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Roman wall paintings characterization from *Cripta del Museo* and *Alcazaba* in Mérida (Spain): chromatic, energy dispersive X-ray fluresence spectroscopic, X-ray diffraction and Fourier transform infrared spectroscopic analysis

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Abstract

The use of visible spectrometry, applied to the chromatic characterization of Roman wall paintings, allows easy and trustworthy grouping of the samples studied. The use of other spectroscopic techniques as Fourier transform infrared (FTIR) and energy dispersive X-ray fluorescence (EDS) in conjunction with X-ray diffraction (XRD) allows good identification of the substances present in the pictorial layers that define and differentiate each chromatic group. In this paper, a study of 21 Roman wall painting samples, from *Cripta del Museo* and *Alcazaba* (Merida, Badajoz, Spain), is described. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Pigments; Roman paintings; Chromatic measurements (CIE64); Energy dispersive X-ray fluorescence (EDS); Fourier transform infrared (FTIR); X-ray diffraction (XRD)

1. Introduction

The study of the different elements that involves the historical–artistic heritage of a people contributes to preserve the historical memory of its citizens and to maintain the own culture. So it is necessary to keep its conservation and to guarantee its transfer to future generations. This paper contributes to the knowledge of these sort of materials in order to undertake their preservation and restoration by the best possible techniques.

In this work, 21 Roman mural painting samples, of the 1st century A.D. and in a good state of conservation, from the buildings *Cripta del Museo* and *Alcazaba* in Mérida, Badajoz, Spain were studied. The study of a mural painting sample involves the characterization of the pictorial surface, that is constituted of the pigment and the substrate in which it is immersed. The chemical composition of both materials is responsible for the end color and it can offer substantial information about the techniques that were used in its manufacture.

With this aim, spectroscopic techniques have been applied for studying the samples, allowing the chromatic characterization and identification of the pigments present.

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2. Experimental

2.1. Chromatic characterization

Usually, the chromatic characterization of the pictorial layer is made using subjective methods based on visual comparison between the sample and a pre-established color table such as, for example, the Munsell code [1,2]. This method may lead to serious errors because it is very influenced by external factors such as the illumination used during the comparison, the subjective abilities of the observer and the chromatic stability of the patterns used for the comparison.

In this work, a spectroscopic method, based on the use of visible spectroscopy, has been carried out and then the data obtained may be reproduced in any laboratory. The spectroscopic reflectance has been obtained by means of an Otsuka MCPD 1100 UV–VIS spectrophotometer. The system, equipped with optical fiber elements, enables the direct study of the pictorial surface of the material without perturbing it.

Using the normalized CIE64 system [3], the chromaticity coordinates x_{10}, y_{10} deduced from tristimulus values X, Y and Z, have been calculated. These variables, that define the tone and the color saturation, have been represented in the color space diagram. The illumination system used was an incandescent tungsten lamp with a filtered emission for simulating the standard D_{65} illuminator. As a white pattern, a polymeric tablet supplied for Top Sensor Systems (model WS-2), was used. The sample chamber has a normalized geometry of 0°/45° for the illumination/observation process (Fig. 1), in order to minimize the specular/diffuse reflectance ratio of the captured radiation [2].

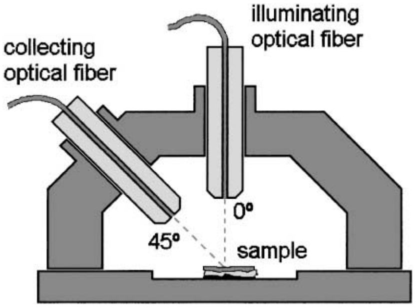


Fig. 1. Sample chamber for chromatic measurements, based on the determination of diffuse reflectance, according to the geometry 0°/45°.

2.2. Chemical analysis

The chemical identification of the pictorial and substrate substances were performed using various techniques. To obtain elemental analysis information a LINK AN10000 energy dispersive X-ray detector system, joined to a JEOL JSM820 scanning electron microscope, was used. To prevent surface charging, the samples were gold-coated; then a constant gold signal appears in all the EDS spectra.

Molecular information was achieved by Fourier transform infrared (FTIR) spectroscopy using a Nicolet lmpact 410 spectrometer with OMNIC software. The FTIR spectral analysis was made using the *search* function of the software, searching in several commercial libraries with 40,000 spectra, including US Geological Survey Minerals, Commercial Material Painter Minerals, Sigma Dyes, Stain and Natural Pigments, and Aldrich Condensed Phase and Coating Technology. In some cases, X-ray diffraction (XRD) was used to confirm the crystal structures of a compound.

3. Results

In this work, c*ri* and *alc* are the generic names that we have associated to the samples from *Cripta del Museo* and *Alcazaba*. A layer, about 1 mm thick, conforms to the pictorial surface of all the samples. The reflectance spectra were registered in the visible range over several points for each one of the different sample colors (Fig. 2), obtaining a statistical representative value. The colorimetric coordinates x_{10}, y_{10}, the saturation and the dominant wavelength achieved are shown in Table 1. A 3D graphic with the x_{10}, y_{10} and Y values are shown in Fig. 3. Regarding the chromatic coordinates and the saturation values, they seem to be very close to neutral white. This behavior is appropriate for Roman mural paintings [4] because of the small concentration of pigments in the pictorial layer.

The samples appear clearly grouped in six zones. The nomenclature used in this article for designing these groups is based on the human visual sensation of the colors; in spite of this chromaticity values obtained can indicate small discrepancies. So, the zones are named as: (1) ochre colors, (2) white colors, (3) black and gray colors, (4) reddish colors, (5) greenish

