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RESEARCH ARTICLE

Identification of 17th-18th Century Pictorial Materials in Church Mural Paintings in the Cuzco Area (Perú) Using Micro-Invasive Analytical Techniques.

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Abstract: The interdisciplinary study of mural paintings in rural churches of the Viceroyalty of Peru, dating from the 17th and 18th centuries, provides critical insights into the availability, provenance, and application of pigments used by colonial artists. This research focuses on murals from the churches of Marcapata and Canincunca in the Cuzco region. Microsamples from these murals were analyzed using micro-invasive techniques, including optical microscopy, scanning electron microscopy coupled with energy-dispersive spectroscopy (SEM-EDS), and Raman microspectroscopy. The study revealed a preparation layer of gypsum/anhydrite on adobe walls and a simple yet diverse palette of pigments. Red pigments included hematite (Fe_2O_3) and organic carmine dye, while green hues featured copper-based pigments such as antlerite ($Cu_3SO_4(OH)_3$) and posnjakite ($Cu_4SO_4(OH)_6 \cdot H_2O$), identified here for the first time as a major component in Andean mural paintings. Blue tones were achieved using indigo and smalt, a cobalt-containing glass pigment whose presence is still scarcely documented in Peruvian murals. These findings deepen our understanding of colonial pigment selection, preparation practices, and their deterioration mechanisms, contributing to sustainable conservation strategies. This study highlights the value of interdisciplinary approaches in advancing knowledge of Hispanic-American colonial art and its preservation as a vital component of cultural heritage.

Introduction

The chemical study of cultural heritage is fundamental to understanding historical pictorial practices, the selection and use of local resources, and technological traditions, especially in Andean colonial contexts. The interaction between European and local pictorial traditions generated works with distinct material characteristics, whose analysis can reveal crucial aspects of the cultural and technological dynamics of the time. In this sense, interdisciplinary studies are particularly important.

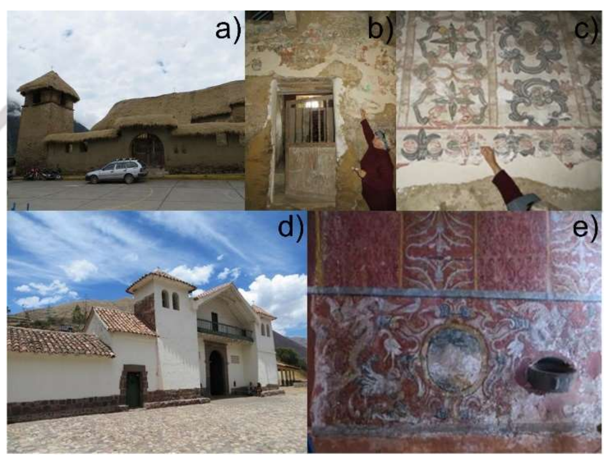

Figure 1. (a) Image of the church San Francisco de Asís de Marcapata; (b) and (c) details of wall painting from this church; (d) Image of the church Nuestra Señora de la Candelaria de Canincunca and (e) detail of wall painting from this church.

During a campaign carried out in 2015, mural paintings were surveyed in two viceregal churches located in the southern Andean region of Peru: San Francisco de Asís de Marcapata and Nuestra Señora de la Candelaria de Canincunca. Both buildings, built in the 16th century, preserve pictorial programmes created

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between the 17th and 18th centuries. Although they are located outside the main commercial circuits, such as the Silver Route (*Ruta de la Plata*)[1], they share stylistic and technical characteristics with other colonial churches in the region. The murals feature floral motifs, scrollwork, and bands that mimic textiles, typical of Andean iconography, adorning both the nave and the sacristy[2,3]. The analysis of these murals allows us to explore the pictorial practices of the region and the availability of pigments used by colonial artists in the rural environment of *Cuzco*.

