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Analysis of Roman age wall paintings found in Pordenone, Trieste and Montegrotto

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Abstract

The aim of the present work is the study of many fragments of wall painting from archaeological excavations in three different Roman age sites dating back to the I Century before Common Era: Pordenone (località Torre); Trieste (Crosada) and Padova (Montegrotto). The techniques used were optical microscopy, scanning electron microscopy (SEM), equipped with a EDS microanalysis detector, X-rays powder diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), Fourier transform Raman spectroscopy (FT-Raman) and electron paramagnetic resonance (EPR) spectroscopy. The identified pigments were: cinnabar, hematite, celadonite, glauconite, cuprorivaite (Egyptian blue), yellow and red ochre, calcite, limonite, coal black.

In general, the mortar preparation did not correspond to the complex procedure suggested by Vitruvius (De Architectura), but generally showed a porous layer, with crushed grains under the pigment layer. In some cases, two superimposed pigment layers were found: yellow superimposed on both red and pink, black on pink, green on black.

The slight differences we found in the use of the pigments in the three studied sites might show that the same technology, culture and taste spread all over the Roman Empire in North Eastern Italy (*X^a Regio Venetia et Histria*).

Keywords: Roman wall paintings; Physico-chemical analysis; SEM–EDS; XRD; FT-IR; FT-Raman; EPR

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1. Introduction

One of the most important questions in the study of ancient paintings, especially as far as Roman age painting is concerned, is the nature of the pigments employed in the painting 'atelier' to create the decorations. The physico-chemical analysis, in fact, gives information useful for defining the gamut of pigments available on local and regional scales and to understand the techniques of colour preparation and application. In addition, the study of the provenance of the pigments allows one to discover the communication and trade exchange lines, or even to define the so-called 'site taste'.

These kinds of studies have been carried out in France, Switzerland [1–5] and Spain [6,7], but in Italy there is still a lack of systematic analyses of the numerous Roman painting finds, although the research is now increasing [8,9].

The aim of the present work is the analysis of many fragments of wall painting coming from archaeological excavations in three different Roman age sites dating back to the I Century before Common Era: Pordenone (località Torre); Trieste (Crosada); Padova (Montegrotto).

The excavations were directed by the Archaeological Superintendence in collaboration with the Universities of Padua and Trieste.

The comparison among the materials, the pigments and the techniques used by the artists in the application of the colours, supplies information on the period, on the social standing of the owner and, more generally, on the diffusion of pictorial techniques during the Augustus age in *X^a Regio, Venetia et Histria*.

For the determination of anionic groups, crystalline phases, structure and elemental composition and to have a complete enough characterisation of the employed pigments and materials, the techniques used were optical microscopy,

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scanning electron microscopy (SEM), equipped with a EDS microanalysis detector, X-ray powder diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), Fourier transform Raman spectroscopy and electron paramagnetic resonance (EPR) spectroscopy.

2. Experimental

2.1. Sampling

The sampling was done by the archaeological team, who collected pieces representative of the variety and differences of colour in each site. The dimension of the fragments analysed ranged from 2 cm^2 to 20–25 cm^2, but it is quite hard to get a precise idea of the dimensions of the whole frescoes because all the painted walls were found as fragments covered by mould and rubble.

2.2. Optical microscopy

The samples were observed by means of an optical microscope Wild–Leitz M8 with a 19.2× to 256× zoom. The samples were illuminated using a movable fibre glass system. This technique was used specially in the examination of the grains, crushed calcite or other tiny stones inside mortars.

2.3. Scanning electron microscopy (SEM)

SEM images were taken using a Jeol (Tokyo, Japan) JSM 5600 LV equipped with an Oxford Instruments 6587 EDS microanalysis detector. The images were taken under low vacuum conditions where samples did not show charging effects; in this way, it was possible to avoid the coating of the samples with a high conductance thin film (gold or graphite films).

EDS microanalysis was contucted to obtain information on the elemental composition of the samples, which were small pieces of un-treated painted mortars.

2.4. X-ray diffraction (XRD)

Powder X-ray diffraction was used to identify the different crystalline phases present in pigments. The smallest pieces of wall paintings were put on the support of the instrument and analysed. As far as the biggest pieces are concerned, we selected an area from which we get a small amount of powder by carefully scratching the painted surface. The powder was then put on quartz single crystal sample holder, which does not give any interferences in the measurement range. A Philips X'Pert vertical goniometer with Bragg–Brentano geometry, connected to a highly stabilised generator, was used for XRD analysis. Cu Kα Ni-filtered radiation, a graphite monochromator on the diffracted beam and a proportional counter with pulse height discriminator were used. Measurements in a 5°–60° range were taken with a step size of 0.05° and 2 s per point.

