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RESEARCH ARTICLE

Characterization of Pigments in a Wall Painting Fragment from the Archaeological Site of Cava Ranieri's Villa 6, Terzigno (Italy)

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Abstract

Spectroscopic techniques were employed to analyze the pigments of a fragment of a stray wall painting from the archaeological excavation in Terzigno, Italy, located approximately six kilometers north of Pompeii. X-ray fluorescence and Raman spectrometry primarily revealed the presence of iron-based pigments—specifically, red and yellow colors composed of hematite and light ochre, respectively. Additionally, calcite and quartz were identified through optical stratigraphy. Compared to our previous findings from wall paintings in Pompeii, infrared spectroscopy and staining procedures strongly suggested that the painting mixture from Terzigno did not contain organic binders. This indicates that a fresco painting technique was likely used.

Keywords: Wall Paintings, Painting Composition, Fresco, Spectroscopy.

Abbreviations: FT-IR: Fourier transform infrared spectroscopy, XRF: X-ray fluorescence, FWHM: full width at half-maximum.

1. Introduction

The Campanian archaeological sites are invaluable for studying ancient remains of significant historical and scientific relevance (Saiz-Jimenez, C., 2003). Among these, the city of Terzigno, a Vesuvian municipality located approximately 6 km north of Pompeii, holds particular importance. In antiquity, its territory, encompassing the southeastern slopes of Mount Vesuvius, formed the northernmost periphery of the *ager Pompeianus*.

The highest concentration of archaeological discoveries in this area is found in Cava Ranieri, specifically in the Boccia al Mauro sector. Here, the extraction of volcanic materials led to the discovery of Roman-era building complexes, buried beneath the eruption of Vesuvius in 79 AD. These structures remained sealed under pyroclastic deposits from subsequent eruptions until 1944 and are now situated roughly 20 meters below the modern ground level. Archaeological investigations in this region commenced in the 1980s, revealing three partially excavated and later reburied complexes, conventionally designated as Villa 1, Villa 2, and Villa 6, based on their discovery order.

These three settlements, dating to the late Republican

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period (late 2nd–early 1st century BC), primarily functioned as wine production and storage facilities (*pars fructuaria*). Centered around a *torcularium* (wine press) and a cellar, they operated as wineries that remained active until 79 AD. It is believed that they produced Vesvinum, a renowned wine traded across the Mediterranean, which may have been the precursor to *Lacryma Christi*. This connection is illustrated in a famous Pompeian painting from the *lararium* of the House of the Centenary, depicting a single-peaked Vesuvius covered in grapevines supported by poles, with Bacchus transformed into a luscious cluster of ripe grapes beside it.

Villa 6 is the most extensively investigated structure in Cava Ranieri, notable for its architectural features and remarkable wall and floor decorations. Discovered in the mid-1980s, it occupies a central location, equidistant (approximately 350 meters) from Villa 1 and Villa 2, with which it shares the same orientation (approximately N 46°E).

Despite their poor state of preservation, the wall paintings of Villa 6 are significant as they represent the only known examples of the Second Style in the extreme northern reaches of the *ager Pompeianus*. These frescoes feature striking decorative solutions, affirming the high artistic standards of the period. Their ornamental schemes, though more restrained, bear similarities to those found in many Pompeian *domus* and suburban residences of the mid-1st century BC (Second Style, Phase II). Notably, the frescoes in the *pars urbana* of the villa exhibit characteristic elements of the Second Style, including: closed painted walls, moderate illusions of depth through superimposed planes (*paratactic design*), and pronounced spatial breakthroughs achieved via painted openings that reveal glimpses of adjoining rooms, *aediculae*, and landscapes.

A particularly remarkable feature of Villa 6 is its use of *megalographia*, a highly evocative pictorial genre depicting human and divine figures within painted architectural perspectives, creating compelling mythological cycles. The *megalographia* of Terzigno revolves around mythological themes, with love serving as the overarching narrative motif. (Cicirelli, C., 2017).

While Pompeian wall paintings have been extensively studied, primarily with a focus on pigment composition using both conventional and advanced chemical analyses (Salvadori, M., and Sbrolli, C., 2021; Orna, M.V. and Fontani, M., 2022), relatively little research has been dedicated to the pigments used in the wall paintings of this equally significant archaeological site. Our study aims to characterize the pigments of a rare mural fragment collected from the Terzigno archaeological site.

