Pigment and Plasterwork Analyses of Nasrid Polychromed Lacework Stucco in the Alhambra (Granada, Spain)

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This paper deals with the description of the stucco (decorative plasterwork) and pigments used in the Alhambra (Granada, southern Spain). The nature, structure and deterioration of these materials are described in detail, as well as the painting and stucco techniques employed. A cast plaster technique is found throughout the Lions Palace (late Nasrid period, second half of the fourteenth century), whereas plaster carved in situ is only found in certain areas of the Partal Palace, one of the oldest constructions still extant in the Alhambra (1302–1309). Two original Nasrid palettes were also identified: an earlier, simpler palette of blue, red and black, and a later, more varied palette of red, blue, green, black and gold. In both cases white was the background colour. The gold decoration technique is described, as are the Christian interventions. Rich pigments were used throughout the history of the Alhambra (e.g. gold, lapis lazuli, azurite, malachite, cinnabar and red lead). Some pigments, red lead and malachite in particular, are severely decayed through introduction of chloride-based materials, resulting in discolorations. Cracking and loss of gold decoration is widespread. The paper gives the first description of the Nasrid palette used to colour the Alhambra stucco, as well as the gilding and the plasterwork techniques. These pioneering results clarify historical and artistic issues.

INTRODUCTION

The Alhambra of Granada is the last and best preserved palace in medieval western Islamic civilization and is a unique monument from an aesthetic point of view. The Alhambra, 'the red castle' (*al-Qal´at al-Hamrā*), was a fortified palatine city whose construction took from the eleventh to the fifteenth century, with the most outstanding palaces built during the Nasrid dynasty (1238–1492). At present, the Nasrid Palaces comprise part of the Partal Palace (Muhammad III, 1302–1309), the Hall of the Mexuar (part of the, now lost, Mexuar Palace of Ismail I, 1314–1325), the world-famous Comares Palace built by Yusuf I (1333–1354) and the Lions Palace constructed by Muhammad V (1362–1391). Within the long tradition of Hispano-Muslim art, the Nasrid period represents the culmination of the evolution of Islamic culture in Europe. The Alhambra was erected on the Sabika hill overlooking what is now the city of Granada (Figure 1). It is surrounded by three concentric walls and includes 23 towers, four gates, seven palaces including a summer palace (the Generalife), a fortress, public and private mosques and spas, a royal cemetery, gardens, quarters, prisons, houses and ateliers [1]. Since the Christian conquest of Granada in 1492, the Alhambra has been subjected to new building (e.g. the Renaissance Palace of Charles V), catastrophe, ruin, restoration and teeming tourism, all definitively altering its original aspect.

The Nasrid period (1238–1492) was the climax of ornamental art in the Alhambra. Only on rare occasions was the sublime beauty of the Alhambra decoration matched or surpassed by later buildings. The basic technique for surface decoration of almost all walls was to use dado tiling at the bottom and cover the upper part of the wall with carved stucco (Figure 2). Stucco is essential to Nasrid art, as it is to most Islamic art. '*Yesería*' is the correct term to indicate the type of wall decoration created with gypsum by the Nasrids. The terms 'gypsum' and '*scagliola*' both refer to the plasterwork containing the mineral gypsum that shapes the Alhambra stucco; they differ in purity of composition and texture. Woodwork is limited to

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Figure 1 General view of the Alhambra and Generalife, a Muslim palatine city erected on the Sabika hill overlooking the city of Granada.

ceilings, doors or architectural elements such as girders and eaves. Stucco and woodwork were polychromed in bright colours [2]. Although the surface decoration of the Alhambra is the most attractive characteristic of the monument, scientific analyses of ornamental materials and polychromy remain limited [3]. Moreover, there is little documentation regarding the Muslim palette and pigments used in the Islamic world, and in particular those concerning Hispano-Muslim polychromy. The few descriptions of Islamic pigments can be found in a Persian codex [4], studies of ceramics [5] and of early Nasrid polychromy (1238–1314) at other buildings in Granada [6, 7]. Systematic analyses to determine the nature of inorganic pigments used in both Muslim and Christian polychrome decorative and structural elements were first performed in the framework of the 1993–1996 project, 'Studies and analyses prior to restoration', conducted by the Alhambra Department of Restoration, led by C. Navarrete-Aguilera. The preliminary results of this pioneering project were presented at the 34th International Symposium on Archaeometry [8].

This paper includes the results of research carried out specifically on the polychrome stucco of different palaces and rooms in the Alhambra. The aims of this study are to characterize the pigments and gypsum support used in the polychromy, to determine their degree of decay and the execution techniques used in the polychromy and stucco. In addition, comparisons can be made between the rooms studied in order to draw historical and archaeological conclusions that help to clarify the confusing chronology regarding the painting of this complex monument. The results have also been compared with data from other Nasrid constructions to correlate decorative principles, and determine the technical evolution of Nasrid polychrome *yeserías*.

Figure 2 Façade of the Comares Palace showing the basic decoration technique used in the Alhambra: dado tiling at the bottom and stucco decoration above, becoming more intricate with height in the muqarnas. Woodwork is restricted to eaves, doors and ceilings. The eaves of the Comares Façade are considered the apotheosis of Hispano-Muslim carpentry.

MATERIALS AND METHODS

The authenticity of stucco polychromy is not always guaranteed, due to frequent rebuilding during the Islamic and Christian epochs and undocumented transfers of stucco work from one palace to another within the Alhambra. Unfortunately, few examples of original Nasrid polychromy on stucco remain in the Alhambra. Many of the paintings were restyled when Christian kings decided to inhabit or visit the Alhambra (e.g. the Catholic kings, Charles V, Philip IV and Philip

V), and remodelled or covered original, deteriorated polychromy with white paint. This paper analyses and compares both polychrome stucco considered by experts to be original Nasrid and those of uncertain chronology. It can be seen that the polychromy covering the stucco was carefully performed even in unreachable areas such as ceilings decorated with *muqarnas* – a honeycomb-like or stalactite decorative motif consisting of numerous niches – (Figure 3a). Moreover, the colour distribution is governed by a symmetrical pattern (Figure 3b) and gold decoration is restricted to the *muqarnas* (Figure 3c). Discoloration, blackening and alteration of pigments is widely found (Figures 3b, 3d and 3e). In the Alhambra, few surfaces were painted black; indeed, this colour was only used to outline the decorative motifs. In addition to this, after six centuries few remains of black are still extant (Figure 3d). Joints between panels of stucco, revealing that moulds were used, are found in diverse rooms (Figure 3f).

Twenty-four samples (Table 1) were taken from the following areas in the Alhambra (Figure 4): three from the east wall of the Hall of the Mexuar (conference room for the Arab rulers) (Mx), three from the façade of the gate of the Mexuar (GMx), ten from the interior wall of the east pavilion in the Lions Courtyard (Lions Palace) (LC) and eight from the upper floor of the González-Pareja House which is one of the three Muslim houses of the Partal Palace (MH). The sampling procedure was guided by:

- location within the monument;
- architectonic or decorative function of the polychrome elements;
- colour observed in the stucco; and
- for a given colour, the different tones that could indicate alteration processes.

The colours studied were white, orange, red, green, blue, black, gold, metallic grey and violet.

