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Review

Combining *in situ* elemental and molecular analysis: The Viceroys portraits in Old Goa, India

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ABSTRACT

The Vice-Roy's Portraits Gallery hosted at the Old Goa Museum of the Archaeological Survey of India in Goa, India, is a unique panel painting collection, spanning from the 16th till the 18th centuries, and representing the Portuguese Viceroys and Governors who administrated the Portuguese provinces in the coastal region of the Indian Ocean. During the Old Goa Revelations project (a collaborative inter-institutional consortium between Evora University HERCULES Lab, Lisbon University Fine Arts Faculty, Archaeological Survey of India and Ghent University), this important collection of panel paintings was examined in-situ using a non-invasive approach with mobile analytical instrumentation. Next to a series of imaging techniques, point analysis has been performed, using both, elemental and molecular spectroscopic methods. On the one hand, handheld X-ray fluorescence analysis and macro X-ray fluorescence imaging was used to obtain the elemental composition and its distribution on pictorial support, while on the other hand mobile Raman spectroscopy was implemented to obtain molecular information. These non-invasive techniques were used to determine the composition of the paint layers and to study the different treatments (e.g. overpainting, changes in compositions, etc.) that the artworks have witnessed since their creation.

1. Introduction

On the first floor of the Archaeological Museum of Old Goa there is a collection of more than a hundred paintings depicting the Portuguese Governors and Viceroys of India (Fig. 1).

These portraits are paintings of large dimensions (about 205 cm x 115 cm) that started to be executed in 1547, when the viceroy D. João de Castro commissioned to a local artist his own portrait and the portrait of his 12 predecessors. Later, in 1585, the paintings were restored for the first time, by demand of the Governor Fernão Teles de Menezes, and at the same time he commissioned ten more paintings [1–6].

In the beginning of the nineteenth century, the collection was retrieved from the Viceroys Palace, in Old Goa, which was then demolished. In 1839, the portraits suffered a poor restoration intervention by a painter who decided to overpaint large missing and or damaged areas. In the end of nineteenth, between 1893 and 1894, the military Manuel Gomes da Costa restored the collection. He repainted the backgrounds with dark colours and the faces and costumes of the Viceroys, the arms, hands and legs positions were also modified. In paintings that were in better conditions, he only repainted the background and has done small retouches [1–6].

Between 1953 and 1957, seven of the paintings came to Lisbon to be restored. During this intervention the overpainting was removed, and after the restoration, three of them were sent back to Goa. From the remaining four paintings, in 1961, the portrait of D. João de Castro was also sent to Goa, while the other three were incorporated in the collection of the Museu Nacional de Arte Antiga [1–6].

In the period between 1974 and 1998, some conservation interventions were done to the Goa portraits, and the last one was in

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Fig. 1. Collection of the Portuguese Governors and Viceroys of India portraits, Archaeological Museum of Old Goa.

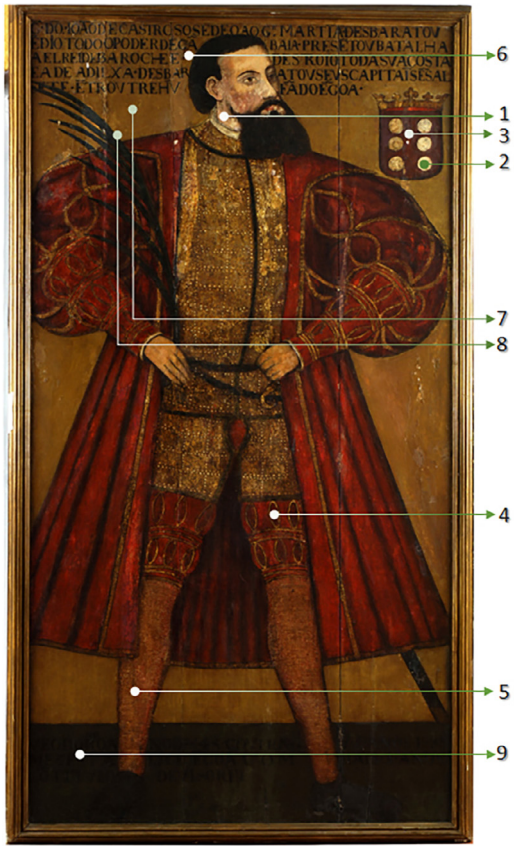

Fig. 2. Portrait of the viceroy D. João de Castro with the mapping of the points analysed by h-XRF and mobile Raman spectroscopy.

the years of 1997 and 1998 following procedures and criteria of the epoch [1–6].