2. Materials and Methods

2.1 Wall Painting Sample

The painting sample was collected from the *Domus* of Villa 6, located within the Cava Ranieri excavation site near the city of Terzigno (Campania, Italy) (Fig. 1). Based on its technical characteristics and the stratigraphic context of the site, it was dated to approximately the 1st century BC by C.P., former Director of the Restoration Laboratory at the Archaeological Museum of Naples (Italy). The sample measured approximately 3 × 3 × 2 cm, with some areas of its surface exhibiting well-preserved coloration. This sporadic fragment was selected for an invasive analysis as it represents the first and only wall painting specimen from this recently discovered archaeological site to undergo archaeometric study (Rigante et al., 2025).

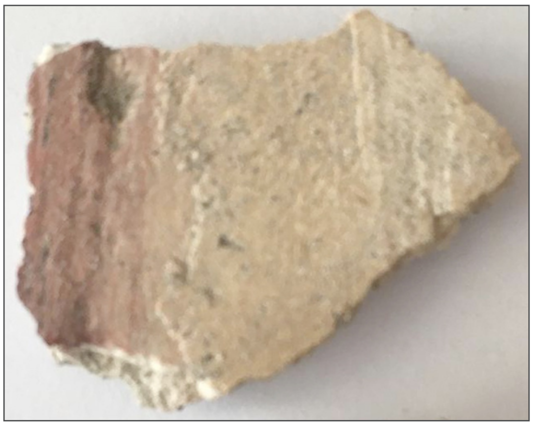

Figure 1. *Wall painting sample from Cava Ranieri's Villa 6*

2.2 Fourier Transform Infrared (FT-IR) and Raman Spectroscopy

Sample preparation for FT-IR spectral analysis was carried out according to the procedure previously reported by us (Gelzo et al., 2019). A small amount of the paint layer and underlying calcite (approximately 50–60 mg) was carefully scraped from the sample surface using a blade. The resulting powder was suspended in distilled water at a concentration of 100 mg/mL and further diluted to 0.3, 0.6, and 0.9 mg/mL. Ten microliters of each suspension (corresponding to 3, 6, and 9 μg) were deposited onto a 3 mm ZnS window, dried under a 60-watt white lamp, and analyzed using a Nicolet iN10MX spectrometer equipped with a ContinuμM microscope (Thermo Fisher Scientific, Waltham, MA). For each sample, three spectra (128 scan acquisitions) were collected in transmission mode, with a sensitivity setting of eight. The microscope focus was adjusted to 100 × 100 μm, with a resolution of 4 cm^{-1}. Peak areas were analyzed using Omnic software (Thermo Fisher Scientific), and peak assignments were made based on reference data (Socrates, G. 2001).

Pigment μ-Raman analysis was conducted using an Alpha300 RAS integrated system (WITec, Ulm, Germany). A 633 nm laser beam served as the excitation source, focused onto the sample surface via a 50× microscope objective (numerical aperture 0.75) in epi-illumination mode. The diffraction-limited focal spot had a full width at half-maximum (FWHM) of approximately 320 nm. Backscattered light was collected using the same objective and transmitted to the spectrograph via a confocal optical pathway.

2.3 XRF and Stratigraphic Analysis

X-ray fluorescence (XRF) analysis of the painted sample was performed using a portable SPECTRO xSORT XRF spectrometer. The technical specifications of the instrument were as follows: Operating voltage range: 9.0–12.6 V. X-ray tube: Rh anode, up to 50 kV, up to 125 μA, max. 2.5 W. Detector: SiPIN or SDD. Power consumption: 11 W during testing, 6 W in stand-by mode. Storage conditions: -20°C to +50°C (-5°F to 120°F). Operating temperature range: +5°C to +40°C (41°F to 104°F).

Stratigraphic analysis was performed using reflected light optical microscopy on polished cross-sections. Imaging was carried out with a Nikon Eclipse L150 reflected light microscope equipped with a Nikon Coolpix camera.

