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SPECTROSCOPY STUDY OF HERITAGE OBJECTS FOR THE DIGITIZATION OF CULTURAL HERITAGE

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Abstract

Cultural heritage objects represent valuable testimonies of the past, which must be kept in the right condition to transmit to future generations. Together with the spectacular progress recorded by humankind regarding technology, opportunities to obtain valuable data on heritage objects so as to preserve them have started to emerge. The current researchers aimed to determine the internal composition of the colors and materials used in some paintings from a historic wooden church monument, from Oradea City, Romania. The samples were taken from icons made in different materials, which were in an advanced stage of degradation. Expert analysis of the samples of the paint layer and plaster base was undertaken using X-ray fluorescent spectrometry. The paper presents the data obtained via spectral analyses of the different sections of the samples taken.

Keywords: conservation, cultural heritage, digitization, paintings, spectrometry

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1. Introduction

As a result of the impressive progress made by humankind in the field of technology, opportunities have appeared to fill the previously exposed gaps. Digitisation appears to be one of the most viable ways of obtaining the required information concerning the heritage objects and storing them as digital presentations (Ruggiero et al., 2012), with the vital knowledge of the conservation of monuments, as well as the appreciation of their future evolution, depending on the particularities of the objects concerned and the environmental conditions under which they are kept. The present study deals with the analysis of valuable objects inside a historical monument wooden church in Oradea, Bihor County, Romania. As may be observed on an inscription in the beams in the pronaos, the construction was carried out between 1760 and 1762 in Letca, Sălaj County, with it later being transported to the University of Oradea campus. From 2010 the item appeared on the list of historical monuments, under the registration code BH-II-m-B-20958 m (Ilieș et al., 2018). The interior paintings, which form the main study object, were located in 1993 on the site of the church itself. To gain an in-depth understanding of the 'hidden' process used in their creation (Legrand et al., 2014), as well as of the internal structure of the paintings and the materials used to create them, X-ray fluorescent spectrometry was used. Determining the internal composition of the

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paintings, in terms of the relevant cultural heritage, offers valuable data that can be used towards developing a new strategy for the conservation and restoration of the paintings concerned (Monico et al., 2011). Starting with the second decade of the 21st century, several studies have been carried out on the digitisation of cultural heritage (Angheluta and Radvan, 2017; Doulamis et al., 2017; Karaszewski et al., 2012; Madanan et al., 2018; Manferdini and Russo, 2013; Newell et al., 2012; Pavlidis et al., 2007; Sotirova et al., 2012), and historical monument churches (Marutoiu et al., 2017; Ruggiero et al., 2012). Paintings on church walls around the world (Bakiler et al., 2016; Clark et al., 2010; Doménech-Carbó et al., 2012; Tomasini et al., 2016), as well as other types of paintings that were considered valuable, and which were in need of conservation and/or restoration (Lengrand et al., 2014; Monico et al., 2011; Zielińska et al., 2013; Vecco, 2010) have been analysed, using X-ray technology. A key feature of the technology is that it is non-invasive (De Viguerie et al., 2010; Mannes et al., 2015; Reiche et al., 2012) for both the internal composition and the external appearance of the digitized heritage objects studied.

The inestimable cultural meaning of wooden churches, and the heightened sense of identity that they give to Romanians, has been confirmed by the large number of works that have appeared on the topic during the last decade (Baias et al., 2015; Ilieș et al., 2011; Ilieș et al., 2016; Ilieș, 2013; Mihincău et al., 2019; Wendt et al., 2018). The wooden church on the Campus of the University of Oradea was examined, taking into account the influence of the microclimate and the contamination of the wooden surfaces with microorganisms and fungi (Ilieș et al., 2018).

2. Material and methods

In order to determine the internal composition of the colours and the materials used to make the interior paintings, the icons that were in an advanced state of degradation were considered. The fragments of seven paintings, with several different types of surface, which lined the walls of the wooden church, were used for the analysis. The samples (1,2,3,4) taken from the iconostasis (Fig. 1) are painted on plaster, with sample no. 4, collected from the bottom right-hand corner of the main entrance to the altar, consisting of a canvas. Samples 6 and 7 (Fig. 2) are pieces taken from wooden paintings.

The three samples of the wall paintings, which were collected from icon 1, were indexed as 1.1 (the grey fragment), 1.2 (the brown fragment), and 1.3 (the blue fragment). The fragments of the second, fourth and fifth icons, numbered 3, 4 and 5 respectively, are pieces of cement, with a dark and light brown pattern. The samples with blue and grey pigments gathered from the wall painting were numbered 6 and 7. The brown piece of canvas was numbered 2 (Fig. 3).