2.5. Infrared spectroscopy (FT-IR)

Absorption spectra in the IR region were collected using a Nicolet Magna 75 FT-IR spectrometer. Thirty-two signal-averaged scans were acquired on the samples. A few milligrams of each sample were diluted in KBr (IR grade, Merck) pellet of a diameter of about 13 mm.

2.6. Raman spectroscopy

Raman spectra of the pigments were collected using a Nicolet Nexus E.S.P. FT-IR spectrometer equipped with a Raman module. To obtain spectra of good quality, 2000 scans were performed with 4 cm^{-1} resolution, necessary to effect a satisfactory signal-to-noise ratio; each specimen required about 8 min to acquire the data. The excitation in the near infrared was effected using a Nd/YVO4 laser with a power of 0.56 W and the scattered radiation was collected with an InGaAs detector. Small amounts of samples (on the order of tenths of a gram) were put into the measurements tubes.

2.7. Electron paramagnetic resonance spectroscopy (EPR)

EPR spectra were recorded at room temperature using a Bruker ER 200D X-band EPR spectrometer equipped with a parallelepiped shaped TE102 microwave cavity. The following parameters were employed: microwave frequency 9.7 GHz; field modulation 100 kHz; klystron power 10 mW; modulation amplitude 2 Gausspp; sweep time 300 s; magnetic field sweep 200–5000 Gauss. The samples, few milligrams of pictorial layer, were treated with diluted HCl in order to eliminate calcium carbonate residues before the analysis. The pigment, thoroughly washed with deionised water, was then put in the resonant cavity inside quartz tubes (3 mm inner diameter) and the spectra were digitally recorded, analysed and deconvoluted by means of the Simphonia and WinEPR software provided by Bruker Co. The separation of pure spectra, concerning any paramagnetic component, was further refined using Microcal Origin.

3. Results and discussion

3.1. Pordenone (Torre)

The analysed samples, the identified pigments and the analytical techniques used are shown in Table 1.

3.2. Black

EDS spectrum of this sample shows the typical components of a coal black: Ca, C, Si, Al and traces of Fe and K.

Table 1

Colour	Identified pigments	Techniques
Black	Coal black	SEM, EDS, FT-IR
Grass green	Glauconite, celadonite	SEM, EDS, FT-IR
Brilliant red	Cinnabar	SEM, EDS, FT-IR, XRD
Wine violet Brick red	Hematite	SEM, EDS, FT-IR
White	Calcite	FT-IR, FT-Raman
Blue	Cuprorivaite (Egyptian blue)	SEM, EDS, FT-IR, XRD
Yellow	Limonite	SEM, EDS, FT-IR

FT-IR spectrum shows the characteristic carbonate bands at 1440, 875 and 715 cm^{-1}.

3.3. Grass green

In Fig. 1 EDS spectrum of a grass green sample is shown. The analysis showed the presence of Si, Mg, K, Cl, Al and Ba, as major components, and Fe and S as trace components. This suggests the presence of natural pigments such as celadonite and glauconite, together with small quantities of chlorite. FT-IR data also confirms the presence of the two minerals glauconite and celadonite showing the presence of small bands at 3550 cm^{-1} due to the characteristic hydroxyl stretching of $K(Fe,Al)_2(Si,Al)_4O_{10}(OH)_2$ [10].

3.4. Brilliant red

The FT-IR spectrum of this red pigment, spread over an orange–yellow layer, showed the presence of calcium carbonate, while the EDS spectrum (Fig. 2) clearly shows the presence of Hg and S, beside traces of Fe. Hg and S peaks are very close, though well resolved, Hg Mβ edge at 2.283 keV and S Kα edge at 2.308 respectively. Moreover, Hg Lα and Lβ edges appeared at 9.987 and 11.823 keV, respectively.

SEM image shows the presence of particles, which seem to be much brighter.

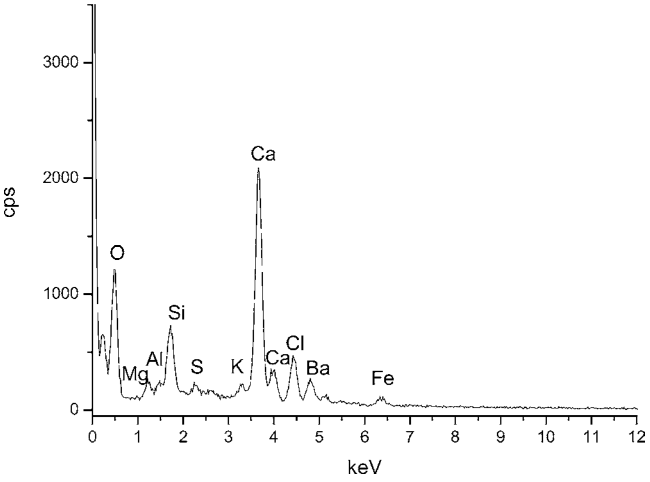

Fig. 1. EDS spectrum of the grass–green sample.