A rigorous visual examination of the polychrome elements was conducted in situ to make a preliminary selection of samples to be analysed. Once the samples were selected and taken, the following instrumentation was employed to ascertain the established objectives. A stereo microscope (Olympus SZH10), equipped for microphotography, was used to examine the overall polychrome structure, the pigment application technique and the conservation state. Polished thin sections were then prepared to study paint cross-sections. Pigment mineralogical composition, texture, manufacture, polychrome microstructure, conservation state and painting technique were examined by polarized light microscopy (PLM) in transmitted and reflected light, with the capability of microphotography (Carl Zeiss Jenapol U). Finally, elemental composition and alteration products were identified with scanning electron microscopy (SEM) using a Zeiss DSM950 equipped with a Link energy-dispersive X-ray (EDX) microanalytical system (QX2000). Despite the conventional analytical techniques used to study the materials, the combined results obtained provide a comprehensive picture of their composition and structure.

RESULTS

In view of the aims of this research, the results from both pigment and stucco characterization are organized according to the different rooms studied in the Alhambra, rather than according to pigment composition or plaster characteristics. This facilitates identification of historical interventions, redecoration, and similarities or differences of painting technique.

Hall of the Mexuar (Mexuar Palace)

The Hall of the Mexuar was probably built by Ismail I (1314–1325) but has undergone substantial alterations, either during the reign of Yusuf I and his son Muhammad V or after the Christian conquest. The colours observed in the stucco were white, orange, red, blue, black, violet, metallic grey and gold (Figure 3c). Sampling was carried out in the frieze of *muqarnas* located on the east wall over the 'Plus Ultra' shield, an ornament resulting from the restyling by Emperor Charles V (1500–1558). The following colours were analysed: blue (Mx-b), black (Mx-B) and violet (Mx-v). In all polychromes, the painting support consists of two white layers divided by a thin reddish-orange layer. The inner white layer is approximately 470 μm thick and composed of finely ground gypsum (average grain size 10 μm) as observed by PLM. Calcium carbonate, clays and iron oxides all showing irregular shapes were identified with PLM and SEM-EDX, as well as gypsum crystals larger than the matrix. Using SEM-EDX, strontium sulphate and titanium were also detected – both typical impurities associated with gypsum (hydrated calcium sulphate). The porosity of this layer is low, consisting of small rounded pores averaging 50 μm in diameter. This intensely white, highly pure, fine-grain gypsum is known as '*scagliola*' (a fine layer of plaster mortar used for priming the plasterwork) by the craftsmen in the Alhambra. Next, a thin (15–20 μm) reddish-orange coat was observed.

Figure 3 *The Nasrid Palaces. (a) The* muqarnas *cupola at The Hall of the Abencerrajes (Lions Palace). (b) The Hall of Ambassadors (Comares Palace). (c) The Hall of the Mexuar (Mexuar Palace). Detail of the colour distribution and gold decoration in the arch intrados. (d) Capital of a column at the Lions Palace. (e) Arch intrados in the Lions Palace showing different tones of green as the result of green malachite alteration forming copper chlorides. (f) Detail of unions between stucco panels in the Hall of Ambassadors (Comares Palace).*

Table 1 Samples analysed in the Alhambra Palaces

Palace	Studied area (see Figure 4)	Sample	Surface colour	Elements identified in surface layer	Pigments	Relevant elements in underlying layers
Mexuar	Mexuar Hall	Mx-b	blue	Na, Al, Si, S, Cl, K, Ca, P	natural lapis	Sr
Mexuar	Mexuar Hall	Mx-B	black	S, Ca, Sr, Ti	carbon black	Sr
Mexuar	Mexuar Hall	Mx-v	violet	Al, S, Si, K, Ca, Fe	gypsum	Sr, Hg, Sn, Au
Mexuar	Mexuar Gate	GMx-wv1	white, violet	S, Ca, Si, Al, Fe	gypsum	Sn, Au
Mexuar	Mexuar Gate	GMx-wv2	white, violet	S, Ca, Si, Al, Fe, Sr, Mn	gypsum	Sn, Au, Pb
Mexuar	Mexuar Gate	GMx-bB	blue	Na, Al, Si, S, K, Ca	lapis, artificial?	
			black	Al, Si, S, Ca, Ti, Mn, Fe	Fe oxide	
Lions	EPLC, W wall	LC-W-DE	dark emerald	Cu, Si, Ba, S	malachite	
Lions	EPLC, W wall	LC-W-LE	light emerald	Cu, Cl, Si, Ba, S	malachite	
Lions	EPLC, N wall	LC-N-R	red	Pb, Ca, Cl, Al, Si, K, Ti, Fe	red lead	
Lions	EPLC, E wall	LC-E-LE	light emerald	Cu, Cl, Ca, Sn, As, Al, Si, P, S, Zn, Mg	malachite	
Lions	EPLC, E wall	LC-E-YG	yellow-green	Ca, Si, Al, Fe, Pb, K, Mg	undetermined	
Lions	EPLC, E wall	LC-E-Ib	intense blue	Na, Al, Si, S, K, Ba	artificial lapis	
Lions	EPLC, E wall	LC-E-Db	dark blue	Na, Al, Si, S, K, Ca, Pb	artificial lapis	
Lions	EPLC, E wall	LC-E-v1	violet	S, Ca, Na, Al, Si, K, Fe	gypsum	Sn, Au
Lions	EPLC, E wall	LC-E-v2	violet	S, Ca, Al, Si, Mg	gypsum	Sn, Au
Lions	EPLC, E wall	LC-E-v3	violet	S, Ca, Al, Si, Fe, Mg, Cl	gypsum	Sn, Au
Partal	Muslim House	MH-J-O	orange	Mg, Al, Si, K, Fe, Ti	mud detritus	
Partal	Muslim House	MH-J-R	red	Hg, S	cinnabar	
Partal	Muslim House	MH-J-b	blue	Cu, Ca, Mg, Cl, As, Zn	azurite	
Partal	Muslim House	MH-P-b1	blue	Cu, As, Zn, Ca, Mg, Fe,	azurite + Fe oxide	
Partal	Muslim House	MH-P-b2	blue	Cu, As, Zn, Ca, Mg, Fe, Cl	azurite + Fe oxide	
Partal	Muslim House	MH-M-R	red	Pb	minium?	
Partal	Muslim House	MH-M-b	blue	Cu, Ca, Mg, As, Zn, Fe	azurite	
Partal	Muslim House	MH-M-wB	white	S, Ca	gypsum	
			black	S, Ca, Si, Al, Mg, K, Fe	carbon black	

EPLC = East pavilion Lions Courtyard. All the samples were studied using stereo microscope, PLM and SEM-EDX.

Figure 4 *Floor plan of the Alhambra and Generalife showing the sampling campaign. 1: Hall of the Mexuar (Mexuar Palace); 2: gate of the Mexuar (Mexuar Palace); 3: Lions Courtyard (Lions Palace) and 4: Muslim House (González-Pareja House) of the Partal Palace.*

SEM element analysis identified silicon, aluminium, potassium, iron, calcium and sulphur, suggesting the presence of iron clays, gypsum and calcite. Over this film another white gypsum layer (≈ 100 μm thick) was observed in cross-sections. This is purer than the *scagliola*, but contains strontium as revealed by SEM-EDX. Its porosity is very low and the small crystal sizes of the gypsum (<10 μm diameter) indicate meticulous preparation. This layer corresponds to the base layer of the polychromy.

