Case report

The Nasrid plasterwork at “qubba Dar al-Manjara l-kubra” in Granada: characterisation of materials and techniques

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Abstract

Dar al Manjara l-kubra (The Royal Chamber of Santo Domingo) is a leisure room from the first period of Nasrid art. Scholars consider that the Chamber is of key importance for defining art during this period and a forerunner of what would subsequently become the palaces of the Alhambra in Granada. This paper presents a study of the materials and techniques used in the plasterwork, carried out before the monument was restored. The study began with a detailed examination of the plasterwork before samples of the more significant areas were selected from what was clearly part of the original work. The study of materials consisted in the identification of the pigments and binding media, as well as the base mortars. Conventional analysis methods were used: optical microscopy, scanning electron microscopy (SEM), and chromatographic methods (gas chromatography, GC and high-pressure liquid chromatography, HPLC) and X-ray diffraction (XRD). The data from material identification and a detailed study of the materials in situ defined the techniques employed and provided key data on Nasrid plasterwork. Very few studies have been made of the materials used in Hispano-Muslim plasterwork, especially in relation to this particular moment in history.

Keywords: Hispano-Muslim art; Nasrid period; Plasterwork; Materials; Pigments; Binding media; Mortars; Technique

1. Introduction

Chronologically, the qubba 1 Dar al-manjara l-kubra (better known as the Royal Chamber of Santo Domingo) (Plate 1) dates back to the first period of Nasrid art, under the reigns of Muhammad I, Muhammad II, Muhammad III and Nasr (1237–1314) [1]. Hispano-Muslim scholars agree to the importance of this monument, considered a key point of reference in Nasrid architecture. Fernández Puertas is of the opinion that its creator gave rise to a school of artisan—artists and calligraphers to whom the greatest achievements of this period can be attributed [2].

We are technical advisors for the team headed by architects Almagro Gorbea and Orihuela Uzal, who have carried out research on the monument and currently direct a project to restore it. They have concluded that, although the Almohad origin of most of the monument’s architecture is evident, it more closely related to the Nasrid palaces of the beginning of the 14th-century than to the Almohad palaces of Seville. Therefore, they consider that it is a forerunner of the palaces built in the Alhambra [3].

Our work is part of the research carried out by the Painting Department in the Beaux Arts Faculty in Granada on the materials and techniques employed in Hispano-Muslim art, and their conservation and restoration. In this study, we seek to provide data on the materials and techniques employed in the execution of the monument’s plasterwork. We contrast these data with the outcomes of previously published and current studies to confirm these relationships in the decoration.

In Nasrid art, the decoration seeks to “dress” the architecture with a “textile” quality. The ceramics, wall paintings and wood are sumptuously adorned and coloured. The decorative plasterwork panels, with their reliefs, and their vibrant carv-
ings and colouring, add an enormous degree of richness to the walls [4].

The materials that decorate the Royal Chamber follow the usual order, which must have depended upon their strength and stability at each point of the wall. Tiled or painted dados covered the lower part of the walls. Since the lower walls were prone to dampness, materials that perform well under damp conditions, such as ceramics or paint on lime mortars, were used for the dados. Next, the dry upper walls were decorated with gypsum, which would not have resisted the damp. In later times, wood or gypsum was used to decorate the ceilings.

In this paper, we will only present the outcomes of the study done on the plasterwork, since the technique used on the painted dados were published earlier [5].

2. Research aims

Very little documentation exists on the techniques used on plasterwork and even less on their polychromy. This is especially true when it comes to the binding media. To date, studies have focused mainly on the historical and formal aspects but very little or not at all on the materials. Therefore, our principal objective was to learn about the materials and the techniques employed in decorating the plasterwork of this interesting monument.

The aim of the study of the materials used in the mortars of the Royal Chamber was to discover the technique used in the polychromy, and therefore the study of the mortars focused on their effect on the painting or its conservation. However, since the mortars could help us to determine specific techniques, and salt migrations could cause changes in the paint and affect pigment identification (the presence of chlorides, for example, could give rise to an erroneous identification of some of them), we felt it was important to have a general idea of the composition of the mortars as well.