Expert analysis of the samples taken from the paint layer and the plaster base were taken at the sites of destruction. The icons of iconostasis (carbonate plaster), the wall painting, the doors and the painting on textile material were investigated using X-ray fluorescent spectrometry (Spectroscan Max G, Spectron, Russia), with the type of spectrometer used being wavelength-dispersive. The spectra of florescence were measured with the Ag anode and the LiF (200) analysing crystal, at 40 kV voltages, with a 0.1 A current, for 8 minutes of exposure time.

Fig. 1. Scheme of icons and frescoes, from which samples were selected for analysis

Fig. 2. The paintings on wood from the church walls, from which samples 6 and 7 were collected

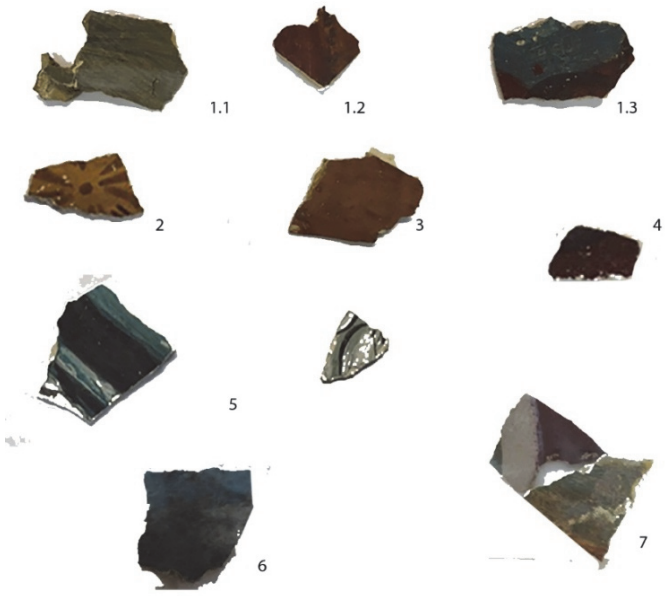

Fig. 3. Samples collected from inside the wooden church historical monument, taken at the sites of destruction

The chemical composition of the objects was investigated using scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX) microanalysis in SEM JEOL JSM – 6390LV, with EDX analyzer Oxford INCAEnergy (Oxford Instrument, England). The system was calibrated for all the observed chemical elements in the samples (C, N, O, F, Na, Mg, Al, Si, P, S, Cl, K, Ca, Cu, Ti, Cr, Fe, Zn, Br, Sr, Ba, W, Pb), with the Registered Standard No. 7366 being supplied by Micro-Analysis Consultants Ltd, England. The samples were cleaned only by blowing them with compressed air, using no liquid solvents (so as to avoid sample degradation). As the samples were non-conductive, immediately prior to the SEM-EDX analysis, the samples were coated with 10-nm carbon-conductive film, in the form of Jeol JFC-560 thermal-carbon coater (Jeol Ltd., USA).

3. Results

The spectra were recorded at four positions for all the studied samples. The data from the spectral analyses at the different points of sampling are shown in Table 1. The results obtained from the same descriptive statistical analysis of the element concentrations in the different samples are the mean, the standard deviation and the coefficient of variation. For a more detailed study of the samples used, the X-ray spectra were repeatedly taken from different positions of the sample, in relation to a range of pigments (Fig. 4).

Sample 2, collected from the icon of Jesus Christ, which was positioned at the altar, was divided into two pieces, which were then numbered and analysed with X-ray spectra, so as to determine the chemical makeup of the brown pigments of which it was constituted. The results of both samples are presented in Fig. 5.

To determine the nature of the blue and grey pigments used, the X-ray technique was applied to the three pieces constituting sample 5. The chemical composition of each sample is presented in Fig. 6. Samples 6 and 7 were collected from the paintings on the wooden walls of the church. Three pieces of the samples, selected for analysis, contained brown, white, blue, green and gold pigment. The results for each piece are shown in Fig. 7. The third piece, analyzed in sample 7, formed part of the aura of the saints, which is characterized by the presence of an intense golden pigment.