Using PLM and SEM-EDX, the blue pigment (Mx-b) was identified as natural lapis lazuli, which is a rock composed mostly of the blue silicate mineral lazurite $(Na,Ca)_8(AlSiO_4)_6(SO_4,S,Cl)$, and commonly containing pyrite (FeS_2), calcite ($CaCO_3$) and other minerals. The elements identified by SEM-EDX in the blue crystals were sodium, aluminium, silicon, sulphur, chloride, potassium and calcium. Crystals containing calcium and phosphorus were also detected. In cross-section, coarse crystals (≈ 20 μm) of varying shapes and colours ranging from vivid blue to white were observed, suggesting a natural origin for the pigment. Lapis lazuli was applied in a 40 μm thick layer. The black colour (Mx-B) applied in a very thin layer (<10 μm) was identified as carbon black using SEM-EDX and PLM. Calcium carbonate and gypsum, considered to be contaminants from the underlying gypsum layer, were also identified using both techniques.

The stratigraphic section of the violet sample (Mx-v) consists of a sequence of ten layers (inner to outer) as detected by PLM and SEM-EDX (Figures 5a and 5b):

1 a base layer of gypsum;
2 a brownish organic layer containing scarce red crystals of mercury sulphide;
3 a severely fissured sheet of tin (≈ 20 μm);
4 a brownish organic layer similar to layer 2;
5 fissured and flaked gold leaf (≈ 5 μm);
6 an organic layer (apparently a coat of varnish) containing calcite and clays;
7–10 as layers 3–5.

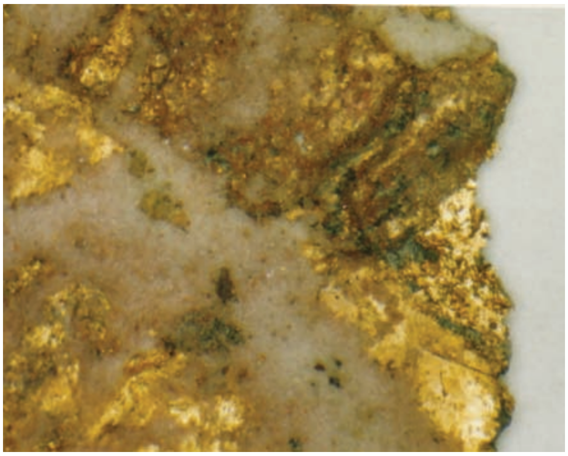
Finally, the above layered sequence is covered by a white layer of finely ground gypsum (crystal size < 10μm in diameter) applied in two coats 120 and 65 μm thick. To the naked eye and using PLM the upper white coat displays a distinctive inhomogeneous violet colour. Rather than being a well-defined paint layer, when viewed in cross-section the violet seems to be a stain within this white coat. Here the elements identified by SEM-EDX were sulphur, calcium, silicon, aluminium, potassium, and iron.

Gate of the Mexuar (Mexuar Palace)

The small doorway that is now the entrance to the Hall of the Mexuar was opened in modern times and the door brought from elsewhere. The mixed results obtained from the samples studied corroborate the theory of stucco replacements of diverse origins. Little polychromy remains in this façade, and a film of red dust further inhibits appreciation of the true colours. The colours studied were white, violet (GMx-wv1 and GMx-wv2), blue and black (GMx-bB). The *scagliola* is similar to that described in the Hall of the Mexuar. Nevertheless, some differences in texture are probably due to the outdoor location, rather than the fabrication process itself. Examination of the outer façade of the gate by PLM showed higher porosity with larger pores (≈ 140 μm) in the *scagliola*, some of which connect with each other, at times leading to fissure formation. The inner walls of the pores are covered with acicular gypsum crystals. In the gypsum matrix, calcium carbonate and iron oxide grains (80–140 μm) were identified, as well as gypsum crystals (≈ 900 μm) showing dissolution borders.

The black and blue colours (G-Mx-bB) each consist of a very thin layer (≈ 10 μm) applied on the layer of base gypsum, as shown by study of the cross-section. For the blue colour, SEM microanalysis identified elements found in lapis lazuli such as sodium, aluminium, silicon, sulphur, potassium and calcium. The small crystal size

(a)



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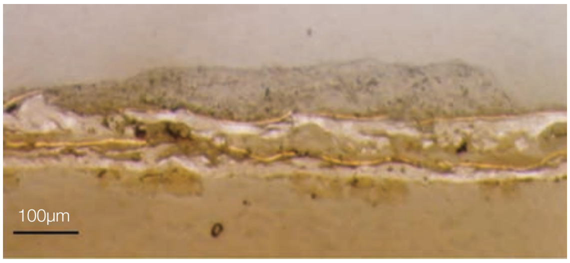


Figure 5 *Sample taken from the Hall of the Mexuar. (a) General view through stereo microscope (2.5×) of a fragment containing gold decoration. (b) Photomicrograph showing the stratigraphic section of this sample. From in to out (from bottom to top in the figure) the sequence of layers is: gypsum base, organic coat, tin sheet, organic coat, gold leaf, organic coat, tin sheet, organic coat, gold leaf, organic coat and finally a gypsum layer displaying a distinctive purple colour in the upper part.*

(<10 μm) indicates intense grinding of the pigment and explains the low intensity of this colour in the polychromy; it might also suggest a synthetic origin. SEM elemental analysis of the black pigment detected iron, magnesium, manganese and titanium, suggesting the presence of iron oxide black. Regarding the violet colour, study of the cross-sections of the two samples revealed differences in the layer sequences. Optical microscopy showed that GMx-wv1 is the only sample taken from the gate where the reddish-orange layer of clays appears above the *scagliola*. In the base gypsum layer, strontium, silicon, aluminium, iron and manganese were identified using SEM-EDX. PLM and SEM-EDX showed differences between sample GMx-wv2 and the equivalent painting stratigraphy in the Hall of the Mexuar. The gypsum base was not observed, but isolated 30 μm crystals of lead oxide (Pb_3O_4) were found directly on the surface of the *scagliola*. On the basis of morphology and size dimensions (grain size ≈ 10 μm) this lead oxide is considered to be minium rather than artificial red lead. The sheets of tin and gold are more severely decayed than in the Hall of the Mexuar. In fact, the intense fissuring and blackening affecting the two tin layers has caused flaking and detachment visible using PLM and by the naked eye. Fissuring also affects the gold leaf, indeed most of the gold has been lost from the polychromy. The outer white layer of gypsum was applied in three coats. Here porosity is higher than in the gypsum base layer with connected pores that result in fissuring. In addition the gypsum crystals are larger, more heterogeneous in shape and show arrowhead twins, all of which indicates that dissolution–recrystallization processes are operating in the surface of the gypsum layer.