Previous studies carried out on murals in colonial churches along the Silver Route, such as those of San Andrés de Pachama in Chile and Copacabana de Andamarca in Bolivia, have shown the use of pigments of local origin and techniques characteristic of the Andean environment[3–5]. Among the most significant materials are basic copper sulphates, such as antlerite ($Cu_3SO_4(OH)_4$) and brochantite ($Cu_4SO_4(OH)_6$), identified as green pigments used in these murals. These compounds indicate not only the use of local mineral resources, but also the continuity of technological practices of pre-Hispanic origin integrated into the colonial context[2–6]. These mineral species, characterised by techniques such as SEM-EDS, attenuated total reflection Fourier transform infrared spectroscopy (FTIR-ATR) and micro-Raman spectroscopies, and, in selected cases, X-ray diffraction (XRD), stand out for their heterogeneous composition, including aluminosilicates and traces of iron oxides, suggesting a local mineral origin in the Andean areas of Chile and Bolivia. These findings reflect both access to and selection of specific resources in the regional context, and their chemical analysis provides valuable information on the properties and behaviour of these pigments under the extreme conditions of the Andean environment[3,5,6].

In contrast, due to their location away from the main colonial trade routes, church murals in southern Peru, such as those at Marcapata and Canincunca, present a unique context. A comprehensive characterisation of materials, including pigments, binders, and substrates, enhances our understanding of the artistic techniques and resources employed in these regions. In addition to providing technical insights into the materials, this analytical approach strengthens the interdisciplinary framework for the preservation and study of South American artistic and material heritage[7,8].

The role of micro-samples and the efficient use of analytical resources in the material study of colonial murals.

In this study, microsamples collected in 2015 from colonial murals in churches in the Cuzco region of Peru were analysed. These fragments, extracted prior to restoration interventions, preserve original materials that may otherwise have been altered or lost during conservation processes. Their analysis offers a direct and minimally invasive approach to investigate the composition, stratigraphy, and painting techniques employed in these works. Moreover, these microsamples serve as a physical archive that ensures the long-term availability of primary material for future inquiries and for the reinterpretation of data as analytical technologies evolve[9]. While non-invasive methodologies are essential for minimsiing the impact on heritage objects[10], representative sampling strategies and the selective application of micro-destructive techniques remain critical in interdisciplinary projects with limited access to advanced instrumentation, especially portable equipment for *in situ* use. The analytical approach adopted here integrates tools from chemistry with expertise from art history, mineralogy, and conservation science, considering the mural paintings samples as material archives. This perspective calls for specific methodological frameworks and a critical interpretation of data obtained through optimised instrumental analysis.

After collection, the samples were analysed using Raman spectroscopy and scanning electron microscopy (SEM). During the interpretation of the initial results, new research questions emerged, prompting complementary analyses. These were conducted using different instruments, reflecting a flexible and adaptive approach based on the availability of analytical resources. While repeating all measurements across multiple platforms would have been ideal, a selective analysis strategy was adopted to ensure both analytical rigour and sustainability. This approach reflects a broader methodological rationale: the efficient use of available resources, the adaptation to logistical constraints, and the pursuit of high-quality results within a realistic and responsible analytical framework[7].

Experimental Section

Painting samples

Microsamples (1-2 mm) were extracted with a scalpel from coloured areas of the different sectors of the mural paintings. The location and the colours of the samples from each church are shown in Tables 1 and 2, respectively. Fragments of samples with an area of less than 1 mm were embedded into a transparent acrylic resin Subiton® (Buenos Aires, Argentina) and polished with sandpaper of decreasing grain size (down to 12 mesh) to prepare the cross-sections (Tables 1 and 2).

Instrumental and methodology

Optical microscopy (OM): Observation and photography of the surface and cross-section of the samples were achieved using a Leica DM 2500M microscope with polarised light.

Scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS): SEM analysis by secondary electrons (SE), backscattered electrons (BSE) and elemental mapping were carried out using a Field Environmental Scanning Electron Microscope Zeiss Supra 40 coupled with an EDS microanalysis INCA X Sight, Oxford Instrument. Analyses were carried out using an accelerated voltage of 20 kV. Cross-sections of the samples were coated by sputtering with a thin (less than 80 Å) layer of platinum.

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Micro-Raman spectroscopy: Raman spectra were acquired using a DXR Smart Raman microscope (Thermo Fisher Scientific, USA) at INQUINOA, Universidad Nacional de Tucumán (Argentina), equipped with 532 nm and 780 nm excitation lasers (5 cm^{-1} spectral resolution). Spectra were obtained by averaging 30–60 accumulations of 0.5 s. Laser power was kept below 5 mW, and the spectral range was 50–3000 cm^{-1}.A 50 µm confocal slit and 10× or 50× objectives were employed. Data acquisition and processing were performed using OMNIC 9.0 software (Thermo Fisher Scientific, USA).