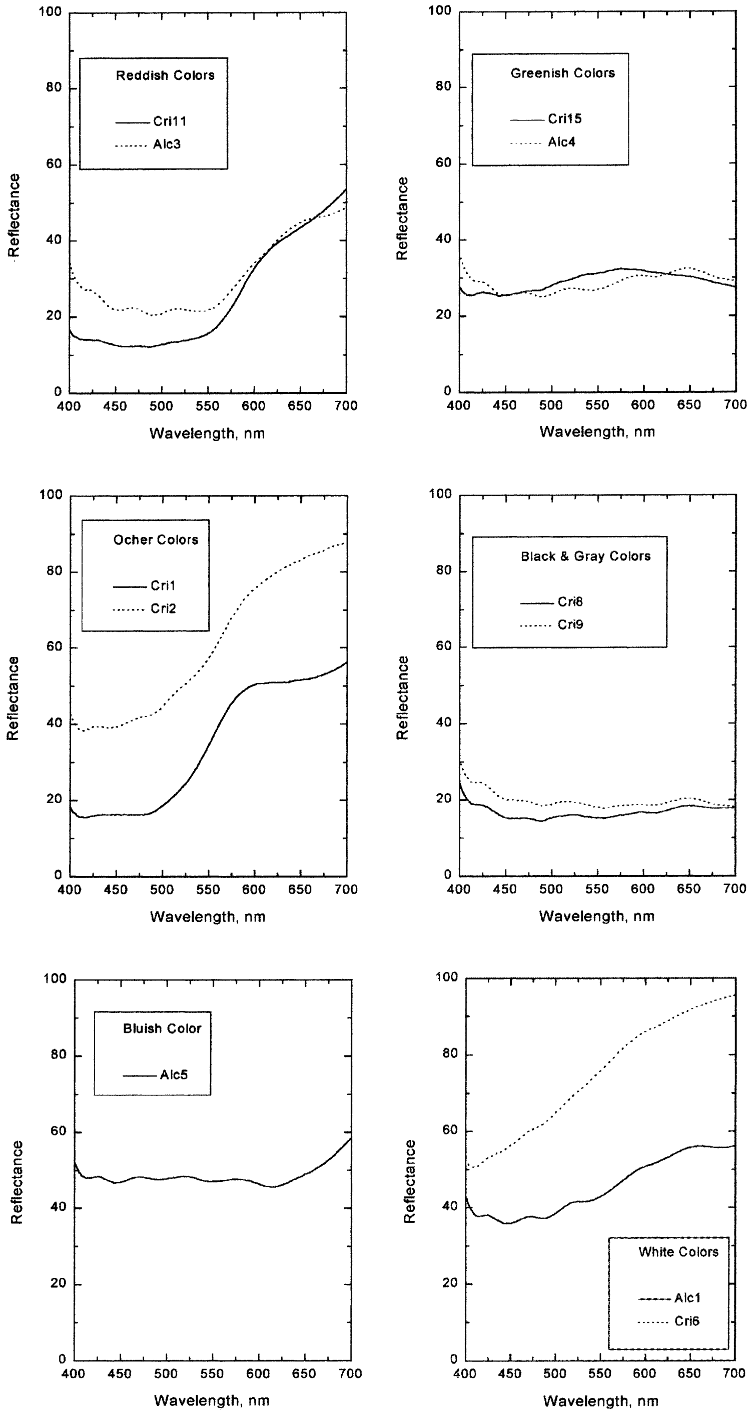


Fig. 2. Reflectance spectra of some samples studied. The samples grouped as bluish and white have a high component of specular reflectance and the spectrum shows an energy distribution not according to the chromatic sensation.

Table 1
General characteristics of the samples studied[a]

Group	Sample	Chemical composition		Chromatic coordinates			$\lambda_{dominant}$ (nm)	Saturation (%)
		Pigment	Base	x_{10}	y_{10}	Y		
Ochre colors	*cri1*	Goethite	$CaCO_3$	0.4143	0.3834	32.992	580	43.8
	cri2	Goethite	$CaCO_3$	0.3745	0.3601	54.696	582	27.5
White colors	*alc1*	Calcite	$CaCO_3/CaSO_4$	0.3519	0.3497	36.409	582	17.5
	cri3	Calcite	$CaCO_3$	0.3491	0.3548	59.019	578	18.4
	cri4	Calcite	$CaCO_3$	0.3521	0.3501	45.318	578	18.4
	cri5	Calcite	$CaCO_3$	0.3507	0.3521	48.012	576	18.4
	cri6	Calcite	$CaCO_3$	0.3436	0.3544	78.742	575	16.0
	cri7	Calcite	$CaCO_3$	0.3496	0.3574	79.990	576	18.4
Black and gray colors	*alc2*	Carbon black	$CaCO_3/CaSO_4$	0.3261	0.3335	25.370	–	–
	cri8	Carbon black	$CaCO_3$	0.3189	0.3256	16.061	–	–
Black and gray colors	*cri9*	Carbon black	$CaCO_3$	0.3084	0.3202	22.056	–	–
	cri10	Carbon black	$CaCO_3$	0.3216	0.3349	37.075	–	–
Reddish colors	*alc3*	Hematite	$CaCO_3/CaSO_4$	0.3950	0.3361	16.407	603	24.4
	cri11	Hematite	$CaCO_3$	0.4124	0.3441	19.132	599	40.0
	cri12	Hematite	$CaCO_3$	0.3671	0.3284	21.688	620	13.8
	cri13	Iron oxides	$CaCO_3$	0.3742	0.3464	48.050	591	21.6
	cri14	Hematite	$CaCO_3$	0.3647	0.3297	25.913	620	14.0
Greenish colors	*alc4*	Green earth	$CaCO_3/CaSO_4$	0.3498	0.3433	18.818	586	14.1
	cri15	Green earth	$CaCO_3$	0.3335	0.3505	33.239	571	11.1
	cri16	Green earth	$CaCO_3$	0.3225	0.3489	37.655	562	7.9
Bluish colors	*alc5*	Egyptian Blue	$CaCO_3/CaSO_4$	0.3075	0.3285	31.748	480	2.6

[a] The main compound of the pigmentary layer and binder are shown. The chromatic coordinates, luminosity, dominant wavelength and saturation of each sample are exposed.

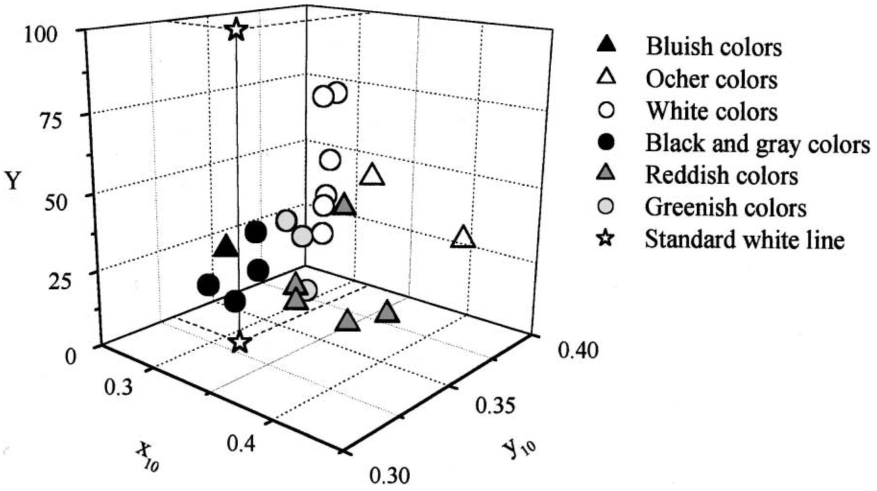


Fig. 3. 3D graphic of the chromatic coordinates measured for the 21 samples studied. Six groups of samples can be observed, each one associated with human chromatic sensations.

colors and (6) bluish colors. The obtained results are described below for each of the groups.

Different crystallographic varieties of calcium carbonate are detected in the binder for all the samples from the *Cripta del Museo*, along with varying amounts of common impurities, which may include quartz, ankerite, dolomite, etc. This has permitted us to deduce that the paintings are made using the "fresco painting" technique, involving lime mortar. By comparison, in the *Alcazaba* samples, besides lime mortar, a thin layer (ca. 500 μm) of calcium sulfate (gypsum) is found. This compound has been identified in other mural painting samples as of *Casa Coupona* from *Pompeya* [5] and *Casa de los Delfines* from *Zaragoza* [5,6]. Typical components of standard silicates appear in many cases. These silicates are associated with impurities due to the mortar, dust and burial remains and, only in some cases, to the pigment used for coloring the layer.

