MATERIAL CHARACTERIZATION OF GILDING AND PAINTING TECHNIQUES OF A POST-BYZANTINE ICON

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Abstract

This study aims to identify the materials used in the production of a post-byzantine icon from the Museum of *Évora's* collection. The icon, representing the "Emperor Constantine and his mother Helen holding the Holy Cross" was once dated as being from the 10th century. Throughout a multi-analytical approach, combining area exams with spectroscopic techniques, this study tried to confirm its actual chronology. The results obtained revealed that it is most likely an icon from the late 17th or 18th century.

Keywords

Orthodox art, Museum of *Évora*, *sgraffito*, punch work, dyes.

CARACTERIZAÇÃO MATERIAL DAS TÉCNICAS DE DOURAMENTO E PINTURA DE UM ÍCONE PÓS-BIZANTINO

Resumo

Este estudo teve como objetivo a identificação dos materiais utilizados na produção de um ícone p*ós-bizantino pertencente à coleção do Museu de Évora. Este ícone, representando o "Imperador Constantino e a* sua mãe Helena manifestando a Cruz", foi datado como sendo do século X. Através de uma abordagem analítica diversificada, combinando exames de área com técnicas espectroscópicas, tentámos confirmar a sua datação. Os resultados obtidos sugerem que será provavelmente um ícone dos finais do século XVII ou do século XVIII.

Palavras-chave

Arte ortodoxa, Museu de Évora, esgrafito, puncionado, corantes.

CARACTERIZACIÓN MATERIAL DE LAS TÉCNICAS DE DORADO Y PINTURA EN UN ICONO POSTBIZANTINO

Resumen

El presente estudio tuvo como objetivo la identificación de los materiales utilizados en la producción de un icono pos bizantino perteneciente a la colección del Museo de Évora. Este

icono tiene representado el "Emperador Constantino y su madre Elena manifestando la Cruz" y había sido datado del siglo X. A través de un abordaje diversificado, combinando exámenes de área con técnicas espectroscópicas, hemos intentado confirmar su datación. Los resultados obtenidos han revelado que será probablemente un icono de finales del siglo XVII o incluso del siglo XVIII.

Palabras clave

Arte ortodoxo, Museo de Évora, esgrafiado, puncionado, colorantes.

Introduction

The Museum of Évora was created by the Portuguese Government in 1915. One of its main cores is the Public Library collection, mainly composed of Friar Manuel do Cenáculo's private collection (1724-1814) which gathers a very diverse and interesting group of objects that help us to understand his notable character. He was a close collaborator of the Marquis of Pombal (1699-1782), minister of King Joseph I (1714-1777) and one of the most powerful men of his time. During the Marquis' term, Cenáculo was chosen for many political offices although the King's death in 1777 and the consequence withdrawal of the Marquis of Pombal, he moved to Beja where he had been nominated bishop in 1770, and dedicated to his activity as a collector and bibliophile. His main passions were books and he contributed to the foundations of many Portuguese libraries, such as the Public Library of Évora, founded in 1805. In this library, willed to the Archdioceses of Évora after his death, he displayed not only books but also his private collection of art and "curiosities", which would later constitute the original core of the Museum of Évora.

Although he did not travel much abroad, he was able to gather a very interesting art collection, mainly through gifts and ordered buys, testified by his many letters. In this diverse collection, there is a unique object: an orthodox icon representing the "Emperor Constantine and his mother Helen holding the Holy Cross" (figures 1 and 2).

Figure 1 - Emperor Constantine and his mother Helen holding the Holy Cross (front), ME211.

Figure 2 – Reverse of the icon "Emperor Constantine and his mother Helen holding the Holy Cross" shows the inscription Jesus Christus Nike, ME211.

Figure 3 – Print of the original engraving plate (19th century), ME2028.