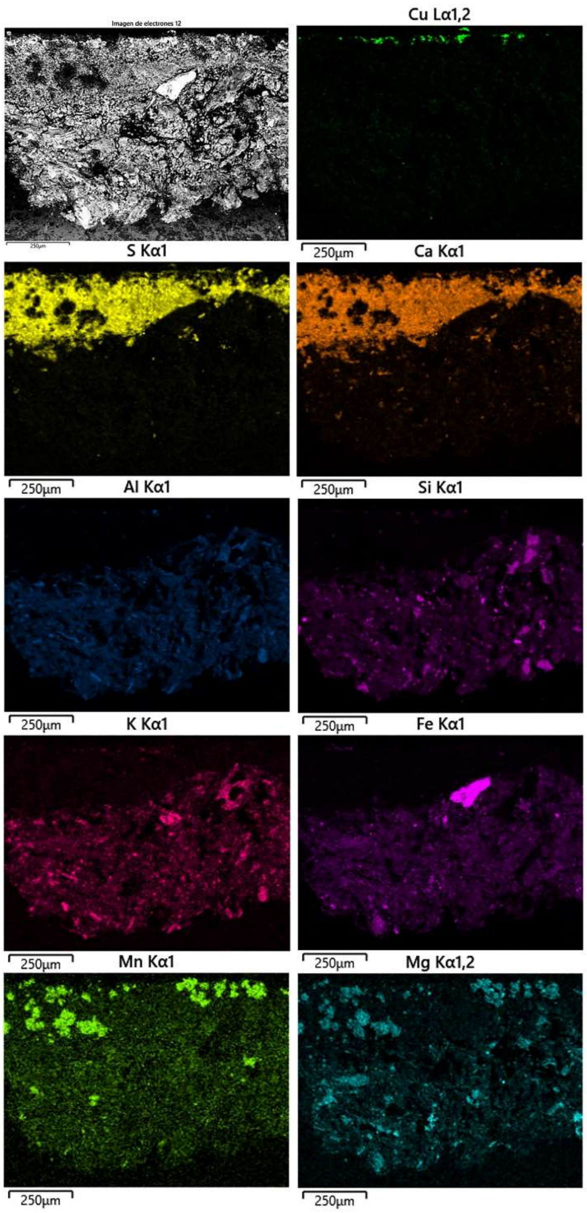

Figure 2. BSE micrography and SEM–EDS results of mapping on cross-section of sample MARCA01.

To confirm or refine specific results, selected samples were reanalyzed using a Horiba LabRAM HR Evolution confocal Raman microscope (Horiba Scientific, France) at INQUIMAE, Universidad de Buenos Aires (Argentina), equipped with 532 and 633 nm excitation lasers, a 600 L/mm grating, and a Peltier-cooled Synapse CCD detector. Spectra were acquired in backscattering geometry with 20× or 50× objectives, using acquisition parameters adapted to each sample (1–30 s exposure, up to 20 accumulations). Laser power was kept below 5 mW, and the spectral range was 200–1800 cm^{-1}.

Results and Discussion

Church of San Francisco de Asís in Marcapata.

Ground, binders and techniques

Optical microscopy (OM) analysis of cross-sections from the samples revealed the presence of a white preparatory layer (Table 1). In sample MARCA01, fragments of the underlying church wall were also observed, consistent with historical sources that describe its construction in adobe, a traditional mixture of earth, water, stones, and *paja brava* straw[4,5]. Elemental mapping also detected manganese and magnesium at the base, together with aluminium (Al) and silicon (Si) from aluminosilicates and iron oxides in the support, possibly pyroxenes that provide a dark colouring. SEM-EDS analysis of the plaster layer confirmed high levels of calcium (Ca) and sulphur (S), pointing to a calcium sulfate system ($CaSO_4 \cdot XH_2O$). Raman spectroscopy carried out on this preparatory layer identified vibrational bands characteristic of calcium sulfate in various hydration states, including gypsum ($CaSO_4 \cdot 2H_2O$), with a main band at 1008 cm^{-1} and anhydrite ($CaSO_4$), showing a characteristic band at 1025 cm^{-1}(Figure 3A)[11,12].These findings are consistent with the use of a gypsum-based ground layer, clearly distinct from the pictorial layer applied over it, an indication of an *a secco* technique. This method involves applying pigments to a dry surface using an organic binder to ensure adhesion[13]. Such technical features agree with results reported for mural paintings in other colonial churches of the Andean region[2–6,14].Furthermore, complementary studies conducted at the University of Buenos Aires by Marta Maier's team, using gas chromatography coupled to mass spectrometry (GC-MS), identified the use of animal glue within the preparatory layer, and egg and plant oils as binders in the pictorial layer[15].