3.1. Ochre colors

These colors appear in the *cri1* and *cri2* samples. Its chromatic coordinates are aligned in the CIE64 diagram, with an average saturation and a dominant wavelength value very much defined; this one is around 581 nm, and it seems to indicate that the samples present the same kind of pigment. In Fig. 4, the EDS spectrum of the *cri1* sample is shown. Apart from calcium, silicon, potassium, aluminum and magnesium, iron is also detected. In Fig. 5, the infrared spectrum of *cri2* sample is shown. In this spectrum, we can identify calcite (bands at 2515, 1439, 874 and 705 cm^{-1}), common clayey silicates like illite (band at 1027 cm^{-1} with a shoulder at 1092 cm^{-1}) and yellow iron oxide (bands at 912 and 799 cm^{-1}). This last compound is considered the pigment that defines the sample color. Some of the band shapes are due to the sum of more than one vibrational band of the aforementioned compounds (i.e. the wide band sited at 3425 cm^{-1} with a shoulder to 3180 cm^{-1}).

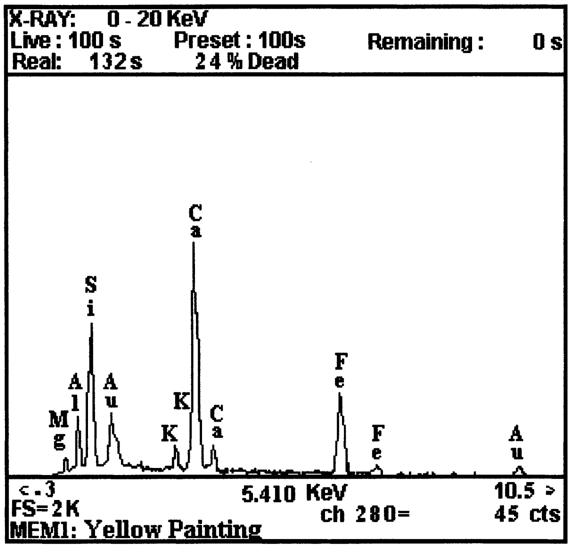


Fig. 4. Energy dispersive X-ray fluorescence spectrum of the *cri1* sample. One can observe the presence of magnesium, aluminum, silicon, potassium, calcium and iron elements. The gold is due to the conductive coating.

Finally, applying XRD to the *cri2* sample (Fig. 6) shows that apart from quartz, calcite and some silicates, the presence of goethite is detected according to the attribution made in the FTIR spectrum.

3.2. White color

It appears in *cri3, cri4, cri5, cri6, cri7,* and *alc1*. Like a defining character, all of them present a dominant wavelength between 575 and 578 nm. These wavelength values indicate us that they are actually situated in the yellowish zone but its low saturation (16.0–18.4%) and high luminosity provoke a white visual sensation. Specially the *cri6* and *cri7* samples present a luminosity of $Y = 78.74$ and 79.9 with a high sensation of white whereas the rest have a medium luminosity so that they present a more grayish appearance. This factor is very common in the measurement of pigments called white, as can be checked in previous studies [7].

From the FTIR spectrum (Fig. 7) a major presence of $\mathrm{CaCO_3}$ varieties, may be deduced because all the bands of these species appear in the spectrum. Specific pigments, associated to the white chromatic sensation, are not detected except calcium carbonate. The presence of minority portions of blue pigments for increasing the white sensation is not detected. These blue pigments have been used in some buildings located in the same metropolis and belonging to the same historical period, as for example in the *Casa del Mitreo* [8].

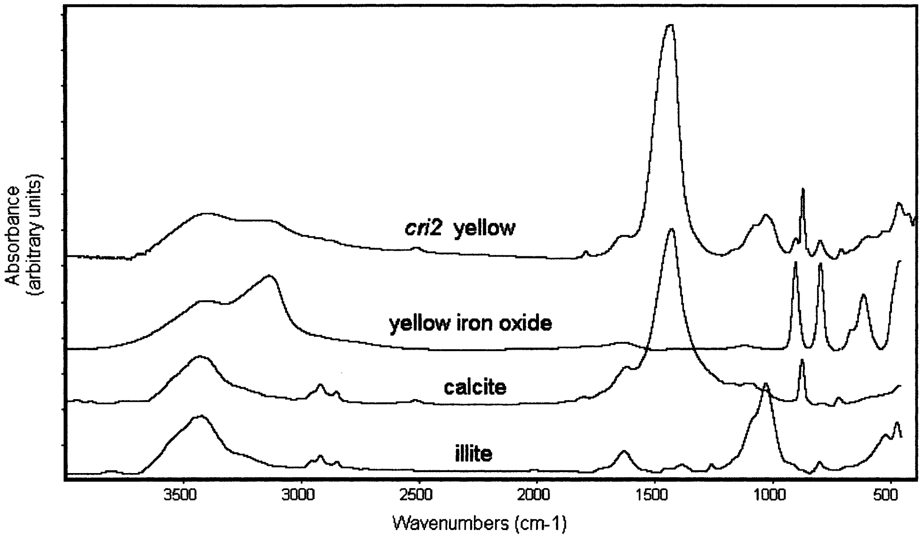


Fig. 5. FTIR spectrum of the *cri2* ochre sample. The spectra of calcite, illite and yellow iron oxide are taken from spectral libraries.

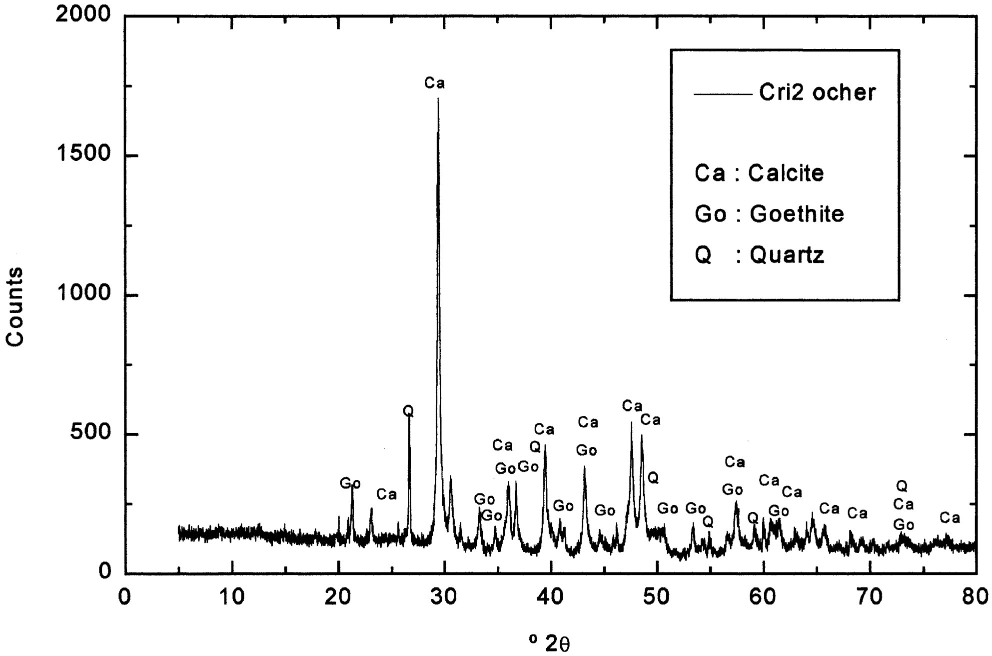


Fig. 6. X-ray diffractogram of the *cri2 ochre* sample. Goethite, calcite and quartz are detected.