This important collection of panel paintings has been examined in-situ using a non-invasive approach with mobile analytical instrumentation in the scope of the project *Old Goa Revelations: New Insights on the Viceroys Portrait Gallery*, a project financed by the Portuguese Science and Technology Foundation, that is focused on the application of non-invasive techniques.

Although invasive methods produce more detailed data on the materials present in artworks, in the context of this project it was not possible to collect any samples as the owner of the collection did not authorize this approach. So, the material characterization of the paintings had to be conducted just with non-invasive analytical instruments.

Considering the various options and the objectives of the study, and after surveying the portraits with imaging techniques such photography, UV fluorescence photography, infrared reflectography and X-ray radiography, for the recognition of altered areas of the paintings by previous conservation treatments, handheld X-ray fluorescence spectrometry (h-XRF), mobile Raman spectroscopy and macro X-ray fluorescence imaging (MA-XRF) techniques were chosen for the purposes of material characterization, expecting to discriminate between original compositions and conservation interventions, and to reveal the underneath layers corresponding to hidden decoration armours, coats of arms, golden decorations among others. The selection of these techniques was based also on availability and on the criteria of combining elemental and molecular analysis instrumentation. h-XRF, mobile Raman spectroscopy and MA-XRF are three of the in situ analytical tools that had significant advances in terms of portability, sensitivity, data acquisition times, readiness of results availability. In the case of MA-XRF, the most recent developed instruments are able to produce a chemical image of a relatively large area, being possible to simultaneously obtain multiple chemical elements maps in a relatively fast way or even in real time [7–26].

2. Research aim

This work is inserted in the scope of the collaborative project *Old Goa Revelations: New Insights on the Viceroys Portrait Gallery*, which integrates a multidisciplinary team of researchers. The main objectives of this project are the material characterization of the Viceroys portraits; to contribute to a better understanding of their history, because along the centuries they suffered profound alterations as a consequence of restoration interventions; and to help in the decisions to be made in future conservation/restoration interventions.

In this paper, the results of combining h-XRF, mobile Raman spectroscopy and MA-XRF in the study of three viceroys' portraits are presented. This are the portraits of D. João de Castro, D. Fernão Teles de Menezes and D. Manuel de Souza Coutinho. This study illustrates the potential of the combined use of these non-invasive analytical tools in the study of areas of the paintings where pictorial alterations (e. g. overpainting, changes in composition, etc.) have occurred, allowing to visualize the original painting that was then overpainted in conservation interventions, and identify the materials in original and non-original layers of the studied paintings. The paintings chosen for presenting in this paper are, among

the viceroys' collection, those that best meet the objectives of this article.

3. Materials and methods

In this work, the portraits of the viceroys D. João de Castro, D. Fernão Teles de Menezes and D. Manuel de Souza Coutinho were initially studied by a series of imaging techniques – photography, UV fluorescence photography, infrared reflectography and X-ray radiography – that allowed to identify areas on the paintings that have suffered pictorial changes.

Following the information obtained by these imaging techniques, the identified altered areas of the paintings were submitted to point analysis using both elemental and molecular spectroscopic methods. Handheld X-ray fluorescence spectrometry (h-XRF) was used to obtain the elemental composition and its distribution on pictorial support, and mobile Raman spectroscopy was implemented to obtain molecular information. Additionally, MA-XRF was used to complement the elemental analysis by h-XRF and the elemental maps obtained revealed some of the major pictorial alterations that the paintings have suffered along the time due to several interventions.

