were collected.

3.3. Fourier Transform-Infrared spectroscopy

Fourier Transform-Infrared (FTIR) spectra were recorded using a Jasco 4700LE spectrophotometer. Measurements were performed in transmission mode using KBr pellets. Usually, archaeological samples are taken from clean areas of the manufacture with a scalpel knife. For the sample preparation, 1-2 mg of the fraction mortar were mixed homogeneously with 100 mg of anhydrous KBr in an agate mortar. Sixty-four signal-averaged scans were acquired on the samples in the 4000-400 cm^{-1} region at a resolution of 4 cm^{-1} . The measure was repeated twice.

3.4. Micro-Raman spectroscopy

Two instruments were used for pigments and mortars characterization. The Raman spectra of the pigments were collected at room temperature using a confocal micro-Raman spectrometer (Jasco, NRS-3100), working with the 514 nm excitation wavelength from an Ar^+ laser (intensity at the sample surface of 3 mW) in a backscattering configuration. The scattered light was analysed with a spectrometer equipped with a Peltier-cooled 1024 \times 128 pixel CCD photon detector (Andor DU401BVI). Raman backscattering was collected using a diffraction lattice of 1200 grooves/mm and 0.01-0.20 mm slits,

corresponding to an average spectral resolution up to 1 cm^{-1} [9]. Wavelength calibration was performed by using cyclohexane Raman signal as a reference. The spectral acquisition time varied from 30 to 90 s, and each measurement was triplicated for scope of reproducibility. Mortars μ -Raman analysis was conducted with the integrated apparatus Alpha300 RAS (WITec, Ulm, Germany). A laser beam at $\lambda = 488\text{ nm}$ was used as excitation light source. The beam was focused onto the sample surface by means of a 50x microscope objective (numerical aperture of 0.75) working in epi-illumination mode. The diffraction-limited focused spot in the objective focal plane had a full width at half-maximum (FWHM) of approximately 320 nm. The light backscattered from the sample was collected by the same objective and sent to the spectrograph through a confocal optical collection path. The samples analysis was conducted in the microregions of the samples scanned over the areas of 25×25 and $50 \times 50\ \mu\text{m}^2$.

3.5. Organic molecules analyses by Gas Chromatography

Samples were fractionated, derivatized and analysed as reported in [10].

3.6. Protein extraction

Because of a significant presence of metals and carbonates (observed by spectroscopic techniques), a demineralization step was performed as a pre-treatment step. In particular, about 300 μL of a solution of 0.5M EDTA pH~8 was added to wall fragments for 10 days at room temperature, refreshing the solution every 2 days. After centrifugation for 2 min at 10000 rpm, the collected supernatant was rejected. After 10 days, the pellet was subjected to a denaturation step with urea. Therefore, 10 μL of a solution of 6M urea were added to micro-samples and incubated for 10 min at room temperature, followed by sonication for 20 min. Urea was then 6-fold diluted with water. Subsequently, trypsin was added to a final concentration of 0.1 $\mu\text{g}/\mu\text{L}$ in Ambic 10mM. After incubation at 37°C for 16 hours, the supernatants were recovered by centrifugation at 10000 rpm, and the peptide mixture was filtered on 0.22 μm PVDF membrane (Millipore), concentrated and purified using a reverse-phase C18 Zip Tip pipette tip (Millipore). Peptides were eluted with 20 μL of a solution made of 50 % Acetonitrile, 50 % Formic acid 0.1 % in Milli-Q water and analysed by LC-MS/MS.

3.7. Liquid Chromatography-MS/MS analyses

Samples were analyzed according to Vinciguerra et al [11]. MS/MS spectra were transformed in Mascot Generic files (.mgf) format and routinely used to query the SwissProt database 2015_04 (548208 sequences; 195282524 residues), with no taxonomy restriction. As soon as collagen was detected, the sample was reanalyzed searching a homemade database, "COLLE" (60 sequences; 88859 residues), that collects the sequences of collagen type I and III for all the common domesticates generally used for animal glues) and that has been deposited to Mendeleev Data [12]. Deamidation of Gln and Asn, oxidation on Met, hydroxylation (K) and hydroxylation (P) were set as variable modifications to consider, like deterioration of the samples and hydroxylation of proline, the specific post translational modification of collagen. Only identifications of proteins with at least two peptides with individual ion score above the significance threshold (> 20), were considered as significative.