3. The plasterwork process

To give an example of the lack of existing references, we will cite Marçais. In his book on Western Muslim architecture, he describes the plasterwork process and states that this
type of work, called *naqch hadîda* (sculpture with iron tools) reached the height of its expansion during the 13th and 14th centuries in Granada, Fez, Tlemcen and Kairouan.

The technique consisted in applying a skim coat and then outlining the design on it with a dry point. Next, the motifs of the design were carved on the fresh gypsum with iron tools. This process required slow setting gypsum that, according to Marçais, was obtained by adding glue or salt, as workers still do in Tunez today. The elements of the motif were placed at different levels to give a feeling of perspective: the main elements are in a higher relief and the secondary elements are lower, forming the background [6].

Fernández Puertas points out that, in the first period of Nasrid art, under Muhammad I and II, the motifs were drawn on smooth panels and then carved in situ with a small chisel, while the blocks of gypsum were still damp. Subsequently, under Muhammad III, the technique was standardised. The width and the scope of the recurring pattern were established and, finally, the module of the pattern. Then it was carved in negative on a plaster of Paris or wooden mould. When the panel dried, its surface was coated with layers of water and lime to soften the rigid borders and angles, blurring the transition between the light and shadows.

Gómez Moreno, in his study of 13th-century Granada, explains that the plasterwork techniques used in the Royal Chamber were not plaster casts. Rather, “like the plasterwork at the Aljafería, they were carved in very hard patent plaster that was already in place, by carving it with iron instruments”. The fresh plaster technique, also used for the Mudejar plasterwork in Seville and Toledo, was still used in

Granada during the 14th-century, in combination with the plaster cast technique, mainly in the Alhambra [7].

Orihuela Uzal [8] relates the carving technique used on the plasterwork in the Royal Chamber to their sequence in time, “the plasterwork was carved on a dark coloured gypsum. Its Almohad origin is clear, although it had evolved considerably, incorporating the advances in decoration employed in al-Andalus during the reign of Ibn Hud (1228–1238). The Granada decorations are the direct heirs of the post-Almohad or proto-Nasrid decorations found in Murcia and the Levant during the second quarter of the 13th-century”.

Regarding the polychromy of the plasterwork, the panels were decorated with a very limited range of colours: red, blue, green, white, black, and occasionally gold or silver. On plain decorations, a few minute details were included, almost miniatures, even in the decorations of the higher parts of the walls where these details could not be appreciated [9]. The cleanliness probes that we took from the Royal Chamber plasterwork during our study evidence some of these virtuoso sketches.

4. Experimental section

4.1. Wall selection

The current whiteness of the gypsum decorations in the Royal Chamber, and of this type of Nasrid decoration in general, is because the original polychromy has been lost or was covered by layers of whitewash over the years. However, a detailed study of the plasterwork shows that the poly-
chromy was very carefully executed, using high-quality pigments.

The qubba has plasterwork on all four walls with almost no differences in their polychromy. The colours are repeated in a very similar way, with a few variations in their distribution due to changes in the reliefs. Therefore, we only ensured that the sample would constitute an original polychromy.

Four colours predominate in the study of the paint layer: blue, red, green, black and the white used for the background. Therefore, we did not need a large number of samples for, although the study surface was very large, the outcomes were repeated constantly.

The majority of the samples were prepared in polished test tubes, except for some missing blue and green areas where we had to map element distribution to identify some of the grains present in the layers of these colours.

4.2. Nomenclature

In view of size of the decorated surface and the number of samples to be studied, we used the same nomenclature system as in previous studies. First come the initials of the current name of the monument, CR for the Cuarto Real, or Royal Chamber de Santo Domingo, next is the initial Y for gypsum (yeso in Spanish), the study material, followed by the number of the panel the sample was taken from and, finally, the initial of the colour studied.

We assigned a number to each wall, from left to right, which in turn was divided according to the clearly differen-
tiated structure of the decoration, following the general outline of the composition.

In this way, each sample is identified by three numbers that clearly indicated which part of the monument it came from.

4.3. Instruments

Three basic techniques were used to identify the inorganic materials: optical microscopy, scanning electron microscopy (SEM) and X-ray diffraction (XRD).