It is not known how this icon came to Évora, who offered it or if it was bought. It is known that it belonged to Cenáculo's collection because he made a print of it in the last book he published: "Graças concedidas por Christo no campo de Ourique", published in 1813 (figure 3). In this book, the image appeared as "*the painting of Constantine and Helen manifesting the Holy Cross that appeared to the Emperor to encourage him to the battle he won, like what happened to our Monarchy Founder. Is in the same Museum of Évora"* (Cenáculo, 1813: Estampa Segunda). The interest of Cenáculo in this icon might be precisely in its iconography because, as it happened to Emperor Constantine before a battle, it is believed that an image of Christ also appeared to the first king of Portugal, D. Afonso Henriques before the important battle of Ourique. Due to this symbolical significance, the cross with the inscription *"In hoc signo vinces"* (similar to the one that appears on the icon) was inscribed in Portuguese coins since the medieval ages until the 19th century. This detail, that is actually different from other icons (figure 4 and 5) with this same theme, might have been what caught Cenáculo's attention, although no reference to this acquisition has been found in his diary or letters.

It would be only in 1882 that there would appear a new reference to this icon, when it was chosen to include the "Portuguese and Spanish Ornamental Art retrospective exhibition" in Lisbon. Back then it was identified as a wood piece, covered in gypsum, gilded and painted with tempera, and dated as a 10th century byzantine icon. This date was established due to the inscription at the back of the icon "IC-XC-NI-KA" (figure 2): *"The characters at the back offer, by their form and arrangement, the elements for the establishment of a date."*

Figure 4 – Icon of Constantine and St. Helen, circa 1800, © Russian Icon Gallery.

Figure 5 – Icon of Constantine and St. Helen, 17th century, © Byzantine and Christian Museum, Greece (BXM: 013233).

Constantine the Great was elected Cesar in 306 of our time, but the use of the phrase: Jesus Christ won *or* winner (Jesus Christus Nike) *only appears, for example, in the coins of Leo V and Constantine VII (813 to 820).*

On the obverse of the coins attributed to John I Tzimiskes (969 to 976) the letters IC-XC-NI-KA can be found bypassing the cross angles exactly as it happens in the painting (…)" (Pereira 1948: 276-277).

Therefore, in the subsequent collection inventories, this information about the materials and the icon dating of the icon would be repeated without being questioned. This might be explained by the lack of knowledge about this kind of art in Portugal, where it is very rare to find artworks like these in museum collections. Probably due to all these reasons, this icon was never analysed before and has mainly been kept in storage, unstudied for over a century.

More recently, amidst a research about Friar Manuel do Cenáculo's collection, interest in this icon was renewed. During carefully analysis of the icon stylistic characteristics, the *sgraffito* decoration of the garments did seem a more recent production, as the ornaments were more similar to those from the 17th and 18th centuries works (figures 4 and 5). However, a more accurate conclusion could only be reached through a material identification of the icon.

Therefore, this study aimed to identify the materials used by the iconographer, in order to confirm the date of its production, but also to allow a deeper knowledge about the production techniques of this work of art.

For the material characterization a multi-analytical approach was used combining area exams with spectroscopic techniques, non-destructively whenever possible.

Description

The depiction of "Emperor Constantine and his mother Helen holding the Holy Cross" is a common representation in orthodox icons since their origin.

Constantine was the roman Emperor responsible for the Edict of Milan (313) that decreed tolerance for Christianity in the empire. This Edict followed his victory over Maxentius, in 312, when Christ appeared to him, guaranteeing his victory if he used the sign of God in the shields of his soldiers (the ChiRho). Helen, his mother, was always a Christian, and was responsible for the discovery of the True Cross, the cross of Christ's crucifixion, in Jerusalem. The depiction of the True Cross became very common, mostly with Constantine and Helen holding it, as it may be observed in this icon, for example.

In this icon, Constantine is on the right side of the cross and Helen on the left, as they are usually displayed. They are wearing red imperial robes, with two different shades, with golden stylized ornaments. These garments' decorations are actually similar to some productions of the Melkite Christians from Syria (17th-18th centuries)[1], but this provenance is not possible to confirm.

