A new insight into the vaults of the kings in the Alhambra (Granada, Spain) by combination of portable XRD and XRF

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a b s t r a c t

In the Hall of the Kings, along the east side of the Courtyard of Lions in the Alhambra (Granada), the vaulted ceilings are clad in painted leather depicting various Kings or Lords on their seats, the fountain of youth, and a Lady playing chess. These artworks are unique masterpieces because of the unusual medium used (leather) and the presence of human representation in 14th century Muslim art.

The colour palette of the paints used was assessed without taking samples, using a prototype apparatus that simultaneously performs X-ray diffraction (XRD) and X-ray fluorescence (XRF), providing an elemental chemical and mineralogical characterization of the areas studied. The curvature of the vaulted ceiling and the size of device restricted analysis to the flattest parts and the most accessible areas.

The results show that the red hues are made from cinnabar and/or hematite. The flesh colour is a mixture of cinnabar and hydrocerussite, while malachite was used for the greens. Blues were achieved using lazurite and azurite, and the ochre tones using clays. Furthermore, XRD detected the presence of red lead in the red-ochre and orange tones, while gold is present together with tin and iron. Moreover, strontium is associated with the plaster preparation, and a compound made with arsenic and sulphur was observed in the green areas.

1. Introduction

The leather paintings that cover the vaulted ceilings in the Alhambra's Hall of the Kings (Granada) are currently being restored. This restoration work has been commissioned by the Alhambra and the Generalife in Granada in collaboration with the Andalusian Institute of Cultural Heritage (IAPH). The intervention is a broad and comprehensive project, responding to the demands of these unique masterpieces, their current state of conservation, their properties, and their environmental conditions.

The three rooms in the Hall of the Kings are located to the east of the Court of the Lions in the Alhambra. From right to left, we first find the image of a Lady playing chess, second, in the center, the Kings, and third, the Fountain of Youth. Traditionally, these depictions were thought to have been created by painting on leather, but it has now been established that they were made on a mixed medium of leather covering the wooden vaults [1]. Stratigraphic sections show a primer layer of gypsum with animal glue followed by pigments with egg yolk and linseed oil in the latest interventions. Over this surface, some areas have been picked out with gold leaf [2].

Our research centered on the vaulted ceiling of the Hall of the Kings, featuring painted scenes depicting various Lords or Kings on their seats (Fig. 1.a) and the Fountain of Youth (Fig. 1.b). These artworks are masterpieces not only because of the medium used (leather) but also for the presence of human representation in 14th century Muslim buildings [3].

At the beginning of the intervention conducted by the IAPH, the integrity of the three vaulted ceilings was at great risk due to the conditions of their conservation, in particular due to ineffective architectural interventions and restoration attempts since 1618 [2].

In 1855, Rafael Contreras divided the original roof (a single space that spanned the three vaults and provided rainwater drainage) into three separate spaces with double-pitched roofs, channelling rainfall through the new division towards the space between the three vaults. This change in rainfall drainage has caused severe water damage to the vaults leading to bio-deterioration in the wood and leather with consequences for the paintings, which displayed disruptions in their primer and subsequent layers of paint, in the form of peeling and cracks [4].

As a consequence of this intervention in the roof structure, several restorations of the paintings have been undertaken to minimize the ef- fects of water alteration on the paintings. These restorations have main- ly focused on treating areas of peeling and scaling paint. For instance,

Fig. 1. XRD/XRF areas of study in the vaulted ceilings of the Hall of the Kings (Alhambra, Granada). (a) Central vault depicting the Kings, (b) Lateral Vault showing the Fountain of Youth.

new preparation layers were added, the vaults were coated, gaps were patched using different materials, and protective products were applied [5].

The most important restoration of the paintings, carried out in 1960 by Gudiol, involved applying fixing treatments based on waxy compounds (wax-paraffin), bringing about major changes in the nature of the original painting and leading to additional forms of weathering (peeling, stains, gaps) [2].

On account of the weathering conditions of the vaulted ceilings, and the need to evaluate the consequences of damage while minimizing the number of samples taken from the paintings, non-destructive techniques were used to determine the mineralogical composition of the artwork, also providing further knowledge of these masterpieces as well as the general state of conservation prior to new interventions. For this reason, the characterization of paintings was conducted *in situ* using a portable device that combined X-ray diffraction (XRD) and X-ray fluorescence (XRF). The working conditions were challenging due to the shape of the artwork and the accumulation of restoration products such as waxes on the surface.