To identify organic materials, we used coloration tests, infrared spectroscopy by Fourier transform, and chromatographic methods (gas chromatography, GC and high-pressure liquid chromatography, HPLC). The choice of one chromatographic method or another depended on the needs and the nature of the problem.

5. Outcomes

5.1. Paint layer

For the polychromy study, we collected 41 samples: first 34 and later seven more from the blue and green layers of paint whose outcomes from the first sampling were not clear.

In total, we took 16 blue samples, first 12 and later four more. For the latter, we prepared thin-polished sheets that were submitted to a detailed SEM study. A considerable number of red samples were taken, 10 in all. We first took five samples of green and later three more which were also prepared by thin-polished sheets. For these, the SEM study focused on the composition of the grains in the layers of paint. Only three samples of black were taken, since their results were all the same.

5.1.1. Blue pigments

Two kinds of pigments were identified in the blue samples. A very thin layer of ultramarine blue that belonged
to a later period was identified in one sample. In all of the other samples, taken from the original plasterwork, only one type of pigment with very similar characteristics was identified (Plate 2). The more numerous grains are composed only of copper of different sizes, mainly large and angular with sharp edges, never round or uniform grains. This granulometry is typical of azurite preparations for the colour is less intense if it is too finely ground. The pigment most frequently used seemed to match the characteristics of a natural azurite, coarsely ground to obtain a more intense colour.

In order to confirm that azurite was indeed used, a sample for XRD was taken. This analysis is conclusive for identifying azurite but it requires a large sample and therefore a considerable amount of background appears in the diffractogram and masks materials that are less represented. The outcomes confirm that, in fact, azurite was used.

In addition, we verified that some of the samples contained small grains of hematites, which could be impurities or possibly they were used to change the tone to obtain a colour closer to lapis lazuli.

On the other hand, most of the blue samples analysed initially had very few, small grains that showed up more brightly in the SEM than the other grains in the layer. We found two kinds: some that were very bright and small and others of an intermediate brightness, halfway between the former and the copper ones in the layer (Plates 3 and 4). There was only a minimal percentage of either kind compared with the copper grains and they were unevenly distributed. Furthermore, they were unevenly distributed inside the layer rather than on its outer surface. They could not be caused by further coats of paint because no instances of several layers were found.

S and Ba appear in the composition of the first type of grains and in the second, we always found As and Cu, although the composition was not constant. In addition, in some of the samples, we found Bi, in others Zn and in the majority S and/or Ca (Plate 3). These latter two elements could have come from salt migration in the mortars or deposits in the superimposed layers and therefore belong to a layer in which they predominated, since they appeared in almost all of the samples we studied.

The chemical composition of both grain types coincides with other pigments used in recent times, which would be Barite in the first case and in the second case, a green such as emerald green (copper acetoarsenite) or Scheele’s green (copper arsenate) [10].

In the second sampling, we could confirm the presence of small Barite grains (S and Ba), as well as a complex and variable composition that always presented As and Cu, and in some cases Bi or Zn (Plates 3 and 4).

As the SEM image shows, these grains were small compared to most of the samples analysed. Their presence was rare and they were unevenly distributed.

To confirm these facts, we made element distribution maps in the part of the sample where there were more grains. The first map, in black and white, shows the distribution of each element (Plate 4). In the first picture, all the components in the layer are visible. The next pictures show the proportion
of the elements and their location. For example, the second picture, shows the presence of As which, as can be seen, is minimal. In the third picture, the lighter areas are Barium, which is also very scarce. The following pictures are of Ca, Cu, and S, which are very abundant. Cu clearly predominates in the paint layer, and S and Ca predominate in the underlying mortar.

We systematically obtained the same outcomes in all of the maps of the other samples we analysed (see pictures).

This system, when applied to the other study samples, gave almost identical outcomes.

In view of the element distribution maps, we think that the presence of these compounds in the blue layers may have been due to natural azurite impurities, since they were present in very low percentages. Since in most cases these materials were inside the paint layer, we can rule out the possibility that their presence was due to subsequent layers of paint. On the other hand, copper carbonate is known to be found in the areas with veins of copper oxidate and that it often contains associated impurities such as As and Zn [11]. Therefore, this would seem to be the most reasonable explanation.