Table 1. Descriptive statistics of the concentration data (atomic %) in the samples studied

Elements, characteristic line series		*1.1*	*1.2*	*1.3*	*2*	*4*	*5*	*6*	*7*
C K	***Mean***	55	42	58	70	55	45	53	47
	SD	9.0	14	9.1	3.0	8.3	20	19	17
	CV	16	35	16	4.3	15	45	36	35
O K	***Mean***	34	46	31	28	26	39	29	37
	SD	5.9	3.8	5.9	3.6	5.5	7.2	11	18
	CV	17	8.2	19	13	21	18	39	47
Na K	***Mean***	10	0.92	2.3	2.0	0.38	2.5	1.0	1.1
	SD	7.0	0.43	0.92	0.94	0.17	3.8	0.36	0.42
	CV	68	47	39	47	44	153	36	37
Mg K	***Mean***	-	0.21	-	-	0.68	-	0.47	-
	SD	-	0.41	-	-	0.41	-	0.18	-
	CV	-	200	-	-	59	-	38	-
Al K	***Mean***	0.065	0.033	0.15	0.51	0.34	0.025	0.093	-
	SD	0.019	0.039	0.026	0.85	0.42	0.05	0.046	-
	CV	29	121	17	168	123	200	49	-
Si K	***Mean***	0.208	0.038	0.485	0.27	1.2	0.41	0.37	0.19
	SD	0.12	0.075	0.19	0.12	1.2	0.11	0.26	0.015
	CV	56	200	38	43	101	26	71	80
P K	***Mean***	-	-	0.055	0.055	-	0.015	0.023	-
	SD	-	-	0.026	0.064	-	0.017	0.005	-
	CV	-	-	48	115	-	115	22	-
S K	***Mean***	0.357	1.45	2.2	0.068	0.76	0.29	0.57	2.1
	SD	0.14	0.89	1.2	0.075	0.33	0.044	0.68	2.2
	CV	38	61	52	111	44	16	118	108
Cl K	***Mean***	0.193	0.022	0.35	0.12	0.37	0.16	0.085	0.42
	SD	0.10	0.026	0.15	0.11	0.28	0.13	0.033	0.28
	CV	51	117	43	87	76	85	39	67
K K	***Mean***	-	-	-	0.0075	0.19	0.028	0.033	-
	SD	-	-	-	0.015	0.044	0.022	0.015	-
	CV	-	-	-	200	24	81	46	-
Ca K	***Mean***	0.46	8.2	1.6	0.15	8.5	11	13	0.52
	SD	0.23	15	0.94	0.14	6.2	10	8.1	0.11
	CV	50	183	58	99	73	88	64	20
Cu K	***Mean***	-	-	-	-	-	-	-	0.42
	SD	-	-	-	-	-	-	-	0.84
	CV	-	-	-	-	-	-	-	200
Ti K	***Mean***	-	-	-	-	0.05	0.28	0.26	-
	SD	-	-	-	-	0.10	0.32	0.14	-
	CV	-	-	-	-	200	113	56	-
Cr K	***Mean***	-	-	-	-	-	-	-	0.073
	SD	-	-	-	-	-	-	-	0.071
	CV	-	-	-	-	-	-	-	98
Fe K	***Mean***	-	0.095	0.54	0.0075	5.1	0.085	0.055	1.1
	SD	-	0.15	0.43	0.0050	4.0	0.11	0.048	0.82
	CV	-	155	80	67	78	125	87	72
Zn K	***Mean***	-	-	-	-	-	0.30	2.1	1.5
	SD	-	-	-	-	-	0.39	0.78	0.36
	CV	-	-	-	-	-	127	37	23
Sr L	***Mean***	-	0.033	0.063	-	0.028	-	-	0.03
	SD	-	0.039	0.024	-	0.015	-	-	0.047
	CV	-	121	38	-	55	-	-	156
Br L	***Mean***	-	0.02	-	-	-	0.02	-	-
	SD	-	0.04	-	-	-	0.04	-	-
	CV	-	200	-	-	-	200	-	-
Ba L	***Mean***	-	0.83	2.8	0.0025	1.2	0.11	0.01	3.9
	SD	-	0.61	1.6	0.0050	0.85	0.19	0.02	4.86
	CV	-	74	57	200	69	170	200	124
Ta M	***Mean***	-	0.01	-	-	-	-	-	-
	SD	-	0.02	-	-	-	-	-	-
	CV	-	200	-	-	-	-	-	-
Au M	***Mean***	-	-	0.015	-	-	-	-	-
	SD	-	-	0.03	-	-	-	-	-
	CV	-	-	2	-	-	-	-	-
Pb M	***Mean***	-	0.21	0.033	0.013	0.023	0.073	0.01	0.19
	SD	-	0.12	0.040	0.019	0.005	0.13	0.011	0.13
	CV	-	58	121	-	22	173	115	61