2. Materials and methods

To characterize the mineralogical pigments, a portable XRD/ XRF device was used. This equipment is capable of performing crystalline phase identification and elemental chemical analysis simultaneously on a single area. This portable XRD/XRF system was developed by the Centre de Recherche et de Restauration des Musées de France (Louvre Palace, Paris), with the support of European projects EU-ARTECH (EU FP6 RII3-CT-2004-506171) [6] and MOLAB CHARISMA (EU FP7–228330) [7]. As this equipment was designed to study various kinds of artworks, in particular canvas paintings [8–10], it was a challenge applying it to this medium and a vaulted ceiling from the top of scaffolding.

To provide a brief explanation of how it functions, X-rays are produced by a Cu anode source. The beam impinges the artwork at a nominal angle of 10° from the surface where it irradiates an area of 3 × 3 mm^2. XRD is obtained from the Cu-Kα line (8.05 keV). XRF analysis is performed with a silicon drift detector (energy resolution of 150 eV at 6 keV) that identifies fluorescence lines between 2 and 30 keV. Because of the Cu source, XRF spectra always include the Cu-Kα line and no Cu-Kβ which is absorbed by the Ni filter on the source window [8–10]. The presence of Cu in the object can be revealed by the presence of the Cu-Kβ line or by carrying out a specific XRF measurement with an Al 750 μm thick filter that eliminates the Cu line from the source [8].

With these conditions, the analyzed depth of materials is directly related to the penetration of X-rays, corresponding in the case of XRD to a 20 μm thick layer of matter for light elements (Al, Si, K,. . .) and 5 μm for heavy elements (Pb, Hg, Sn,. . .). In the case of XRF, penetration depends on the energy of the fluorescence line, which can be less than 1 μm at 2 keV for Pb-M and as high as 50 μm at 25 keV for Sn-K [10].

A layer of organic matter that is 100 μm thick does not significantly absorb X-rays above 5 keV [9]. Therefore, the presence of wax layers should not be a problem in this study. The challenge in this case is to position the XRF-XRD equipment on a concave surface and to maintain its alignment when it is located on scaffolding that moves under the weight of the operators. Nevertheless, the possibilities of the prototype meant it could be adapted to the conditions and cope with such difficulties. It also proved to be a valuable experience to improve the equipment.

The concave surface and the geometrical constraints meant that the XRD–XRF system had to target greater distances than it does when analyzing flat objects such as canvas [9,10]. This has no effect on the collection of diffracted beams, but it does mean that the XRF detector is no longer situated in an optimized position, with possible XRF intensity loss at low energy levels (below about 5 keV). Bearing this in mind, qualitative interpretation of XRF spectra is still useful, particularly when interpreting XRD diagrams. For XRD, the main difficulty is caused by the instability of the scaffolding, making it necessary to determine the center of the diffraction pattern for each measurement. This can be done by trial and error because the artwork generally includes known minerals (quartz, cerussite, hydrocerussite, etc.) that can be identified in the diagrams at the expense of additional work for the operators.

When studying the section of vaulted ceiling depicting the Kings, eleven points were analyzed using XRD–XRF (Fig. 1.a), whereas fourteen points were analyzed using XRD–XRF (Fig. 1.b) for the Fountain of Youth. In both cases, evaluating the colour compositions of the paints and the mineralogical phase of the pigments beneath the weathering agents were the main aims of the research. The points were selected depending on the palette, the restorations, the curvature of the vaults, and the experimental conditions, which meant that only accessible parts of the vaults could be selected.

3. Results

Table 1 summarizes the results of the chemical and mineralogical analysis performed on the vaulted ceiling depicting the Kings. On-site XRF spectra detected the presence of Ca, S, Fe, Hg, Cr, K, Cu, Co, Au, Si, As, Ba, Pb, Sn, Zn, and Sr. Most of these elements were also confirmed by taking sample and analyzing them with SEM-EDX [11]. However, Al, Na, and Mg could not be detected using such non-destructive techniques and under on-site XRF conditions, as their emission lines

Table 1
Results from XRD and XRF into the vaults of Kings (Alhambra, Granada).