2.4 Methylene and Coomassie Blue Sample Staining

A small amount of the sample was resuspended in 10 μL of distilled water to create a thin smear, which was then applied to a glass slide and allowed to dry under a 60-watt white lamp. One to two drops of a 0.1% dye solution were directly added to the sample and left for 1–2 minutes to allow staining. The sample was then gently rinsed with distilled water to remove excess dye and background staining. Observations were conducted using an Axiovert 40 CFL microscope. The white balance was set at 5500K. Pictures were taken at a focal length of 56 mm with and Optical of 10x. A positive control was prepared using a suspension of melanoma A2058 cultured cells (approximately 1 × 10^6 cells). Organic materials, such as cells, microbes, and plant matter, absorbed the stain, appearing blue against the unstained background.

3. Results

3.1 Pigment Analysis by XRF

X-ray fluorescence (XRF) analysis was performed to examine the pigments present in the surface paint layer (Fig. 2). Samples were taken separately from areas displaying red and yellowish hues. The results indicated the use of ochre as a pigment in both color regions. Ochre is a naturally occurring pigment, with its color determined by the hydration level of the iron oxides it contains. Red ochre primarily consists of hematite (Fe_2O_3), while yellow ochre is derived from limonite and goethite, both hydrated iron oxides ($Fe_2O_3 \cdot H_2O$ and α-FeO(OH)) (Giachi et al., 2009; Fermo et al., 2013; Marcaida, I., et al., 2017, 2019).

Spectral analysis of the red region (Fig. 2A) revealed peaks corresponding to the Kα and Kβ lines of calcium, which were primarily attributed to the carbonate support, along with iron in varying concentrations across the red and yellow regions. The presence of iron confirmed the use of an earth-derived pigment rich in iron oxides (ochre).

In the yellow region (Fig. 2B), XRF detected a low-intensity potassium Kα peak, as well as more intense calcium Kα and Kβ peaks compared to the red region. Conversely, the iron peak in the yellowish region was significantly weaker than in the red region. In Roman wall paintings, potassium content is typically evaluated by comparing the area of the potassium Kα peak to that of the calcium Kα peak, with potassium levels averaging about 4% of the calcium content (Sciutti et al., 2001). The presence of both potassium and calcium in the colored layers suggests that a painting technique similar to that used in Pompeian wall paintings was employed. As described by Selim Augusti in the 1950s (Augusti, S., 1967), this technique involved dissolving pigments in a solution of lime, soap, and water, with the soap acting as a stabilizing agent to counteract the alkaline effects of lime on mineral-based pigments.