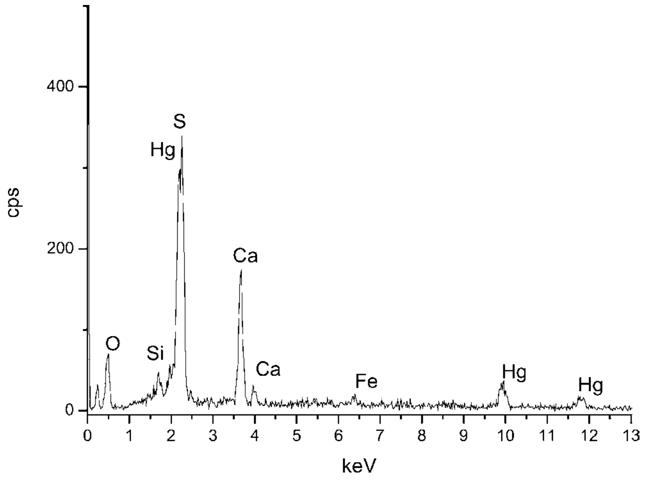

Fig. 2. EDS spectrum of the Brilliant red sample.

This phenomenon is due to the presence of heavy metals, which have a bigger cross section for the electron backscattering, since they have a higher atomic number.

3.5. Wine violet and brick red

The FT-IR analysis of the wine violet sample showed that the most important bands, at 472 e 558 cm^{-1} due to hematite. The same happens for the brick red sample, although the peaks are less marked [6].

3.6. White

The white samples, studied with the FT-IR technique, showed the characteristic carbonate at 1440, 875 e 715 cm^{-1}. The EDS analysis also showed only the peaks corresponding to calcium.

3.7. Egyptian blue

SEM image (Fig. 3) clearly shows the brighter crystals of cuprorivaite (average dimension ≈30 μm) scattered on the surface of the sample together with calcite crystals. In Fig. 4, the EDS spectrum of the blue sample in which the peaks of Cu, Ca, Si are present, is shown.

FT-IR analysis of the same sample showed, beside carbonate bands, two intense bands at 1005 and 1052 cm^{-1} and two medium intensity ones at 1160 and 1250 cm^{-1}, respectively, which are all typical of the Egyptian blue [8].

Also XRD analysis confirmed the presence of the crystalline phase cuprorivaite together with small quantities of silica and calcite.

3.8. Yellow

The SEM observations showed a homogeneous distribution of small crystals. FT-IR analysis showed, beside the carbonate bands, the presence of two small bands at

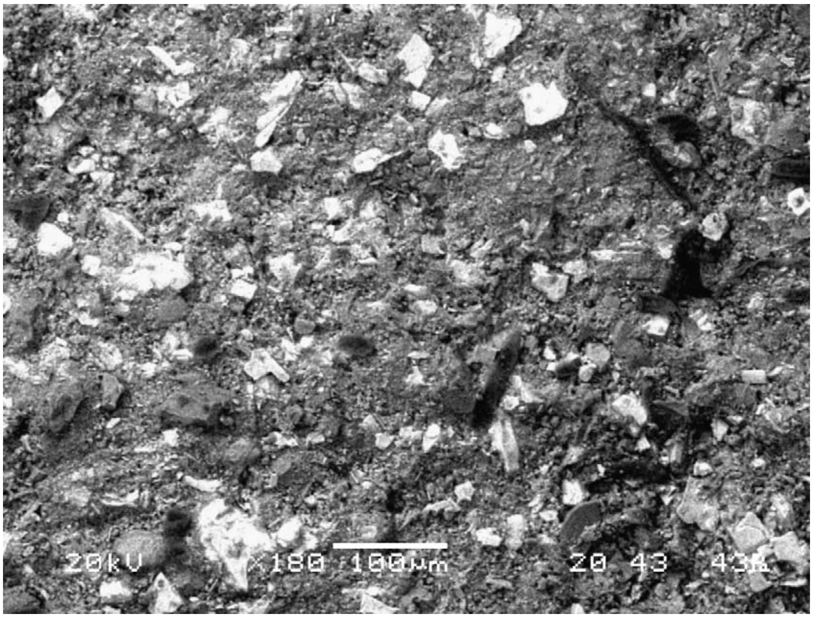

Fig. 3. SEM image of the blue sample.

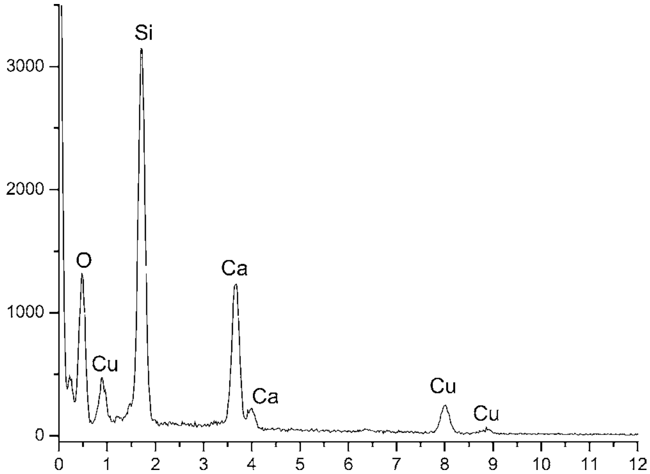

Fig. 4. EDS spectrum of the blue sample.