East pavilion of the Lions Courtyard (Lions Palace)

Two small pavilions jut onto the east and west sides of this courtyard, each housing a small fountain (Figure 6a). The courtyard has undergone numerous interventions and remodellings that have altered its original appearance, in particular the pavilions. The following colours were observed in the stucco covering the interior walls of the east pavilion: white, orange, red, different tones of green and blue, violet, metallic grey and gold (Figure 6b). In order to establish whether the tonalities are due to artistic intention or caused by alteration processes, several of these tones were analysed by SEM-EDX, including the most intense ones considered to be original, unaltered pigments. Isolated violet patches with different tints are observed in the *muqarnas* in association with sheets of tin. Samples were taken from the north, east and west walls of the interior of this pavilion. The SEM microanalysis of the *scagliola* shaping the stucco did not detect strontium. Examination by PLM revealed that grain sizes (≈ 140 × 375 μm) of the impurities such as clays, iron oxides and calcium carbonate were larger than in the rest of the *scagliola* studied.

On the west wall, two tones of green were sampled from the Kufic script (angular calligraphy used by early

(a)

(b)

Figure 6 *(a) The Lions Courtyard (Lions Palace) and the east pavilion (sampled) seen from the west pavilion. (b) Detail of the sampled area of* muqarnas. *Note the advanced deterioration of the remaining polychromy, particularly the violet spots that appear in localized zones where gilding was identified, and the emerald colour of the original malachite green.*

Muslims): dark emerald (LC-W-DE) and light emerald (LC-W-LE). The pigments of the dark green layer (50 μm thick) were applied directly over the reddish-orange layer of clays (Figures 7a and 7b). Anisotropic light bluish-green crystals of different sizes (5–50 μm) and angular shapes were observed with PLM and identified as malachite ($CuCO_3{\cdot}Cu(OH)_2$). SEM-EDX also detected barium sulphate (barite) and silicon (quartz), corresponding to extender minerals added to give more body to the paint layer. The light green layer (10 μm thick) was composed of opaque green crystals recognized as malachite under transmitted light using uncrossed polars (Figures 8a and 8b). Barium sulphate and copper chlorides were also identified by SEM-EDX.

A vivid red colour from the epigraphic frieze on the north wall was studied (LC-N-R). The cross-section of this paint shows two orange-reddish layers totalling 30 μm thick. PLM and SEM-EDX analyses revealed

(a) (b)

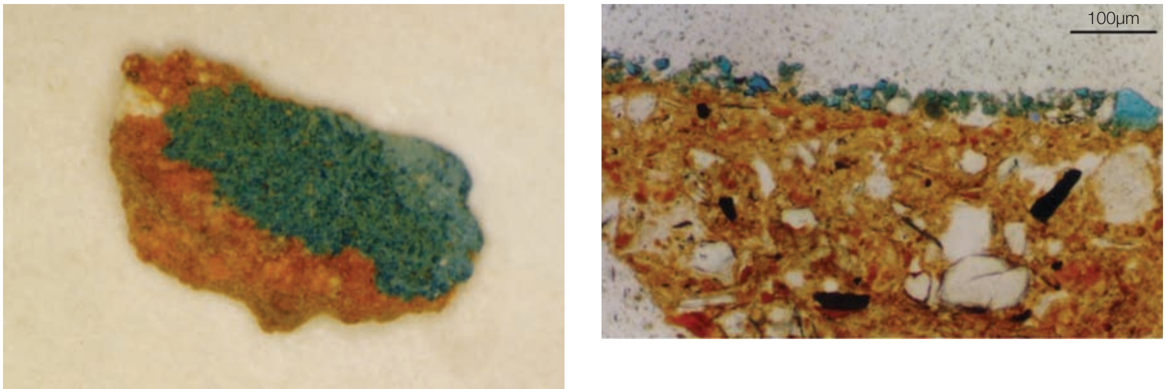


Figure 7 Sample from the Lions Courtyard, east pavilion. (a) General view under stereo microscope (3×) of the dark emerald green sample (LC-W-DE); (b) Photomicrograph of stratigraphic section. Large malachite crystals were applied over a reddish-orange clay layer. Extenders of barite and quartz were identified in the green polychromy.

(a) (b)

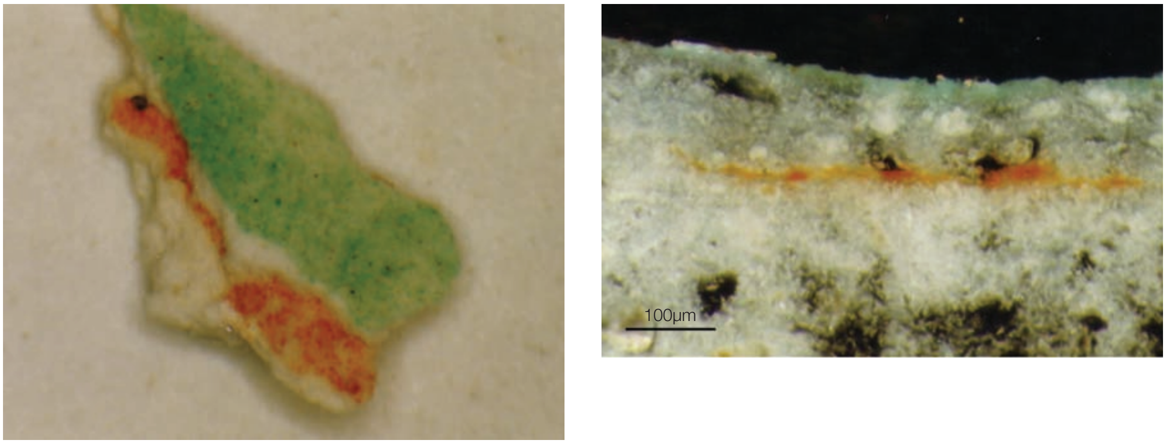


Figure 8 Sample from the Lions Courtyard, east pavilion. (a) General view under stereo microscope (3×) of the light emerald green sample (LC-W-LE). (b) Photomicrograph of stratigraphic section. Green pigments are opaque under uncrossed polars. Malachite, fast white and copper chlorides were identified. Notice the reddish-orange layer between the scagliola and the gypsum base indicating that moulds were used in the manufacture of the stucco.

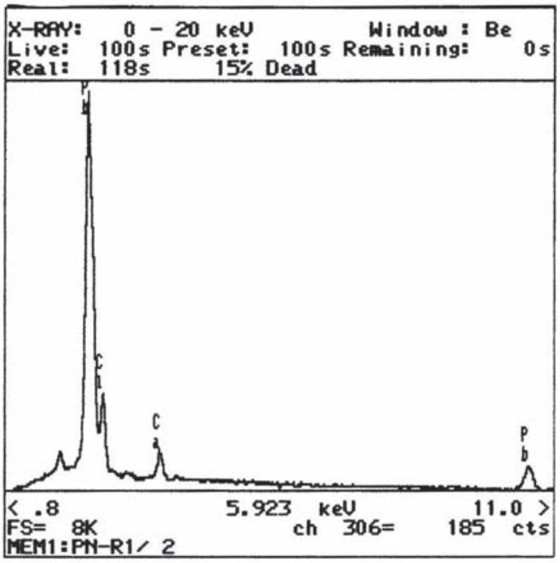


Figure 9 SEM-EDX spectrum showing the elements identified in the acicular crystals growing perpendicular to the red paint layer (red lead) in the east pavilion of the Lions Courtyard (Lions Palace).