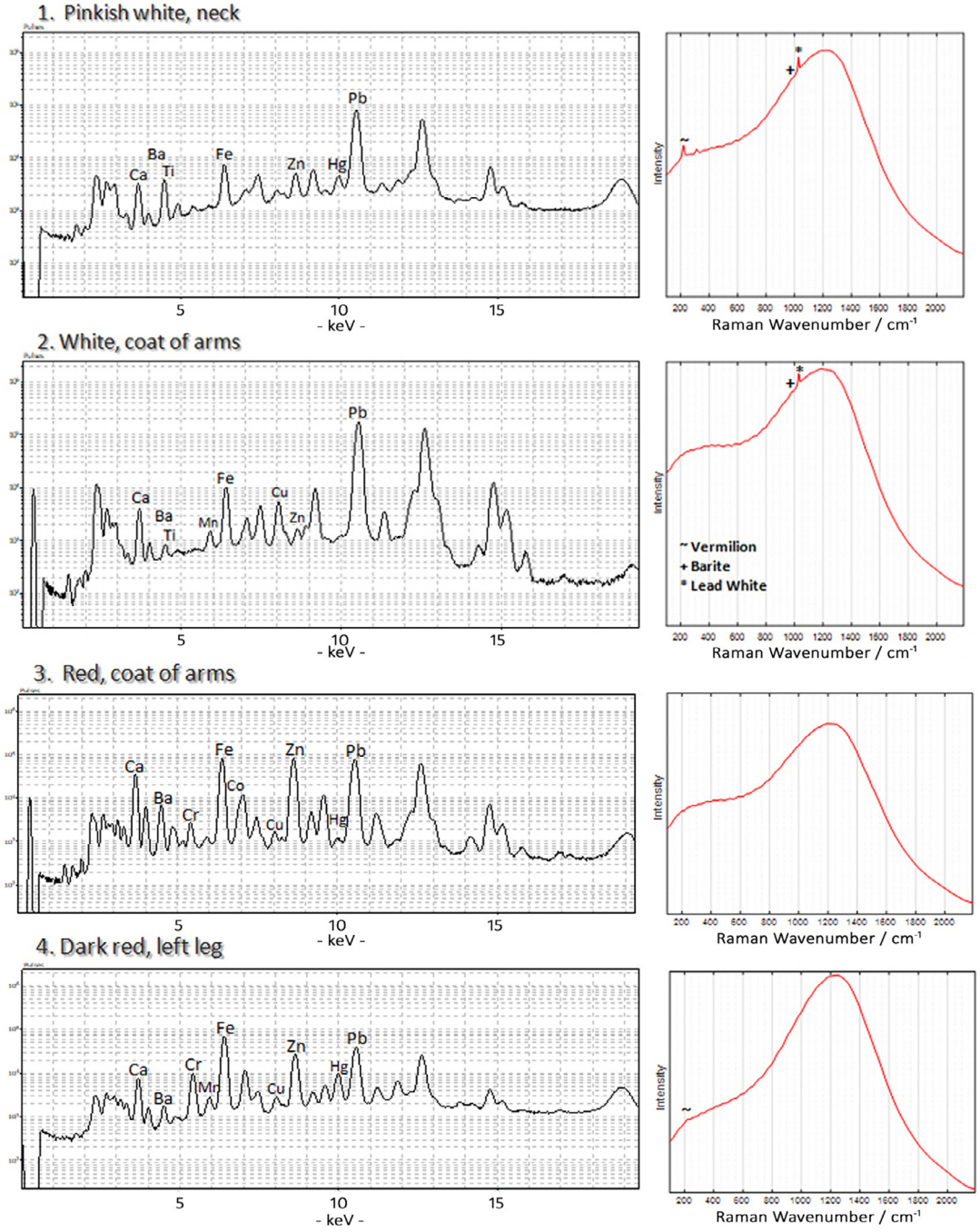

Fig. 3a. XRF and Raman spectra obtained in the analysis of the viceroy D. João de Castro (points 1 to 4). The symbols ~, + and * correspond respectively to vermilion (254 and 343 cm^{-1}), barite (986 cm^{-1}) and lead white (1056 cm^{-1}) [27–29].

3.1. Handheld X-ray fluorescence spectrometry (h-XRF)

A Bruker Tracer 5i handheld X-ray fluorescence spectrometer (spot size of 3 mm), equipped with a rhodium X-ray tube and a X-Flash SDD detector. The spectra were obtained using a voltage of 40 kV and a current intensity of 30 μA, with an acquisition time of 60 s. After acquisition, the spectra were processed using ARTAX (Bruker) software.

3.2. Mobile Raman spectroscopy

Mobile-Raman analyses were performed with a Enwave EzRaman-I Dual (DR) portable spectrometer equipped with a fibre optic head allowing an analysis spot size of ~0,5 mm, and a thermo-electrically cooled CCD detector. Spectra were acquired using a near-infrared 785 nm excitation in the spectral region of 100–2350 cm^{-1}.