1 Information gently suggested by Dr. Mariella Lobefaro (ICONE Association, Biella, Italy).

Both figures are wearing the *loros*, the imperial embroidered scarf. Constantine is depicted with beard, as usual since the 9th century (Teteriatnikov 1995: 174), with an imperial crown and holding a sceptre. Helen is wearing a green scarf covering her hair, with a crown on top. She is holding a red flower, which is not very common in this iconography, and for that no explanation was still found. They are holding a green medallion with a small golden cross enclosed, with the Latin inscription *"In hoc signo vinces"* written in a red band surrounding the medallion. The background is divided in three levels: the red ground, the gilded background and the green sky, with some streamlined clouds. On the top of the icon, there is a small semicircle in blue, with a few stars, that probably represents the Holy Spirit or the presence of God. The names are written in Greek beside each figure.

In other icons with this same iconography, Constantine and Helen are depicted with a Latin or an Orthodox cross, usually with the same height as them (figure 4 and 5). In this case it presents a different cross, which is not common. Interestingly, the figures of Constantine and Helen were many times used in church decorations as a way of displaying the cross, varying its size in relation with the space where it should be depicted. Therefore, in some churches in Cappadocia, due the architecture of the vaults, it is possible to find them holding a small cross, enclosed in a medallion (Teteriatnikov 1995: 175) similar to the one represented in this study that shows an interesting variety of depictions of the Holy Cross.

As for the artistic techniques of this icon, the iconographer used the *sgraffito* technique on the garments and punch work on the saints' halos, on top of the red floor, below the medallion with the cross, and also all around the edges. The artist also exploited the variety of tones and shades through the use of strokes of brush, conferring a more volumetric shape namely in Constantine and Helen's face or in the green sky.

The used techniques and the iconography of this icon follow the traditional rules of production of these devotional objects defined by specific treaties, such as the 18th century treaty of Dionysius of Fourna. These treaties are still used among the Orthodox Church as icons' stylistic characteristics remained mainly unchanged and its contemporary production still obeys the same strict rules, since they preserve their ritual importance. However, in the history of the production of icons, there are small details that would evolve during the centuries, not only reflecting the styles and artistic features of each period, but also the use of new available materials (pigments, dyes, binders).

Experimental

Area exams were used for the first approach to the material characterization: ultraviolet fluorescence photography, to identify possible restorations; radiography, to analyse the inner structure and painting composition; infrared reflectography, to reveal possible preparatory drawings.

Point analysis involved the use of in situ X-Ray Fluorescence using a Bruker Tracer III SD with a Rhodium target and a silicon drift detector (SDD) used as a preliminary elemental characterization of materials used in the icon. The resolution was 152 eV (Mn Ka -FWHM at 6.4 keV) and all the spectra were collected in S1PXRF software developed by Bruker. Instrumental settings were: 20 KeV, 35 μA and an acquisition time of 90 second for each point. A Ti filter was used, which did not allow Rh L line from the tube to reach the sample. This eliminates superimposition between Rh L lines and S, Cl, and P K lines. A vacuum pump was used to increase the sensibility of light elements. The spot of the analysis is 7 square millimeters. ARTAX 7.0 was used for qualitative spectra analysis.

Micro samples were collected and mounted in epoxy resin for cross-section observation. The cross-section observation by Optical Microscopy (OM) was performed with a stereo zoom microscopy Leica M205C, with camera Leica DFC290HD for image acquisition. The material characterization of the different strata, including ground layers, bole, golf leaf and paint layers, was performed with a Scanning Electron Microscope HITACHI S-3700N with energy dispersive X-ray spectrometry with a Bruker Xflash 5010 SDD spectrometer (SEM-EDS). The samples were also analysed by a confocal μ-Raman spectroscopy instrument from Horiba Xplora (638 and 785 nm laser, spatial resolution 1 μm, spectral resolution 5 cm^{-1}, maximum power 1 mW, CCD with 1026 x 256 pixels). For the analysis of the green colour of the cross, Fourier Transform Infrared spectroscopy (μ-FTIR) was performed using a Bruker spectrometer Tensor 27 model at medium infrared region (MIR). The spectrometer uses a microscope Hyperion 3000 controlled by software OPUS 7.2 from Bruker, with a Mercury Cadmium Telluride detector. The sample was analysed in transmission mode using a 15x objective and a diamond compression microcell EX'Press 1.6 mm, STJ-0169. The spectra were performed in the 4000-600 cm^{-1} region, with 64 scans and spectral resolution of 4 cm^{-1}.

