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Archaeometric Non-Invasive Study of a Byzantine Albanian Icon

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Authors' contributions

This work was carried out in collaboration between all authors. All authors read and approved the final manuscript.

Research Article

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ABSTRACT

The present study is part of a project concerning the characterization of Albanian Byzantine and post-Byzantine icons, through the identification of pigments, of painting technique and the state of conservation of the artworks.
The Albanian iconographers produced an incredible number of icons from the 14th to the 19th century and about 6000 of them are conserved in the Museum of Medieval Art of Korça (Albania). The study of these artworks is conducted by non-destructive methods, X-ray fluorescence, visible light reflectance spectrophotometric measurements and UV fluorescence analysis, according to an analytical procedure developed in our Laboratory. With this procedure we can recognize the inorganic pigments from their typical features. Moreover, the study of the optical properties of paintings is of fundamental importance for correct restoration. Eleven areas were selected in order to carry out the measurements. The present work, concerning the study of an icon of 14th century, has allowed us to recognize the palette and the painting technique used in this artwork by the anonymous painter. We have also compared this icon with other Byzantine works housed in the same Museum.
Aims: The main purpose of this research was to identify the original pigments, those added during restoration interventions, the restored areas, as well as the painting technique used by the anonymous painter; and together provide useful information to art historians and restorers.
Study Design: The present study is part of a project concerning the characterization of

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Albanian Byzantine and post-Byzantine icons, through the identification of pigments, of painting technique and the state of conservation of the artworks.
Place and Duration of Study: Museum of Medieval Art of Korça, Albania, between June 2008 and July 2009.
Methodology: The study is conducted by non-destructive methods, namely, X-ray fluorescence, visible light reflectance spectrophotometry and UV fluorescence analysis.
Results: The non-destructive study allowed us to identify the pigments' palette of the anonymous 14th century artist. For the ground, the painter employed calcium white coloured with brown ochre, ivory/bone carbon and copper based compounds. We also detected the previously restored areas of the icon and the pigments utilized by restorers.
Conclusion: The set of non–destructive techniques employed in this study has proved to be very useful for recognizing pigments, including those not detectable with certainty by means of X-ray fluorescence.

Keywords: Byzantine icons; Albanian icons; X-ray fluorescence; UV fluorescence; reflectance spectrophotometry.

1. INTRODUCTION

Entire Albania maintains countless artistic treasures, especially related to the Byzantine and post-Byzantine icons and wall paintings. The Museum of Medieval Art of Korça, located in the southeast Albania, near the border with Greece, in addition to many other notable artistic objects, keeps over 6000 icons. Among the icons, we mention the masterpieces of leading painters such as Onufri, Onufri Qiprioti, Konstantin the Teacher, Konstantin Jeromonaku, Konstantin Shpataraku, David of Selenica, and the Çetiri brothers, a family of painters from Grabovë, a village in the Korça district.

Onufri, who was a leading exponent of icon and mural painting of 16th century, is the most celebrated painter in this museum. He painted in many churches in Elbasan, Berat, Kastoria (Greece), Zerze (Prilep-Macedonia) and elsewhere. Albanian artworks, for various reasons, are not sufficiently studied in terms of preparation technique and materials used. Some research, on Albanian Byzantine and post-Byzantine icons, have been done in recent years, which led to the publication of the results [1,2,3,4,5], but these are only few studies considering the large amount of not yet studied artworks held in the Museum of Medieval Art of Korça and in the other Albanian art sites.

This article reports the study of a Byzantine icon, painted on a wooden panel by an anonymous artist of the 14th century, with the tempera technique. The wooden table is carved out forming a frame, adorned by a band in red. This icon, shown in Fig. 1, is famous and known as the *Albanian Mona-Lisa* [6].