During the analysis, the probe head of the Raman spectrometer was manually positioned and kept in contact with the panels' surface.

3.3. Macro X-ray fluorescence imaging (MA-XRF)

The MA-XRF examinations were made with a CRONO Bruker spectrometer. The system is constituted by a measurement head mounted on a motorized stage capable of scanning areas of 60 cm x 45 cm at a speed of 42 mm/s (imaging lateral resolution of 0,5 mm). The scans were made in a non-contact mode with focus distance of 5–7 mm. The spectrometer is equipped with a rhodium X-ray tube and a large area 50 mm^2 SDD detector with CUBE technology. The spectra were obtained using a voltage of 50 kV and a current intensity of 200 μA. Data acquisition and processing was made with CRONO and ESPRIT Bruker softwares.

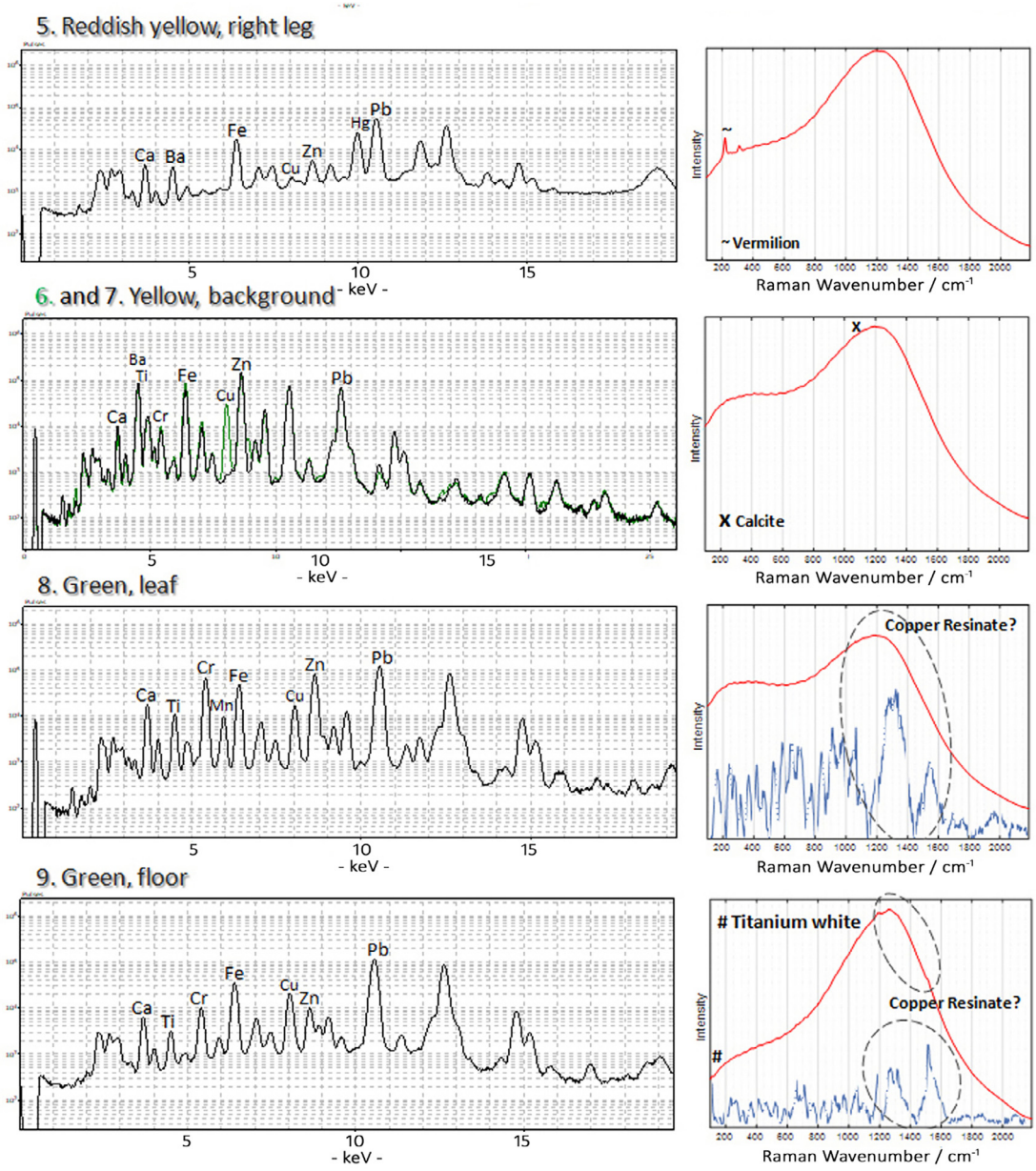

Fig. 3b. XRF and Raman spectra obtained in the analysis of the viceroy D. João de Castro (points 5 to 9). The symbols ~, x and # correspond respectively to vermilion (254 and 343 cm^{-1}), calcite (1086 cm^{-1}) and titanium white (142 cm^{-1}) [27–29]. (The blue curves in each Raman spectrum correspond to the same spectrum after polynomial baseline correction).

4. Results and discussion

4.1. Portrait of D. João de Castro

The portrait of the viceroy D. João de Castro is one of the paintings that was restored in the middle of the twentieth century in Lisbon and returned to Goa. Fig. 2 depicts the portrait of D. João and the points analysed by h-XRF and mobile Raman spectroscopy, and in Figs. 3a and 3b are presented the XRF and Raman spectra obtained.