800 and 910 cm^{-1} due to the presence of iron hydroxides of the mineral limonite, which does not have characteristic peaks in X-rays diffraction due to its non-crystalline nature.

It is worth noting that the EDS analysis showed the presence of a small quantity of iron, together with Ca, Si, and Al, coming from the mortar below the pictorial layer.

To investigate also the range between 400 and 200 cm^{-1}, some FT-Raman measurements were done. Fig. 5 shows the FT-Raman spectrum made on a white mortar [11]. The carbonate bands are clearly visible at 1086, 712 and 281 cm^{-1} and the silica band at 461 cm^{-1}. In this case, however, the additional technique does not give important information with respect to FT-IR spectroscopy.

3.9. Trieste (Crosada)

The analysed sample, the stratigraphic unit, the identified pigments and the analytical techniques used are shown in Table 2.

3.10. Brilliant red

In Fig. 6, the SEM image of the red sample belonging to the SU 463 shows the heterogeneity of the pictorial layer. It is made of a mixture of calcium carbonate and small cinnabar

Table 2

Colour	Stratigraphic unit (SU)	Identified pigments	Techniques
Brilliant red	463	Cinnabar	SEM, EDS
Pink–red	526	Cinnabar	SEM, EDS
Black (wall)	463	Coal black	FT-IR
Gray–black (ceiling)	463	Coal black	FT-IR
Brown red	936	Hematite	SEM, EDS, FT-IR
Green	463	Glauconite, celadonite	EDS, FT-IR
Yellow	215	Limonite	EDS- FT-IR
Blue	882	Egyptian blue	FT-IR, XRD, EPR
Tri-colour (brown red, white, green)	463	Hematite, calcite, glauconite	SEM, EDS, FT-IR
Tricolour (black, white, orange-red)	463	Coal black, calcite, iron oxides	SEM, EDS, FT-IR

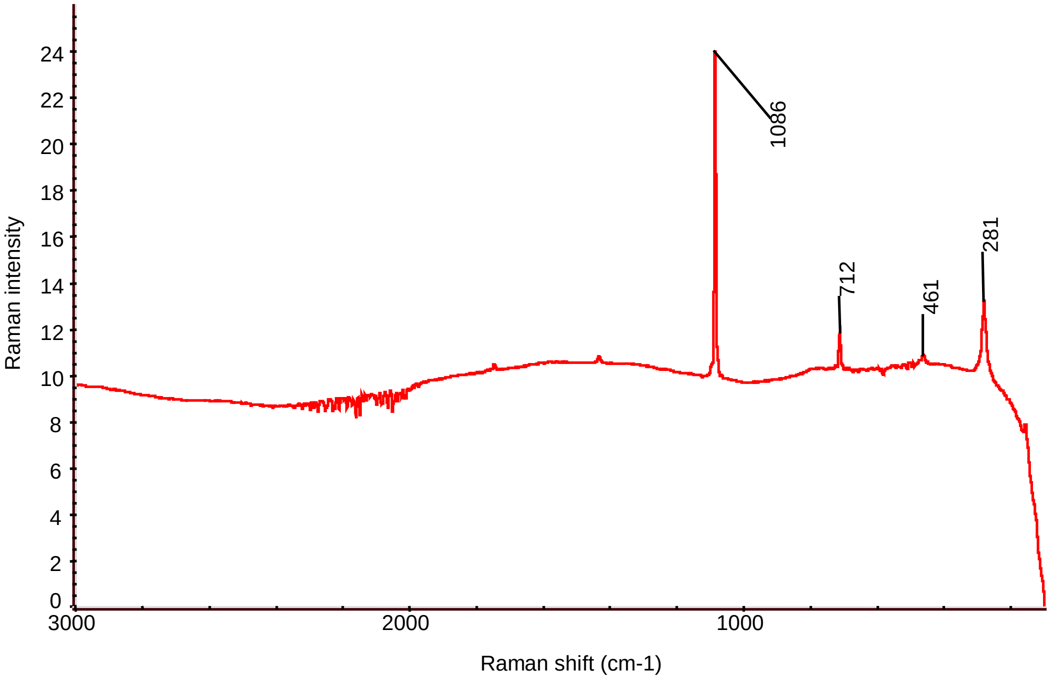

Fig. 5. FT-Raman spectrum of a mortar sample.

crystals, which are brighter. EDS analysis confirmed the presence of Hg and S as major components and of Fe, Ca and Si as minor components.