Sample	Colour	XRD	XRF	SEM-EDX [11]
R01	Red-brown	Hydrocerussite, hematite, gypsum, minium	Fe Cu Pb Sr	–
R02	Red	Cinnabar (hydrocerussite, gypsum)	K Ca Fe Hg Pb	Ca S Fe Hg
R03	White	Gypsum, hydrocerussite, (cerussite), others	S Ca Fe (Pb)	–
R04	Green	Hydrocerussite, gypsum, Malachite	Ca Cr Fe (Co) Cu Pb Sr	Pb S Interventions Ti Ba Zn
R05	White	Hydrocerussite	Fe Pb Sr	–
R06	Golden	Gold, gypsum	Ca Ti Fe Au (Pb) Sr	Au
R07	Carnation	Hydrocerussite, gypsum (Cinnabar)	K Ca Fe Pb (Hg) Sr	K Ca Fe Hg S Pb
R08	Carnation (Dark)	Hydrocerussite (Cinnabar)	Pb, (Hg), Sr	–
R09	Carnation (dark)	Hydrocerussite, cinnabar (Gypsum)	K Ca Fe Pb Hg Sr	–
R10	Orange	Minium, gypsum	K Ca Fe Pb (Hg) Sr	Pb Hg Ca Fe
R11	Green	Gypsum (oropiment)	S Ca Fe As Sr	–
F01	Blue	Hydrocerussite, lazurite, gypsum	K, Ca, Fe, Pb, Sr (Co)	Pb, Ca, Si, Al, Na, S
F02	Red-brown	Hydrocerussite, gypsum, quartz (clays)	(Si) K, Ca, Fe, Pb, Sr	–
F03	Blue	Hydrocerussite	K-, Ca-, Fe, Pb, Sr	Pb, Ca, Si, Al, Na, S
F04	Brown	Hydrocerussite, minium, gypsum	K, Ca, Fe, Pb	–
F05	Green	Gypsum, quartz, oropiment	S, K, Ca, Fe, (Hg), As, (Pb), Sr	–
F06	Red	Gypsum, barite, hematite, cinnabar	S, Ca, Ba, Fe, Hg, (Pb)	Pb, Hg, S, Fe,
F07	Blue	Azurite, gypsum (Hydrocerussite)	K, Ca, Ti, Fe, Cu, Pb, Sr	Pb, Ca, K, Si, Al, Mg, S, Cu, Fe
F08	Leather	Azurite, gypsum, Hydrocerussite	K, Ca, Ti, Fe, Cu, Pb, Sr	–
F09	Carnation	Hydrocerussite, Cinnabar Gypsum	K, Ca, Fe, Hg, Pb	Pb, Si, Al, Ca, Fe, S, Hg
F10	Red	Gypsum, cinnabar	K, Ca (Ba) Fe Hg (Pb)	Pb, Hg, S
F11	Yellow	Hydrocerussite, Cerussite, gypsum, gold	K Ca Ba? Fe Au Pb (Sn)	Au, Al, Si, K, S, Ca, Fe
F12	Leather	Gypsum, hematite	S K Ca Fe	–
F13	Dark blue	Gypsum, hydrocerussite	K Ca Ti Fe Pb	
F14	Blue	–	Ca Ti Cr Fe Co Zn (Pb)	Ca, Ti, Cr, Fe, Co, Zn, Al

Note: Gypsum ($CaSO_4$),hidrocerussite ($2PbCO_3 \cdot Pb(OH)_2$), hematite (Fe_2O_3),minium (Pb_3O_4), cinnabar (HgS), malachite ($Cu_2(CO_3)(OH)_2$), oropiment (As_2S_3), lazurite ($Na_3CaAl_3Si_3O_{12}S$), gold (Au), cerrussite ($PbCO_3$), quartz (SiO_2), clays (aluminosilicates with iron), barite ($BaSO_4$), and azurite ($Cu_3(CO_3)_2(OH)_2$).

are below 2 keV, therefore they are absorbed by air and the detector entrance window.

On-site XRD detected the following minerals: hydrocerussite ($Pb_3(CO_3)_2(OH)_2$), cerussite ($PbCO_3$), hematite (Fe_2O_3), gypsum (Ca_2SO_4 $2H_20$), minium (Pb_3O_4), cinnabar (HgS), malachite ($Cu_2CO_3(OH)_2$), gold (Au), oropiment (As_2S_3), lazurite ($Na_3CaAl_3Si_3O_{12}S$), quartz (SiO_2), clays (likely aluminosilicates with iron), barite ($BaSO_4$), and azurite ($Cu_3(CO_3)_2(OH)_2$). The results of XRD and SEM-EDX indicated the presence of azurite for blue, earth and vermilion for red, and azurite and earths for green [11].