Fig. 1. Anonymous, 14th century, *The Archangel Michael*, 108.5x83.5 cm, inventory number 2764, painted for the church of Ascension in Mborje (Korça district). Numbers indicate the spots where X-ray fluorescence and spectrophotometric measurements have been performed

The image represents the archangel Michael, against the originally gold ground, in armour and with his sword in his right hand, as guardian of the church. The helmet, held in his left hand, completes the traditional iconography of the archangel. On the green-grey helmet, between the arms of the cross, in white lettering, is the inscription:

IC XC
N K

indicating the victorious Christ, **I(HCOY)C X(PICTO)C N(I)K(A)** (see detail in Fig. 2).

Fig. 2. *The Archangel Michael*, detail showing the inscription on the helmet and the carved frame

In addition, from the detail shown in Fig. 2, it is possible to observe, between the damaged areas and the detachments of the paint film, the brown colour of the ground. The conservation state of the painting is fairly good, but the icon has been restored in the past without performing analyses on pigments and preparatory layers.

The main purpose of this research was the identification of the original pigments used by the painter, those added during restoration interventions, the restored areas, as well as the painting technique used by the anonymous painter. The study should provide useful information to art historians and restorers.

The comparison, with previous studied icons of 14th and 15th century [3,4], is of interest in highlighting analogies and differences in the iconographic Albanian technique.

2. METHODOLOGY

Given the historical, artistic, religious and cultural importance of this icon, we decided to use non-invasive methods in this study. A variety of areas were chosen for the purpose of identifying the pigments and the painting techniques of the anonymous artist. The following archaeometric techniques were applied.

2.1 Optical Microscopy and Macrophotography

Optical observation and photographic documentation was achieved using a Dino-lite portable digital microscope and a Canon EOS 350D camera equipped with a Canon Zoom Lens EF-S 18-55 mm.

2.2 UV Fluorescence

This analysis was carried out using a ceiling light with four Sylvania black light-blue F18W/BLB-T8 tubes. The digital camera used for recording images is the Canon EOS 350D without barrier filter. It is a non-destructive superficial analysis that identifies the presence of one or more film-forming substances, such as varnishes applied on the work (resins, oleoresins, proteins, etc.) and, generally, every previous intervention.

This technique allows assessing the condition of the paint, enhancing the presence of restorations, biological attacks, even when they appear indistinguishable to the naked eye. Also, it can give some information on pigments that may have their own particular fluorescence, for example: the yellow green of arsenic trisulphide, the brightness of lead white, the darkening of iron oxides.

2.3 X-ray Fluorescence Spectrometry

Elemental analysis was performed using a Lithos 3000 portable system and an appropriate Lithos program by Assing to process the data. The apparatus consists of a molybdenum tube, a zirconium filter and a semiconductor silicon (Li) detector, cooled by Peltier effect. The operating parameters were: 25 kV, 0.1 mA, and 240 seconds of acquisition time. The elements with the highest intensity detected on the paintings, such as lead, iron or mercury, have been used as internal standards.

2.4 Reflectance Spectrophotometry

Reflectance spectrophotometric and colour measurements have been performed using a Minolta CM-2600 portable spectrophotometer, provided with a Xenon lamp to pulsate the light on the sample surface and with an integrative sphere inside the apparatus. Light is reflected by the pigment with an angle of 8°. It is captured by a silicon photodiode that measures the colour spectrum between 360 and 740 nm with an interval of 10 nm. Colour coordinates are based on the CIEL*a*b* system using an illuminant D65 with an observer angle of 10º. In this system, L* represents colour lightness, while a* and b* are the coordinates of chromaticity. Coordinates +a* and -a* indicate red and green values, while +b* and –b* indicate the yellow and blue values, respectively.

The methodology, followed in this work, was set up during the studies conducted on various kind of paintings, both frescoes or paintings on different supports (paper, parchment, stone, copper plates, canvas and wood panels). In these studies it was always pursued a non-destructive approach, based on the interaction between electromagnetic waves and the artworks in question. This has led to the achievement of a methodology based on the use of different complementary techniques. The processing and comparison of the data obtained have led to the databases about the behavior of pigments, mainly inorganic, and the different instrumental responses.