The green and the blue colour samples were analysed with pyrolysis gas chromatography mass spectrometry (PY-GC-MS) with TMAH derivatization. Liquid chromatography with diode array detector and mass spectrometry (LC-DAD-MS) was used for the analysis of organic material.

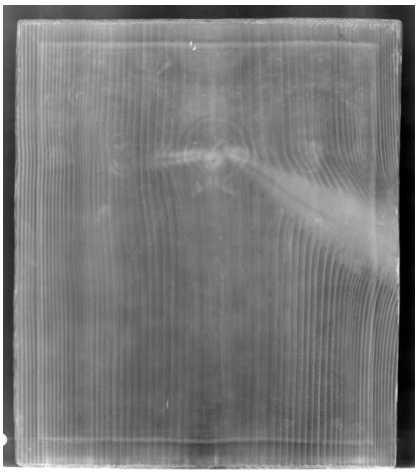

Figure 6 – Radiography of the icon.

Results and discussion

Support

Given that the icon is painted in both sides, radiography was performed to analyse the support (Sotiropolou, 2010:879). As seen in figure 6, the support is composed of a single wood panel, without assemblies or slats, which often appear on icons to prevent cracking due to the hygroscopic movements of the wood. The radiography also allowed us to find that the support is in a very good conservation condition, without cavities provoked by wood boring insects.

Ground

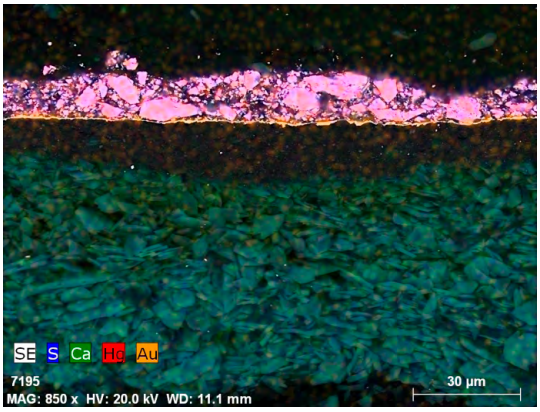

Figure 7 - SEM-EDS of ground layer of CH7 sample.

By optical microscopy (OM) it was visible that the ground layer has white appearance and was applied over the entire front surface. By OM it was not possible to identify the different strata in this white layer. The higher thickness was around 180µm. Although the treaties (Dionysius of Fourna, Francisco Pacheco, Filipe Nunes) recommend the application of several strata, they can never be distinguishable through this technique (Barata, 2012:44). This could be due to the use of sandpaper and the successive hot applications of the material which promotes a fusion of the materials from different applications (Cardoso 2014:173).

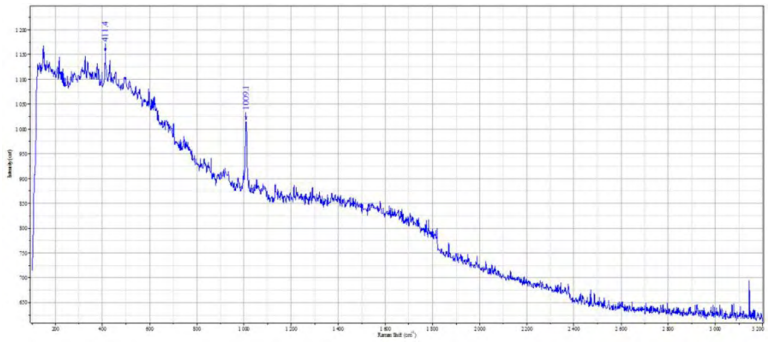

Figure 8 - µ-Raman spectra of gypsum of CH2 sample Peak identification: 414m, 1008vs (Wavelength 785 nm, 6.5 mW, 10 s).

Nevertheless, SEM-EDS analysis confirmed the presence a layer composed of calcium associated with sulphur, suggesting calcium sulphate, with lamellar homogenous particles (figure 7). Some Si, Al and Cl were also identified but in very residual proportions. Due to its natural, origin those minor elements can be associated to gypsum (Coroado, 2013:82). Although some of them can be helpful in material provenance studies (Gómez, 2005:48), these elements could also be residual from the technical procedures, such as polish materials (Le-Gac 2015:73).