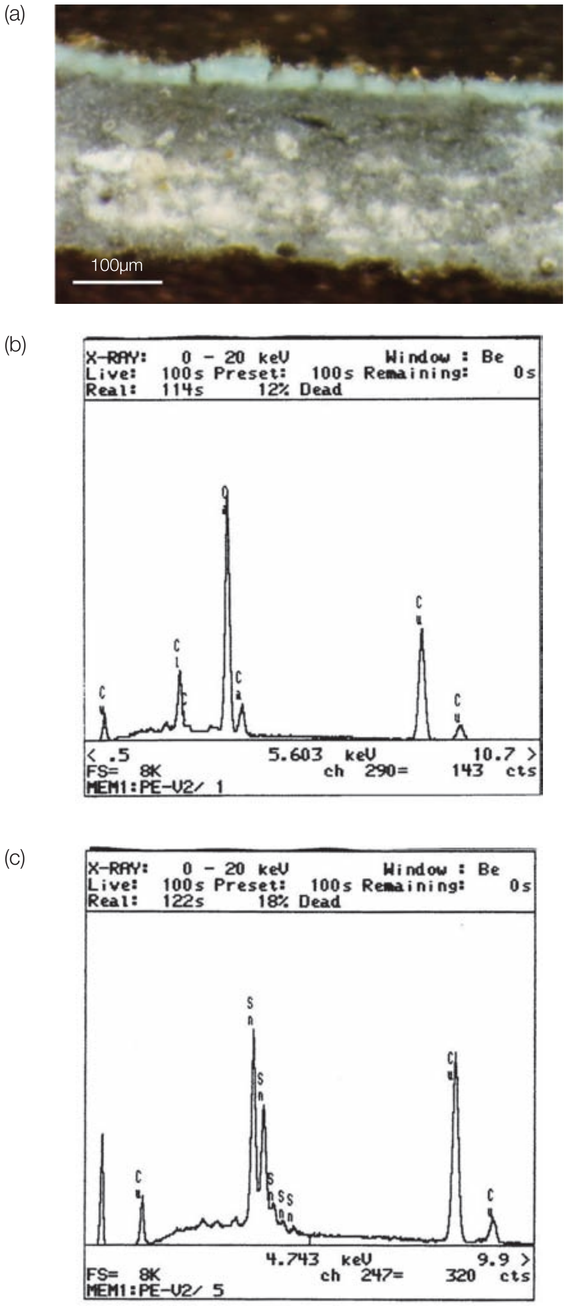


Figure 10 Sample from the Lions Courtyard, east pavilion. (a) Photomicrograph of stratigraphic section of the light emerald sample (LC-W-LE) taken from the Kufic script. Note the vertical fissures that result in flaking. (b) SEM-EDX spectrum of chloride, copper and calcium identified in some grains in the above green layer. (c) SEM-EDX spectrum of unaltered grains composed of tin and copper identified in the previous sample.

the presence of small red and white lead crystals (<10 µm) that were considered to be red lead and basic lead carbonate respectively. Since red lead (artificial minium) is produced by roasting white lead ($2PbCO_3{\cdot}Pb(OH)_2$), the coexistence of these crystals and their particle sizes suggest that red lead (Pb_3O_4) was the pigment used. Calcium carbonate, laminated crystals of biotite and chlorite, and acicular crystals containing lead, calcium and chloride ions (Figure 9) were also identified. The acicular crystals grow perpendicular to the painting surface, indicating their secondary origin.

Seven samples were studied from the east wall. A light emerald (LC-E-LE) and a yellowish-green (LC-E-YG) colour were taken from the Kufic inscription. Under the polarized light microscope the emerald layer (≈ 20 µm thick) shows intense physical and chemical alteration. It is crossed by numerous fissures and the layer appears to be forming flakes (Figure 10a). PLM and SEM-EDX analysis revealed the presence of calcium carbonate, white lead and gypsum. Basic copper chlorides and mixed calcium-copper chlorides (Figure 10b) were identified using SEM-EDX, as well as unaltered tin spots (tin metal) with trace amounts of copper (Figure 10c). In the yellowish-green layer a few green pigments were found. The SEM-EDX did not identify copper, but instead revealed the presence of abundant lead and potassium, and lesser amounts of iron, silicon, aluminium and magnesium, which could correspond to white lead and dust particles. Barite and white lead were identified by PLM and SEM-EDX in the base layer.

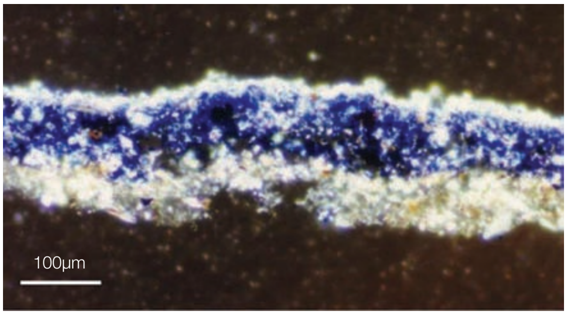
Two blue colours were studied. The first was an intense blue (LC-E-Ib) consisting of a layer ranging from 75 to 100 μm in thickness, as shown in Figure 11a. SEM analysis revealed that the blue pigment is made of rounded fine-grained crystals (≈ 10 μm) identified as lapis lazuli including barite. The particle size and morphology of the blue pigment and the presence of barite indicate an artificial origin for the lapis lazuli. The dark blue colour was taken from the *muqarnas* (LC-E-Db). SEM-EDX revealed that the lapis lazuli pigment was applied as a very thin layer (≈ 15 μm) over a thin layer of white lead (Figure 11b). PLM showed rounded anisotropic crystals of homogeneous sizes (≈ 10 μm), suggesting the artificial origin of the lapis lazuli. Potassium, iron and lead (contamination from the lead-based base layer) were also identified by SEM-EDX.

Three samples of violet were analysed (LC-E-v1, LC-E-v2 and LC-E-v3). Their paint cross-sections are similar to those studied in the Mexuar Palace. From inner to outer layers, the painting section is as follows: organic layer, tin sheet, organic layer, gold leaf, varnish, tin sheet and on top gold leaf (both tin and gold sheets are quite altered showing fissures, flakes and detachments), and finally the white layer of gypsum displaying a non-homogeneous violet tint. Here chlorides were detected by SEM-EDX. The optical microscope showed large pores (≈ 185 μm in diameter) with inner walls sealed by secondary gypsum.