In particular, by processing X-ray fluorescence data, it is possible to determine the elemental composition of the investigated materials, obtaining indication of the nature of the pigments and of the underlying layers, until the ground. The latter should correspond to needs related, also, to the nature of the different pictorial supports. On the other hand, the examination of macro and UV images gives information about the conservation state of the artwork and the restoration interventions. Some suggestions on the nature of the pigments, including also organic pigments, such as several lakes and indigo, can be obtained. The reflectance spectrophotometry is able to give information on the composition of the paint layer, highlighting, also, any pigment mixtures. For the evaluation of the reflectance curves we used a second-derivative processing, with the opposite sign, which focuses the chromatic behavior of pigments and allows a better comparison with the curves of reference pigments.

3. RESULTS AND DISCUSSION

Looking at the edges of the table and at the cracks in wooden planks, it is evident that no intermediate canvas was used beneath the painted surface.

The inorganic pigments employed for the painting could be identified by means of the principal characteristic elements, their relative abundance and by comparing their reflectance spectra with the literature data and with those of a pigment database, developed in collaboration with the *Soprintendenza per i Beni Architettonici e Paesaggistici della Liguria*.

The results, helpful to identify the pigments and the painting technique of this artwork, are discussed, showing the contribution that each technique has provided.

3.1 UV Fluorescence

Under the UV illumination, areas containing cinnabar or vermillion become purple; iron oxide presents a typical darkening behaviour, clearly showing the painting technique used by the artist, with strokes applied to form continuous layers as painting basis and thin strokes as overlayers (Fig. 3).

The presence of lead white is clearly evidenced by the strong response to UV illumination [7]. The use of arsenic sulphide, orpiment, is indicated by a greenish yellow UV fluorescence [4]. The fresh, more or less recent varnish appears as black areas, denoting restoration interventions [8].

The flesh tone was obtained by the use of brown and dark-brown brushstrokes and a series of highlights, consisting of almost pure lead white (Figs. 3 and 4), in addition to the brown colour of the ground.

Looking at Fig. 4, where a detail of *Archangel Michael*, corresponding to the face and the background, is shown, we can make some considerations, regarding the different areas:

- In the areas where the ground layer is emerging, due to the loss of metal coating, we can notice the preparatory drawing;
- The presence in the background of iron oxide (bole residue?) can be seen here and there, in the areas with lower UV fluorescence;
- Areas, containing lead white, are revealed by the strong UV fluorescence;
- Cinnabar strokes, under UV radiation, change to a red-purple tone;
- Dark brown colour reveals the presence of iron oxides that darken under UV radiation.
- The green colour, where copper compounds are present, turns from green to black.

Fig. 3. UV image of *The Archangel Michael*

Fig. 4. Comparison between visible light (left) and UV (right) images. Detail from *The Archangel Michael*

Observing the areas, corresponding to detailed wings of the Archangel, in the Fig. 5, one can note the overlap of the brushstrokes with various shades of colour formed by overlays of arsenic sulphide to cinnabar. The mantle is obtained using cinnabar with broad dark lines to give the shadows that, under the UV radiation, appear to have a different response with a darker colour.

The colour of the wings (Figs. 5 and 6) was obtained with ochre, as a base, realgar (dark red strokes) and orpiment (yellow points and lines); the latter giving a green fluorescence in the UV image. The blade of the sword and the areas containing iron oxides become darker under UV light.

3.2 X-ray Fluorescence

From the data obtained by XRF measurements processed as discussed in the literature [9], we could detect the presence of various elements in the different layers of the painting.

The experimental data, obtained by X-ray fluorescence measurements, are collected in Table 1 and visually summarized in Fig. 7, where the occurrence of the principal elements - detected on the basis of the counts measured for the main peak of each element - in the different analysed spots is plotted. In the case of simultaneous presence of arsenic and lead, their principal peaks As Kα and Pb Lα are superimposed at about 10.5 keV. In order to obtain a correct evaluation, in the case of simultaneous presence of the two elements, we considered the counts of Kβ (11.73 keV) and Lβ (12.61 keV), respectively. The counts of the peaks of the elements have been normalized for graphical presentation.