The μ-Raman results showed the presence of dihydrate calcium sulphate ($CaSO_4 \bullet 2H_2O$) as showed in figure 8.

Above the ground layer the artist projected the composition with a preliminary drawing, visible through infrared reflectrography. The drawing was evident on the flesh tones only (faces and hands of the figures). The fact that the drawing is not visible in other areas of the icon does not mean that it does not exist. There are limitations related to the reflectography technique (Weiner 1999) and it is just possible to detect drawing in areas where there is no bole layer and gold leaf above it.

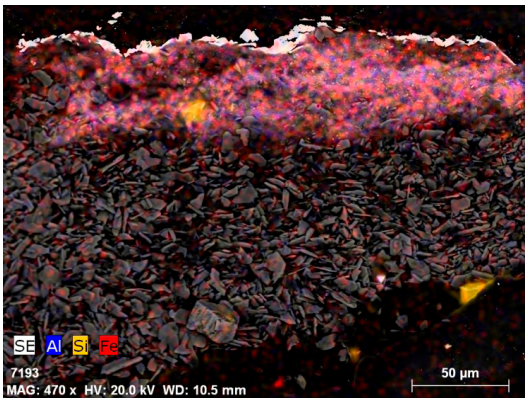

Figure 9 - SEM-EDS of bole layer of CH1 sample.

Bole

The bole layer has an orange colour and is very homogeneous. The layer is applied over the ground, except on the flesh areas. The bole layer was applied even under the cloudy green sky, which is not gilded, which could be interpreted as regret during the gilding execution or just a way to simplify the whole process. It was perceptible by EDS that this layer is composed mainly of iron (Fe), aluminium (Al) and silicon (Si) (figure 9) and a very dispersive quantity of particles of titanium (Ti) were also traceable, usually associated in clay minerals.

Gold leaf

The gold leaf was applied over the bole layer except in the cloudy sky area. An exception was identified in the cross-section of the green cross in the middle of the icon, which is gilded although no bole was detected. This might be due to the proximity of the flesh tones, or it

could be a regret: the artist projected not to gild but, at the end, decided otherwise. The SEM-EDS spectra collected from the gold alloy show the presence of gold (Au) and silver (Ag). The semi-quantitative analysis undertaken in three different points of three samples revealed a mass proportion of 95,5% of gold and 4,5% of silver in average. This average demonstrates the use of a very high purity gold leaf of about 23 karats.