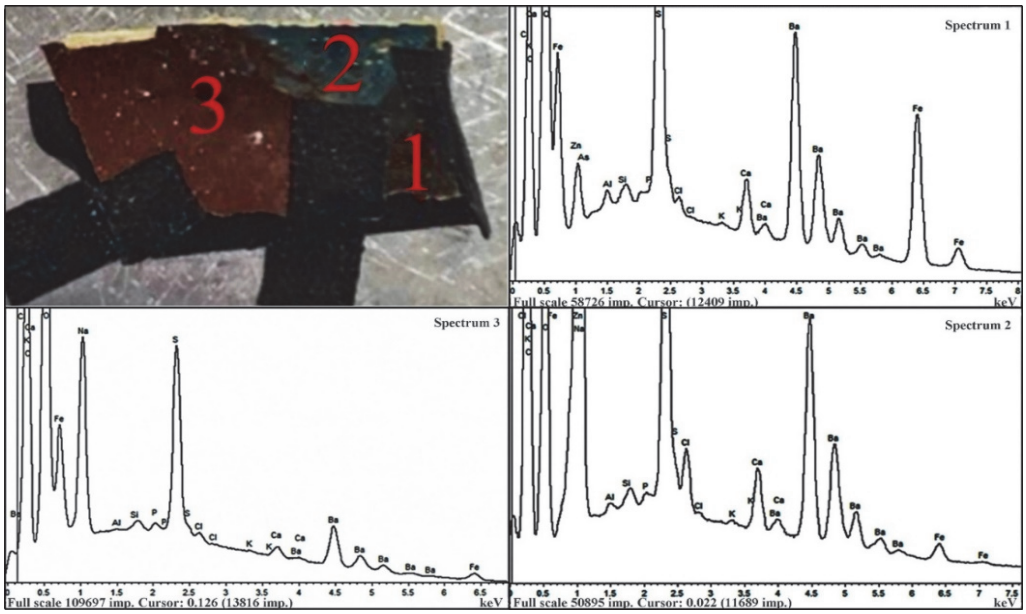

Fig. 4. Location of the detecting points in sample 1.3, and the X-ray spectrum sample segments

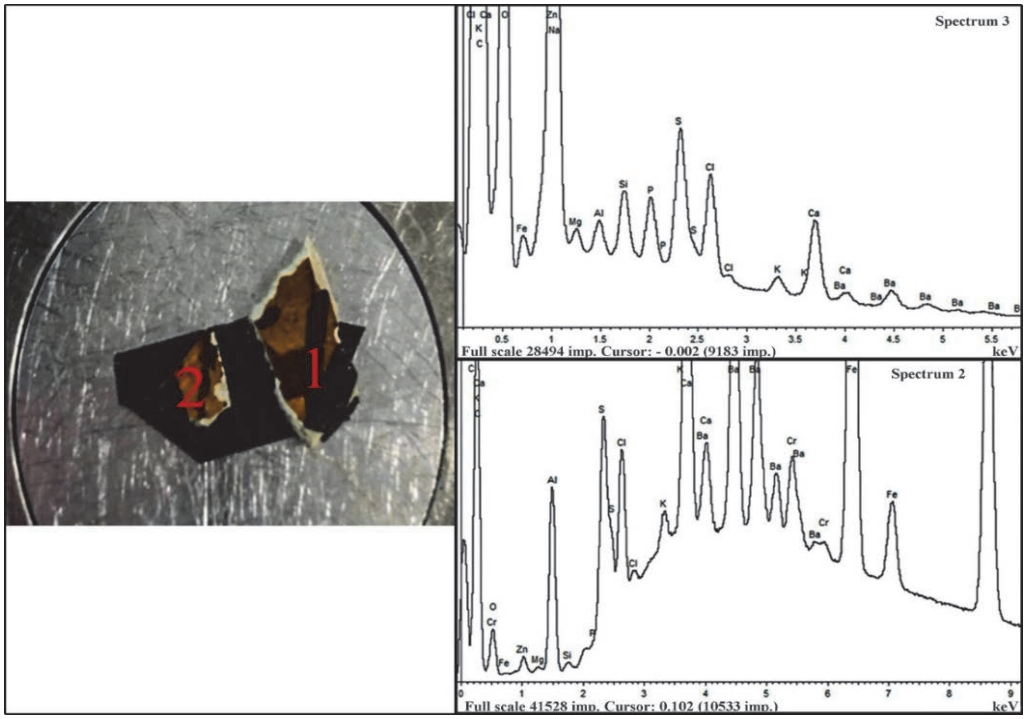

Fig. 5. Location of the detecting points in sample 2, and the X-ray spectrum of the sample segments

The electronic image and the spectral results, obtained for the green and gold segments, are presented in Fig. 8. Sample 3 is a piece of brown paint taken from the lower right-hand area of the canvas, which appeared on the main entrance door to the altar. The electronic image of the sample and the X-ray spectrum that were obtained from the analyses are shown in Fig. 9.