Fig. 5. Comparison between visible light (top) and UV (bottom) images. Detail showing *The Archangel Michael's* clothing and wing

Barium was found in six spots (1,2,4,6,9,10) and zinc in three (6,9,10), thereby indicating the occurrence of restoration interventions. In fact, these elements have to be attributed to white pigments $BaSO_4$, *blanc fixe* [10] and ZnO, zinc white [11] or lithopone, white pigment developed from 1870, mixture of barium sulphate and zinc sulphide: such pigments have been used starting from the 19^{th} century. The visual observation of painting's surface permitted the detection of extensive colour detachment (spot 1); we can observe that, from these data, we achieve the elemental composition of the ground, containing mainly phosphorus, calcium and iron. Strontium is generally low or absent (this is another difference between the two contemporary icons of 14^{th} century, since the *St. Nicholas* has significant values of strontium [4]).

Fig. 6. Comparison between visible light (top) and UV (bottom) images. Detail showing *The Archangel Michael*'s red wing. Yellow lines are obtained by using orpiment. Red strokes are probably obtained with realgar

Table 1. The counts of the main peaks of the elements detected in the analysed areas of *Archangel Michael*

Spot	Colour	P	K	Ca	Ba	Mn	Fe	Co	Ni	Cu	Zn	Au	Hg	As	Pb Lα	Pb Lβ	Sr
1	Ground	9		868	6	5	54		3			4		14 Ka			30
2	Brown hair	4		126	11		1600						1992		300	100	32
3	Dark flesh		12	167		20	730						1140		655	376	40
4	White flesh	10	10	20	14		103	17		11			610		4537	2950	
5	Flesh tone			10		15	130	13		13			945		4245	2670	
6	Brown	6		333	25	4	171			7	58			tr	56	28	
7	Red brown	4	21	265			2655			10			45	275 Kβ	tr		20
8	Red		7	43		6	32	6	4				2390		820	512	
9	Dark green			74	8	16	106			5400	56		6	32 Kβ	1187	786	
10	White			15	2	5	48	11	8	10	10				5050	3435	
11	Light brown	12		640		3	77			6					23		25

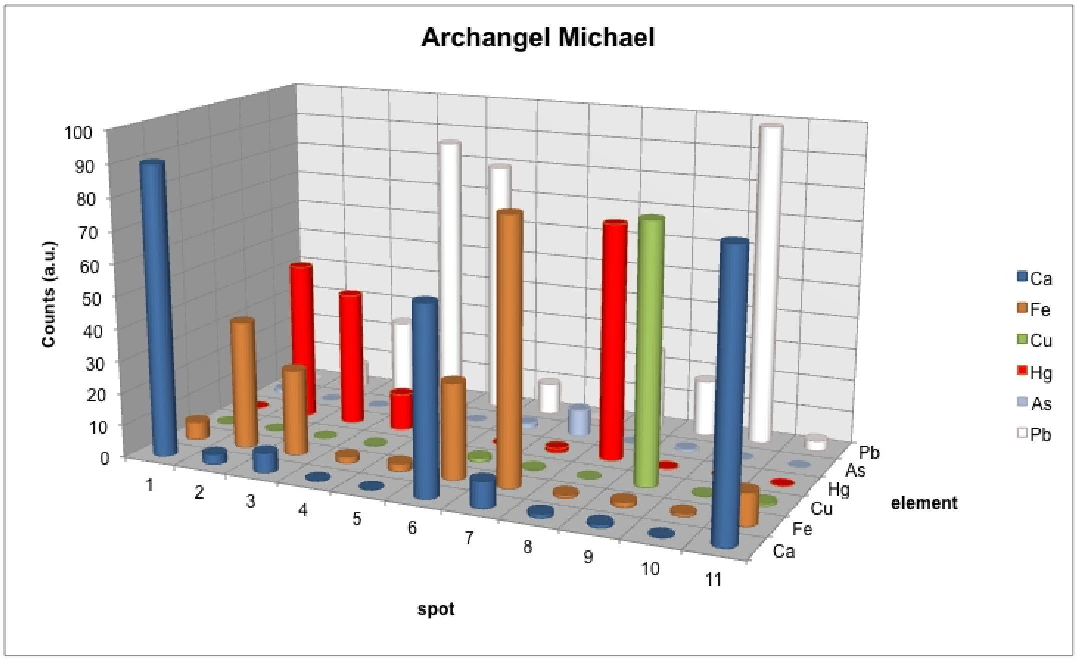

Fig. 7. The occurrence of the most significant elements in the 11 analysed spots of *The Archangel Michael*