Muslim House of the Partal Palace

The Partal Palace was almost certainly built by Muhammad III (1302–1309). In its day, this area was one of elegant houses surrounded by beautiful gardens, a mosque, palaces, streets, pools and so on. Several Muslim houses are adjacent to the Tower of the Ladies (Figure 12). The polychrome stucco studied in the so-called González-Pareja House is darkening due to a coating of dust and other particles related to domestic heating. The palette is more limited that in the palaces described above: white, orange, red, blue and black. To establish whether pigment composition on ornamental elements varies with architectural function, samples were taken

(a)



(b)

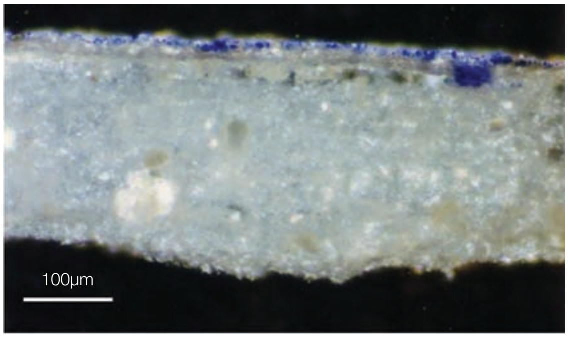


Figure 11 *Sample from the Lions Courtyard, east pavilion (Lions Palace). (a) Photomicrograph of stratigraphic section of the intense blue sample (LC-E-Ib). Observe the thickness of the layer of about 100 μm made of ultramarine. Barite was identified particularly in the upper part. (b) Photomicrograph of stratigraphic section of the dark blue sample (LC-E-Db). Ultramarine with crystal size <10 μm was applied in a very thin layer.*

Figure 12 *The open portico of the Tower of the Ladies in the Partal Palace, with the adjacent Muslim houses.*

from the *muqarnas* and around the windows from the jambs and the pilasters. The *scagliola* of the stucco shows small differences in the three architectonic elements. The SEM microanalysis did not identify strontium (related to celestine) in any of them. PLM shows that in the jambs the *scagliola* is composed of white, homogeneous, well-ground gypsum (<10 μm in diameter) containing occasional calcium carbonate and iron oxide nodules. Its porosity is very low. Next was a thin reddish-orange layer of clays in which SEM-EDX detected copper, iron, magnesium, manganese and titanium. Finally a coarse-ground layer (≈ 140 μm thick) of gypsum was observed with very low porosity and no calcium carbonate. The *scagliola* from the pilasters and *muqarnas* shows a distinctive pinkish colour due to a higher amount of iron oxides. PLM and SEM-EDX showed that the *scagliola* consists of a matrix of coarse-grained gypsum and calcite with embedded large gypsum and calcite crystals (≈ 90 μm in diameter). Pores are scarce and elongated (≈ 18 × 45 μm). Then follows, not the orange clay layer, but instead a layer of gypsum (≈ 280 μm thick) with pores over 100 μm in size.

Regarding the paint layers, SEM microanalysis showed that in the jambs the orange colour (MH-J-O) corresponds to the clay-rich orange layer exposed here. Mercury sulphide was used to provide the red colour (MH-J-R). The optical microscope showed that particle size ranges from 10 to 25 μm in diameter with mainly elongated and irregular shapes, suggesting that natural cinnabar was used. In cross-section the red layer, ranging from 15 to 45 μm thick, is intensely decayed with blackening and numerous fissures and flakes, also visible to the naked eye. PLM and SEM-EDX identified the blue colour (MH-J-b) as azurite ($2CuCO_3 \cdot Cu(OH)_2$) mixed with calcium-magnesium carbonate grains. It was not possible to distinguish between dolomite and huntite given the amount of sample available and the instrumentation used. However, huntite outcrops are not found locally, whereas dolomite deposits are very common. Azurite crystal sizes vary from 10 to 45 μm and show irregular shapes. SEM microanalysis also identified calcium, magnesium, chloride, arsenic and zinc in this layer; these last two are natural impurities normally associated with copper carbonates. Regarding the calcium-magnesium carbonate, it is a common extender mineral added to blue paint layers to give them volume, since the medium-to-low covering power of blue pigments requires application in thick layers.

Two samples of blue from the pilasters were studied (MH-P-b1 and MH-P-b2). In both cases the optical microscopy and SEM microanalyses identified the blue pigment as azurite applied in a layer 100 μm thick. Azurite crystals of irregular shape were on average ≈ 10 μm in size. Calcium-magnesium carbonate (extender), isolated red pigments of iron oxides, green crystals of copper chloride, arsenic and zinc were also identified using PLM and SEM-EDX.

In the *muqarnas,* SEM microanalysis identified the red colour (MH-M-R) to consist of lead oxide with approximately 10 μm grain size, applied in a layer 25 μm thick. PLM shows the blue paint layer to be similar to the above-mentioned blue layers on the jambs and pilasters. The blue layer is around 300 μm thick and shows an excellent state of conservation. Chlorides were not identified with SEM-EDX. The white colour (MH-M-wB) was found to be calcium sulphate using SEM-EDX. PLM and SEM microanalysis determined the very thin (<10 μm) layer of intense black colour (MH-M-wB) to be carbon black with particles about 5 μm in size. Other elements identified with SEM-EDX were silicon, aluminium, magnesium, potassium and iron.

DISCUSSION

In the Alhambra, two plaster techniques have been identified based on stylistic studies [9]. During the first Nasrid period (1238–1314), stucco was sketched on smooth panels, a design was incised, and then carved in situ using chisels while the gypsum blocks were still damp. Later, a plaster cast technique was used where stucco was cast in plaster of Paris, clay or wooden moulds (carved in negative) and afterwards placed on the walls.

However, until now, compositional and textural studies had not been carried out to corroborate this supposition. It makes sense that in the late Nasrid period working methods became systematized, using casts to produce more complicated decorative motifs than before; the Kufic and cursive epigraphs with geometric and vegetal motifs are more time-consuming in their execution (Figure 3f).

By determining the characteristics of the gypsum used in the preparation of the stucco, the two plaster-work techniques described above could be recognized. An indication of the casting technique is given by the presence of rounded pores, which suggests that the casts were manufactured on a horizontal rather than a vertical plane, since the exertion of force has a tendency to produce elongated pores. A second distinguishing trait is the small gypsum particle size (<10 μm), a requisite for the gypsum to enter and fill all cavities in the cast. It has been shown that best quality gypsum was used to model the stucco. This well-ground gypsum of deep white colour and very pure composition is termed '*scagliola*' in the Alhambra. References from the Archive of the Alhambra specify the use of selenite for the manufacture of the stucco. Finally, the reddish-orange layer identified on the surface of the *scagliola* must be the result of mud remains from the casts used in the plasterwork. Therefore, the presence of this layer may be used as a chronological indicator to distinguish different periods of building. As a consequence, the only *yesería* of this study that can be assigned to an early Nasrid period are those of the *muqarnas* from the Muslim House of the Partal Palace and some pieces placed in the gate of the Mexuar, as suggested by the absence of the orange layer, the coarse-grained, dark-coloured gypsum, and the elongated pore shapes, all characterizing an in situ stucco carving. This is consistent with the references ascribing the Palace of the Partal to Muhammad III (1302–1309) and thus to the first Nasrid period [1, 10].

In the case of the cast plasterwork, once the stucco was placed on the wall, one or more layers of fine gypsum were applied to the whole panel to hide plate joints and imperfections and to provide a uniform bright white base upon which the polychromy could be applied. These white layers correspond to the base layer of the polychromy. Regarding the stucco composition, the presence of calcite and celestine indicates raw material origin and stucco manufacture, but cannot be used for dating purposes. The presence of celestine suggests that the gypsum originates from an area near the city of Granada (i.e. from celestine outcrops in the Granada basin), as indicated by the Hispano-Muslim gypsum mortars in the Alhambra containing high strontium levels [11]. The calcium carbonate may be either a natural impurity of the gypsum or an extender mineral intentionally added to lighten the gypsum or to make it more resistant to moisture. The stuccos containing barite (*blanc fixe*), a pigment introduced in the market from the beginning or middle of the nineteenth century in France [12–14], are not authentic Nasrid art.