FT-IR spectrum showed the presence of carbonate bands (1451, 872, 709 cm^{-1}), the small bands of silicates (1025 e 1082 cm^{-1}) and traces of iron oxides (542 e 467 cm^{-1}). Cinnabar does not have IR characteristic signals in the frequency range we used.

As already noted by the authors [12], this “precious” pigment is spread over a porous mortar with crushed calcite crystals, maybe to give to the pictorial layer a high degree of permeability and a certain degree of resilience to avoid cracks and crazes during the carbonatation process. The pink–red sample, belonging to the SU 526, was also made of cinnabar.

3.11. Black

This colour, belonging to the SU 463, seems to have been applied in large quantities on a vertical surface, since, during the carbonatation fixing phase, the colour slid towards the bottom, forming “waves”.

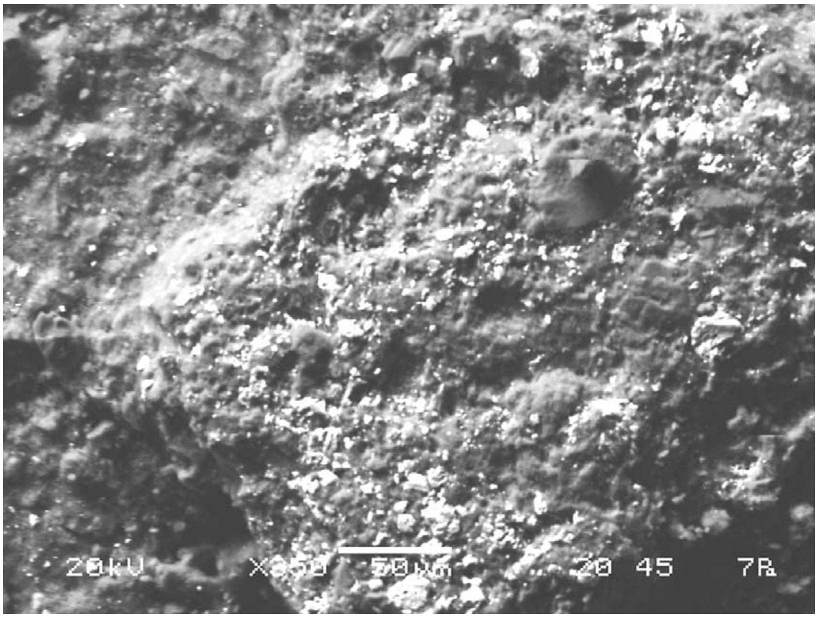

Fig. 6. SEM image of the cinnabar sample.

The outer preparatory layers, below the plaster, are made of 1.5 cm of mortar mixed with calcite fragments, while the inner level of *tectorium* is given by 1 cm of mortar made of $CaCO_3$ and fine sand.

EDS spectrum showed the typical components of a coal black: calcium, carbon, silicon, aluminium and traces of iron and potassium.

3.12. Grey–black

The sample of the SU 463 was spread on a different mortar. Under the compact veil of pigment, from which small reflecting $CaCO_3$ crystals emerge, it is possible to recognise the plaster. This is a thin layer (about 1 mm) of smooth plaster, followed by 1 cm of mortar with fine sand, followed further by calcium carbonate, without sand and stuck to the canes of the ceiling support. This different kind of mortar should have been light, as part of a ceiling.

As with the black sample, FT-IR spectrum showed the calcium carbonate characteristic bands.

Black and grey–black samples were put in a muffle oven at rising temperatures up to 500 °C.

Starting from 400 °C, the colour began to disappear, leaving at 500 °C just the ivory colour of the mortar. These colours are not made of metal oxides, which would have remained unaltered, but of coal black, which combines with air oxygen to create carbon dioxide and disappear at temperatures higher than 400 °C.

3.13. Brown red

Low magnified SEM image of the monochromatic brown red sample belonging to the SU 963. One can see vertical brush strokes and the compact spreading of the colour without crystals appearing on the surface.

EDS analysis of the sample showed the presence of iron as major component, as well as calcium.

The FT-IR spectrum of the sample shows, as usual, the presence of the carbonate bands, and at 540 and 470 cm^{-1} the typical bands of iron hydroxides [6], which confirm the nature of this pigment, made of hematite.

The mortar on which the pigment is spread over is made of 2.5 cm of lime with pieces of crushed calcite.

Green and yellow samples from Trieste gave the same results and spectra of the grass green and yellow of Pordenone.

3.14. Blue colour

FT-IR spectrum of the intense blue sample, spread over a mortar preparation with crushed calcite crystals followed by a layer of mortar with fine sand showed, besides hydroxyl stretching bands at 3447 cm^{-1}, the carbonate ones at 1456, 874 and 715 cm^{-1} due to the mortar. The typical cuprorivaite, or Egyptian blue, bands appear at 1157, 1052 and 1006 cm^{-1} [8].