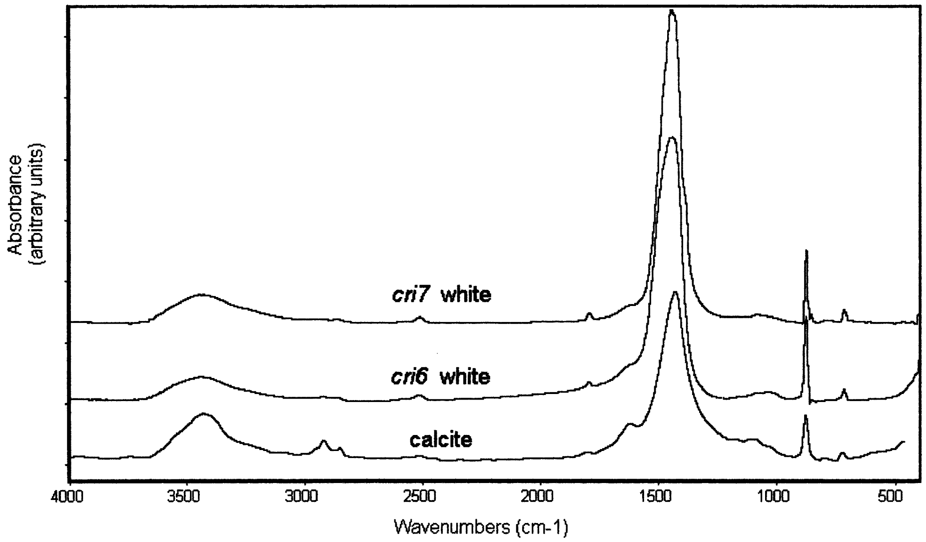


Fig. 7. FTIR spectrum of the *cri6* and *cri7* white samples. The calcite compound only is detected and no other pigment is present.

3.3. Black and gray colors

The black color appears in the *cri8, cri9* and *cri10* samples whereas the gray color appears only in the *alc2* sample. All the samples are very much grouped around the achromatic point and then it is not suitable to define a dominant wavelength.

The bands that define the FTIR spectrum of these black samples permit calcium carbonate compounds (bands at 3425, 2515, 1439, 874 and 705 cm^{-1}) and common silicates (band at 1027 cm^{-1} and his shoulder at 1092 cm^{-1}) to be detected. Any band associated with some organic or inorganic pigment is not detected, as we can observe in the *cri9* sample shown in Fig. 8. Because the graphite and amorphous carbon cannot be detected by the FTIR technique, we have studied the samples by XRD. With it, the presence of calcite and basic silicates is confirmed and also we can detect a very small quantity of carbon in the form of graphite (Fig. 9), but we assume that it is too little to generate all the black color. The existence of another carbon morphology as amorphous carbon is feasible. The greatest probability is the presence of vegetal carbon because we cannot detect parallel substances like calcium phosphate with bone carbon or iron oxide with mineral carbon.

3.4. Reddish colors

This group is made up of: (a) the *cri11*, *cri12*, *cri14* and *alc3* samples associated with a red chromatic sensation and (b) the *cri13* sample with a pink-yellowish chromatic sensation. The chromatic coordinates show that the red samples are defined by a dominant wavelength >599 nm and the pink sample of 591 nm.

The FTIR spectra of the *cri14* and *alc3* samples are shown in Figs. 10 and 11. In the *cri14* sample spectrum we can detect, besides calcium carbonate compounds (bands at 2515, 1439, 874 and 705 cm^{-1}) and clay silicates (bands at 1081 and 795 cm^{-1}), iron oxide like hematite (bands at 642 and 534 cm^{-1}). The last is the main pigmentary compound. These data are corroborated by energy dispersive X-ray fluorescence (EDS) spectra (Figs. 12 and 13), where the presence of Fe and O are detected, elements that can be associated with iron oxides. Of course, we can detect Ca, Al, Mg and Si, chemical elements associated to common silicates and calcium carbonates. However, in the *alc3* sample FTIR spectrum, apart from the aforementioned compounds, we can detect calcium sulfate in the crystallographic form of gypsum, characterized by the 3549, 3406, 1142 and 1117 cm^{-1} bands. The presence of gypsum is corroborated by the S detection in the EDS spectrum.

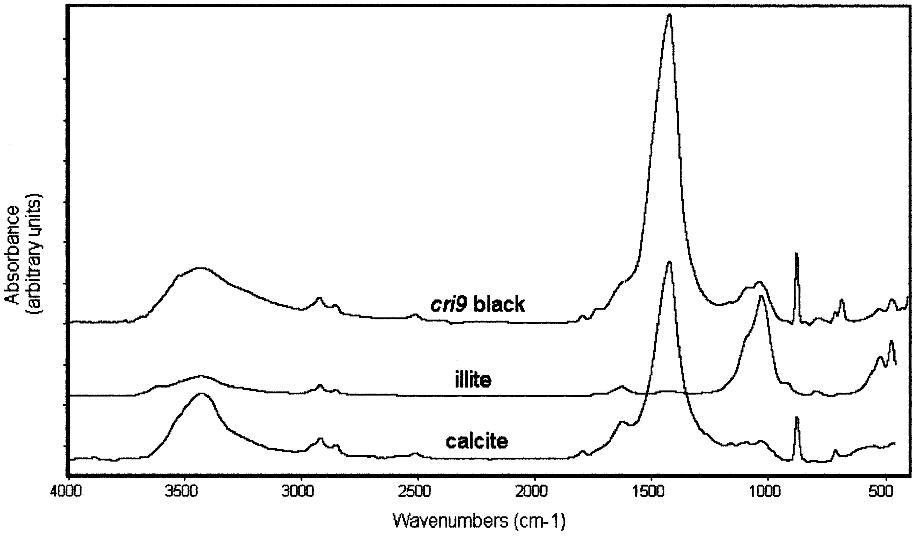


Fig. 8. FTIR spectrum of the *cri9* black sample. On it, one can detect a mixture of calcite and common silicates as illita. The reference spectra are obtained from commercial libraries.