However, when considering the barium, we cannot reject the possibility that it may have been used intentionally to improve the quality of the layer of blue paint. Data are available on the use of barium in Western paintings, which does not imply that it was unknown in to Eastern painters or that it was not used to improve paint, in which case the use of barium in Hispano-Muslim paints would have come from the East.
Therefore, although we consider that the barium could be an impurity in the mineral used to prepare the pigment, to reach a conclusive opinion we would need to determine the origin of the pigment and of the mineral these materials came from.

This information, although interesting, is not the object of this paper, and therefore it could lead to further research to determine the origin of the materials used in the paint layer and in the mortars.

5.1.2. Red pigments

We found three different pigments in the red samples. The composition of the most plentiful pigment included Hg, identified in seven of the nine samples analysed. It evidenced the use of cinnabar–vermilion (Plate 5). The colour of the second pigment was due to the presence of Fe in the paint layer. This pigment was red earth or hematites (Plate 6). Finally, the third pigment was a red that was obtained by mixing two pigments, minium and an iron red similar to the one described above (Plate 7).

The iron-oxide red sample was taken from the background of the sebka or panel. It was easy to see the difference in tone compared to the other reds in the panel was easily appreciated. This pigment could be natural or artificial but its chemical composition always consists of iron oxi-hydroxides whose degree of purity and hydration determine the colour of the layer, whether it is a more intense red, violate or orange.

The most abundant red pigment was composed mainly of Hg and evidently, it was identified as cinnabar or vermilion (Plate 5). Cinnabar and vermilion are names assigned to a single chemical compound of different origin: in the first case, it is a natural compound and in the second case, it is an artificial pigment. In both cases, there were no differences in the structure or composition, which complicates its determination in paint layers. In nature, cinnabar is associated with heavy metal sulphides, pyrite, and marcasite, and often the rock matrix is composed of quartz, gypsum, calcite, dolomite, barite, fluorite, etc. [12]. In Roman times, the cinnabar mines in Almeden were already known and the first references to the use of vermilion (artificial mercuric sulphide)
occur in the eighth-century, although we know that in China it was used since antiquity [13]. The ninth-century Arab alchemist, Geber, refers to the artificial preparation of this pigment [14] and to introduction of the artificial variety to the West by the Arabs [15]. None of the samples analysed during this study show indications of elements that can be attributed to the presence of heavy metallic sulphides or to the remains of the materials we found as rock matrix. Only Hg was identified. We must bear in mind that the S peak was overlapped by the mercury peak and, therefore, the SEM only identified the latter. Therefore, since vermilion began to be used in Europe after it was introduced by the Arabs in the eighth-century, and since no impurities were identified in these paint layers—although considerable quantities of this material was found, as Plate 5 shows—we think that it could be vermilion (artificial mercuric sulphide). However, we do not reject the possibility that cinnabar was used, given the quality and importance of the Spanish cinnabar mines.

Finally, we also identified minium mixed with iron-oxide red (Plate 7). At first there seemed to be no difference between this colour and the more abundant red, but the identification of Pb in the outcomes of the analysis is decisive (Plate 8).

5.1.3. Green pigments

Although the green colour seems very similar in all the samples, there are a few differences.

First, we must highlight the changes in the green samples, in which the layer of paint is usually very fragmented (Plate 9), loose and practically lost in some areas. In some instances, there are only a few isolated grains of the pigment. Second, we must take into account that the samples were taken from the middle and upper part of the walls, since there were no remains of green in the lower part. Of course, this does not mean that there is no green there, since the layers of whitewash prevent us from visualising the polychromy as a whole. However, the green in the more accessible areas has been thoroughly scanned.

All of the samples were taken from original areas, which can be recognised by the mortar and the layers of paint. Newer areas of mortar look whiter and their granulometry is smoother, whereas the polychromy of the original layers of paint is denser and more consistent. However, in some layers, the green has changed so much that the layers are almost transparent and the colour is much less dense than in the other pigments we studied.