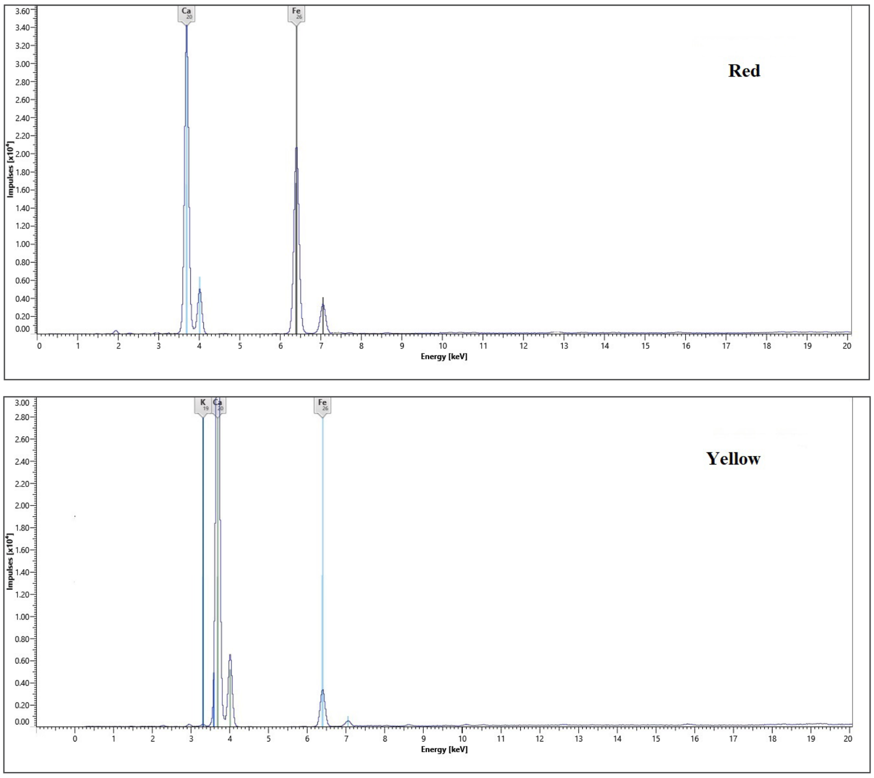

Figure 2. *XRF spectra of Terzigno wall paintings decorations.*

3.2 Sample Stratigraphy

Stratigraphic analysis, conducted using reflected light optical microscopy (Fig. 3), revealed the presence of a preparatory layer with a carbonate binder. This layer contained an aggregate composed of angular to sub-angular and rounded grains, primarily consisting of quartz, calcite crystals, and small clay lumps. The layer exhibited moderate porosity. A discontinuous red surface layer was identified, predominantly composed of iron oxides (ochre) with small carbon residues. The visible pictorial layer had a residual thickness ranging from 20 to 70 microns (Angelini et al., 2019).

Figure 3. *Reflected light microscope image of the sample (100X)*

3.3 Fourier Transform Infrared (FT-IR) Microscopy

Representative FT-IR spectra of the scraped red, yellow, and calcite powders are presented in Fig. 4. The FT-IR spectra of the paint sample closely resembled that of the underlying calcite layer. This suggests that the red and yellow powders contained very low amounts - or possibly none - of organic material.

Several characteristic absorption bands were observed: The hydroxyl (-OH) and Fe-O stretching vibrations were identified. A peak at approximately 3467 cm^{-1} was attributed to hydrogen bonding, likely due to residual surface water (Huang et al., 2018). The peak at 599 cm^{-1} corresponded to Fe-O bond vibrations, characteristic of hematite. A band around 1000 cm^{-1}, considered by some researchers as indicative of iron oxides, was also detected (Maity & Agrawal, 2007; Neto, F.N.S., 2012). Peaks at 2986 cm^{-1} and 2869 cm^{-1} were attributed to the asymmetric stretching of CH ($\nu_{as}CH_3$) and CH_2 ($\nu_{as}CH_2$), respectively. A band at 1612 cm^{-1} in the clay sample was likely associated with C=O stretching (Mesquita et al., 2003). A band at 1137 cm^{-1} corresponded to CH_2 twisting vibrations.

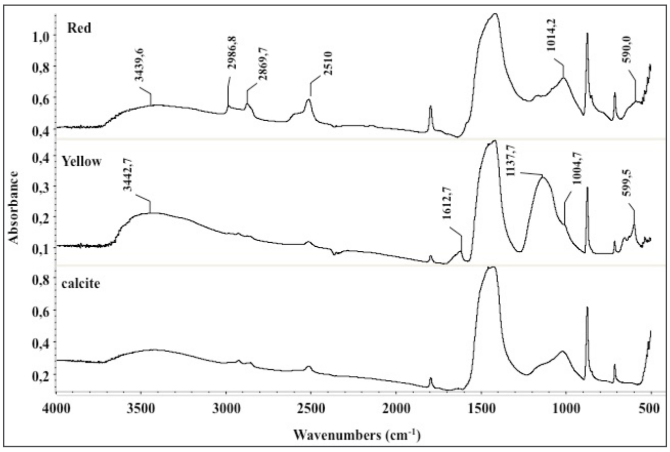

Figure 4. *FT-IR spectra of the Red, and Yellow regions of Terzigno samples and corresponding undelying Calcite.*

Given the residual thickness of the pictorial layer, an attempt was made to quantify the actual amount of pigment present in the sample. The quantification was performed using FT-IR spectroscopy by comparing the calcite peak area in the paint stratum scraped from the sample surface with that of the underlying calcite layer, which served as a reference. The wavenumber selected for analysis, around 2510 cm^{-1}, was chosen to avoid spectral overlap with other infrared signals from compounds present in the sample (Gelzo et al., 2019). The calculated amounts of paint powder and corresponding underlying calcite for each sample are reported in Table 1. For each painting sample and its corresponding underlying calcite, FT-IR peak areas were measured at increasing material deposits on the ZnSe window (3, 6, and 9 µg). The peak areas at 2510 cm^{-1} were then plotted to facilitate calculation (Fig. 5). Using the calibration equation derived from the calcite reference, the calcite content in each paint powder sample was determined. This allowed for the estimation of the average percentage of actual painting materials present in the samples, which likely consisted of pigments along with degradation or contaminant compounds (Table 2). Extensive research has been conducted on the pigments used in Pompeian frescoes. While systematic quantification of pigments may not have been performed for all frescoes, scientific studies have provided detailed insights into their composition and application (Giachi et al., 2009; Aliatis et al., 2010).