Paint layer

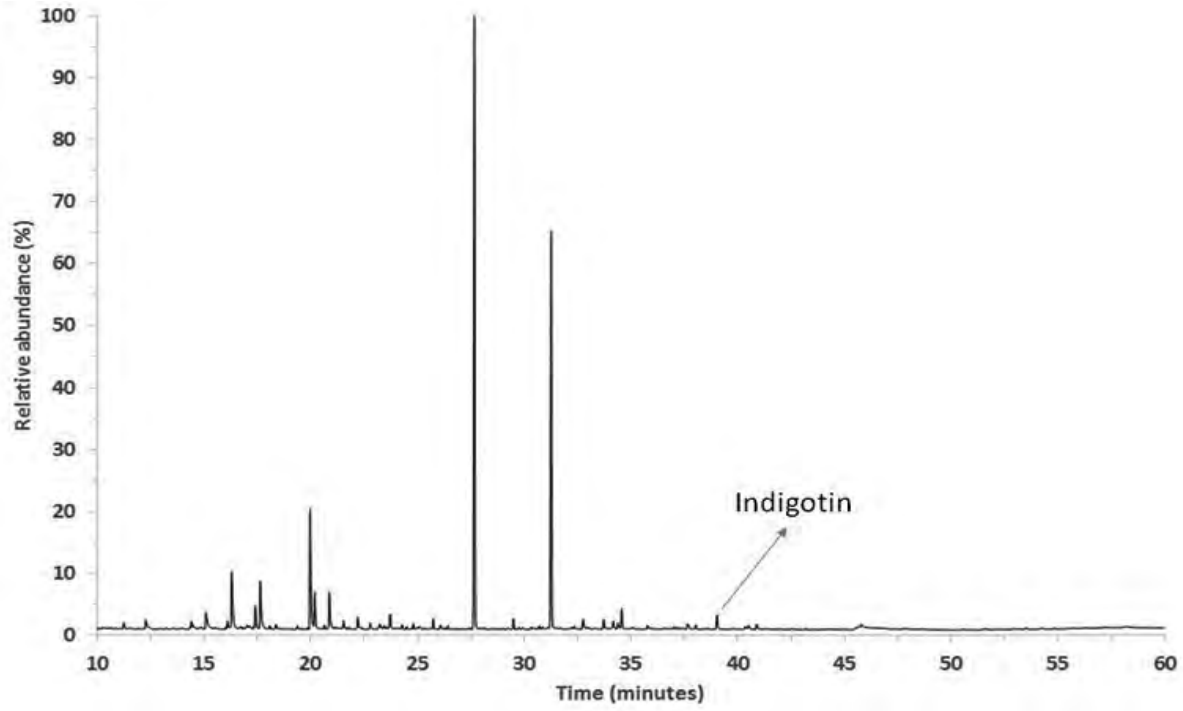

Figure 10 - PY-GC-MS chromatogram of blue sky sample. Peak identification as follows: 1-azelaic acid; 2 – palmitic acid; 3 – stearic acid; 4 – indigotin (TMAH derivative).

The icon was painted after the gilding process. Gold was left in the middle part of the background, in the halos of the figures and in the frame. In these cases, the punch work was used to create different shades of gold.

The study involved the characterization of all the colours used: green, blue, red, white and black. The flesh tones were not considered because by UV photography was possible to perceive that Helen's face was overpainted and Constantine's face was in a very good condition for sampling (Sandu, 2006).

Two different greens were used, one for the cloudy sky and another for the central cross and some other details. The first one is opaque and mixed with white to create different grades of light and shadow. By SEM-EDS, a copper-based pigment was identified in a mixture with lead white and calcium carbonate. Malachite was identified by μ-Raman spectroscopy. The second green is translucent, used as a lake, allowing the gold layer to appear below. Through SEM-EDS a copper-based pigment was identified but analysis by *μ*-Raman and

μ-FTIR were not conclusive about the chemical nature of the pigment, for it could be, for example, copper resinate.

The white colour present in the *loros* is calcium carbonate, identified through SEM-EDS.