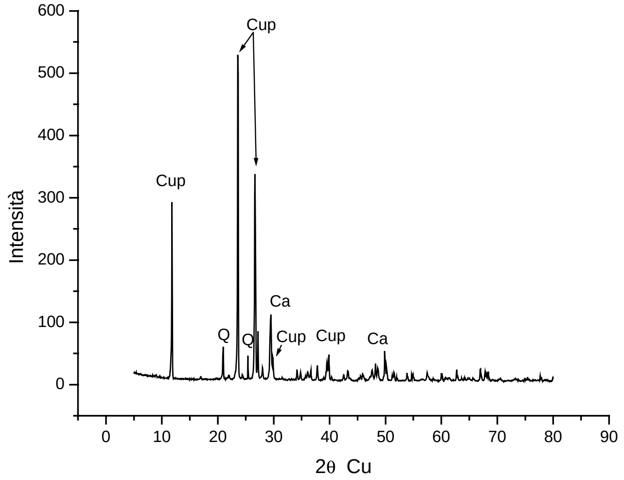

Fig. 7. XRD spectrum of the blue sample with the crystalline phases calcite, cuprorivaite and quartz.

In Fig. 7 the XRD spectrum of the Egyptian blue is shown. One can see the peaks due to the presence of cuprorivaite, as well as the calcite and quartz peaks.

In Fig. 8 the EPR spectrum of the Egyptian blue sample is shown. The spectrum consists of two absorption lines corresponding to the spectral components perpendicular and parallel to the external magnetic field respectively, in agreement with the occurrence of a unique copper(II) paramagnetic species [8]. The parameters indicate that the cupric ion is coordinated to other ligands in an octahedral geometry (bi-tetragonal, bi-pyramidal) distorted along the longitudinal axis.

3.15. Tricolour samples

Some samples with green and white bands painted on a brown red ground were investigated.

The white ones were made of calcium carbonate, while the green ones were identical to the monochromatic sample already studied.

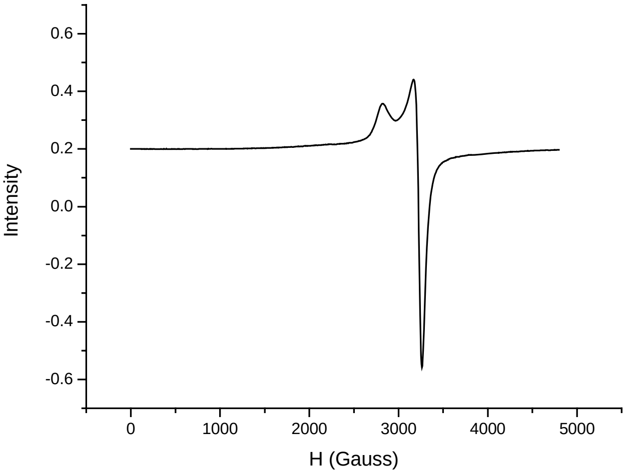

Fig. 8. EPR spectrum of the Egyptian blue sample.

Table 3

Colour	Stratigraphic unit/year	Identified pigments	Techniques
Orange red	90	Hematite, red ochre	SEM, EDS, FT-IR
Brick red	104	Hematite, ochre	SEM, EDS, FT-IR
Wine violet	316	Hematite	SEM, EDS
Gray–green	216	Glauconite, celadonite	SEM, EDS, FT-IR
Yellow	90, 89	Limonite	EDS, FT-IR
Brilliant red	90	Cinnabar	FT-IR, XRD,FT-Raman
White	90	Calcite + traces of iron and silicates	EDS, FT-IR
Wine brown	90	Hematite	EDS, FT-IR
Blue stripe on brown–grey ground	104	Cuprorivaite	SEM, EDS, FT-IR, XRD
Green stripes	‘90	Glauconite, celadonite	FT-IR, EDS

In one case, below the brown-red one, a pink layer of about 1 mm was present.

The FT-IR analysis of this colour did not show any new finding: it was just calcite with traces of hematite.

The second tricolour sample, instead, was a number of thin white and orange red lines partially superposed on a black ground.

While the black pigment was typically coal black and the white lines were made of calcium carbonate, the orange red lines were made of a mixture of hematite (red) and limonite (yellow) on a carbonate basis.

3.16. *Padova (Montegrotto)*

The analysed sample, the identified pigments and the techniques used in the analysis of the samples found in Montegrotto (PD) during the excavations in the year 1990 are shown in Table 3.

3.17. *Orange red and brick red*

The SEM analysis revealed a number of crushed calcite crystals covered with a fine red ochre powder with traces of hematite.