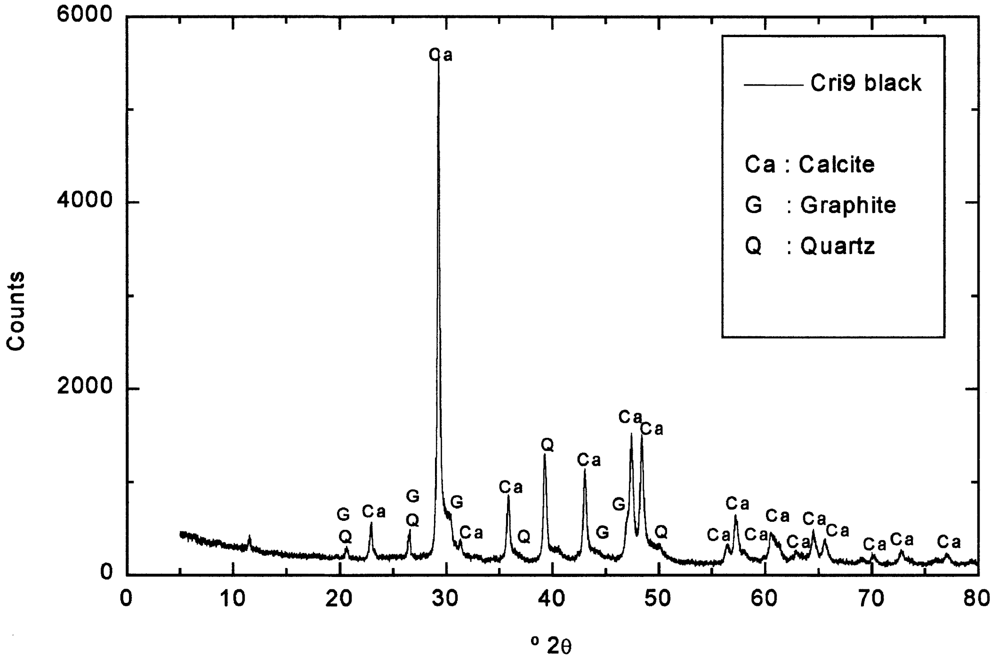


Fig. 9. X-ray diffractogram of the *cri9* sample. Calcite, quartz and a little graphite are detected.

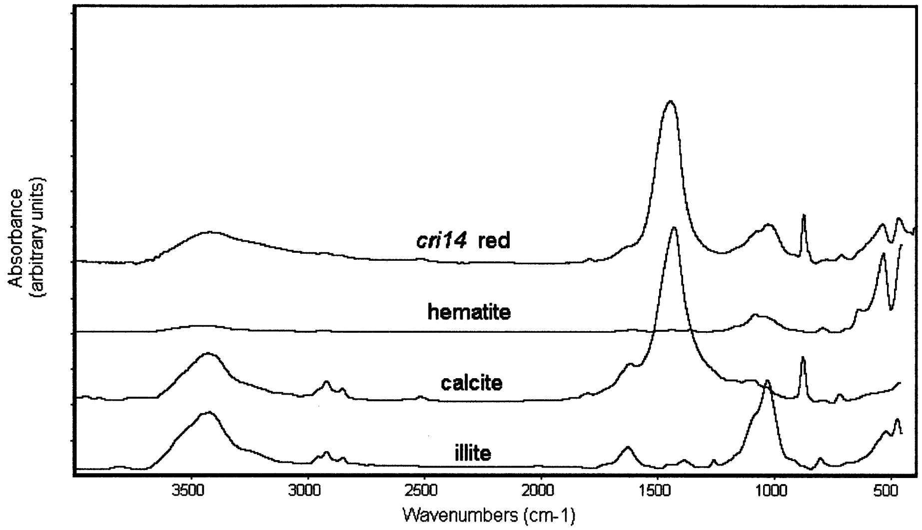


Fig. 10. FTIR spectrum of the *cri14* red sample. On it, one can detect hematite as pigment and a mixture of calcite and common silicates as illita as binder.

As the iron oxide pigment is the only pigmentary substance is detected, we assume that the color differences between the red samples and the pink-yellowish sample are due to mixtures of iron oxides in different oxidation states. This means that the resultant color is intermediate between the red ochre and the yellow ochre.

3.5. Greenish colors

They appear in *cri15, cri16* and *alc4*. As we can observe in Table 1, the two samples from the *Cripta* show a different dominant wavelength, 571 nm for the *cri15* and 562 nm for the *cri16*, both wavelengths in the greenish-yellow zone. The longer wavelength of

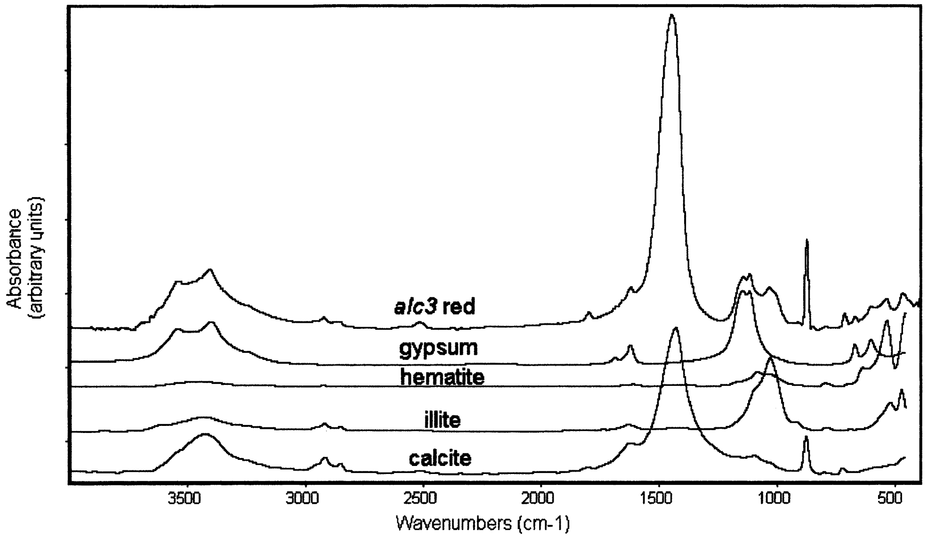


Fig. 11. FTIR spectrum of the *alc3* red sample. The pigmentary substance is iron oxide in hematite form. The binder compounds are similar to the *cri* samples but we can detect a moderate quantity of gypsum.

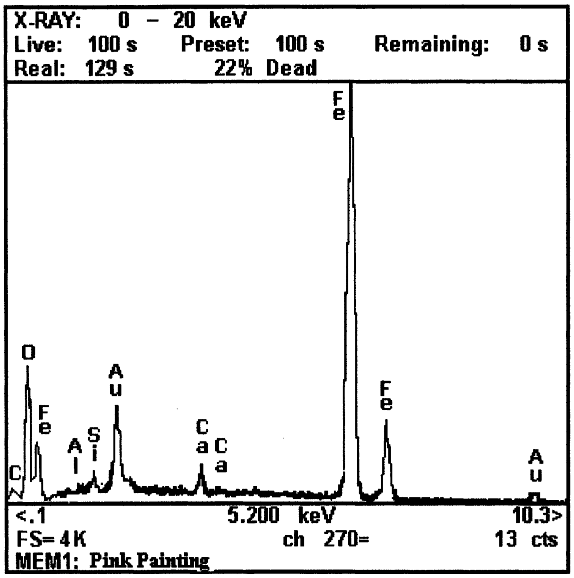


Fig. 12. Energy dispersive X-ray fluorescence (EDS) spectrum of the *cri13* pink sample. The presence of iron is shown.

the first sample is explained because the green coloration is placed on a red layer that functions as a base color, while in the second sample the green layer is directly on the mortar.

In contrast, the *Alcazaba* sample shows a higher dominant wavelength (586 nm), corresponding to the yellowish-orange zone. As well as in the *cri15* sample, in the *alc4* sample the pigment is on a red layer that behaves like a base color.