One can note the presence of arsenic in spots 1, 6, 7, 9 and gold in spot 1: the latter is a remain of the upper layer, formed, probably, by a gold leaf, as it was in the Byzantine traditional technique. The presence of phosphorus, in addition to calcium, suggests the use, in the preparative layer, of ivory/bone black. The presence of calcium, in correspondence of light brown colour of spot 11, indicates the use of calcium white for the particular shade. Moreover, considering the iron content (iron is accompanied by manganese in this icon, indicating the use of a brown earth), we can note that the painter used it, in small quantity, to obtain the colour of the ground. As we found the same ensemble of elements in an icon, St. Nicholas, of the same period coming from the same church [4], we took the suggestion of a traditional use of brown earth for the purpose to obtain a dark ground for the next paint layer, in the style of that age [12]. In addition the presence of iron derives from successive paint layers, in particular to give brown and dark flesh tones (spots 2,3,7).

The association of iron and arsenic is found in correspondence with the dark red colour of the wings (spot 7). The presence of arsenic indicates the use of an arsenic sulphide (realgar, As_4S_4, or orpiment, As_2S_3). Orpiment and realgar are known to occur, in some Orthodox countries, in paintings of icons during a long period of time, lying from 12th to16th century [13]; recently their use has been reported in 14th and 15th century Albanian icons [4]. Noteworthy is the association of these pigments with a copper compound in correspondence of the armour (spot 9).

Mercury is present in many areas, indicating the use of cinnabar (HgS), to obtain shades in brown hair and the intense red colour of the mantle of the Archangel. To achieve flesh tones and to give highlights, lead white was also used (spots 4,5,10).

3.3 Reflectance Spectrophotometry

The reflectance Vis spectrophotometric measurements were made on all the areas examined by X-ray fluorescence. On this basis, it was possible to confirm some results obtained by the other techniques or to establish new as following:

3.3.1 Red and flesh tones

The hue range of red pigments varies from light orange to dark red/brown. The red inorganic pigments used in this icon were red ochre, realgar and cinnabar (HgS). In some cases, red tonalities were obtained from appropriate mixtures of these pigments with lead white. There is no evidence of the use of lakes.

The graph obtained by data processing in the first derivative [14] improves the reading of data, and highlights the behaviour of the examined reflectance spectra. The method may allow identifying the pigments that mainly contribute to the perception of colour. The technique, which offers the best results in the examination of mural paintings, where there is little or no paint binder on surface film, poses more difficulties in easel painting, as in almost all the icons of the Korça Museum, veiled with thick, unequal and misleading varnishes. For a better comparison, we used the second derivative of the curves, as discussed in [3].

The Fig. 8 shows the results obtained by measuring three different red spots of *The Archangel Michael*, 4, 7 and 8, and comparing them with the spectra of cinnabar [15], red ochre [16] and red lead [17].

Cinnabar is revealed in four of the examined areas. The contemporaneous presence of other pigments, with their less or more strong spectral response, is evident. One can explain the XRF signal of copper found in the spot 4; the presence of a copper compound is revealed by the deformation of the second derivative curve in the range 400-500 nm. The curve, corresponding to the dark red wings (spot 7), reflects the predominant presence of red ochre and ivory/bone black. Finally, the red tone of the mantle was obtained using cinnabar with lower amounts of lead white (note the slight shift of the peak in the second derivative curve toward higher wavelengths).

3.3.2 White

Areas containing lead white are clearly evidenced by the strong response to UV illumination [7] (Fig. 4) and by XRF in various spots. This compound was used both as white colour and in mixture with other pigments. White pigments ZnO and $BaSO_4$ (or lithopone), as already remarked, indicate restoration interventions.