Regarding the pigments, two reds were identified in the polychromes studied: mercury sulphide and lead oxide. Mercury sulphide was only found in the window jambs of the Muslim House (Partal Palace) and in the Hall of the Mexuar under the gilding. In the Muslim House the morphology of the red pigment suggests that natural cinnabar was used, though lack of evidence in the Mexuar makes it impossible to suggest the origin of the pigment. Lead oxide was found in the *muqarnas* of the Muslim House, the gate of the Mexuar and the Lions Palace. In the last, the morphology of the crystals together with the presence of white lead grains suggests a synthetic origin for the lead oxide (i.e. red lead). By contrast, on the basis of the textural results obtained by optical microscopy, the nature of the lead oxide in the Mexuar and Muslim House cannot be readily specified.

As can be seen, it is difficult to draw conclusions regarding the attribution of each red pigment to a particular art period, since they have been used indiscriminately from the early Nasrid epoch to the most recent interventions of the nineteenth and twentieth centuries. Unlike the results of the polychrome stucco from other constructions dating from the first Nasrid period [7], iron oxide red pigments were not found in the Alhambra. The high instability of lead oxide and mercury sulphide to light and humidity may explain the advanced state of decay [15]. In general the red colour is lost from the polychromy, and what remains either lacks intensity or is darkening and shows fissures.

Three kinds of blue pigment were identified, namely natural azurite in the Muslim House of the Partal Palace, and natural and artificial lapis lazuli in the rest of the areas studied. As in other early Nasrid polychromes characterized in Granada, iron oxide was identified along with azurite. Considering the above attribution of the Muslim houses located in the Partal Palace to Muhammad III (1302–1309), and the fact that azurite was not found in the rest of the palaces studied, it might be inferred that azurite was the blue pigment used in the Alhambra during the first Nasrid period. While it is tempting to rationalize the absence of lapis lazuli in the Muslim House in terms of cost, hypothesizing that expensive materials were reserved for the most magnificent palaces in the Alhambra, it must be noted that an adjacent house (the so-called 'House of the paintings') displays one of the most outstanding Muslim mural paintings made of rich pigments including gold [8]. Lapis lazuli, as expensive as gold, was principally used in miniatures, frescos and canvas painting, and always in rich decorations. Its use in decorative architecture was restricted to the most prestigious palaces [14], such as the Alhambra complex.

Both natural and artificial lapis lazuli are the best conserved of all pigments in the polychromes in the Alhambra. In the Mexuar Palace (hall and gate) natural lapis lazuli was found as deduced from the textural characteristics of pigment. In contrast, artificial lapis lazuli was used in the Lions Courtyard, as inferred both by morphological characteristics and the use of barite. Here barite was extensively found as extender, not only in the blue layers made with lapis lazuli, but also in the green layers made of malachite. This result is consistent with the references concerning the numerous interventions performed here during the ninth and the twelfth centuries [16].

The only green pigment found was malachite, which was the most altered of all the pigments studied. Different tonalities of green were observed. In the resulting tones two factors have been considered of key importance. The first is the underlying white or reddish layer that influences the final luminosity of the green colour in the surface. It is known that a white base reflects incident light contributing to brightness of the surface paint layer, whereas a dark base reduces reflection and darkens the surface colour [12, 17]. In the course of this research it was found that malachite was applied in some cases over the white base layer of gypsum and in other cases directly over the reddish-orange layer of clays.

The second factor, which has nothing to do with artistic design, is the severe alteration observed in some green layers, where the original dark green malachite colour progressively discolours to the typical emerald colours of the copper chlorides, as apparent to the naked eye and widely observed in the polychromes (Figure 3e) [18, 19]. Lack of homogeneity in the emerald colours is observed by visual inspection as well. Moreover, it was evident to the touch that the pigment hardness varied from solid green crystals to a powdery dull green mass. PLM showed changes in crystal sizes from 30 μm in the unaltered malachite pigments to approximately 5 μm in the copper chlorides. In addition, the latter do not form a well-defined paint layer in cross-sections. These characteristics (morphology and texture of the green grains) observed by optical microscopy, and the fact that only malachite was found in some parts of the polychromes, and chlorides were identified in other pigments besides green (i.e. red and white), support the hypothesis that malachite was the green pigment used by the Nasrids. Therefore copper chlorides must be products of alteration. In fact, in the Lions Courtyard mixed copper-lead chlorides, calcium-lead chlorides and lead chloride-phosphate minerals were identified by SEM-EDX in the red and green layers, which indicate that chloride might have reacted with the paint layers containing gypsum and lead, thus causing changes in these colours. The above minerals, though infrequent in nature, have been identified as corrosion products in bronze objects [18, 20]. Mixed chrome-lead chlorides and lead chlorides have also been reported as alteration products in mural paintings [17].

Two black pigments were identified – carbon black and iron oxide black. The absence of phosphorus allows the use of bone ash to be excluded, but further analyses should be performed to establish whether coal or soot was used instead. Regarding the superimposed white layer that covers vast areas of the polychrome stucco, its application could be justified aesthetically, to mask the deterioration or the partial lack of the original polychromy. Similar to other findings regarding stucco from the early Nasrid period in Granada [7], the composition of this layer has been identified as gypsum (note that this is not 'whitewash') [21, 22]. In the Alhambra, the white layers display a distinctive violet colour in their outer part, a characteristic that is not mentioned elsewhere [7]. The violet shows neither a painting pattern nor a regular tonality (Figure 6b), and it appears in localized damp areas where tin and gold sheets were detected, thus suggesting that the violet is not a pigment and that it could be related to the alteration of those metallic sheets. The lack of gold decoration in other Nasrid stucco might explain why the violet colour is not observed [7]. Elsewhere, a similar violet colour associated with altered tin sheets has been detected in murals in the Monastery of Saint Jerome in Granada [17]. Despite the fact that the SEM-EDX microanalyses found localized areas of iron-rich clays and manganese (a metallic element that may cause this colour), haematite (ferric oxide) was never identified. It is well known that haematite, physically associated with clays, can display a distinct violet tint in some cases (i.e. highly crystalline haematite with crystal size of about 1 μm) [23]. Further analyses of the violet colour will be necessary to suggest a reasonable hypothesis regarding its origin.

Many records indicate that the interior decoration of the Alhambra (woodwork, stucco, tiling, etc.) included bright polychromes and gilding to give opulence to the walls [24–26]. Unlike other palaces in Granada from the first Nasrid period [7], in the Alhambra gilding was found in the *yesería*s of all the palaces, excluding the Muslim House of the Partal Palace. Specifically, in the Alhambra, gold was used to decorate *muqarnas* and the arch intrados. Regarding the gold technique, the disposition of gold leaf over a tin sheet may have both practical and aesthetic motivations. In practical terms, a sheet of gold is so thin (<10 μm) that it required very experienced handling in order to avoid wasting this expensive material. The intricate tracery and ornaments of the stucco, as well as the height at which they are placed demanded a technique to facilitate their gilding. Therefore, the gold leaf was first applied to a tin sheet, a support that is more flexible, consistent and easier to handle. In terms of aesthetics, the metallic brightness of the gold is enhanced over another metallic surface compared to a gypsum base. Cennino

Cennini describes this technique in his *Il libro dell'arte* [27]. As to why the gold decoration was applied twice, the PLM and SEM-EDX results have revealed the severe deterioration of the first gilding, where fissures, flakes, detachments and loss could be widely observed, justifying the second gilding which in turn is also extensively altered. The similarity between the two gold decoration techniques suggests that either the second gilding was applied not long after the first, or that the technique was well documented and transferred among artists.