4. RESULTS AND DISCUSSIONS

All the samples were analysed using a multi-methodological approach, including optical and electronic microscopies, vibrational spectroscopies (Fourier-transformed infrared, FT-IR, and micro-Raman) analysis and mass-spectrometry based techniques.

4.1. Pigments and binders

Based on optical microscopy observations, for all the samples analysed the first layer consists in a pictorial layer with variable colours. The most common colour is red, in various shades, but there are also green, yellow, black, blue as well as white slightly yellowed. A careful analysis with PM and EDS analyses, revealed that the pictorial layers are characterized by the presence of cryptocrystalline and microcrystalline minerals, sometimes associated to minor amount of lime binder.

The Raman spectra demonstrated that the blue colour is made of cuprorivaite ($\text{CuSi}_4\text{O}_{10}$) known as Egyptian blue, related to bands at 430 and 1015 cm^{-1} . Green colour shows signals belonging to the "Green earths", as also confirmed by FTIR data showing glauconite (data not shown). The samples 151A, 151B and 151C show different shades of red, assigned to cinnabar, HgS ($254, 287$ and 344 cm^{-1}), and both hematite (bands at $406\text{ cm}^{-1}, 607\text{ cm}^{-1}, 1318\text{ cm}^{-1}$) and goethite. Black colour can be assigned as amorphous carbon (broad band around 1611 cm^{-1}), while white can be easily assigned as calcium carbonate (290 and 1090 cm^{-1}). To evaluate the organic content a multistep protocol was applied to all the samples to separate the polar fractions, including sugars and compounds of medium polarity, from the unpolar fraction, composed mainly of lipids.

No sugars were detected in any sample, whereas flavonoids, steroids and degradation products of abietic, considered as a marker of pinaceous resin, were identified.

Moreover, steroid compounds were detected in samples 151A and 151B. Several fatty acids were detected as methyl ester derivatives (FAMES) in the unpolar fraction. Palmitic and stearic acid, two of the most common molecules that can be found in many matrices of both animal and vegetable origin, were identified in all samples. Based on literature data [13], the ratio C16:0/C18 in the samples 151 A and 151 B is compatible with the presence of animal fat, although the relative ratio can be affected by ageing.

A proteomic approach with trypsin digestion in heterogeneous phase was performed to investigate the presence of any proteinaceous binder with the protocol described in Vinciguerra et al [11]. The chains of collagen type I from *Bos taurus* were detected in samples 151 A and 151 B in agreement with the identifications of lipids of animal origin reported above. No significant proteins were detected in the samples 151C and 187.

4.2. Mortars

The samples were analysed in two different perspectives, the first is a macroscopic observation on 3D materials using a stereo microscope (SM), the second is a petrographic (PM) observation on thin section whit polarized microscope, back scattered images (BSE), and quantitative chemical analyses (EDS). The samples are made up of wall fragments inclusive of the two or three layers of mortars. On the basis of SM and PM observations, the fragments are generally composed of three layers. The first layer is the pictorial one showing variable colours (Figure 2a), the second layer has a very light colour and shows binder mixed to crystals fragments at first sight (Figure 2a, b, c and d), the third