In this icon we did not found the presence of a filler layer formed by lead white [1] or the presence of a second thick white or slightly coloured layer, known as *imprimatura* [16].

As one can see, the ground (spot 1), composed primarily of calcium white, behaves differently from that of lead white [18], as highlighted, in Fig. 9, by the second derivative shapes. With regard the curve of spot 10 (white wings), we can note two important signals: in the shortest wavelengths the deformation is linked to the presence of copper and cobalt, while, in the red region, the peaks are relative to the underlying paint layer.

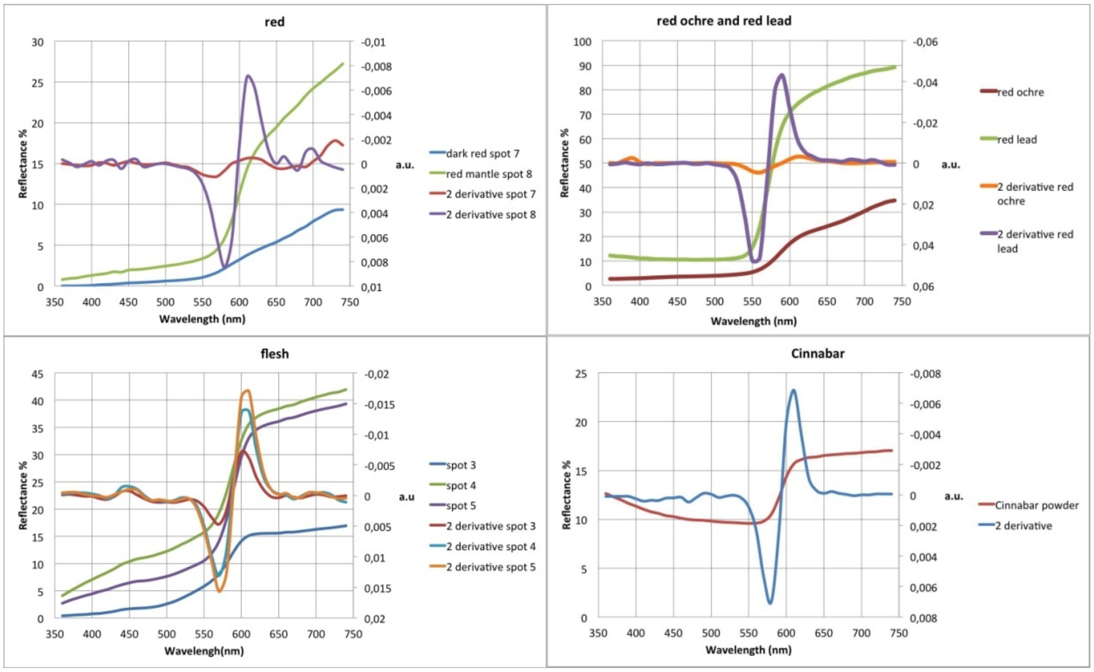

Fig. 8. Reflectance curves referring to *The Archangel Michael's* two red spots 7 and 8, and three flesh tones, spots 3, 4, 5, comparing them with the spectra of red ochre, red lead and cinnabar and their second derivatives

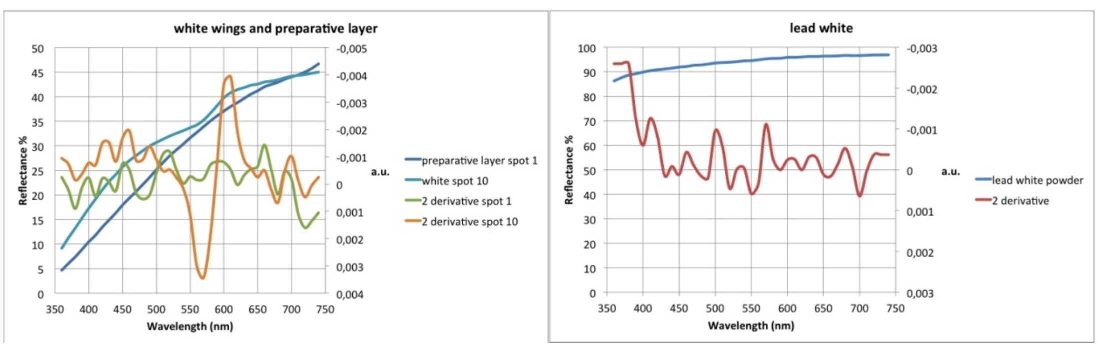

Fig. 9. Reflectance curves referring to spots 1 and 10 in *The Archangel Michael*, compared with the curve typical for lead white and its second derivative