The analysed points correspond to the painting's main colours: white, red, yellow and green.

In relation to the white colour, the coat of arms and the neck were analysed (points 1 and 2), and the neck exhibits a pinkish white colour. For both, h-XRF reveals the presence of calcium, barium, titanium, manganese, iron, copper, zinc and lead. The XRF spectrum of the pinkish white neck (point 1) also shows a peak for mercury. The Raman spectra acquired in the same points as h-XRF exhibit peaks attributed to barite (986 cm^{-1}) and lead white (1056 cm^{-1}) for both cases [27–29]. The pinkish tone of the neck is due to the presence of vermilion, as the spectrum shows absorptions at 254 and 343 cm^{-1} [27–29].

The presence of zinc and titanium may be attributed to the use of zinc white and titanium white in twentieth century interventions, calcium is due to the ground layer (made of gypsum or chalk), manganese and iron are related to ochre pigments applied in layers under the superficial one, and finally copper may be attributed to a blue or green pigment originally applied in the execution of the coat of arms and that was covered by the white paint.

In relation to the colour red, all XRF and Raman spectra in Figs. 3a and 3b (points 3, 4 and 5) account for the presence of vermilion, except the Raman spectrum of point 3. As it can be seen in the corresponding XRF, the peak for Hg has a very weak intensity and vermilion was not detected by Raman spectroscopy (the spec-