Green paint

SEM-EDS analysis of the green paint layer revealed the presence of copper (Cu) and S, consistent with a basic copper sulphate compound of general formula $Cu_xSO_4(OH)y \cdot zH_2O$. These results are comparable to those observed in previous studies of Andean mural paintings, where antlerite ($Cu_3SO_4(OH)_4$) and brochantite ($Cu_4SO_4(OH)_6$) have been identified in many cases [3–6]. These compounds have been interpreted as natural mineral phases, likely sourced from local deposits, and their presence has been

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associated with the continuity of pre-Hispanic material traditions. However, the Raman spectrum obtained for this sample (Figure 3B) presents a distinct pattern. While the most intense band appears at 975 cm⁻¹, corresponding to the symmetric stretching of the SO_4^{2-} group in brochantite according to some authors [16,17], the relative intensity and position of the remaining bands do not match those of that mineral. Based on the Raman bands observed in the mid-frequency region (366, 398, 420, 484, 610 and 620 cm⁻¹), and their correspondence with those reported for hydrated copper sulfates, the spectral pattern is consistent with either posnjakite ($Cu_4SO_4(OH)_6 \cdot H_2O$) or langite ($Cu_4SO_4(OH)_6 \cdot 2H_2O$) reported by Frost et al.[18–20]. The absence of bands near 606 and 629 cm⁻¹ suggests that brochantite and antlerite are not present as major phases[21]. Notably, weak signals attributable to antlerite were also detected, with its characteristic band at 989 cm⁻¹, indicating its presence as a minor phase, unlike in previous studies where it had been identified as the dominant component[18]. To support this interpretation, Raman analyses were repeated using a different instrument and a 633 nm laser excitation, yielding consistent results (Figure 3C). Although compounds such as antlerite are known to be sensitive to laser power and susceptible to degradation, previous studies have shown that such effects do not produce Raman shifts below 980 cm⁻¹[6]. Additionally, the paint layer appeared extremely thin under optical microscopy, reinforcing the hypothesis that pigments were applied using an *a secco* technique, where the colour is deposited over a dry preparatory layer.

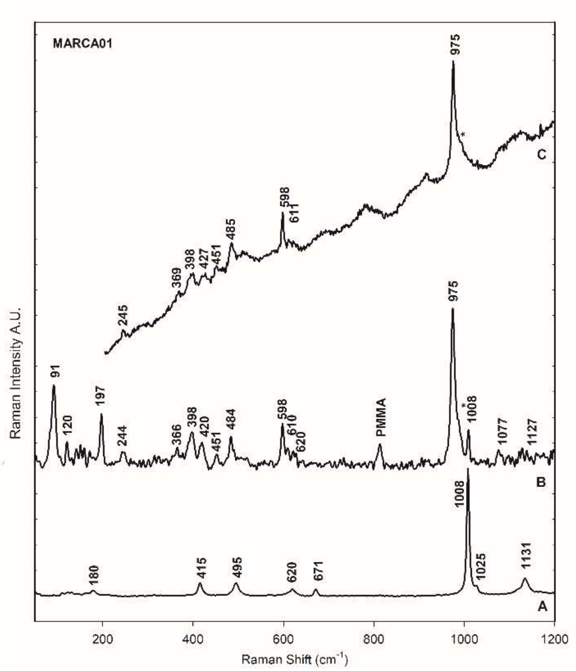

Figure 3. Raman spectra of the cross-section of sample MARCA01: (A) Ground layer – 532 nm, (B) green layer – 532 nm, (C) green layer – 633 nm. The asterisk indicates 989 cm⁻¹ characteristic band of antlerite.