4. Discussion

The analysis suggests that the ground consists of calcium carbonate used with an organic binding agent (flaxseed oil varnish). The difficulty of determining the nature of the organic materials was exacerbated by the natural aging processes that had occurred with some of them, and by the need for more of the substance to have been available for the analysis than was accessible at the time.

A key reason for the change in the mechanical properties of the plasters over the course of time is the seasonal fluctuation experienced in temperature and humidity, during which time the plaster is alternately moistened and dried. The saturation with water, and the drying of plaster, which has been occurring for many decades, results in fatigue of the material from repetitive internal stresses, as a result of which the internal bonds are broken, and the strength of the

material decreases. Radiant heat (e.g. from the burning of candles), released for a long time around the painting, can convert into calcium carbonate, leading to the periodic adsorption of moisture, which causes the plaster to disintegrate, and alters the tone of some pigments (Devina et al., 2000).

The high carbon content found in the samples also suggests that protective organic varnishes, possibly based on gum, were used for the coating of the painting, but to obtain a more accurate analysis than the above, a larger sample would have to be taken.

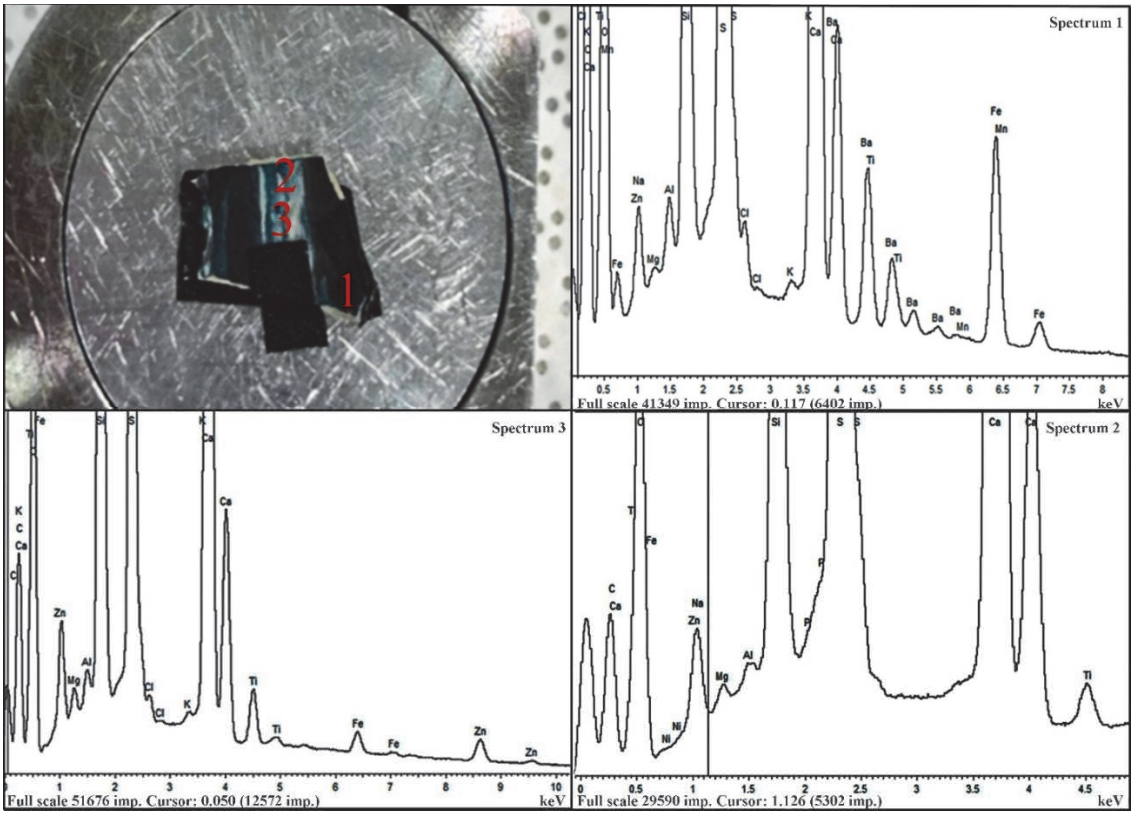

Fig. 6. Location of the detecting points in sample 5, and the X-ray spectrum of sample segments