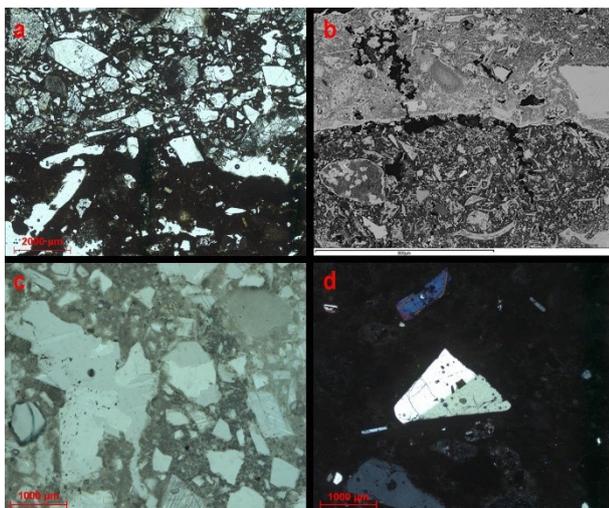


Figure 2. Sample 151. a) Images at polarized transmitted light (//). Contact layer 2 (with fragment of calcite crystals) and layer 3. Sample 151 B. b) Photomicrograph in BSE. Contact between layer 3 and layer 4. Sample 151 B. c) Image at polarized transmitted light (//). Fragment of calcite in lime matrix. Sample 151C. d) Fragment of diopside, k-feldspar, apatite crystals and feldspatized pumice. Image at cross polarized transmitted light (+).

layer is characterized by a brownish binder mixed to volcanic fragments (scoriae, pumices and crystals) with different dimensions (Figure 2c), probably pozzolanic mortar. In the 151 C sample, there is a fourth layer (Figure 2a and b) that can always be traced back to the pozzolanic mortar. This layer shows a darker and less porous binder (Figure 2b) respect to third layer. The 187 sample shows the first two layers strongly altered (Figure 2d).

The first layer is the pictorial one. The second layer is white grey in colour and its thickness varies greatly from 4 to 8 mm. In a thin section, it appears to be made up of a lime mortar with fragments of calcite (CaCO_3) crystals of highly variable dimensions, even millimetres (Figure 2c). Only in the sample 187 there are also dolomite ($\text{CaMg}(\text{CO}_3)_2$) crystals fragments in lime mortar. The third layer could be a “preparation layer” of a typical Roman plaster [14]. It is generally separated from the second one in a sharp way and this is particularly visible when observing the sample in thin section in optical microscopy (Figure 2a). This layer was formed by a micro and cryptocrystalline matrix with some fracture and shrinkage bubbles, and fragments with different dimension. Matrix quantitative chemical composition (via SEM) shows variable amount of lime and silicon (up to 8 %) contents. The fragments are pumice, volcanic glass shard, minerals of feldspar group crystals, plagioclase, clinopyroxene, mica, strongly altered leucite, apatite minerals group, oxides and ceramic fragments in smaller quantities (Figure 2d). Layer 4, when it is present, is a part of plasters.

It was detected only in the 151 C sample. It appears very similar to the third layer but shows darker matrix with sub rounded pores and fissure, crystals fragments and higher concentration of pumice and glass shard compared to third layer (Figure 2b). Chemical analyses have shown that the matrix has a silicate component of up to 18 % and mineralogical composition of crystal fragment are the same of third layer. The fourth layer is a part of plasters or bedding mortars [14]. Infrared spectroscopy (FT-IR) was used to obtain preliminary qualitative information on chemical composition of the mortars and on some of the characteristic materials contained in it (calcium carbonate, dolomite, calcium and magnesium hydroxides, etc.).

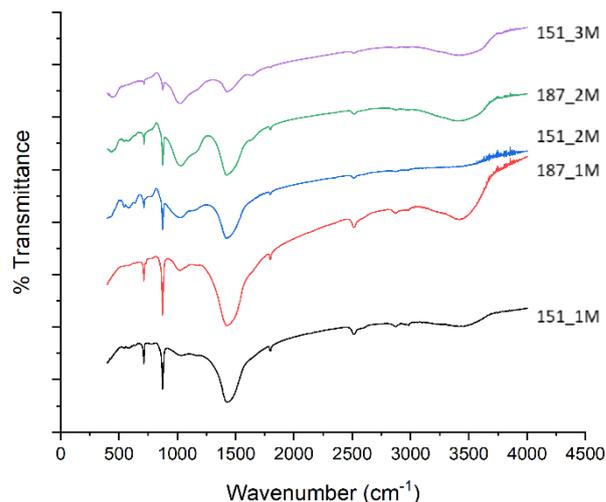


Figure 3. FTIR spectra recorded for mortars sample 151 and 187. The coding refers to first mortar (layer 2, 1M); second mortar (layer 3, 2M); third mortar (layer 4, 3M).