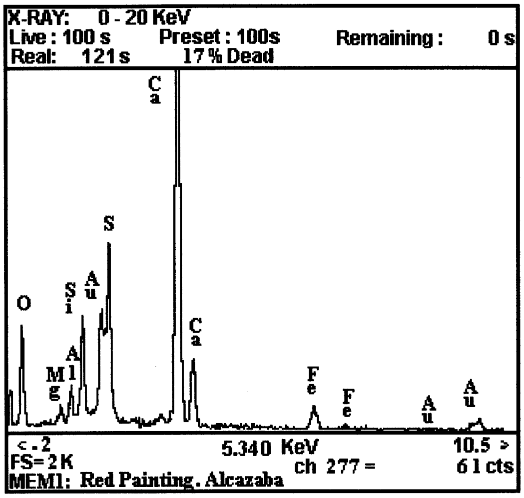


Fig. 13. Energy dispersive X-ray fluorescence (EDS) spectrum of *alc3* red sample. The sulfur detection confirms the presence of gypsum also identified by FTIR spectroscopy.

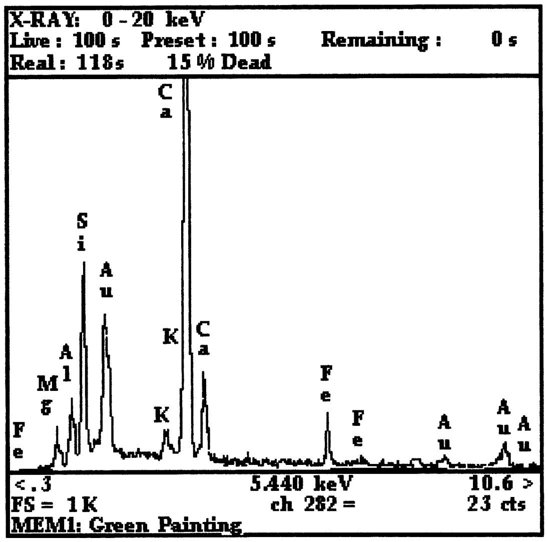


Fig. 14. Energy dispersive X-ray fluorescence (EDS) spectrum of the *cri15* green sample. The detection of magnesium, aluminum, silicon, potassium and iron can be associated with the presence of green earths.

As an example, the energy dispersive X-ray fluorescence (EDS) spectrum of *cri15* sample (Fig. 14) is shown. In it, the presence of silicon, aluminum, iron and potassium is observed and these elements can be associated with the so-called *green earth*. Under the name of *green earth* [9] a mixture of clays is embraced and the most important of them are glauconite, celadonite, and the chlorite group (clinochlore and penninite); all of them are aluminum silicates with impurities of Fe^{2+}, Fe^{3+}, K^+ and other ions.

In the Fig. 15, the FTIR spectrum of the sample of *alc4* is shown. The bands associated with the presence of calcium carbonate compounds, calcium sulfate (gypsum) and clinochlore (green earth) are detected. This last is characterized by: (a) a small band at 662 cm^{-1}, (b) a wide band with two peaks at 978 and 1009 cm^{-1} mixed with some of the gypsum and calcium carbonate peaks, and (c) a band that appears at 3406 cm^{-1} with two shoulders at 3549 and 3680 cm^{-1} [10].

Finally, using XRD on the *cri16* sample (Fig. 16), apart from quartz, calcite and some silicates, the

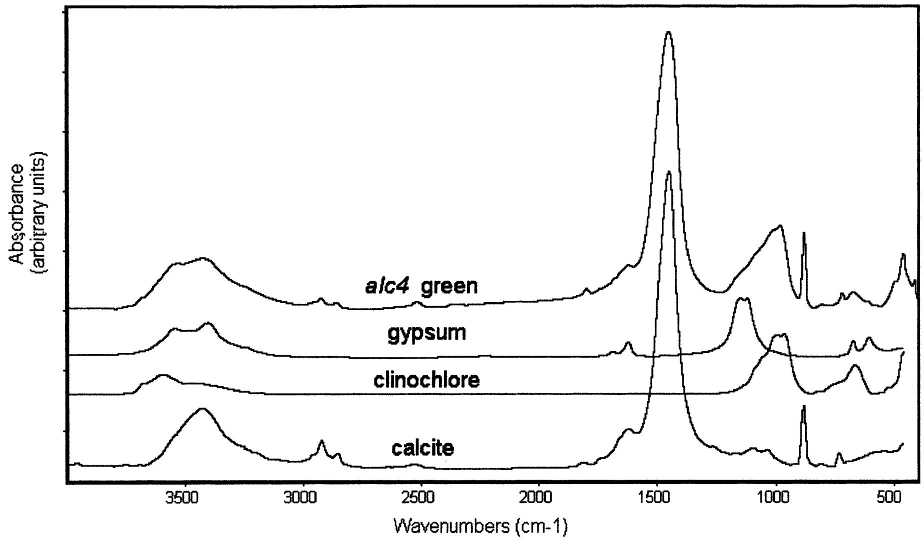


Fig. 15. FTIR spectrum of the *alc4* green sample. Besides the typical substances of the binder, one can detect clinochlore, a typical compound of the green earths.

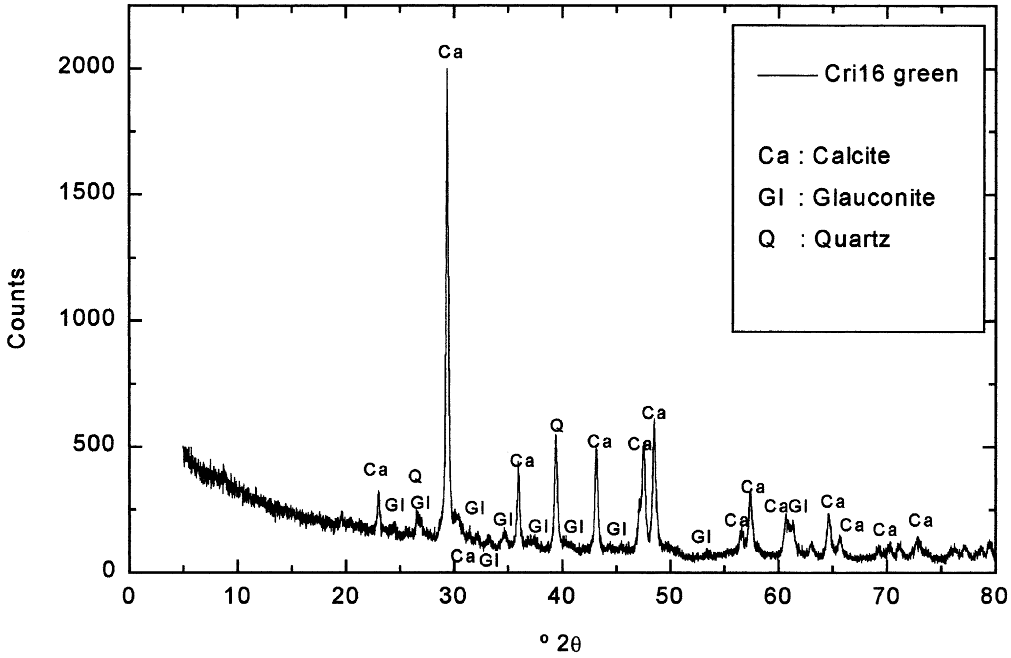


Fig. 16. X-ray diffractogram of the *cri16* green sample. The presence of calcite, quartz and glauconite is detected.