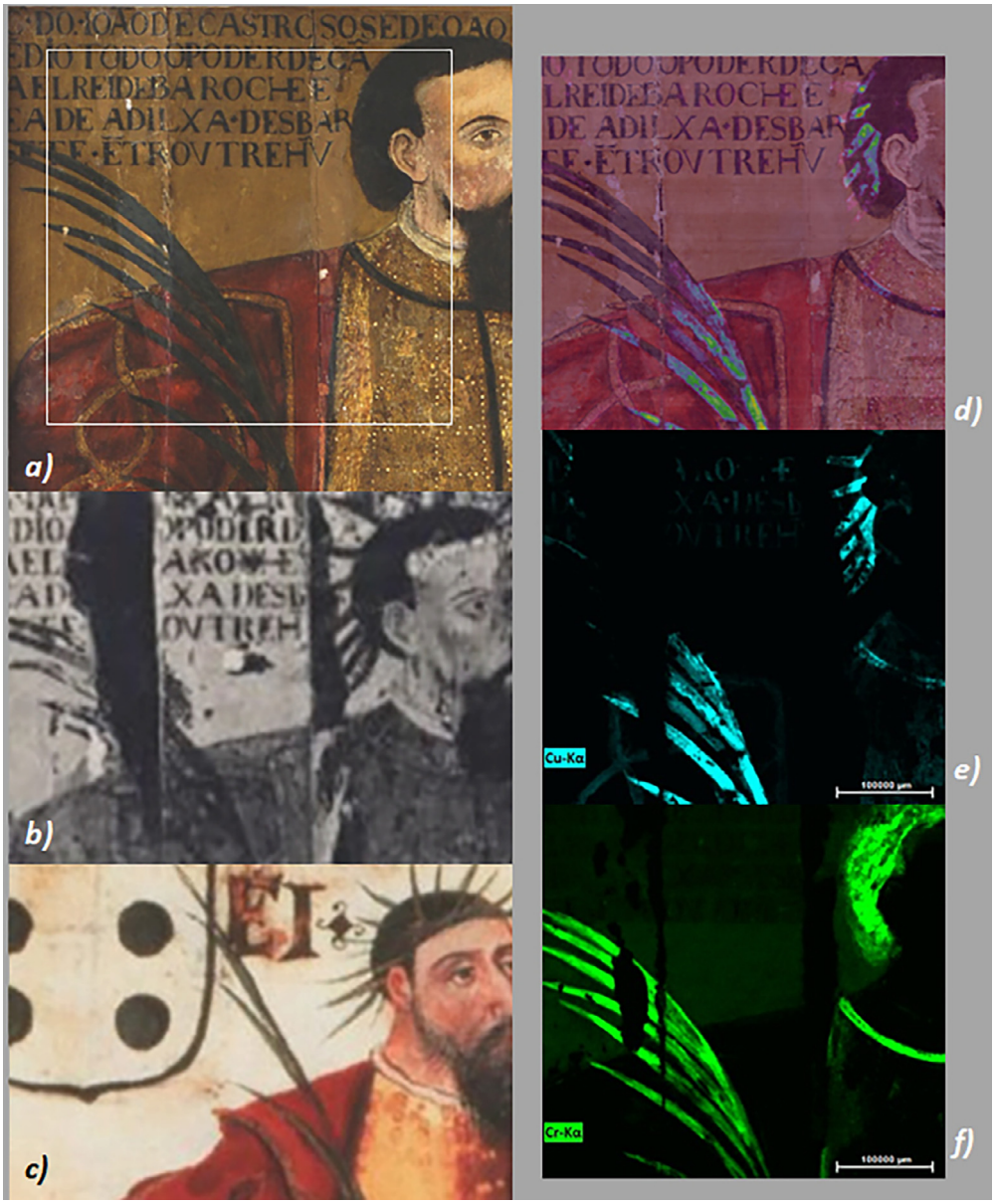

Fig. 4. Detail of the portrait of D. João de Castro with the indication of the area analysed by MA-XRF (a); corresponding detail of a photograph from the time of the restoration of 1954 (b); detail of a reproduction of the painting dated from 1560 (c); MA-XRF combined map of the elements Cu and Cr, overlaid in the visible image (d); MA-XRF map of Cu (e); MA-XRF map of Cr (f).