CONCLUSIONS

The results of this research are of particular interest because this is the first study of its kind to ascertain the nature and characteristics of the inorganic pigments and carved stucco of Nasrid art in the Alhambra. The following conclusions can be drawn:

- The fresh plaster technique (plaster carved in situ) was found in localized areas in the Partal Palace (Muslim House) and the gate of the Mexuar. The plaster cast technique was widely identified in the Mexuar Palace and the Lions Palace, thus suggesting that the Muslim House corresponds to an early Nasrid decoration period in the Alhambra, and that the two other palaces were decorated during the later Nasrid period.
- Two Nasrid palettes were identified in the Alhambra. The simpler palette consisting of blue (azurite), red (cinnabar and red lead) and black (carbon), was identified in the Partal Palace (Muslim House) and attributed to the first Nasrid period (Muhammad III, 1302–1309), in agreement with the plasterwork results. The second, more ample palette also consists of a limited range of colours: red cinnabar and lead oxide, green malachite, blue lapis lazuli, carbon black and gold (over tin leaf). This palette appears in the *yesería*s located in the Mexuar Palace and the Lions Palace, which were both decorated during the late Nasrid period and might be assigned to Muhammad V (1362–1391).
- The pigment composition results obtained in the gate of the Mexuar confirm that replacements from diverse unknown origins took place.
- Christian interventions were identified thanks to the pigment manufacture (morphology and composition), principally in the Lions Courtyard (Lions Palace).
- The use of expensive pigments such as lapis lazuli, gold, cinnabar, azurite, malachite and tin by Nasrids and Christians confirms the high esteem in which the Alhambra has been held throughout time.
- The gold decoration technique was widely used by Nasrid artists on *yesería*s in the Alhambra, unlike other Nasrid palaces in Granada.
- Discolouring of vivid colours in original pigments (e.g. red and green) is due to salt crystallization of chloride-based minerals. However, efflorescences are not observed in the painted surfaces.
- Pigment composition on ornamental elements varied with architectural function.

The results of this study have implications regarding the perception of the original colours as well as for future cleaning and conservation interventions. Future research should focus on the identification of the binding media and comparison of pigments and painting techniques on the various supports including woodwork, murals and marble.

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***Résumé* — Cet article décrit les stucs sculptés et les pigments colorés utilisés à l'Alhambra de Grenade (Espagne). On décrit en détail la nature, la structure, et la détérioration de ces matériaux, ainsi que les techniques de fabrication des stucs et de peinture employées. La technique de moulage est observée partout dans le Palais des Lions (période Nasrid tardive, 2ème moitié du XIVe siècle), alors que les plâtres sculptés in situ ne se trouvent que dans certaines parties du palais Partal, une des plus anciennes constructions existant encore dans l'Alhambra (1302-1309). Deux palettes Nasrid originales ont également été identifiées : une plus ancienne, palette simple de bleu, rouge, et noir, et une autre plus récente et plus variée, de rouge, bleu, vert, noir, et or. Dans les deux cas le blanc est la couleur de fond. La technique de décoration à l'or est décrite, ainsi que les interventions de l'époque chrétienne. De riches pigments ont été utilisés tout au long de l'histoire de l'Alhambra (par exemple l'or, le lapis-lazuli, l'azurite, la malachite, le cinabre, le minium). Quelques pigments, notamment le minium et la malachite, sont très dégradés en raison de l'usage de minéraux à base de chlorures, provoquant des décolorations. Les craquelures et les pertes de dorure sont généralisées. L'article fournit la description de la palette Nasrid utilisée pour la coloration des stucs de l'Alhambra, ainsi que les techniques de dorure et de moulage. Ces résultats novateurs clarifient certaines questions historiques et artistiques.*

***Zusammenfassung* — In dieser Arbeit werden die geschnitzten Stuckornamente (Gipsarbeiten) und die verwendeten Pigmente der Alhambra (Granada, Südspanien) beschrieben. Die Beschaffenheit, die Struktur und die Zerfallserscheinungen der Materialien werden ebenso detailliert beschrieben wie die Mal- und Stuckierungstechniken. Gipsabrucktechniken sind im gesamten Löwenpalast (späte Nasrid Periode, erste Hälfte des 14. Jahrhunderts) vorhanden, während in situ bearbeiteter Gips nur in einigen Bereichen des Partal Palastes, einem der ältesten erhaltenen Teile der Alhambra (1302–1309), gefundenen werden konnte. Zwei originale Paletten aus der Nasrid Periode konnten gefunden werden: eine frühere, einfachere, die aus Blau, Rot und Schwarz bestand und eine variantenreichere, spätere mit Rot, Blau, Grün, Schwarz und Gold. In beiden Fällen war Weiß die Hintergrundfarbe. Die Golddekorationen werden ebenso wie die späteren christlichen Veränderungen beschrieben. Kostbare Pigmente wurden während der gesamten Baugeschichte in der Alhambra verwendet (z.B. Gold, Lapislazuli, Azurit, Malachit, Zinnober und Mennige). Einige Pigmente, besonders Malachit und Mennige, sind durch die Verwendung chloridhaltiger Mineralien abgebaut und entfärbt. Risse und Verluste sind bei den Golddekorationen häufig. Diese Arbeit ist die erste Beschreibung der in der Nasrid Periode zur Färbung der Stuckierungen verwendeten Palette sowie zur Vergoldung und den Gipsarbeiten. Die Ergebnisse erhellen historische und künstlerische Fragestellungen*

***Resumen* — Este artículo trata sobre la descripción de los estucos tallados (yeserías) y pigmentos usados en la Alambra (Granada, Sur de España). La naturaleza, estructura y deterioro de estos materiales se describen en detalle, así como las técnicas de policromía y estuco empleadas. La técnica del vaciado en yeso es detectada en el Patio de los Leones (periodo Nasrid tardío, segunda mitad del siglo XIV), mientras que los yesos tallados in situ se encuentran sólo en ciertas áreas del Palacio del Partal, una de las construcciones más antiguas existentes todavía en la Alambra (1302-1309). Se identificaron dos tipos de paletas empleadas originalmente en el arte Nasrid: una más temprana y simple que incluye azul, rojo y negro, y una segunda más tardía de rojo, azul, verde, negro y oro. En ambos casos el blanco es el color del fondo. Se describe también la técnica de las decoraciones con oro, como en el caso de las intervenciones cristianas. En la historia de la Alambra se utilizaron ricos pigmentos (por ejemplo oro, lapislázuli, azurita, malaquita, cinabrio y rojo de plomo). Algunos pigmentos, el rojo de plomo y la malaquita en particular, se han degradado severamente por la introducción de materiales basados en el cloro, resultando en decoloraciones. El cuarteado y la pérdida del oro están muy extendidas. Este artículo aporta la primera descripción de la paleta usada en el periodo Nasrid para colorear los estucos de la Alambra, así como el dorado y las técnicas de yesería. Estos resultados pioneros aportan luz a cuestiones históricas y artísticas.*