Red paint

SEM-EDS analysis of sample MARCA02, corresponding to a red-painted area, revealed a high concentration of iron (Fe) near the pictorial layer, suggesting the use of an iron oxide-based pigment as the main chromophore (Figure 1S and 2S). This type of pigment is typically found associated with aluminosilicates such as clays, which explains the presence of Si, Al, and potassium (K). Ca and S were also detected and are attributed to the underlying gypsum preparation layer.

The presence of carminic acid, the main component of cochineal dye, was identified in previous organic studies carried out on this same sample[15]. This animal-derived dye is obtained from insects of the genus *Dactylopius*[22] and was widely used during the colonial period for its intense red hue[23]. However, it could not be identified through Raman spectroscopy, as its strong fluorescence under visible excitation prevents the acquisition of clear spectra[24].

The combination of hematite and carminic acid within a single pictorial layer is particularly distinctive and has not been frequently documented in previous studies of Andean mural paintings. This finding suggests the intentional mixing of materials of different nature, both mineral and organic, to achieve specific tones, offering valuable insights into colonial chromatic practices in the region.

Nuestra Señora de la Candelaria de Canincunca

Ground, binders and techniques

Cross-section analysis using optical microscopy, SEM–EDS, and micro-Raman spectroscopy confirmed the presence of a white preparatory layer mainly composed of calcium sulphate phases (Tables 2 and S1). SEM–EDS analyses of all samples from the Canincunca church ground layers confirmed the presence of elements consistent with the $CaSO_4 \cdot xH_2O$ system[11] (Figure 4), in agreement with previous findings and comparable to observations in other Andean churches along the Ruta de la Plata[3–5]. The Raman spectrum of CANI02 (Figure 5) showed bands attributable to gypsum and anhydrite, including a characteristic signal at 1025 cm⁻¹ corresponding to anhydrite type III. Although no organic analysis was performed on these samples, the combination of elemental composition and stratigraphic features observed by OM supports the same interpretation proposed in earlier studies: the use of an *a secco* technique, consistent with practices observed in colonial Andean mural painting.

Red, ochre and orange paint

Elemental analysis by SEM-EDS of samples CANI01 and CANI05 revealed significant Fe content in the pictorial layer, accompanied by Si, Al, K, and magnesium (Mg). These results are consistent with the use of mineral mixtures containing iron oxides and aluminosilicates, commonly found in natural deposits of the Andean region. Raman analysis of sample CANI01 revealed a red pigment with characteristic bands at 226, 294, 407, 493, 611, and

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1325 cm⁻¹,(Figure 5) attributed to natural hematite (α-Fe_2O_3) with a disordered structure[5,25,26], while sample CANI05 exhibited spectral features consistent with goethite (α-FeOOH), confirming its use in ochre tones. These iron oxides, including limonite, goethite, and hematite, have been widely documented in colonial Andean mural paintings[27–29]. Their presence is often associated with natural mixtures containing quartz and clay minerals, which contribute to a variety of tonalities ranging from yellow to dark red and are readily available in Andean geological contexts.

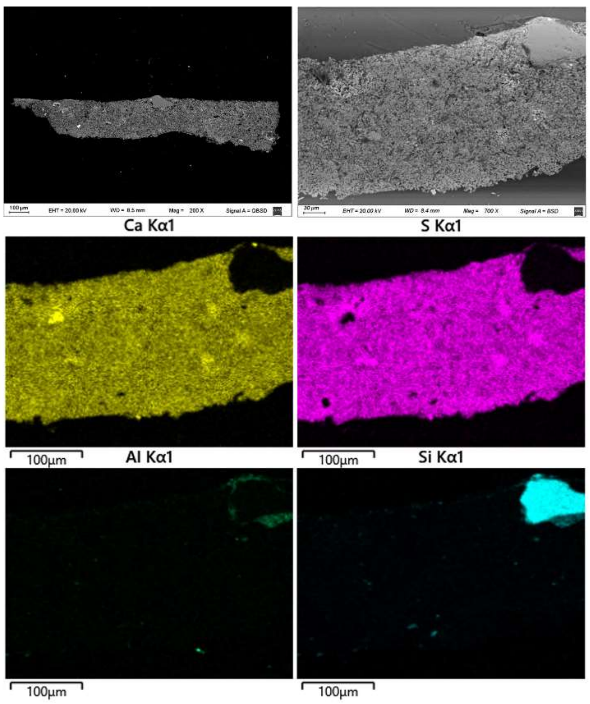

Figure 4. BSE micrograph and SEM–EDS mapping results of the cross-section of sample CANI02.