Point R03 corresponds to plaster, while point R05, corresponding to the white preparation layer, is characterized according to XRF by the presence of S, Ca, Fe, and Pb (low concentration). XRD shows the presence of hydrocerussite ($Pb_3(CO_3)_2(OH)_2$), gypsum (Ca_2SO_4 $2H_20$), and cerussite (PbCO3). They correspond to gypsum and lead white from the stucco and preparation layers, respectively (Fig. 2).

Transformation between hydrocerussite and cerussite is possible due to continuous water filtration and relative humidity variations in the conditions of the vaults [12]. This transformation is accompanied by a considerable increase in volume that could cause cracks and peeling.

The presence of lead white implies that restorers cannot use acid treatment during the intervention due to the presence of carbonates.

Gypsum appears in most of the areas analyzed using XRD/XRF technology. It might be from the support layer of the paintings, which implies that X-rays would provide information on all the stratigraphic layers at each point of analysis. Furthermore, discontinuities in the paint layers might reveal gypsum from deeper layers. However, in a palace such as the Alhambra, with its plaster stuccos, the surface may be contaminated with gypsum dust. For this reason, it is not possible to guarantee that all the stratigraphic layers have been detected, in spite of the presence of gypsum.

Non-destructive analysis (XRD) indicates the use of vermilion, red lead, and red earth for red tones, whereas SEM-EDX analysis conducted on samples only detects vermilion and red earth (Table 1). Fig. 2.a shows the XRD and XRF results of a red area with hydrocerussite ($Pb_3(CO_3)_2(OH)_2$), hematite (Fe_2O_3), gypsum (Ca_2SO_4 $2H_20$), and red lead (Pb_3O_4). Furthermore, Cu and Sr were detected by XRF (Fig. 2.b). Other XRF spectra (Fig. 2.b) contain Hg. The dots on the Debye–Scherrer Rings (Fig. 2.c) indicate the presence of 'large' particles in the preparation layers and in the pigments. This was observed in most of the measurements.

According to traditional techniques, the colour blue is obtained using azurite, but XRD reveals the presence of lazurite (lapis lazuli or ultramar) and azurite. The usual coarse grains typical of ground mineral such as lazurite or azurite produce dotted Debye–Scherrer Rings [10]. Point F01 shows the presence of lazurite, and dotted rings are suggesting that this blue may be natural (lapis lazuli) rather than handmade (ultramar), which is fine grained.

The colour green also yields differences. XRD–XRF results show malachite and oropiment, whereas samples suggest the use of a mix of azurite and earths.

Areas R04, R11, and F05 correspond to green tones. They are characterized by XRF showing the presence of Ca, S Cr, Fe, (Co), Cu, Pb, As, K, (Hg), and Sr (low concentration). The X-ray diffractogram indicates the presence of hydrocerussite ($Pb_3(CO_3)_2(OH)_2$), gypsum (SO_4Ca_2 $2H_20$), and malachite ($Cu_2CO_3(OH)_2$) for R04, gypsum and oropiment for R11, and gypsum, quartz, and oropiment for F05.

SEM-EDX results [11] detected azurite mixed with earths, whereas malachite was not detected. Malachite green might initially be azurite and undergo mineralogical changes due to environmental conditions [13–15]. Nevertheless, malachite was used in other gypsum paints of the Alhambra Palace [16,17], so we cannot reject its use originally.

Points R11 and F05 corresponding to green, characterized by the presence of S, Ca, Fe, As, and Sr according to XRF, and revealing the presence of plaster preparation (SO_4Ca_2 $2H_20$) and oropiment (AsS) according to XRD. The analysis of green surfaces using a ZARBECO magnifier with visible light shows that these green colours consist of mixtures of blue and yellow in some areas, as it might be in the case of R11 or F05, whereas in other areas the colours are more homogeneous and created using green pigments, probably corresponding to malachite. Fig. 3 shows different surfaces of green pigments [18].

Cobalt, titanium, and chromium (F13 and F14) have not been associated with any of the minerals. Ti may be associated with modern interventions, and cobalt could be linked to blue tones.