Table 1. *Amount of painted paint powder and corresponding calcite powder scraped from Terzigno's wall sample*

Sample	Paint color scraped	Paint layer (mg)
1	Red	40
2	Yellow	20
3	Whitish (Calcite)	60

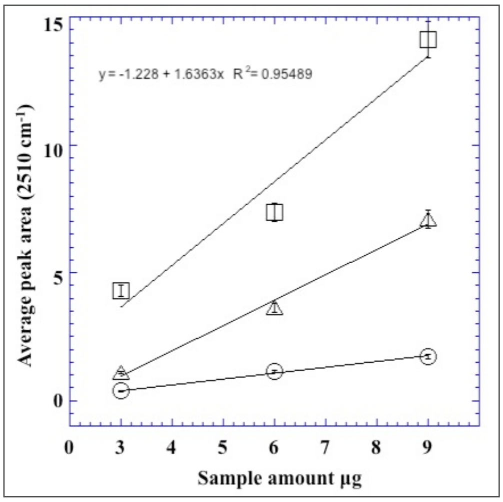

Figure 5. *Increasing amounts of paint and underlying calcite powder from paint sample used to calculate the FT-IR peak area at 2510 cm-1. For each sample spot deposited on ZnSe window, three spectra were collected and the average peak area and standard error was calculated. Calibration curve was obtained from calcite layer spectra. (□) Calcite layer. (○) Red layer. (Δ) Yellow layer. Y error bars correspond to standard error.*

Table 2. *Calcite and effective paint amounts in the paint layers*

	Measured calcite in scraped paint powder (µg)					
Paint samples	**3 µg spot**	**6 µg spot**	**9 µg spot**	**Average**	**SD**	**CV%**
Red	0.97	1.43	1.81	1.40	0.41	29.0
Yellow	1.41	2.97	5.03	3.14	1.81	57.0
	Measured true paint in scraped paint powder (µg)					
Red	0.67	0.76	0.79	0.74	0.06	8.52
Yellow	0.52	0.50	0.44	0.49	0.04	9.28
Average true paint (µg %)						
Red	67.55	76.02	79.98	74.52	6.35	8.52
Yellow	52.91	50.39	44.06	49.12	4.55	9.28

3.4 Analysis of organic components

To determine whether the Terzigno wall paintings were created using the fresco technique (where pigments are applied to wet plaster) or the secco technique (where pigments are applied to dry plaster) (Evershed, R.P., 2008), we investigated the presence of organic substances in the red and yellow pigment powders, which could indicate the use of binding agents. These painting techniques were typically carried out by ancient workshops consisting of multiple artisans led by a master craftsman, who negotiated with clients regarding the project scope, cost, and deadlines (Bragantini, I., 2004).

To assess the presence of organic binders, the red, yellow, and calcite powders were subjected to a qualitative staining test using methylene blue, and coomassie blue, dyes commonly used for detecting organic materials in biological and chemical applications (Raymer & Smith, 2007; Khan et al., 2022). Compared to a control melanoma cell line, no blue coloration was observed in the analyzed samples (Fig. S1 and S2), thus suggesting an absence of organic binders.

3.5 Raman Spectroscopy

Optical microscopy was employed to identify micrometric areas suitable for Raman spectroscopy. The Raman spectra were recorded to determine the chemical composition of the pigments. The red-painted zone was analyzed, and its spectrum is presented in Fig. 6, while the yellow zone (spectrum not shown) exhibited fluorescence interferences from trace compounds, making identification challenging (Perez-Alonso et al., 2005; Pessanha et al., 2012). The red pigment spectrum displayed characteristic Raman bands of hematite (α-Fe$_2$O$_3$) at 225, 293, 410, 613, and 1319 cm^{-1} (Simões et al., 2020; Qureshi et al., 2022). Hematite, a natural mineral pigment, was widely used in ancient paintings due to its affordability compared to cinnabar (HgS), a more expensive red pigment.