In contrast, the Raman spectrum of sample CANI07, which corresponds to an intense orange layer, revealed a distinct formulation based on lead compounds. Two lead oxides were identified: minium (Pb_3O_4)[30], with bands at 122 and 551 cm⁻¹, and massicot (PbO)[31], with bands at 144, 290, and 390 cm⁻¹. (Figure 5) These pigments cannot be distinguished by SEM-EDS alone due to their shared elemental composition, highlighting the importance of molecular identification techniques. While compounds such as hematite, goethite, or even vermilion and orpiment have been previously used to obtain orange hues in colonial murals, the combination of lead oxides found here had not been previously identified in murals of similar manufacture. A comparable formulation was documented in a mural behind the altarpiece of the church of Curahuara de Carangas in Bolivia, which revealed distinct pictorial and stylistic features and the same lead-based pigment mixture[32].

Green and blue paint

The analysis of the blue and green samples from the Canincunca mural paintings revealed a diverse use of materials and pigments, both organic and inorganic. Sample CANI02, originally taken from a green area, did not show in SEM-EDS any chromophore elements typically associated with green or blue hues, such as Cu or Fe. However, Raman spectroscopy detected the presence of indigo[33], an organic dye, through its characteristic vibrational bands at 133, 253, 546, 599, 675, 759, 1223, 1311, 1459, and 1573 cm⁻¹ (Figure 5). This finding aligns with previous observations in Andean mural paintings, where indigo was commonly used to produce blue tones in the absence of copper-based pigments like azurite or synthetic pigments such as Prussian blue[3–5]. Indigo, a natural dye derived from the fermentation of plants of the families Papilionaceae, Brassicaceae, and Polygonaceae (notably *Indigofera tinctoria*)[34], was widely used during the colonial period and appears to have been available through both long-distance trade and local production. The detection of indigo in a sample originally *categorised* as green also raises the possibility that its combination with a yellow pigment or binder could have resulted in a green hue, a common strategy in colonial artistic practices. However, in this case, no additional chromophores were identified that could account for the green tonality, making the result particularly intriguing.

In contrast, samples CANI03 and CANI04 displayed under optical microscopy the typical morphology of vitreous blue particles, further confirmed by SEM-EDS analysis showing the presence of Si, K, cobalt (Co), and arsenic (As) (Figure 6). This elemental composition is characteristic of smalt, a cobalt-based blue glass pigment[35]. Though Raman spectroscopy was not conducted on these samples, the combined evidence of particle morphology and elemental composition supports the identification. As commonly reported in colonial mural and easel paintings, the cobalt in smalt gives it its intense blue tone, and the manufacturing process involved melting cobalt oxide with quartz and potash, followed by rapid cooling. Since smalt was not produced locally in the Viceroyalty of Peru, its presence in Andean artworks suggests that it was imported and reflects the circulation of materials through colonial trade networks. The presence of smalt in Canincunca confirms its use in Andean mural painting, as previously documented in churches along the *Ruta de la Plata* in northern Chile. Its identification here reinforces the evidence for the circulation of imported pigments and their integration into local pictorial practices[3].

The use of indigo and smalt in these murals reflects the coexistence of local and imported materials in colonial pigment palettes. While indigo was likely sourced locally or regionally, smalt points to European origins.