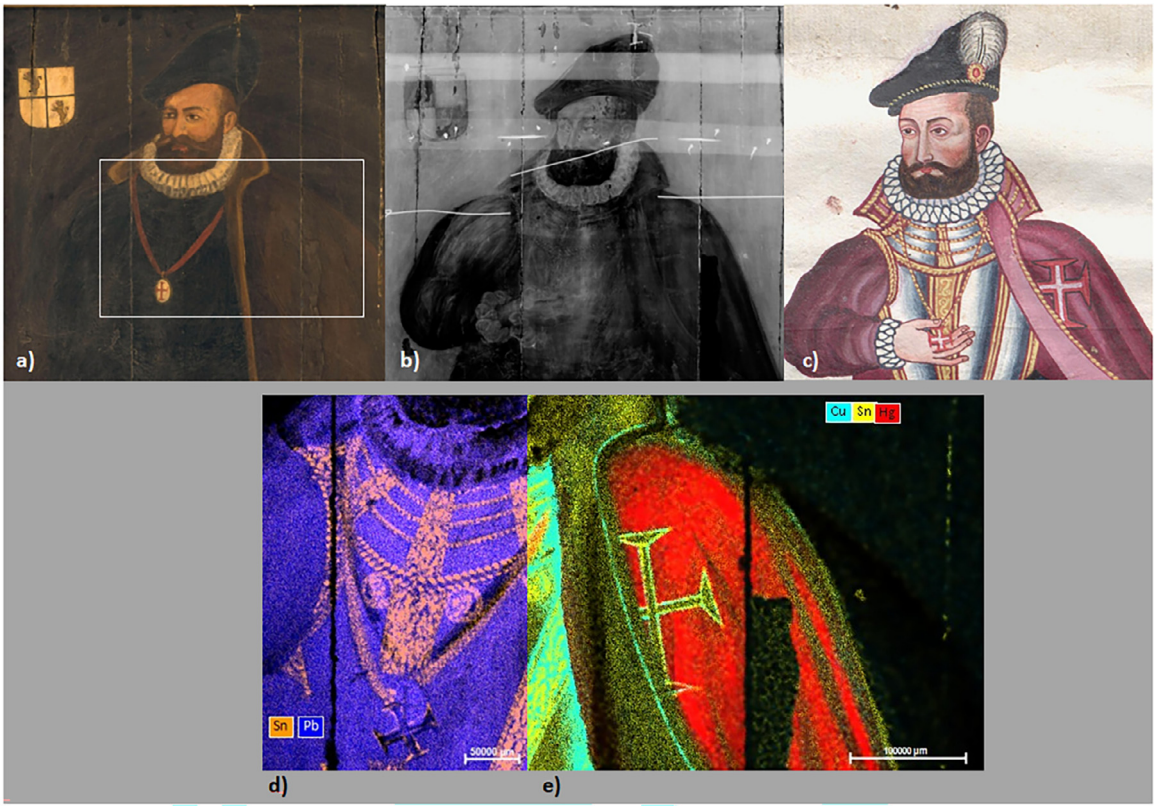

Fig. 5. Portrait of D. Fernão Teles de Menezes with the indication of the area analysed by MA-XRF (a); corresponding radiography (b); detail of a reproduction of the painting dated from 1646 (c); MA-XRF combined map of the elements Sn and Pb (d); combined map of the elements Cu, Sn and Hg (e).

trum does not show any absorption at 245 and 343 cm^{-1} as do spectra responding to points 4 and 5). Besides this, in the coat of arms, Raman spectrum reveals a lot of fluorescence, and it was not possible to identify any red pigments by this technique. It is possible also that a red lake has been applied in this case or an iron oxide pigment, as iron was also detected by h-XRF. Furthermore, chromium was detected in points 3 and 4, suggesting the presence of chrome yellow. Taking into account the removal of repaints in the twentieth century, we could assume that the chrome yellow was used in the intervention of 1953.

In what concerns to the yellow colour of the background, h-XRF reveals again the presence of chromium, in both analysed points (6 and 7), suggesting the use of chrome yellow. This element is also present in the colour green of points 8 and 9, and it can be also due to the presence of this pigment in the layers under the green layers, which contain a copper pigment, according to h-XRF results.

It is interesting to verify that copper was also identified in the point 6 corresponding to the yellow background, but in point 7 there is no copper. This result might be surprising, but by using MA-XRF analysis (Fig. 4) this could be better understood.

In a photograph from the time of the restoration of 1954 (Fig. 4b), it is visible the remains of a crown of leaves in the head of D. João, which is very similar to the one depicted on a reproduction of this painted dated from 1560 (Fig. 4c).

The map of copper obtained by MA-XRF (Fig. 4d and e) reveals the existence of remains of this crown of leaves of D. João. On the other hand, MA-XRF shows the presence of chromium in a semicircular area around D. João's head. This corresponds to a chrome yellow paint applied to cover the remains of the crown of leaves.

Also, copper is present in part of the leaves that D. João holds in his right hand. This part coincides just with the remains of the original leaves that we can see in the photo of 1954.

These original leaves are covered by a paint made of a chromium green pigment such as viridian, chrome oxide green or chrome green as it can be seen in the MA-XRF map. Chromium is also present but in less quantity in the rest of the yellow background.

4.2. Portrait of D. Fernão Teles de Menezes

A reproduction of the portrait of the viceroy D. Fernão Teles de Menezes dating from 1646 suggests that his painting was greatly modified in a restoration intervention (Fig. 5).

The costume of D. Fernão in the reproduction is completely different from that of the portrait and X-Ray radiography (Fig. 5b) confirms that the painting was modified.

In this case, the MA-XRF demonstrates the existence of the original painting under the actual one. The elemental maps (Fig. 5d and e) produced by MA-XRF clearly exhibit the same features depicted in the reproduction. The original costume of Fernão Teles de Menezes still exists, at least partially, as it may be seen in the maps of MA-XRF for tin, lead, copper and mercury, which account for the presence of lead-tin yellow and vermilion.

4.3. Portrait of D. Manuel de Souza Coutinho

In the case of viceroy D. Manuel de Souza Coutinho painting, by comparison with a reproduction from 1646 it is possible to verify an alteration on the cape of D. Manuel Coutinho.