Layer 2 of all the samples analysed is composed exclusively of calcium carbonate.

The FTIR spectra of all the samples show characteristic bands of calcium carbonate (1430 , 873 , and 712 cm^{-1}) as well as the IR band at 3436 cm^{-1} due to free hydroxyl groups of water observed in variable intensity in almost all the samples reported (Figure 3). In particular, the layers 3 and 4 show a characteristic peak (in addition to the presence of calcium carbonate bands) attributable to silicates, i.e. the Si-O-Si band at 1030 cm^{-1} . Micro-Raman imaging allows to collect spectral data from discrete sections of an area of the sample. This method was used to obtain micro-Raman images of the wall paintings focusing the area, containing both the pictorial layer and the first mortar (layer 2).

The samples analysed are 151B and 151C, consisting in two parts of the same wall painted with different shades of red, and 187. Results on 187 (made of cuprorivaite) were difficult to obtain because of the inhomogeneous and degraded coloured surface of the sample and the fluorescence coupled, in some cases, with saturation of the signal due to the laser line. The sample 151B has a brighter shade of red, typically associated with cinnabar (HgS). Micro-Raman image clearly shows the different chemical composition existing between the pictorial layer and the underlying mortar. The extracted spectra (Figure 4, A) of the pigment exhibit bands at 256 cm^{-1} and 347 cm^{-1} attributable to cinnabar and confirmed the presence of calcite in second layer as expected from previous measurements [15], [16]. As to sample 151C, the obtained data including the optical image, micro-Raman image and extracted spectra are reported in Figure 4, B. Dark red pigment shows bands at 283 cm^{-1} , 395 cm^{-1} , 600 cm^{-1} , 1302 cm^{-1} typically attributable to hematite (Fe_2O_3) confirming the above results, while the first mortar (white area in the optical image) is composed of calcite with a diagnostic peak at 1090 cm^{-1} . These measures strongly strengthen the hypothesis of a *secco* pictorial technique because of the absence of calcite content in the coloured area. For second and third mortars (corresponding to third and fourth layers) only Raman single point spectra were recorded, due to the challenging focusing process required for mapping of such inhomogeneous material. For second and third mortars, it was possible to identify calcite and sporadically anatase, TiO_2 , (Figure 5).