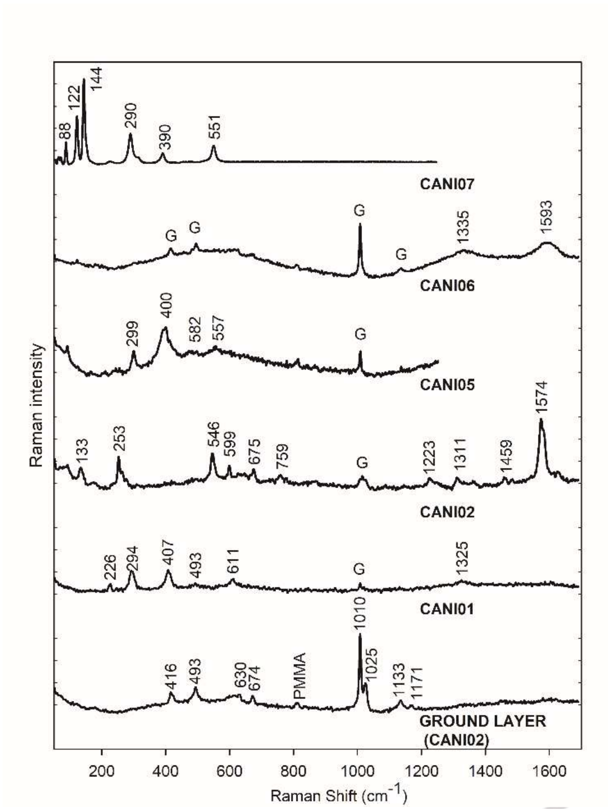

Figure 5. Raman spectra (532 nm) of the cross-sections of samples CANI01, CANI02, CANI05, CANI06, and CANI07. G = Gypsum.

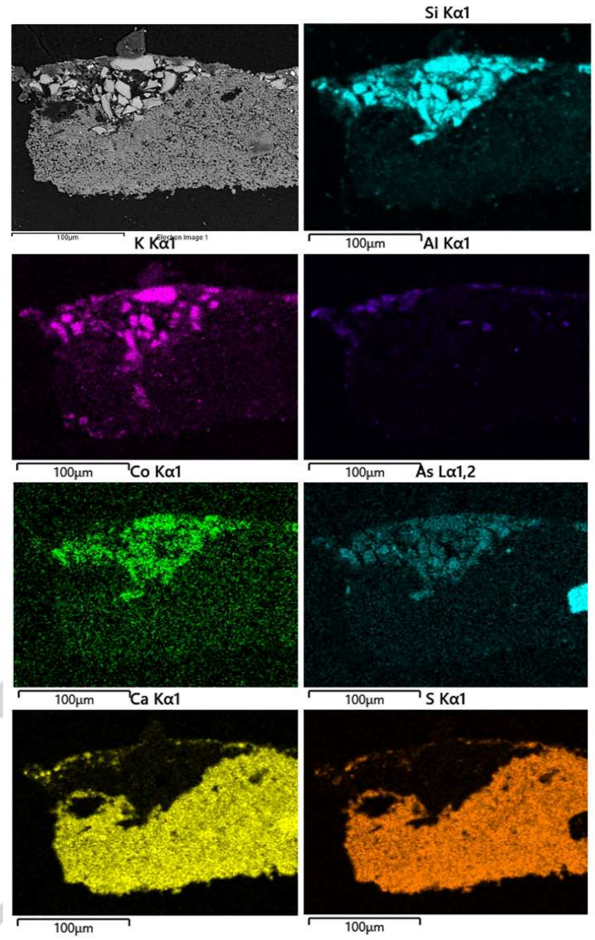

Figure 6. BSE micrograph and SEM–EDS mapping results of the cross-section of sample CANI03.

Black paint

Sample CANI06, corresponding to a black-painted area, showed in SEM-EDS a high carbon content along with minor levels of Ca, S, Al, and Si, and no detectable Fe or Mn. These results point to the use of a carbon-based black pigment. Raman spectroscopy confirmed this identification by showing the characteristic D and G bands of disordered carbon materials at approximately 1335 and 1593 cm^{-1}(Figure 5). This profile is consistent with charcoal-based pigments previously identified in other Andean murals, such as the Copacabana de Andamarca in Bolivia[36] and widely used in colonial painting practices and loaded with symbolic connotations in the visual culture of the period[37,38].