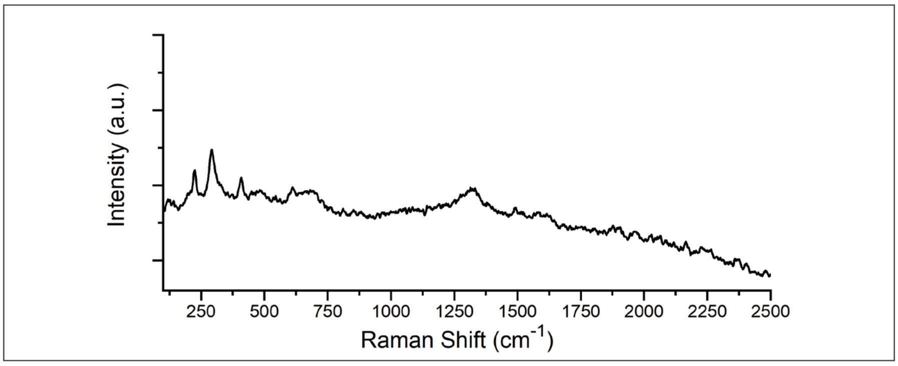

Figure 6. *Raman spectrum (λ = 633 nm) of the red pigment on the sample under investigation.*

4. Discussion and Conclusions

This study presents an archaeometric analysis of a wall painting fragment dated to the second half of the first century BC, discovered in the Cava Ranieri excavation during the 1980s. The small sample contained red and yellow painted areas, which experts believe were part of a wall decoration from the east wall of Cubicolo Diurno 9 (Fig. S2). This decoration featured painted pilasters with Corinthian capitals set against a backdrop of large yellow panels adorned with leaf garlands. The panels were centered with a Maenad's mask and a tambourine set against a red background (Cicirelli, C., 2017).

In 2011, following the excavation campaign of Villa

6, the site was backfilled to protect it from looting, as the Superintendency lacked the personnel to provide continuous surveillance. The wall paintings and numerous other artifacts were restored and transferred to the Terzigno Archaeology Museum (MATT) for conservation.

The findings suggest that hematite was deliberately used for its red hue, rather than as a byproduct of material degradation. This choice was likely driven by economic factors, as hematite was significantly cheaper than cinnabar (Miriello et al., 2018).

Using FT-IR spectroscopy, an attempt was made to quantify the pigment content in the painted layer. The analysis estimated that the red area contained approximately 74% hematite, while the yellow area contained around 49% yellow ochre. However, accurately determining the exact pigment quantities used in Pompeian frescoes is challenging due to the lack of historical records and material degradation over time. While scientific studies have identified pigment compositions, precise measurements in grams or milligrams are not consistently documented in the literature.

This study underscores the importance of systematic analytical methods in examining polychrome artworks. A critical aspect of this approach involves careful material recovery, minimizing damage while ensuring representative sampling. Cross-sectional analysis of paintings is particularly valuable, as it reveals layered structures and allows for the identification of inorganic and organic components through techniques such as optical microscopy and staining tests. These investigations provide crucial insights into the materials, techniques, and preservation state of historical paintings. Maintaining an accurate and minimally invasive sampling strategy is essential for meaningful art-historical and conservation studies (Corso et al., 2013; Sandu et al., 2012; Magrini et al., 2013).

Supplementary Information

1 Methylene Blue and Coomassie Blue Staining of Terzigno Sample

Small amount sample powder was suspended in distilled water and 10 µl were spotted on a microscope slide and let it dry under a 60 watt white lamp for about 5 minutes. Two drops of methylene blue solution (0.1% aqueous) were then added directly onto the sample and let it sit 1-2 minutes to allow staining. Subsequently, gently was rinsed the excess stain with distilled water to remove background staining. After drying, the microscope slide was observed at the microscope. Organic material will take up the stain and appearing blue against the unstained background.

For staining with coomassie blue, a staining solution made of Aceti Acid 10%, Methyl alcohol 50%, Comassie blue R250 0.25%, was used.