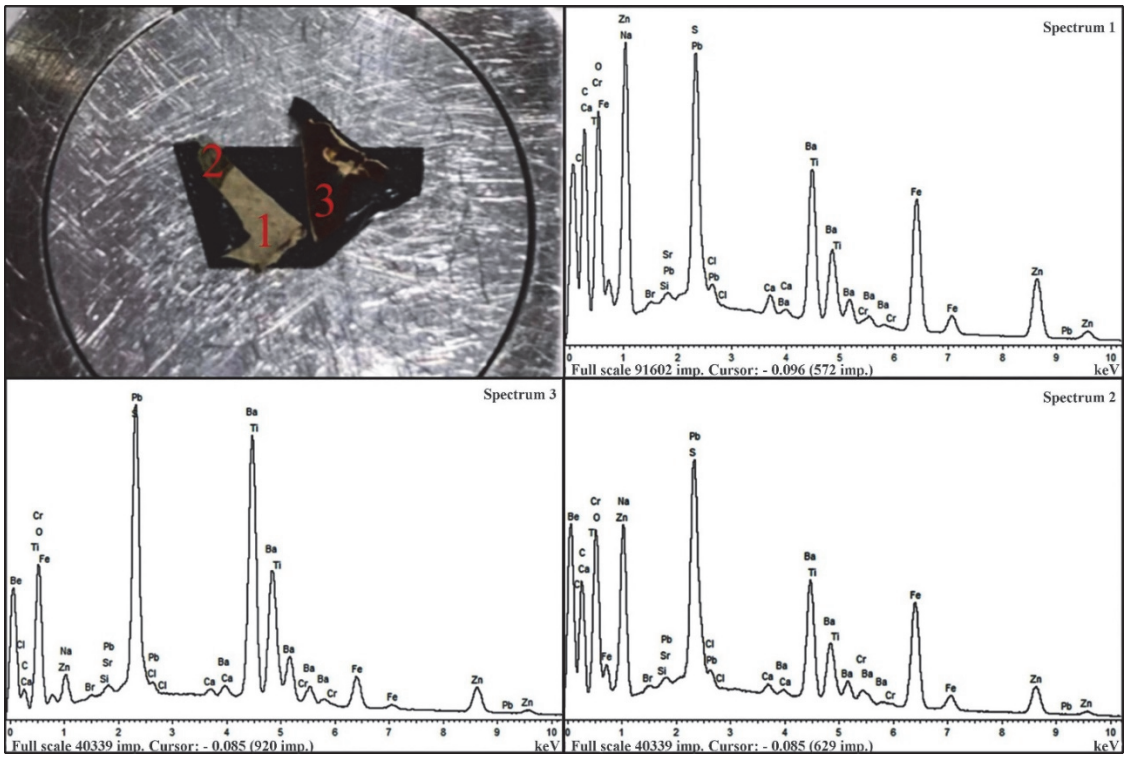

Fig. 7. Fragments of the wall painting, sample 7, showing the spectra of the white, grey and brown pigments