Conclusion

The interdisciplinary analysis of colonial mural paintings using analytical techniques not only enables the identification of historical materials and technologies but also provides valuable insights into manufacturing practices and the circulation of knowledge in the Andean colonial context. The characterisation of copper-based green pigments supports the hypothesis of a persistent technological tradition in the region, rooted in the use of local minerals and their transformation into pictorial materials. Notably, the identification of posnjakite ($Cu_4SO_4(OH)_6 \cdot H_2O$) as a major component in one green sample constitutes a significant finding, as this compound had previously only been reported in trace amounts. This study documented a wide range of pigments in the murals of the churches of Marcapata and Canincunca, including both mineral and organic compounds such as hematite, goethite, carmine, minium, massicot, indigo, and smalt. The detection of smalt in the wall from the church of Canincunca, along with mixtures based on lead oxides to produce intense orange tones, such as the combination of minium and massicot, indicates the integration of European-origin materials through colonial circulation networks.

The results contribute to expanding the understanding of the materiality of Andean wall painting and highlight the importance of approaching it as a material archive. The study of these murals makes it possible to recover information about local technologies, colonial exchanges, and the production and use of colour in the context of colonial artistic production in the Andes.

Supporting Information

The Supporting Information includes complementary data related to the analyses described in the manuscript

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Keywords: Pigments analysis • Mapping SEM-EDS • Raman spectroscopy • Wall painting • Spanish American Colonial

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Table 1. Image of the samples, sampling location, colour and cross-section of the wall of the Church of San Francisco de Asís de Marcapata

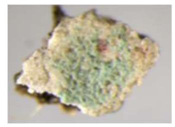
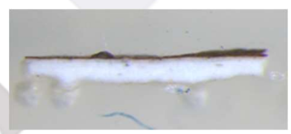
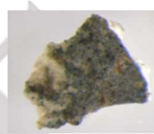
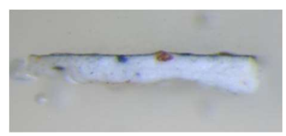
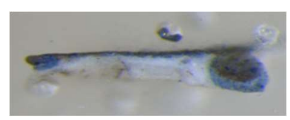
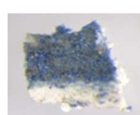
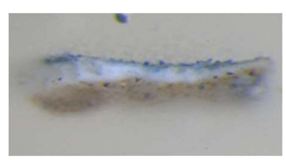
Code	Sample collection	Localization	Colour	Sample	Cross Section
MARCA01		Light green (decoration of the sotto Coro wall)	Green		
MARCA02		dark pink (decoration of the sotto Coro wall)	Dark pink		

Table 2. Image of the samples, sampling location, colour and cross-section of the wall of the Church of Nuestra Señora de la Candelaria de Canincunca.

Code	Sample collection	Localization	Colour	Sample	Cross Section
CANI01		Flower (Mural Decoration)	Dark Pink		
CANI02		leave (Mural Decoration)	Green		
CANI03		Escutcheon edge (Mural Decoration)	Blue		
CANI04		Escutcheon. (Mural Decoration)	Blue		
CANI05		Escutcheon (Mural Decoration)	Ochre		

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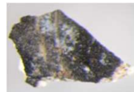
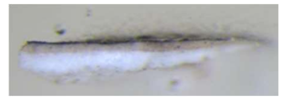
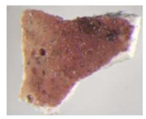
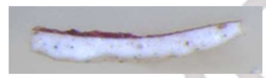
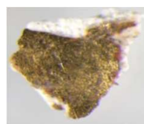
CANI06		phytomorphic human figure (Mural Decoration)	Black		
CANI07		phytomorphic human figure (Mural Decoration)	Orange		
CANI08		Mural damask fabric	Dark pink		
CANI09		Mural damask fabric	Golden		

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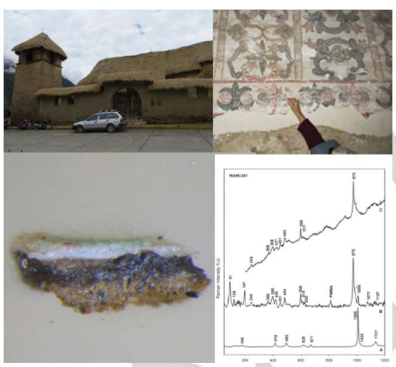

This study identifies pigments and materials in 17th–18th century mural paintings from two churches in the Cuzco region (Peru, Viceroyalty of Peru) using SEM-EDS and Raman spectroscopy. The results reveal both local and imported materials, including the first major identification of posnjakite in colonial South American murals, offering new insights into Andean technological practices and material circulation.