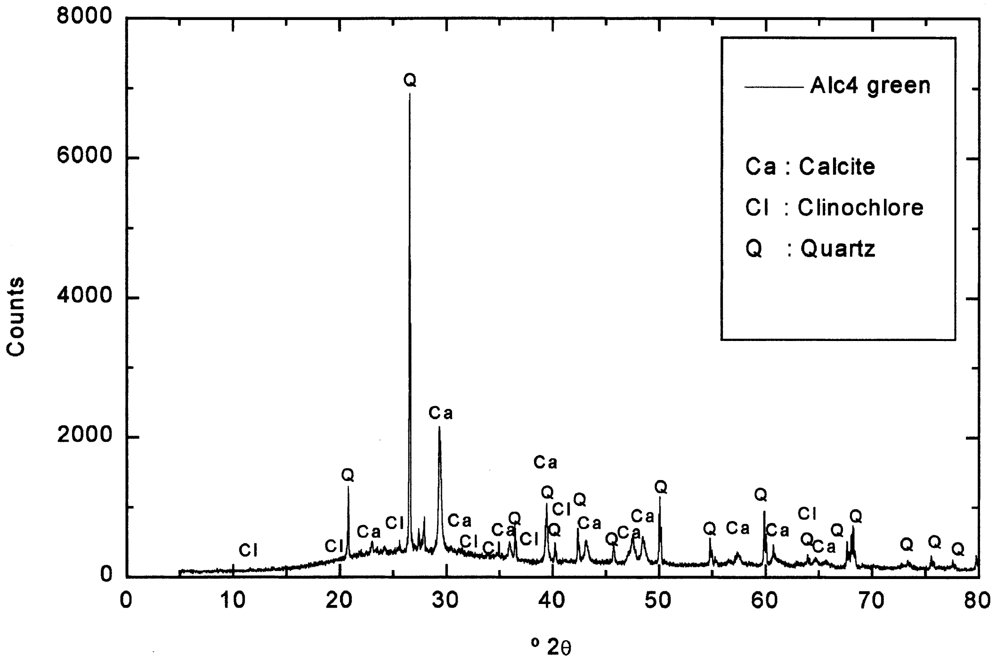


Fig. 17. X-ray diffractogram of the *alc4* green sample. The presence of calcite, quartz and clinochlore is detected.

presence of green earth as glauconite is detected. In the *alc4* sample (Fig. 17), apart from quartz, calcium carbonate as calcite and some silicates, the presence of green earth as clinochlore is observed. The data corroborate the deductions made using FTIR.

3.6. Bluish colors

This color only appears in the *alc5* sample with a dominant wavelength of 480 nm. This data is similar to the value shown by an artificial calcium copper silicate, the so-called Egyptian Blue pigment. Because of the coincidence of XRD data of this pigment with the mineral cuprorivaite, the accepted formula for this pigment ($CaCuSi_4O_{10}$) is that of cuprorivaite [11]. This has been detected in other samples of Roman mural paintings in Merida [8]. Studies by IR, UV and visible spectroscopy of this pigment are very scarce [11] and, for this reason, we decided to synthesize the pigment using the Ullrich [12] recipe. The compound obtained has been positively identified as cuprorivaite by XRD (Fig. 18). The FTIR, UV and visible spectra of the synthesized pigment have been used to corroborate its presence in the *alc5* sample.

Fig. 19 shows the XRD analysis carried out on the *alc5* sample. In this we can observe the presence of cuprorivaite together with calcite, gypsum and quartz. In Fig. 20, the FTIR spectrum of the sample is shown. This spectrum is compared with the others ones shown by calcite, gypsum and the synthesized pigment Egyptian Blue, the two first from the spectral library of the system. Besides the $CaCO_3$ characteristic bands, a group of bands centered at 1150, 1116, 1055 and 1010 cm^{-1} is observed, attributed to Egyptian Blue. The bands at 1150 and 1116 cm^{-1} show an increase in intensities because of the contribution of the gypsum bands at 1150 and 1121 cm^{-1}. The presence of this last compound is corroborated for the appearance of two bands at 3405 and 3544 cm^{-1}.

In order to confirm the presence of Egyptian Blue the X-ray fluorescence spectrum of one blue grain of the sample has been obtained (Fig. 21). It shows the presence of silicon, calcium and copper, characteristic elements of the pigment.

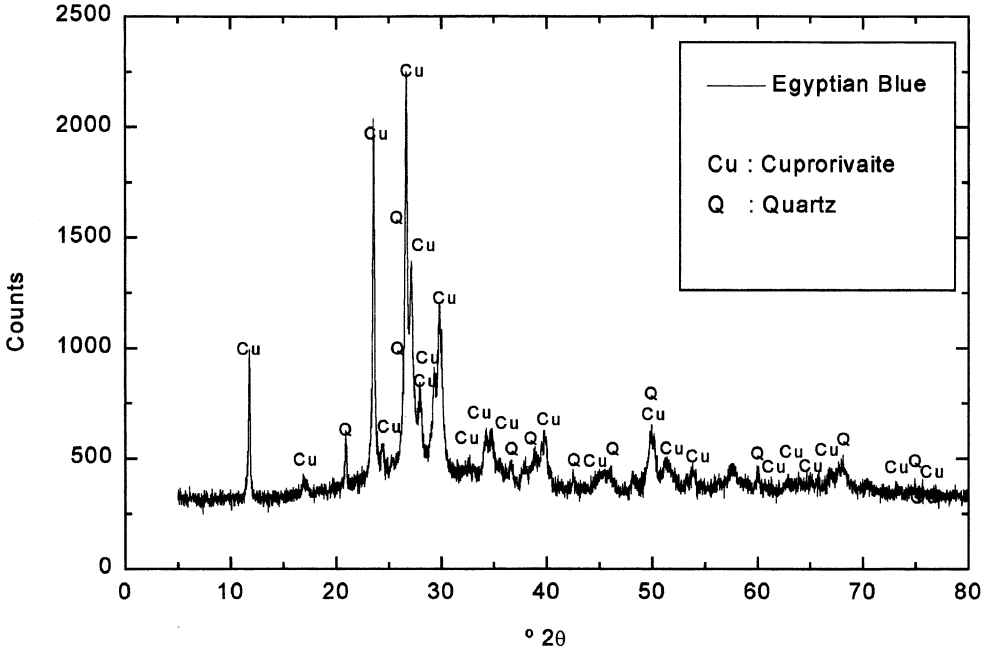


Fig. 18. X-ray diffractogram of Egyptian Blue, synthesized following the Ullrich recipe. The pigment is identified as cuprorivaite.