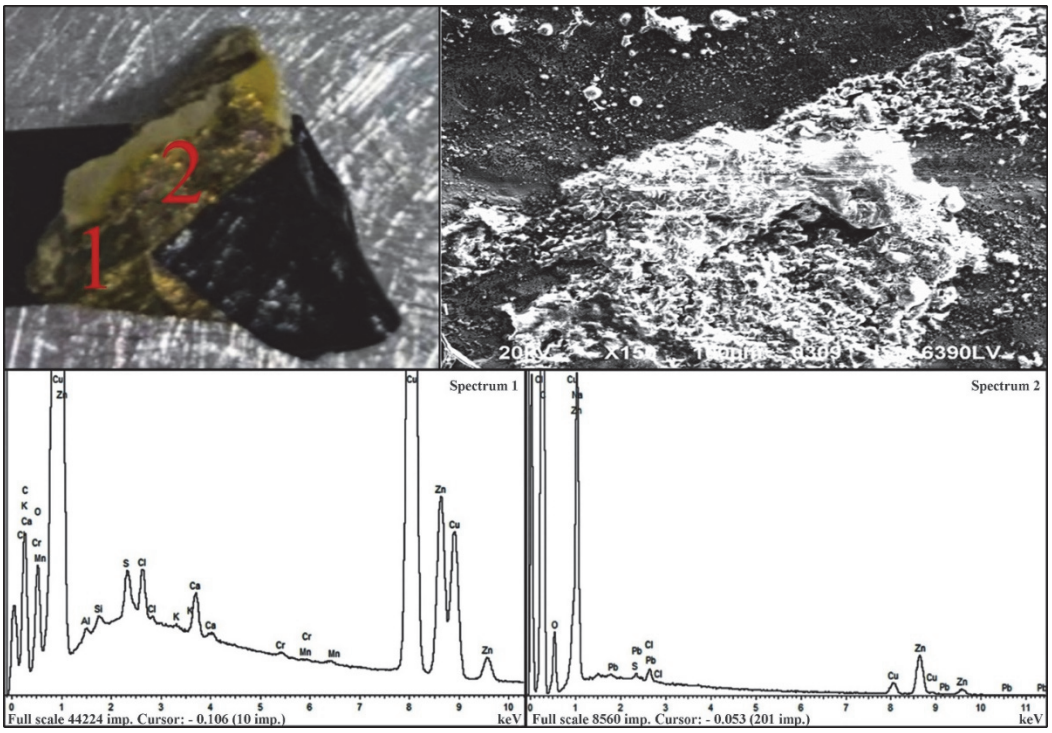

Fig. 8. Sample 7, point 1 – green segment; point 2 – gold drop. Electron microscope image of gold pigment and spectra of green and gold segment

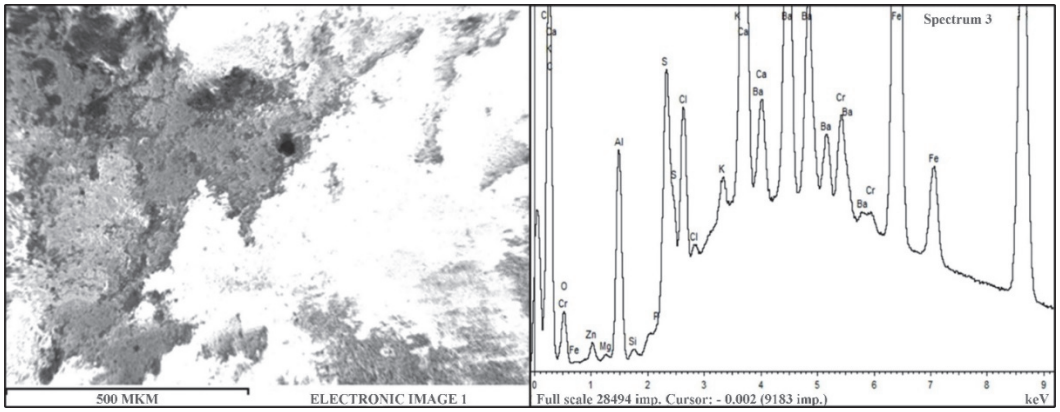

Fig. 9. Electron microscope image, and the X-ray spectrum of a segment of sample 3

Blue (ultramarine) pigments are represented exclusively by sodium aluminum silicate, including sodium polysulfide $Al_6Na_3O_{24}S_3Si_6$ or $(Na,Ca)_8(AlSiO_4)_6(S,SO_4,Cl)_{1–2}$. Depending on the ratio of the components involved, the hues vary from blue-green to violet, which, when mixed with white pigments, emit hues ranging from pale blue (paleo) to grey-blue (Slansky, 1953). Excluding the acidic components (SO_2, CO_2, NO_x), which first cause yellowing, and then discoloration; the pigment is resistant to UV radiation and weather impact. At temperatures above 250°C (candlelight), the pigment may darken, sometimes acquiring a greenish tint.

Over time, the ultramarine coloring tends to develop the so-called ‘ultramarine sickness’, with the ultramarine pigments having the disadvantage of absorbing, and condensing, moisture from the air. Moisture violates the homogeneity of the system. As a result of moisture disrupting the homogeneity of the system, changes may occur in the appearance of the paints, which can become turbid, grey or colourless.

The mixing of ultramarine with lead pigments (browning the colors of the paint) and with earthy colors (ochre, umber, etc.) causes the pigment involved to turn grey. Ultramarine pigment is present in the icons of Archangels Michael and Gabriel. The sample also contains gold (Au), with small particles of gold having possibly been applied to the ultramarine background (used for the blue coating of the Archangels) (Fig. 1). In sample 5 (the icon of Jesus Christ – Fig. 1), ultramarine green was employed as an intermediary to obtain artificial ultramarine blue (Slansky, 1953). The chemical composition of the ultramarine green present is expressed by the formula, $NaAl_6Si_{62}O_{24}$.