From the beginning, the presence of Cl in five of the six study samples made us think that a pigment similar to verdigris was used (Plate 9) since, according to Kühn, viride salsum was a mixture of basic copper chlorides and copper carbonates or basic copper chlorides and copper acetates. This pigment was much used since antiquity and can lead to many different variations depending on how it is obtained [16]. Vitruvio’s [17] eruca was obtained by putting vine
shoots, sheets of copper and vinegar in large earthenware jars, sealing them so it would not evaporate.

On the other hand, in the study layers of paint, in addition to copper chlorides, we find grains that only contained copper and that do not have verdigris characteristics. Contrary to what could be expected in a semi-organic pigment (copper acetate), a lower number of counts in the SEM was not detected.

In the sample CR-Y-361-V (Plate 9), differences in granulometry and colour appear in the paint layer. On the lower part, the grains are more clearly defined and larger. On the outside, the pigment grains cannot be differentiated and a change in tonality is clearly visible. Given the process for making verdigris [18] and the fact that Cl only appears in the green samples and not in any of the other pigments, it would seem logical that this compound constitutes the original composition and that it is not due to subsequent degradation.

Some samples (Plate 9) suggest a mixture of pigments, in which case the green would contain a mixture of copper and malachite.

As in the case of azurite, to obtain an intense blue, malachite should not be ground too much. In spite of the study samples’ poor state of conservation, it can be seen that the size of the pigment grains is considerable.

Finally, we must point out that the presence of grains containing Pb and Sb in one of the analysed samples could be due to Naples yellow. In another sample, grains containing Pb and Sn were identified. These elements can be attributed to the use of lead–tin yellow in the paint layer. This type of pigment has already been identified by X-ray fluorescence (XRF) and XRD in late Roman paintings and glazed ceramics.

These two pigments must have been used to lighten colour and to obtain a greener tone when pigments were too dark or bluish.

5.1.4. Black pigments

The black samples show the same characteristics. All three come from the relief outline and, as will be seen below,
they share the same composition. Since black is often used to highlight reliefs on surfaces, it is worth mentioning that here very few surfaces are painted in this colour (see Plate 10, picture 2, a detail of the Sebka showing the pearling done with this pigment). Therefore, although black was found in many places in the decoration, few remains of it are visible because they are hidden by superimposed layers of whitewash.

The outcomes, as mentioned above, are constant. In all three cases, atomic numbers drop, apatite (P and Ca) is identified and an earth containing iron (Plate 11). All this indicates that this colour is a mixture of pigments consisting of a bone black and an iron-oxide black. This mixture of pigments was already used in first-century Roman paintings [19]. We know that, although plant and, in this case, animal lampblacks are very long-lasting and have great colouring power, they are not very solid.

Ivory black, or elephantion according to Pliny, was discovered by Apelles [20]. Mayer [21] states that most of the ivory black used by artists is high quality bone black, characterised by the presence of an important quantity of calcium phosphate (apatite), as well as calcium sulphate and other impurities. Iron-oxide black is a dense, opaque, heavy and very permanent pigment, usually used in watery environments because it is long-lasting although the black pigment used is not Mars black, pure ferrous–ferric oxide, but an earth containing iron oxides that, in this case and due to their black colour, was mixed with bone black to give it more body and, consequently, a more solid paint layer.

5.1.5. White pigments

In the white samples analysed, only S and Ca were identified. As we found in the other whitewashed samples, these samples consist of a layer of much smoother, whiter gesso.
than the mortars that served as a base (Plate 12). This layer must have been applied to obtain a smoother, whiter surface and to soften the sharp outlines of plasterwork carvings [22].

5.1.6. Gold decoration

No elements were found that indicated the use of gold or, at least, a ground for applying gold, although we thought that it was highly probable that gold would be used in the Royal Chamber of Santo Domingo. However, it is possible that positive results will be obtained at other points because, as mentioned above, most of the surface was hidden.

The outcomes of a sample of gold decoration from the Alhambra (a fragment of gesso deposited in the Alhambra Museum with entry number 10686) served as a guideline for our search. The base for the gold on this sample was tin. At the time this paper was published, no gold or tin had been identified in any of the study samples from the Royal Chamber.