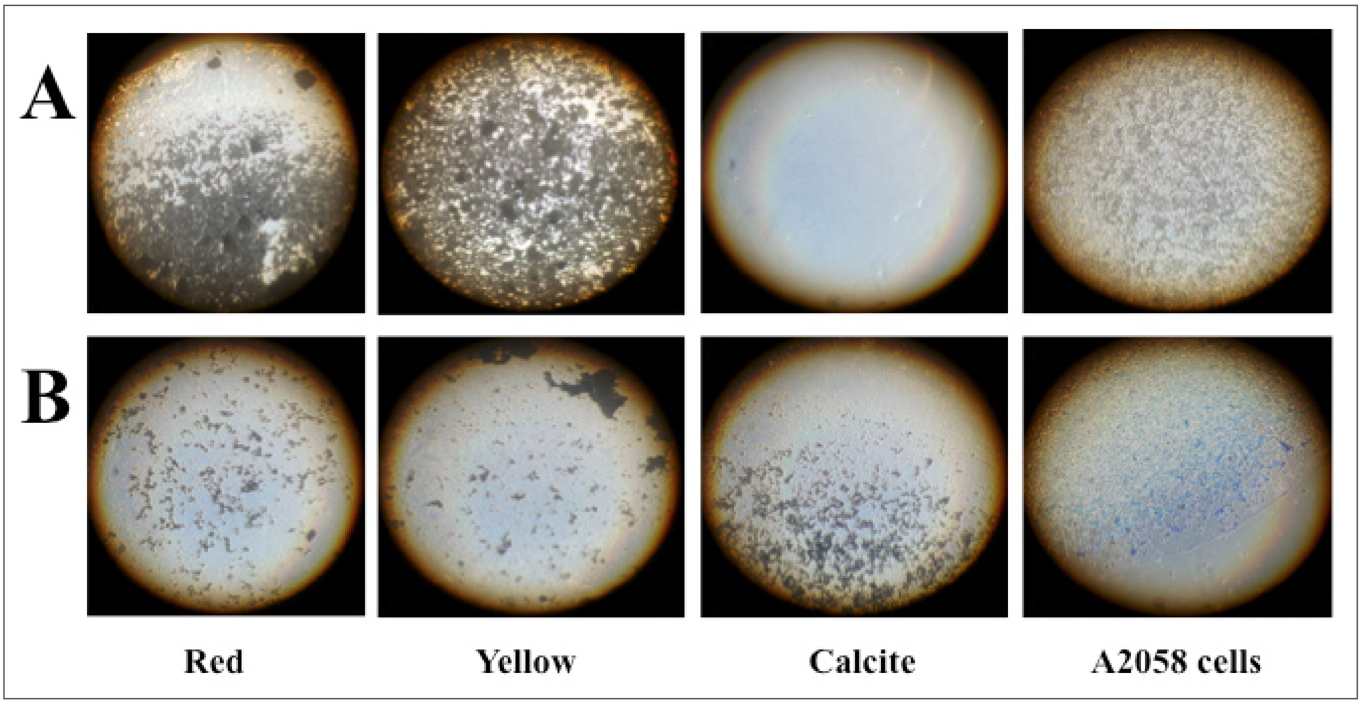

Fig. S1. *Terzigno wall painting powders before (raw A) and after (raw B) methylene blue staining of the samples. Positive control: A2058 cell. Images were acquired with a Axiovert 40 CFL*