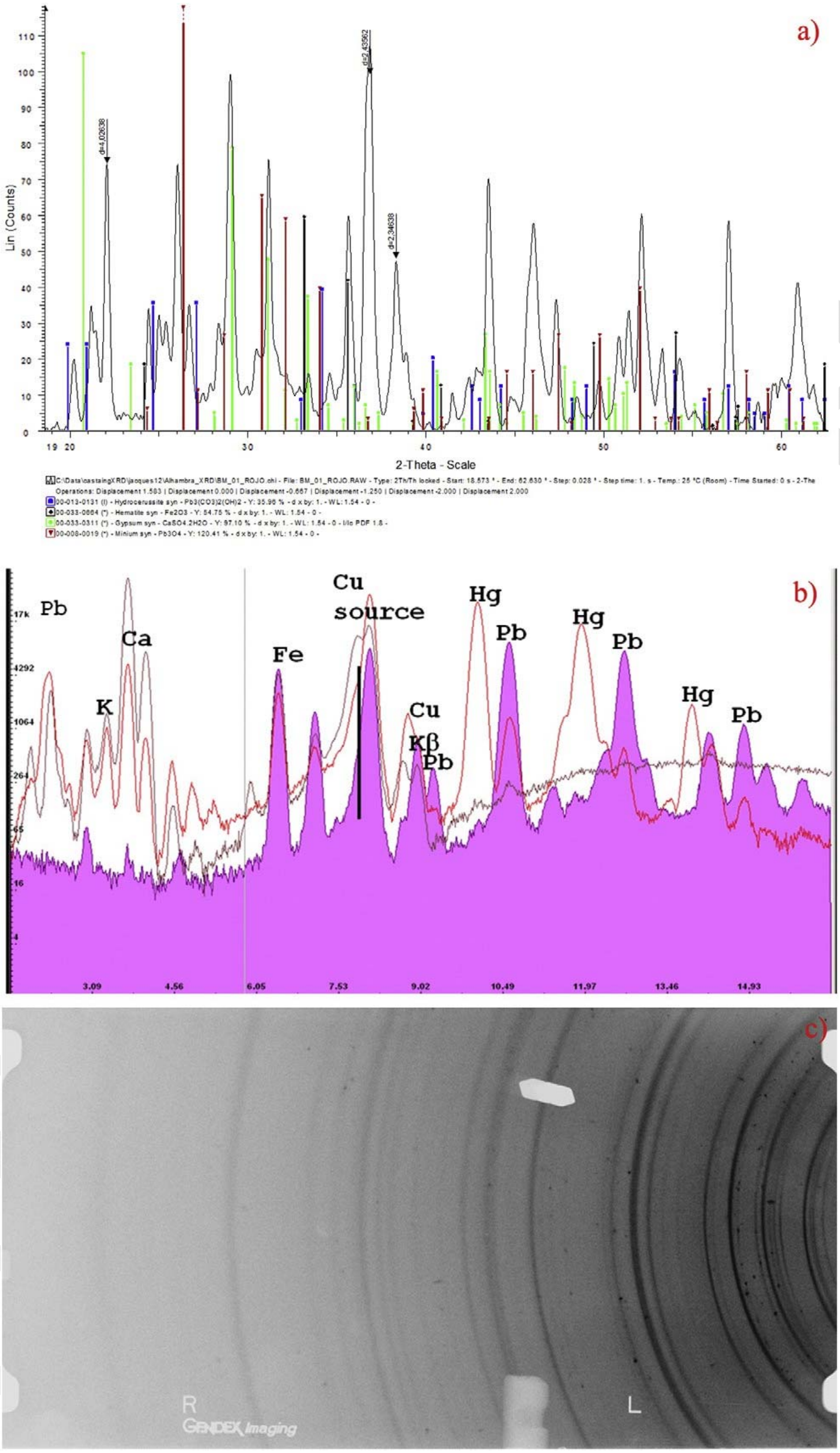

Fig. 2. XRD/XRF of a red zone: (a) The X-ray diffractogram (top left) shows the presence of hydrocerussite ($Pb_3(CO_3)_2(OH)_2$), hematite (Fe_2O_3), gypsum (Ca_2SO_4 $2H_20$), and red lead (Pb_3O_4); (b) Fe, Cu, Pb, and Sr are detected by XRF. No peaks are visible below Fe because of poor positioning of the detector. The other two XRF spectra correspond to red at point number 2, which contains Hg, and to a measurement on an uncoloured zone (leather and preparation with Ca and K; (c) Debye–Scherrer Rings of point number 1 in the Hall of the Kings.

The points corresponding to R06 and F11, yellow and golden colours, were shown by XRF to contain Au. The X-ray diffractograms detected the presence of plaster (Ca_2SO_4 $2H_20$) from the preparation layers and gold. Gold leaves are badly cracked and evolved in numerous points of the artwork. The presence of Sn has been described by Cardel [16] under the golden layer for similar artworks.