No materials that can be attributed to the use of gold were found in the plasterwork, although they have been identified in the plasterwork of other Granada palaces, such as the ones in the Alhambra, and therefore we cannot affirm that they were not used. These materials were used in for the gold reflections in the tiles in the entrance arch and, given the perfect integration of colour found in plasterwork and tiling, it is highly probable that gold was used.

5.1.7. Binding media

To study the binding media, the whitewash base and the paint layer of samples were studied, as well as the superimposed layers of whitewash in those samples where they were present.

The data obtained coincide in all cases and relate to the use of same technique in all of the pigments analysed.

The binding media identified in all of the samples is gum arabic (Plates 2 and 9), which coincides with the outcomes obtained from the majority of Muslim paintings that we studied during previous research, including the dados in the Alhambra and, obviously, the painted dados in the Royal Chamber of Santo Domingo.

There is no evidence of the use of glue in the paint layer or in the whitewash base. In the latter, only traces of gum arabic were identified, which we attribute to the way the paint impregnates the base when it is applied on a dry surface.

As will be seen in the section on mortars, no organic material was identified in the last mortar that could have been used to delay the setting.

Only the presence of animal glue and linseed oil was detected in the superimposed layers of whitewash. They were probably used to strengthen these layers and to make them shinier.

In conclusion, we could say that the polychromy identified must have been used to make the decoration richer, to highlight the relief, to integrate all the wall decoration materials
(tiled dados, plasterwork, and wooden framework) and to highlight the carved motifs. Furthermore, the colours were applied in such a way that they order and clarify the compositions in the relief. Their distribution is always governed by an element of symmetry.

That painted decorations were used in the areas of higher relief has been demonstrated by the fact that, although most of them have been lost, they were identified in several areas of the plasterwork. It is worth noting that this type of paint was carefully made, even though it was used in inaccessible areas. In the places where this decoration was found, it consisted in outlines, pearl and lines that emphasise the movement of the decorative elements.

5.2. Mortars

To study the mortars, samples were taken from different areas and, from each area, from the mortar levels to which we had access. Therefore, in some instances, we could only analyse the outside mortar and the whitewash base (Plate 13).

We found two levels. One was a thicker mortar whose gravel was essentially composed of silicates, calcite and dolomite to which some lime was probably added to make it harder and damp-proof, as well as to delay the setting. The other level had similar characteristics but the grain was finer, making it possible to make the decoration of some areas stand out more. The use of gypsum mortars to which gravel has been added is frequent throughout Spanish architecture. Slaked lime is added to make the mortar harder through carbonation. The carbonation is enhanced by the fact that the presence of gravel makes the mortar more porous [23].

The presence of celestina (strontium sulphate) in all of the samples analysed is constant. It was found in the XRD and the SEM. This compound can be identified in most of the plasterwork in this geographical area [24].

The composition of these mortars indicates that their binding media is a mixture of gypsum and calcium carbonate. A study of the gravel reveals that it is composed of quartz, silicates, calcite, dolomite and pieces of gypsum. The proportions vary according to the sampling area but it is worth...
noting that the components are repeated regularly. These changes in the proportion of gravel are not strange, given that the samples are small and the materials are natural and mixed by hand.

Regarding organic material, some gum arabic was found in the samples that had some paint, which suggests that the liquid adhesive was absorbed when it was applied. No other set-retardant organic material was found that could have been added to the mortar.

All these data indicate a characteristic technology for applying mortars in this geographical area, which is known for adding slaked lime to gypsum mortars to make them more resistant to the damp caused by calcium carbonate formation and to delay setting, thus offering more time to carve the reliefs in the decoration. Therefore, although Marçais states that glue or salt was added to the gypsum in the Royal Chamber to delay setting, in fact lime was the set-retardant.

The work process was probably as follows: first, a thick layer of mortar was applied. The composition would be outlined in the mortar. Next, the second mortar would be added and then carved. Finally, the entire surface would be covered with a whitewash base. Marçais [25] describes the plasterwork process for the convent of San Francisco in the Alhambra and his description exactly matches what we deduced for the plasterwork in the Royal Chamber.

No lines indicating a union between panels were found at any point, which would have been the case if moulds had been used. This confirms that the plasterwork was carved. Furthermore, slight irregularities appear in the repeated themes, which otherwise would not exist.

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