FT-IR spectrum of this sample showed the typical carbonate bands (1450–874 and 714 $\mathrm{cm^{-1}}$) due to the mortar beneath, the silicate bands at 1035 $\mathrm{cm^{-1}}$ and the iron oxides ones at 540 and 470 $\mathrm{cm^{-1}}$.

All the collected data confirms that the used pigment is mainly a red ochre [6].

3.18. *Wine violet*

The wine violet samples, found in the SU 316, are made of pure hematite, as can be observed in the FT-IR spectrum (not shown), in which two intense iron oxide bands are present at 543 and 473 $\mathrm{cm^{-1}}$. In this spectra the carbonate bands, the O–Si–O bands and the ones due to the water traces of the sample at 3450 $\mathrm{cm^{-1}}$ are also present.

3.19. *Gray–green*

The EDS spectra of the samples belonging to the SU 216 showed the presence of Mg, K, Fe and Al, suggesting the use of pigments like glauconite and celadonite, largely used in Roman age wall paintings, while the lack of Cl leads to the exclusion of chlorite.

3.20. *Yellow*

Two samples from the excavation were made of a yellow pigment which, examined by means of FT-IR, showed just two small bands at 800 and 900 $\mathrm{cm^{-1}}$ as well as the usual presence of carbonate bands. From literature data [6], we can state that this pigment is again limonite.

3.21. *Brilliant red*

Among 1990 findings, there were also some brilliant red samples. As previously seen, the SEM analysis showed the presence of brighter particles, made of heavy metals, while FT-IR spectrum showed the only presence of carbonate bands. By means of EDS, we found the presence of S and Hg, confirming the use of the precious pigment cinnabar (HgS), together with traces of Fe, Ca and Al due to the mortar below.

In Fig. 9 a FT-Raman spectrum of the cinnabar sample is shown. The more evident bands at 251, 281 and 341 $\mathrm{cm^{-1}}$ are characteristic of the cinnabar, as reported in literature data [13].

3.22. *White*

Some ivory white sample were found, which were simply calcium carbonate like the mortar below, with traces of silicon and iron oxides.

3.23. *Wine brown*

In the same excavation, the archaeological team also found some pieces of a wine brown colour, which was

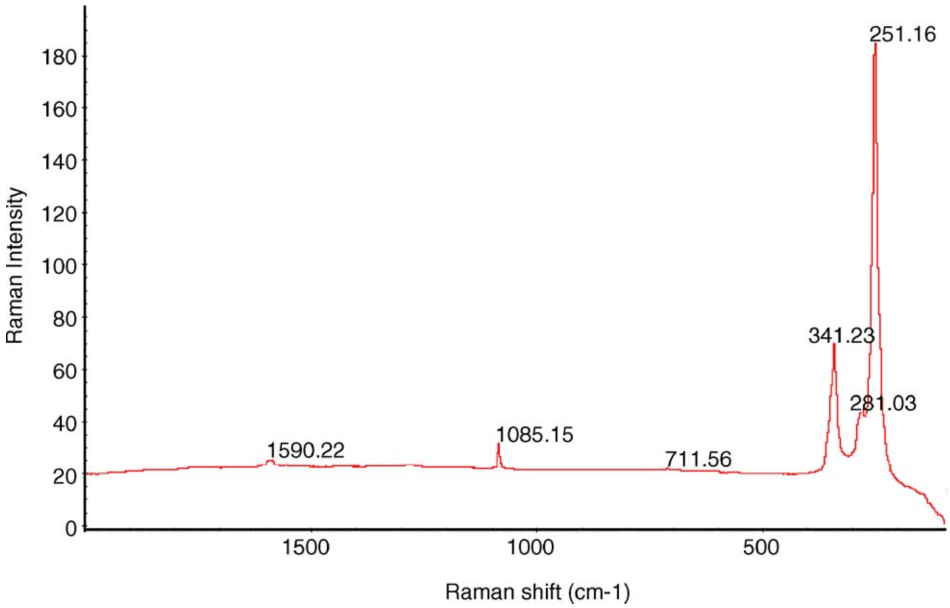

Fig. 9. FT-Raman spectrum of the cinnabar sample.

made of hematite, as clearly demonstrated by the FT-IR analysis.

One of the samples was different: the wine brown colour was spread on a compact bluish grey layer of 2 mm, an interesting peculiarity.

EDS analysis of this pictorial “sous-couche” showed the presence of small quantities of silicon and aluminium together with calcium.

FT-IR analysis did not show any meaningful bands, except for carbonate.

XRD analysis, Fig. 10, showed the presence of the crystalline phases of calcite and calcium aluminate, which could be due to the presence of the *pozzolana* used as plaster.

3.24. Blue stripe

A grey sample belonging to the SU 104 showed a blue stripe, which, analysed by means of EDS, indicates the presence of copper and calcium, suggesting the presence of cuprorivaite together with traces of Fe, K, Cl, and Al.