The brown pigments present could be identified as Marsyas brown iron oxide pigments (which were present in all the samples, excluding the

wooden part of the iconostasis, featuring the saints and the floral borders, as well as elements of the clothing). The pigment was obtained by igniting a mixture of iron sulphate and aluminum oxide with soda.

Iron oxide pigments were also used to produce the red and dark-brown shades (Mars red, Mars yellow, mulberry dye, etc.) (Pfaff, 2017; Slansky, 1953). Iron oxide paints are durable and resistant to light and weather. The intensity of their color depended on the amount of chromophore present, with it being especially high in the iron-rich ores of hematite (red), hydrohematite (mummy or red-brown), goethite (yellow), and others. The brown and red-brown paints were found to contain impurities of baryta white. The black pigment is most likely bone black, with such a finding being indicated by the presence not only of carbon, but also of magnesium and calcium phosphates (Christie, 2015; Pfaff, 2017). Since the black pigment consists of amorphous carbon, it is resistant to air and light, and in mixture with other paints. The yellow pigment is most likely strontium yellow (Slansky, 1953), with its chemical composition being that of strontium chromate, $SrCrO_4$ (sample 7, wood). The white pigments are represented by the presence of baryta white (blanc fixe, $BaSO_4$). Although the pigment is resistant to heat, it eventually begins to break down, becoming subject to chalking (Pfaff, 2017).

5. Conclusions

The physical condition of the historic wooden church in Oradea, Romania continues to deteriorate, as a result of various negative phenomena and processes, in which the environment plays a special role. For example, such effects as the air pollution caused by industrial facilities and automobile transport contribute to the formation of a chemically aggressive environment, which causes the destruction of natural building materials, as well as of layers of paint, plaster and decor. Also, important issues involved are the biological damage of the wooden building structures by fungi and microorganisms, and by the presence of excessive moisture inside the church, due to inadequate surface drainage.

The current study showed that traditional inorganic pigments were used to paint the church icons. Their colouring ability is less than that which is obtainable with the use of organic pigments, but they tend to offer increased light resistance, and to have a heightened density. The art masters responsible for the paintings involved used toxic copper-containing pigments that actively interacted with the plaster elements, and, when they were exposed to the flame of a candle, they decomposed as a result of the formation of copper oxide, causing blackening of the paintings.

Redox processes (thermo- and photo-oxidation) are seen to have been responsible for the alteration of a number of the properties of some inorganic pigments, including ultramarine blue. As many of the reactions involved included the taking of a photoactivation step, it was necessary to avoid direct contact with sunlight. Currently, the main destructive factor in terms of interior paintings is high humidity.

The color of most pigments used for textiles consisting of cotton and linen fades relatively quickly under conditions of high humidity (Devina et al., 2000). Very low humidity leads to the drying out and the increased brittleness of the soil used. With daily or seasonal fluctuations in temperature and humidity, hygroscopic materials easily absorb and release moisture, undergoing swelling and compression, which accelerates their destruction.

The above effects might, in the present instance, have resulted from the deterioration of the vertical and horizontal waterproofing, which probably occurred during the relocation of the building, which resulted in leaks in the roof, as well as it possibly being due to the formation of condensate, especially resulting from insufficient air exchange. In addition, many types of molds tend actively to populate slightly alkaline substrates, such as the mineral pigments studied.

One of the first steps taken towards the conservation of the paintings involved should be a detailed engineering survey of the building concerned, and further restoration of the waterproofing, which should serve to stabilize the temperature and humidity conditions. Treatment with fungicidal and bactericidal agents that are safe for use with both people and paintings should assist in avoiding the further destruction of the wood, textile and plaster coatings and pigments involved.

Thus, the following priority areas for protection of the cultural heritage from negative environmental consequences can be identified. First of all, carrying out regular monitoring of the condition of the paintings, icons and wooden structures is necessary, as such supervision could help ensure the reliability and timely updating of information about the target indicators observed (the area and nature of the damage to the wooden base and plaster layer, the safety of the pigments and paint layers, etc.).

In addition, environmental monitoring within the precincts of the church is required (in terms of air pollution; the landscape and climatic conditions of the territory, including the temperature and humidity, the atmospheric pressure, the amount and time distribution of precipitation, the frequency of change in wind direction, and the snow cover value; and the engineering and geological conditions).

At the next step in the study, the necessary recommendations regarding the preservation and partial restoration of the church should form part of the monitoring of the data analysis.

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