2 Coomassie Blue Staining of Terzigno Wall Paintings

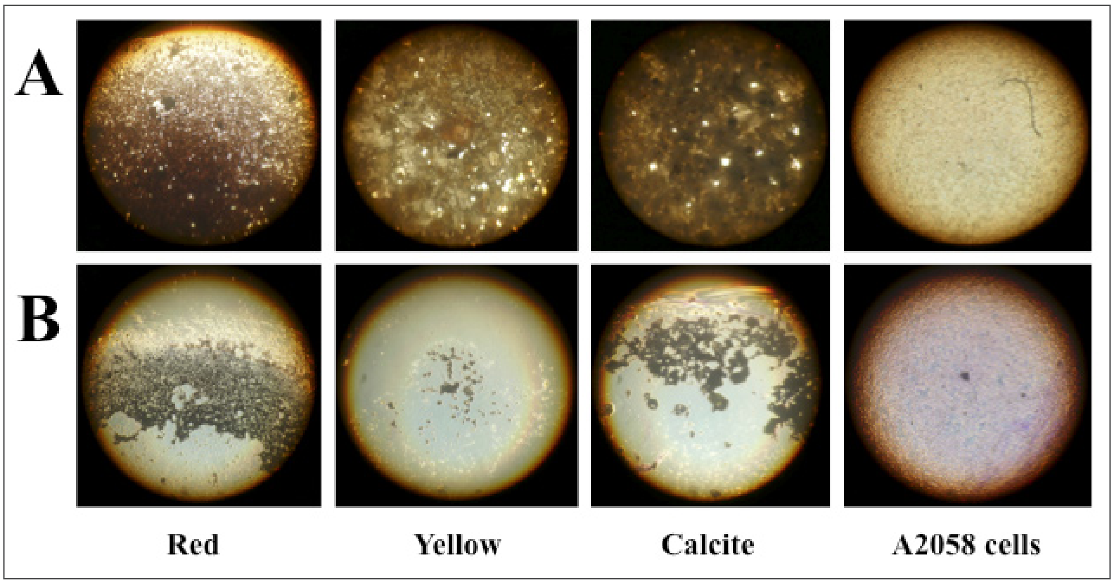

Fig. S2. *Terzigno wall painting powders before (raw A) and after (raw B) coomassie blue staining of the samples. Positive control: A2058 cell. Images were acquired with a Axiovert 40 CFL*

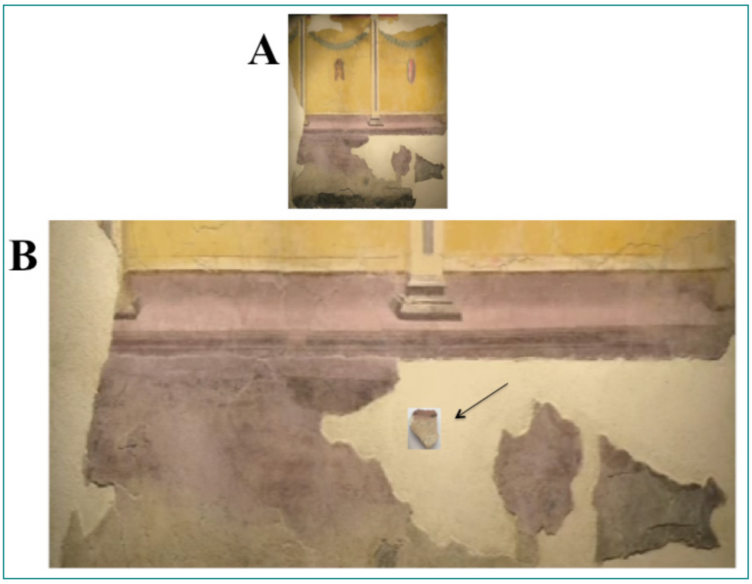

Fig S3. *This figure illustrates a possible association based on color similarity between a Terzigno wall painting fragment (indicated by an arrow) and the detached and restored wall decoration found on the east wall of cubicolo diurno 9 in Villa 6, housed at the Archaeological Museum of Terzigno (MAT). The cubicolo diurno 9, located to the east of portico 8, opens along its entire width (2.45 x 2.40 m) and is preserved up to a maximum height of 1.80 m. A) Painted decoration. B) Enlargement of the lower section of the painting, with the arrow indicating the portion of the analyzed fragment.*

*(***Source:** *Cicirelli, C. Terzigno 1981-2016. Dalla ricerca archeologica ad una fruizione delle ville romane di Cava Ranieri. Nicola Longobardi Editore, 2017, p. 43. ISBN 978-88-8090-480-9.)*

Authors' Contributions

CP and OA were involved in sample collection and archaeological study. AV, MA, and MS were involved in Raman spectroscopy, GT take care of the XRF analysis and stratigraphy. PA and GC were involved in FT-IR spectroscopy data acquisition and data analysis and made substantial contributions to interpretation of data, drafting, and revision of the manuscript. All authors read and approved the final manuscript.

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We wish to acknowledge this work to the memory of Dr. Caterina Cicirelli who dedicated almost her carrier to the escavation, study, classification and historical relevance of the Cava Ranieri archaeological remains in Terzigno.

Competing interests

The authors declare that they have no competing interests.

Availability of Data and Materials

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

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