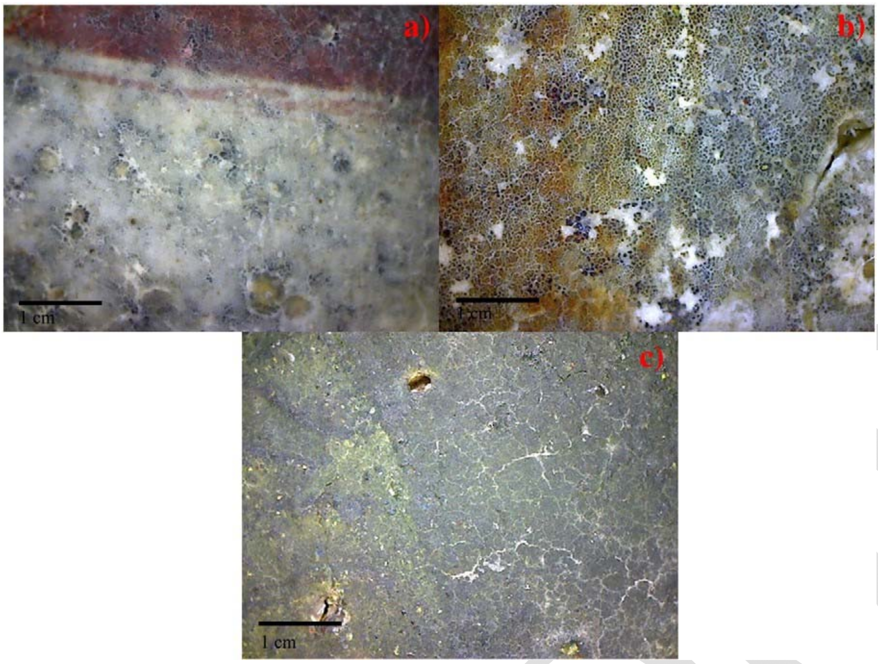

Fig. 3. Microphotographs taken using a ZARBECO magnifier on green surfaces. a) and b) are mixtures of blue and yellow grains; c) more homogeneous green due to pigments, which probably correspond to malachite.

The presence of Sr, in gypsum stuccos of similar periods, is usually associated with gypsum that is particularly rich in Sr and it could be due to celestine [19,20].

X-ray fluorescence of carnation colours revealed the presence of K, Ca, Fe, Pb, Hg, and Sr. XRD detected the presence of hydrocerussite ($Pb_3\ (CO_3)_2\ (OH)_2$), gypsum ($Ca_2SO_4\ 2H_20$), and cinnabar (HgS) in varying amounts depending on the tone and measuring points.

The results obtained by XRD–XRF indicate that the pigments used in polychromies from the Hall of the Kings in the Alhambra (lead white, cinnabar and red lead for red, and lapis lazuli for blue) are similar to the pigments identified in the Partal Palace (Alhambra) [16] (azurite for blue, cinnabar and red lead for red) and in the Mexuar Palace and the Lions Palace (cinnabar and lead oxide for red, lapis lazuli for blue, malachite for green, and gold over tin leaf for golden) [17,21].

4. Conclusions

The results show that reds are made using cinnabar and/or hematite. The flesh colour is a mixture of cinnabar and hydrocerussite, whereas malachite and oropiment were used to create greens. Oropiment has been detected for the first time in this gypsum paint and means that restorers must take special care due to its toxicity. Blues are made using lazurite or azurite, and ochre by clays. Furthermore, XRD has detected the presence of red lead in the red-ochre and oranges, while gold is present together with tin. Moreover, strontium is associated with the plaster preparation.

Knowledge of the original pigments allows restorers to intervene in accordance with international standards and to evaluate the vulnerability of the new conditions. This analysis was performed without taking samples, thanks to the prototype used that allows for X-ray diffraction (XRD) and X-ray fluorescence (XRF) to be performed simultaneously. The combination of these two X-ray techniques, without damaging the artwork, enables elemental chemical and mineralogical characterizations to be performed of the areas studied.

Acknowledgements

This study has been partially supported by the agreements IAPH & UPO and the Projects of Junta de Andalucia RIVUPH (HUM-6775).

Thanks to the Alhambra and Generalife Council for the collaboration and the permission to carry out this research.

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