3.3.3 Dark Green

The XRF measurement on the dark green of the armour reveals the high presence of copper with iron, arsenic and lead. The colour should be attributed to the strong presence of copper, while the darkness can be related with an iron based pigment, like the burnt umber [16], but it is also possible the use of carbon black, not identifiable by means of this technique.

It is not possible, by the simple comparison with typical reflectance spectra of malachite and verdigris, to assess the actual nature of the copper based pigment. We have also considered the possibility of the use of a blue pigment, like azurite, with the yellow orpiment, to obtain the green tonality. The use of *Vergaut* (a mixture of orpiment and indigo) in medieval paintings is well known. On the other hand, it is possible to recognize a close similarity between the mixture of pigments found in another previously analysed Albanian icon, St. George with scenes of 15th century [4] and that of the present work (see in particular the second derivative of the reflectance spectra, on the left of Fig. 10).

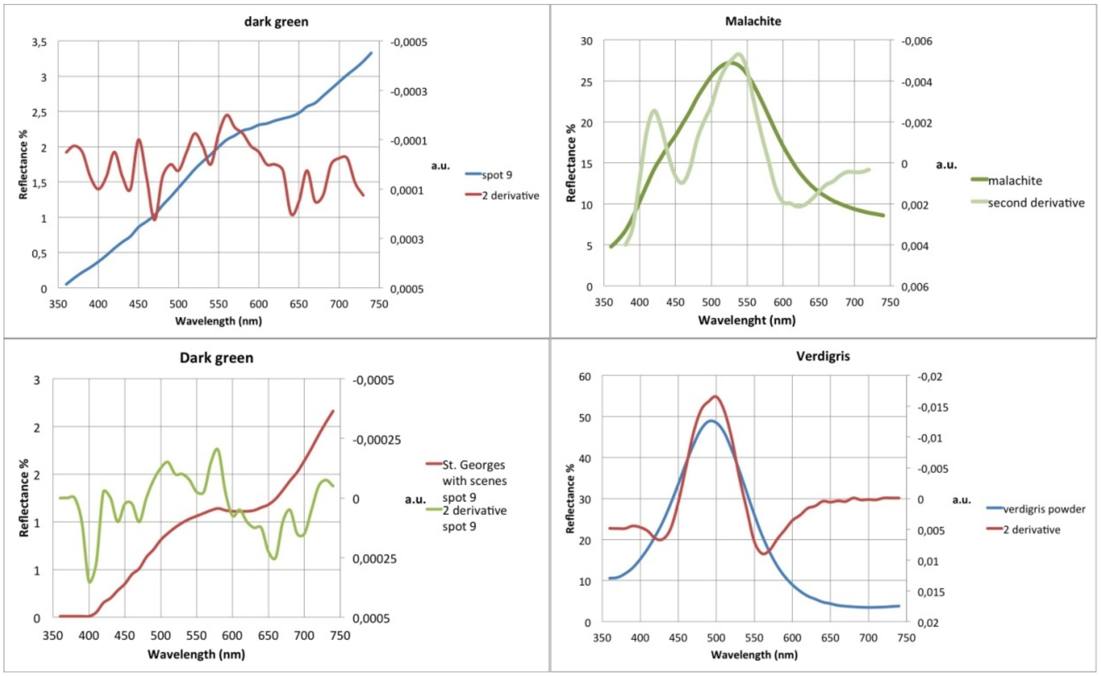

Fig. 10. Reflectance curves (on the left) referring to spots 10 in *The Archangel Michael* and 9 in *St George with scenes* [4], compared with the curves typical for malachite and verdigris and their second derivatives (on the right)