This evidence could help the interpretation of the h-XRF analysis (Fig. 6) in these altered zones. The presence of Hg in point 1 referred to the location of a red cross that is overpainted. Testimony of this can be observed with the naked eye or by using ranking light, as relief is visible on the surface of the painting.

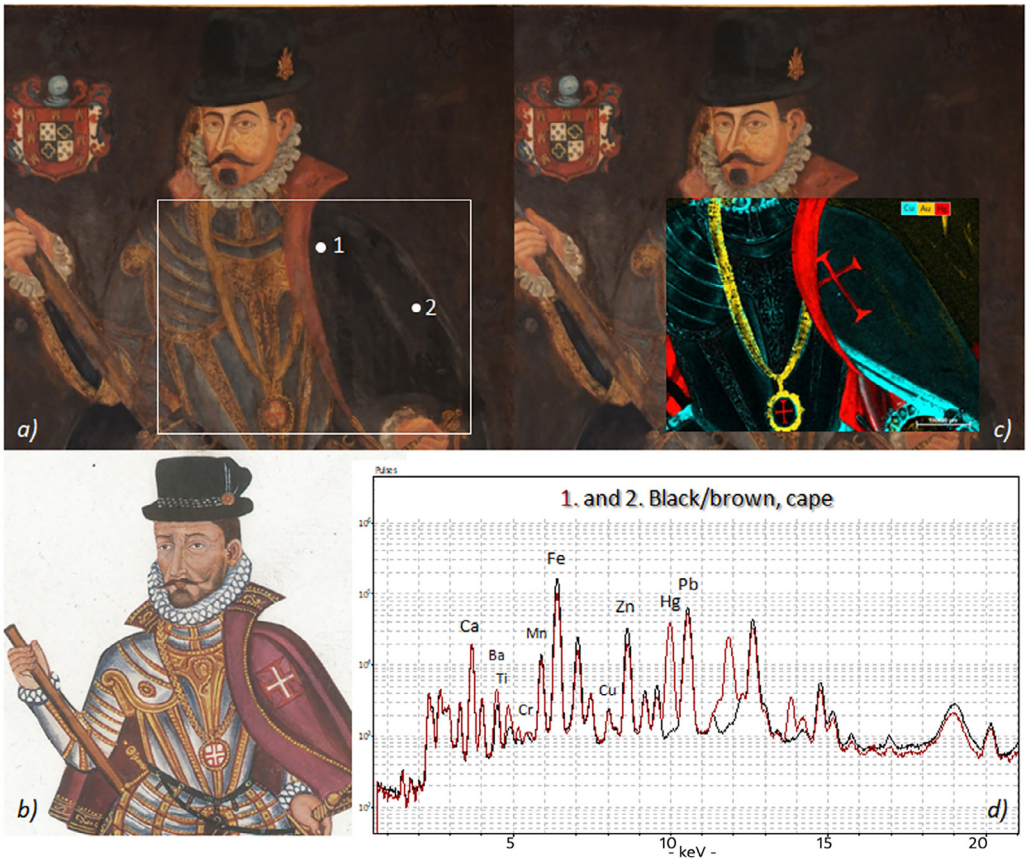

Fig. 6. Portrait of D. Manuel de Souza Coutinho with the mapping of the points analysed by h-XRF (a); detail of a reproduction of the painting dated from 1646 (b); portrait with MA-XRF combined map of the elements Cu, Au and Hg (d); h-XRF spectra of points 1 and 2 (d).

The combined map of copper, gold and mercury obtained by MA-XRF (Fig. 6c) also unveils a cross in the cape of D. Manuel de Sousa Coutinho.

5. Conclusions

The study of these three paintings from the collection of the Portuguese viceroys of India, belonging to the Archaeological Museum of Goa, shows that combining in-situ elemental and molecular analytical techniques (h-XRF, MA-XRF and mobile Raman spectroscopy) is a powerful approach for the material characterization of a painting, as it furnishes a great deal of information that considerably reduces the number of samples to be collected, in the case of being strictly necessary to collect samples, depending on the objectives of the study. In this study, the main pigments were identified only with the use of non-invasive techniques, though it was not possible to confirm the ground layer filler. Also, these techniques did not allow the identification of the binders and varnishes in the paintings.

Moreover, the information produced by these techniques allow mapping the materials on a painting, accessing to the composition of the underlayers and discriminating between original and non-original materials applied, in cases where a painting was profoundly modified in restoration interventions.

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