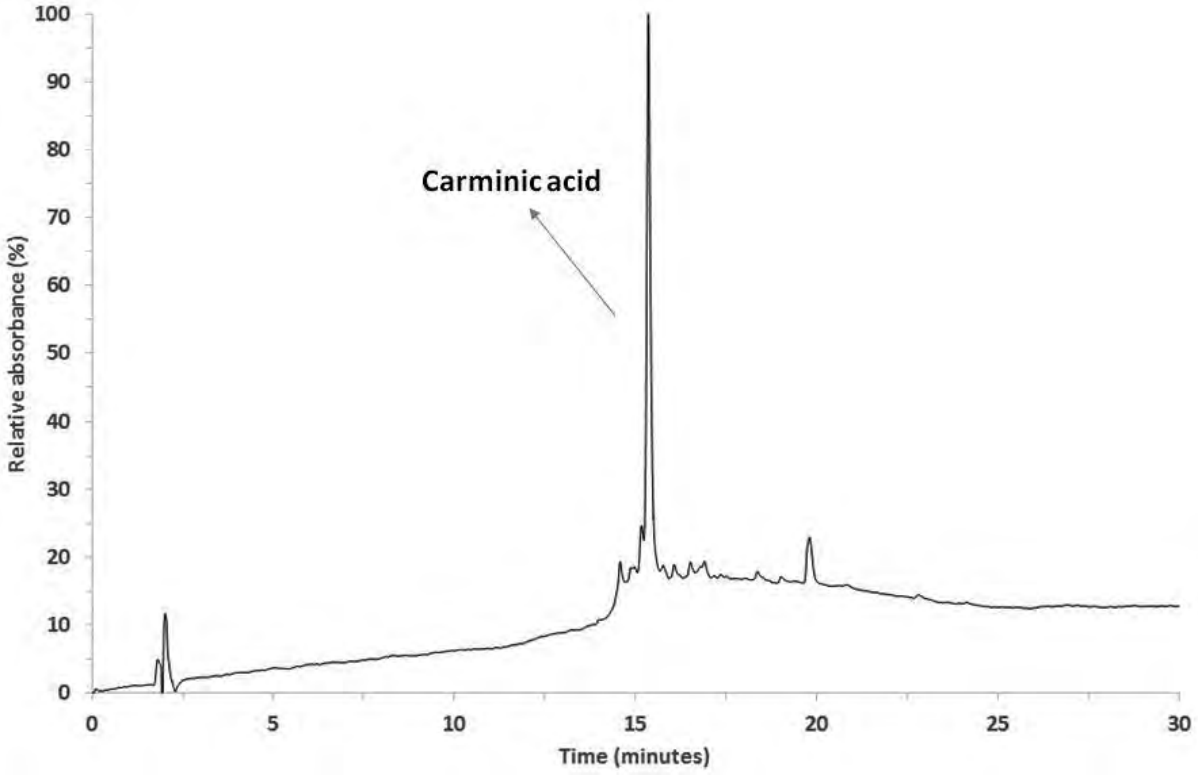

Figure 11 - LC-DAD-MS chromatogram of the red sample from Constantine's garments. Peak identification: 1-carminic acid.

For the blue colour at the round starry sky at the top, the results by SEM-EDS did not reveal any element compatible with an inorganic blue chromophore and therefore were not conclusive regarding the blue pigment, allowing only the identification of the white pigments used in the mixture: lead white and calcium carbonate, based on the identification of Pb and Ca and the absence of S. PY-GC-MS allowed the identification of indigo, confirming the organic nature of the blue colour (figure 10).

Three different reds were analysed: the floor, Helen's garment and Constantine's garment. The first one was identified by SEM-EDS as an aluminosilicate with iron oxide in a mixture with lead white and calcium carbonate. The red in Helen's garment is composed of mercury, which suggests the use of vermillion. The red in Constantine's garment was analysed through LC-DAD-MS due to its translucent appearance that suggested an organic compound. The results showed the presence of carminic acid, the main constituent of cochineal lake (figure 11). Kermesic and Flavokermesic acids are minor constituents, acting as markers of Polish and Armenian cochineal (Eastaugh, 2004:118). Since no evidence of these acids was found, it is possible that the type of cochineal used was South American cochineal, which

was only introduced in Europe in the late 16th century by Spanish navigators. The pigments and dyes identified in the paint layer are in consonance with other analytical studies about icons (Avillez, 2008; Karapanagiotis, 2008; and Daniilia, 2002).

The binders were analysed in the green area of the cross and the blue sky. No evidence of proteinaceous binder was found through *μ*-FTIR. Through PY-GC-MS the presence of linseed oil was identified based on the palmitic to stearic (P/S) ratio, which was 1,57 for the blue sky sample and 1,71 for the green sample.

Conclusion

The results of the analyses performed in this study allowed to verify that all the materials used by the iconographer are frequent in the production of icons throughout the last five centuries. The material characterization of this icon of "Emperor Constantine and his mother Helen holding the Holy Cross" identified gypsum in the ground layer and aluminosilicate containing iron in the bole layer. In the paint layer, with linseed oil as binder, white lead, calcium carbonate, iron oxide, vermillion and malachite were identified as pigments and indigo and cochineal as dyes used in the production of lakes.

Therefore, the identification of what seems to be South American cochineal, introduced in Europe by Spanish navigators, locates the execution of the icon after mid-16th century, revoking the 10th century date originally attributed. Overall, this data corroborates the art historian's opinions that according to its stylistic characteristics, like the garments' ornaments, this is a post byzantine icon probably produced between the 17th and the 18th centuries, perhaps a contemporary work of Friar Manuel do Cenáculo.

This study also confirmed the importance of the material characterization as an important tool for a more accurate knowledge of artworks in museum collections, helping to fulfil one of the main purposes of these institutions: the research.

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