3.3.4 Brown

We have compared three different brown areas (Fig. 11), corresponding to the spots 2 (brown hair), 6 (brown sword) and 11 (light brown of the sword handle). Appropriate layers containing ivory/bone black, calcium carbonate, ochre, red lead and cinnabar give the different tones. Looking at the Fig. 11, we can infer the high content of cinnabar used to render the hair hue (spot 2). One can see the same feature for points 6 and 11, where the slope of the reflectance curves is dominated by the ratio of calcium carbonate and black in the mixture. The presence of red ochre, on the layer under the sword handle (spot 11), is shown by the slight curvature around 570 nm.

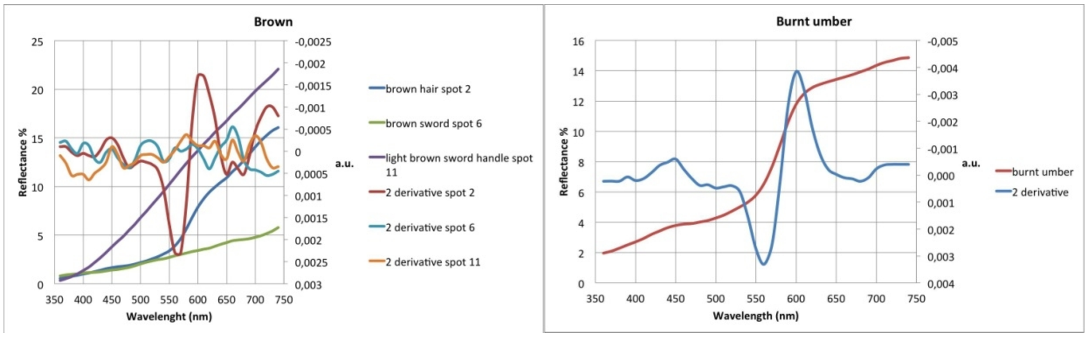

Fig. 11. Reflectance curves referring to spots 2, 6 and 11 in *The Archangel Michael* and their second derivatives compared with the spectra of burnt umber

4. CONCLUSION

The non-destructive methods, that we have used, provided information to identify the pigments and the preparation of the icon worked by the anonymous artist. For the ground, the painter employed calcium white coloured with brown ochre (as suggested by the contemporary presence of manganese and iron) and, in limited areas, ivory/bone carbon (as indicated by the contemporaneous presence of calcium and phosphorus) or copper based compounds. The absence of sulphur suggests the main use of calcium carbonate rather than the more usual gypsum. A further difference is related to the nickel content: this element is present only in three areas, at the contrary it always accompanies iron in ochre in the other artwork of 14th century, St Nicholas, painted for the same church of Mborje, investigated in a previous study [4].

The anonymous painter's palette of the 14th century studied in a previous work [4] includes a limited number of pigments, as lead white, cinnabar, red and yellow ochre, brown earth, realgar, ivory/bone black and probably indigo. The icon of the same century, object of the present study, appears more richly coloured, although the artist has used practically the same type of pigments. The wide variety of colour combinations could be obtained through the use of mixtures with different proportions and superimposing layers of different pigments. To be noted the presence of green with different shades obtained using a copper compound with arsenic sulphide.

The icon studied in the present work represents an interesting example of the Albanian Byzantine art of 14th century. Its anonymous author follows the iconographic tradition, with some characteristic features, as discussed above.

Through our study, we also detected the previously restored areas of the icon and the kind of pigments utilized by restorers in a period of time from the first half of 1800 (as denoted by the presence of *blanc fixe* and zinc white). Furthermore, the spectral reflectance measurements of the different pigments gathered in this work, in addition to recognizing the conservation status of the examined artwork, will also be useful for future restoration intervention, offering a contribution towards the accurate restoration of the colours. In conclusion, the set of non–destructive techniques employed in this study, has proved to be very useful for recognizing pigments, including those not detectable with certainty by means of X-ray fluorescence.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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