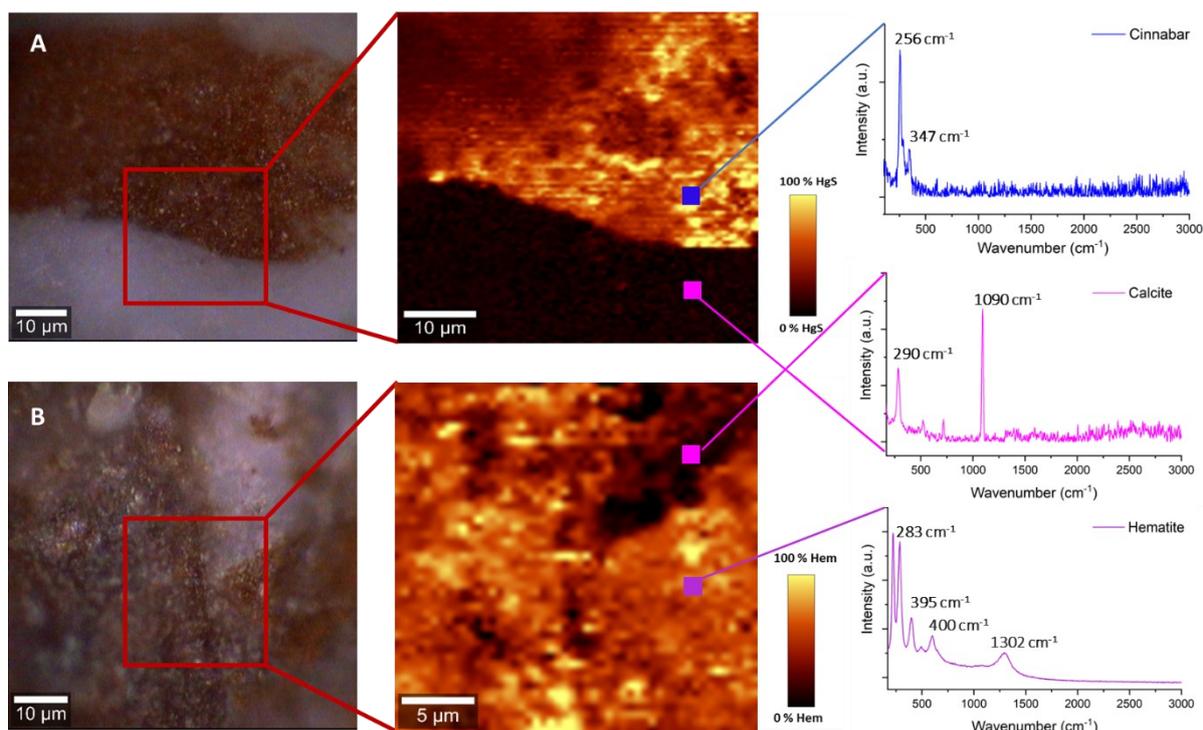


Figure 4. Bright-field image of the investigated sample region collected by 50x microscope objective, micro-Raman image and spectra of 151B (panel A) and 151C (panel B) sample. The Raman spectra results from 200 ms acquisition time, while the Raman images of the analysed regions were reconstructed integrating for each scanned position the Raman signal in a spectral window of 40 cm^{-1} in width, centred at the 256 cm^{-1} peak (panel A) and a spectral window of 50 cm^{-1} , centred at the 280 cm^{-1} (panel B).

5. CONCLUSIONS

An analytical and chemical study of wall painting of Roman origin was performed. To the first analysis of pigments and their inorganic and organic composition it was added an insight on historical mortars. Raman and SEM spectroscopy analyses allowed to characterize the inorganic components of the different layers of the wall paintings of the *domus* in S.M. Capua Vetere. The pigments identified are: i) “Egyptian Blue” the mineral cuprorivaite for blue shades [17]; ii) “Green Earth” the mineral Glauconite [18] for green; iii) cinnabar (HgS), goethite and hematite (Fe_2O_3) for different red shades [18]-[20]. The complex mortars preparation (that goes from two to three layers) results of raw material blends as calcium carbonate, silicates, anatase and others, as shown from the optical microscopy, FTIR

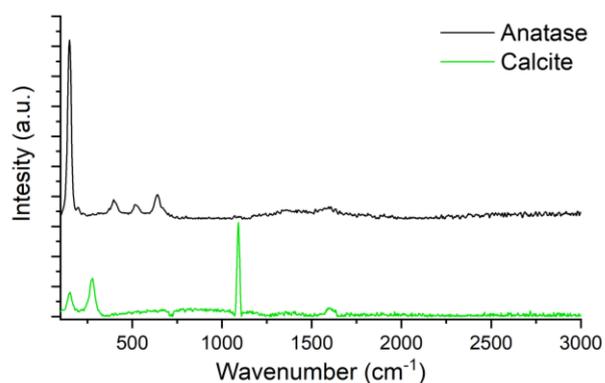


Figure 5. Micro-Raman single point spectra of recurring elements of second and third mortars (layer 3 and 4).

measurements and micro-Raman analysis. The conducted analysis has shown that in the preparation of the wall paintings, resins and animal glue have been extensively used, so they can be classified as *a secco* paintings, where the colour was applied directly on dry mortar. The rich palette and the abundance of binders suggests an aristocratic *villa*.

ACKNOWLEDGEMENTS

This work was financially supported by PNRR PE5 CHANGES (PE00000020).

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DOI: [10.1002/jrs.1518](https://doi.org/10.1002/jrs.1518)