FT-IR analysis of the stripe showed that the band between 1000 and 1200 cm^{-1} had typical Egyptian blue peaks [8].

Thus, this precious pigment was used also in the Montegrotto site, even if monochromatic samples have not yet been found.

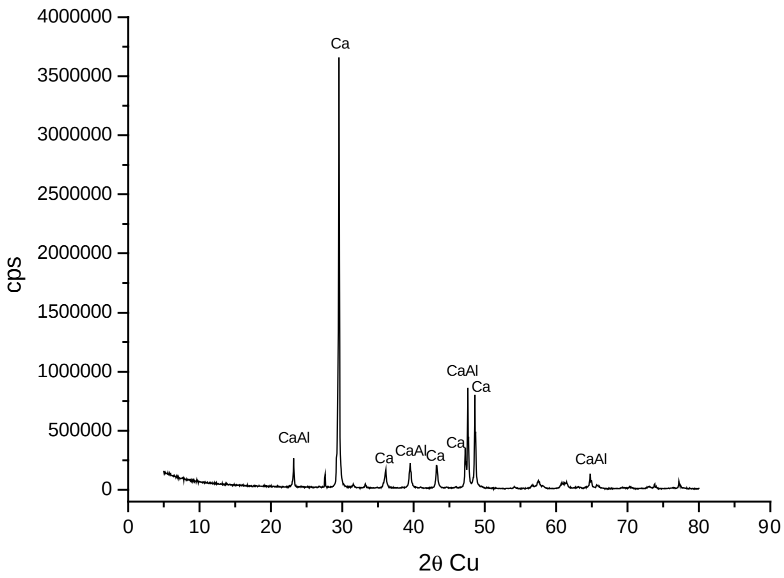

Fig. 10. XRD spectrum of the sous-couche with the crystalline phases tricalcium aluminate (CaAl) and calcite (Ca).

3.25. *Green stripes*

Among the 1990 findings, some fragments were decorated with green stripes on a white ground.

The FT-IR analysis of this sample showed, beside the carbonate bands, the typical hydroxyl stretching bands at 3450 and 3550 cm^{-1}, due to the OH group of glauconite and celadonite.

Also EDS analysis confirmed that the green pigment used is made of glauconite and celadonite.

4. Conclusions

Even if modest with respect to the variety of colours found in other archaeological sites such as Rome or Pompeii [14,15], the palette we found in these archaeological sites is fairly rich and includes, besides the ordinary pigments, precious pigments like cinnabar and Egyptian blue and sophisticated paintings in all the examined villas; we also found the use of different combinations of pictorial layers. We did not find the use of lead as orange (minium, Pb_3O_4) or as white (cerussite, $PbCO_3$; white lead, $2PbCO_3{\cdot}Pb(OH)_2$), nor As-based pigments which, on the contrary, were found in Gallia, in Argentomagus [5], while we found practically the whole gamut given by hematite (orange red, red, brick red, violet) and by cinnabar (brilliant red and pink).

We did not clearly identify organic ligands nor organic pigments which could lead to pictorial techniques different from frescoes as already verified in previous works [10] and [12].

Different kind of mortars were used for the walls of those sites, but, in general, the preparation of these supporting materials was not that suggested by Vitruvius in De Architectura [16]. The mortars below the pigment layer were investigated using FT-IR techniques to see whether in-site and/or among-site differences were present. Some FT-Raman measurements were done, but this additional investigation does not give important information with respect to FT-IR spectroscopy, which anyhow was revealed to be insignificant with regard to the differentiation of the origin of the mortar.

On the other side, using EPR technique, which was attempted also for the investigation of the mortars, seemed suggest a fruitful direction for further investigation. This technique is not very common in the field of the cultural heritage, even if its sensitivity to paramagnetic ion traces can supply a kind of fingerprinting of the analysed samples, providing useful information in the comparison of different samples/sites. In the analysis of the Egyptian blue of the Trieste site, we could confirm the suitability of this technique, allowing us to understand the structure of the synthesised product, which is very similar to the one reported by Mirti et al. [8].

The employed analytical techniques were suitable to give the information required for the characterisation of the pigments. On the one hand, the most useful information came from the EDS spectroscopy as concerns the elemental analysis; from FT-IR for the anionic group determination and XRD spectroscopy for the analysis of the crystalline structure. On the other hand, EPR measurements seemed to be a promising tool to attain information both on the definition of the structure and to define the within-site and between-site variability of the samples.

We found strong similarities among the three sites studied here, and other sites in different regions of the Roman Empire (France, Switzerland [1–5]), witnessing the active trading exchange that took place in Augustus times.

The slight differences we found in the use of the pigments might show that in the North Eastern Italy (*X^a Regio Venetia et Histria*) the same technology, culture and taste spread all over the entire region, with clearer differences with respect to the centre of the Empire like Rome or Pompeii [14,15].

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