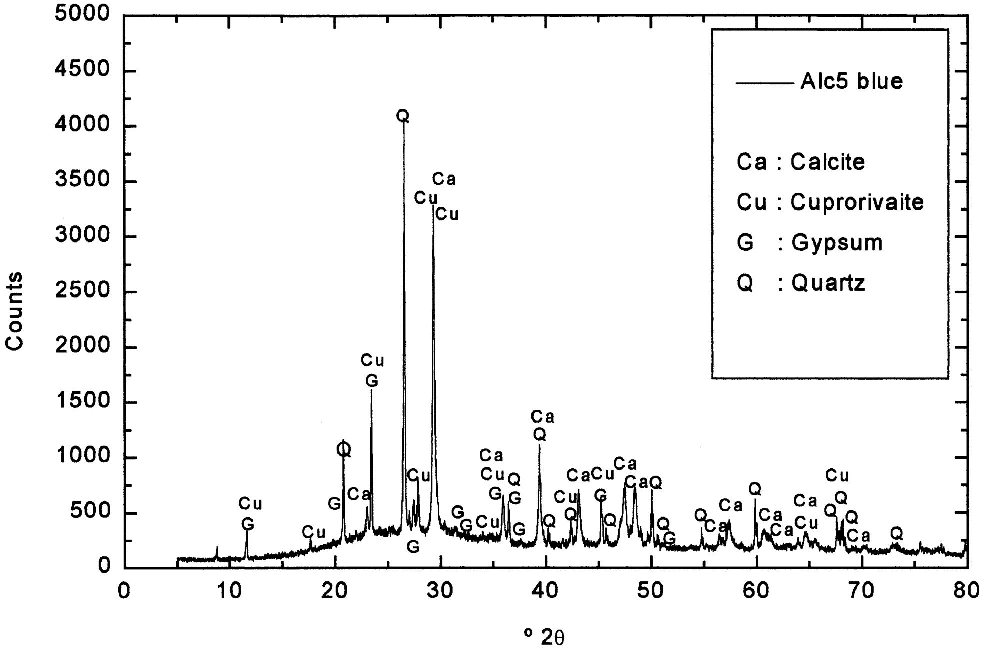


Fig. 19. X-ray diffractogram of the *alc5* blue sample. The presence of cuprorivaite, calcite, quartz and gysum is detected.

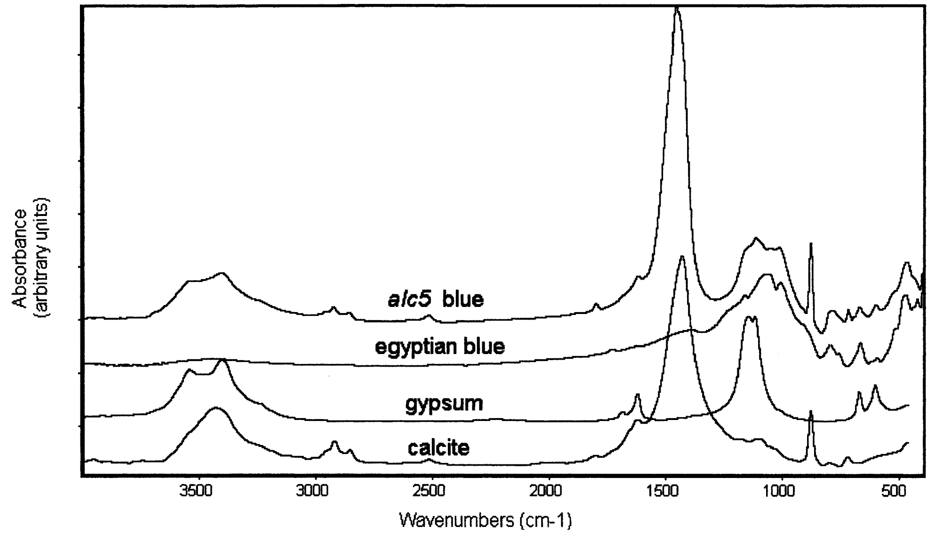
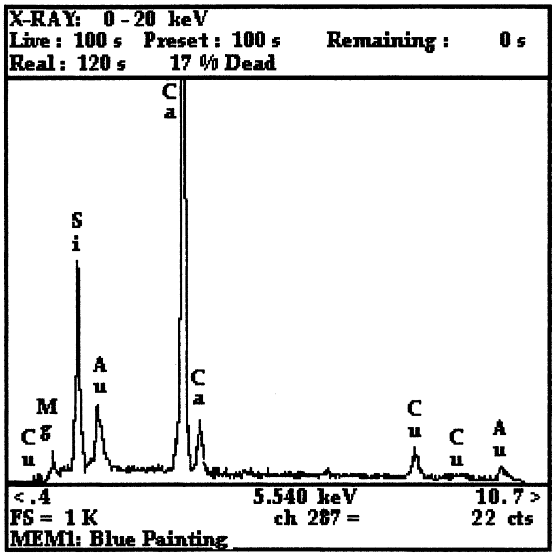


Fig. 20. FTIR spectrum of the *alc5* blue sample. The main compounds detected are calcite, gypsum and Egyptian Blue. The last one is associated with the pigment.



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X-RAY:  0 - 20 keV
Live : 100 s  Preset : 100 s     Remaining :      0 s
Real : 120 s     17 % Dead
< .4                  5.540 keV             10.7 >
FS = 1 K               ch 287 =             22 cts
MEM1: Blue Painting
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Fig. 21. Energy dispersive X-ray fluorescence spectrum of a pigmentary particle from the *alc5* blue sample. The copper, calcium and silicon elements are related to the presence of Egyptian Blue pigment.

4. Conclusions

To accomplish the compositional analysis of Roman wall paintings, a convenient method is to group the samples by chromatic criteria. The best way to determine the chromatic characteristics of the sample is to use the reflectance spectroscopy in the visible region and then to apply a normalized process of chromatic coordinate calculus as defined by CIE64. Atomic and molecular analysis, by energy dispersive X-ray fluorescence and FTIR spectroscopy and the crystallographic analysis by XRD, applied to each chromatic group allow identification of the pigments as well as the pictorial techniques that were used.

The application of these techniques has allowed us to ensure that the mural paintings studied from *Cripta del Museo* have a high quality [13]. In all the cases, the pigment is supported in lime mortar (calcium carbonate), in comparison to the samples from *Alcazaba* in which the presence of a thin layer of gypsum (calcium sulfate) on the pigmentary layer has been detected. Regarding the pictorial technique used, there is no indication of the presence of organic substances like wax and soap. It allows us to deduce that the called "fresco painting" technique has been used for applying the colors.

Regarding the different color types that appear in the samples, we can deduce the following.

- The ochre and yellow colors have mainly been obtained from yellow iron oxides.

- The white color is mainly due to own calcite substratum. No sample shows the presence of blue pigments for increasing the white sensation, as has been encountered in other Merida samples.
- The black and gray colors appear to be black carbon. The most probability is the presence of vegetal carbon since the existence of collateral substances like calcium phosphate from bone carbon and iron oxide from mineral carbon have not been detected.
- The reddish colors are made of diverse mixtures of iron oxides as hematites, limonite, goethite, etc.
- The greenish colors have been obtained using green earth. Glauconite is the main pigment for the *Cripta del Museo* samples and clinocholore for the *Alcazaba* samples.
- The blue color is due to the artificial pigment, the so-called Egyptian Blue.
- All the pigments found are according to those mentioned in [14] for that historical epoch.

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