Multidisciplinary Investigations of a Double Sided Wooden Icon from Nicula Monastery, Romania

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A two-sided wooden icon from a monastery in Transylvania was submitted for multidisciplinary investigations involving X-Ray Fluorescence, Radiographic Photographyand Fourier Transform Infrared Spectroscopy. The most important part of the icon is St. Nicholas wooden icon, painted over forty years ago. The spectroscopic methods used revealed the painting materials composition, the status of the wooden stage, and the presence of resins as varnish (Fourier Transform Infrared Spectroscopy). On one side, the St Nicholasicon was painted over an old icon, St. Arch. Michael, which was evidenced by X-Ray Photography. The obtained data can serve for the preservation and the restoration of these wooden icons.

Keywords: XRF; X-Ray Radiography; FTIR, patrimony object, painting materials, wooden icon

Cultural patrimony objectsare an important part of our cultural and historical heritage. They are studied using different experimental techniques, in order to characterize the constituent materials. FTIR (Fourier Transform Infrared Spectroscopy) [1-11] and XRF (X-Ray Fluorescence) spectroscopy [12] are employed to investigate various samples taken from significant areas of the artwork. Recently, the destructive techniques were replaced by non-destructive ones (XRF, FTIR [13], portable Raman [14]). The constituent materials composition data are used to appropriately plan the conservation treatments [15] and restoration and also to support the historic and artistic study of the paintings. There is a great amount of information [16-24] concerning the investigation of painting and binding materials of religious art objects.

The investigated artwork in this study is a holy wooden icon painted on both sides. On one side St Nicholas is painted and on the other side is painted Jesus Christ. *Saint Nicholas*' icon is a precious work of Transylvanian medieval art from the Seventeen century that survived the vicissitudes of time, unseen and forgotten for more than two centuries. Discovered as a result of recent research, together with other related stylistic vestiges and dating from the same time period, the icon gives testimony about an artistic phenomenon not at all accidental [25], very little known and researched in Central and Northern Transylvania, but often noticed in Maramures County [26].

The composition faithfully follows the Orthodox iconographic tradition, in the centre we can see the bust of Saint Nicholas blessing with his right hand and holding the Holy Gospel in the left. Beneath his red cape he is wearing a blue sticharion and the white Omophorion on top, over the lower part of arm that holds the Holy Book. On each side is flanked by the Saviour Jesus Christ and the Virgin Mary, represented in the smaller scale, but painted in a full stature – a fact that is less usual and that represents an interesting peculiarity of the *rustiphication* phenomenon. Holy people offer him the Archbishop insignia: The Gospel and the Omophorion. The icon has been only partially preserved, the wooden support being adjusted at the top, during the second painting.

The colour palette is restricted to basic colours - red, yellow, blue; to which white and black have been added, as well as a mixture of them in order to get several shades. Red is the predominant colour used for Saint Nicholas clothing, for the aureole and skin tone, in contrast to the blue and green colour of the clothing, and the background used at the bottom of the representation. The initial brightness of colours is altered by colour reversion and browning of the robe, the blue color of attire is perceived in natural light as being green, and blue in the flashlight.

The reverse of the icon was painted in the Eighteen century, most likely around 1771, when the church was sanctified [27] and repainted in the following decades by an amateur.

As it can be readily seen from the color gaps, the wooden panel was first puttied with light gray color putty and then prepared with a layer of compact primer, applied thickly enough to cover the slopes of the panel. The drawing was executed directly on the primer; traces of incisions can be observed only at the aureole contour and under the Saint's name inscription.

The pictorial layer shows numerous gaps, most of them small, on the entire surface. The most notable losses are situated at the bottom of the panel and in the lower left corner. With few exceptions, the colour layer losses are correlated with the losses of the primer layer.

Due to contractions and expansions of the wood mass and the aging of the materials, in addition to the fine networks of cracks that cover the entire pictorial surface, small breakawayscan also be observed. The varnish layer is brown and has traces of adhering dirt: dust, fumes, lanolin, wax, etc.

On each side, Jesus Christ is flanked by Saint John the Baptist and by the Virgin Mary. The only part that was not entirely repainted is the Holy Gospel, which has the inscription done in Romanian, with Cyrillic letters: *Come My Father blessed ones, to inherit My Kingdom which is ready for you* [28]. The mentioned composition and the inscription made possible the classification of the icon in the post-Brancovenesc artistic style that reached Transylvania in the first decades of the XVIIIthcentury [29].

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The panel was tangentially chipped and is made from a single board. During back-painting, massive slats were added, bounded with wood dowels on each side, and also a metal belt clip. The panel rings appear to be oriented with the concave side facing backwards, which would explain the slight curve on the edges. There are no traces of biological attacks or xylophages insects.

The aim of this paperis to investigate the painting materials and the wooden stage of these icons in order to preserve and restore them.

Experimental part

The panel dimensions are: length = 665-670 mm; Width = 455-460 mm; Thickness = 30 mm.

The painting materials sampling procedure is presented in figures 1a and 1b. X-ray fluorescence measurements (XRF) were performed using an Innov-X Alpha-6500 portable instrument (35 kV voltage, 15 μA intensity, 3 mm filter, Be window, 2 square mm spot size and Pin Si detector). Integration time was set for 60 s , in two consecutive runs of 30 s each.

X-ray radiography was performed with a Bucky RTG device (using 30/40, 35/43 or icon size dependant film cassette), image processing devices and a computer. The technique employed for the X-ray was the following: the icon was laid on the X-ray table; the light spot was centred and diaphragmed in order to cover the icon as much as possible. The central ray must be perpendicular to the film centre. Work parameters: 45-50 kV and 12-14 mAs. No right or left labelling was used.

FTIR spectra were registered with a resolution of 4 cm^{-1} using a Jasco 6100 FTIR spectrometer in the 4000 to 400 cm^{-1} spectral domain by employing KBr pellet technique.

Results and discussions

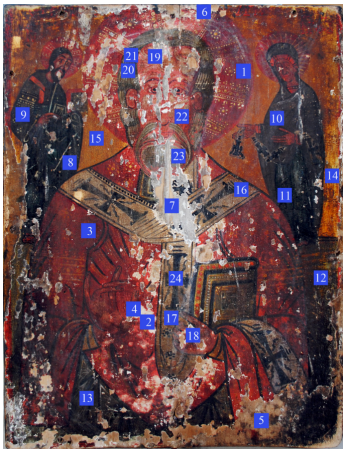
Characterization of St. Nicholas icon

XRF analysis

XRF data for the icon is presented in table 1.

Based on XRF analysis, one can propose the following possible composition:

a



b

Fig. 1a) St Nicholas wooden icon: XRF sampling points. b) Jesus Christ Imperial (Kingly, Royal) wooden icon: XRF sampling points

mg/kg	K	Ca	Fe	As	Hg	Pb
A1	3828	32086	2079	1124	91651	2170
A2	<LOD	70047	1038	316	47298	818
A3	8469	31198	3184	894	108730	1784
A4	<LOD	163088	410	208	1230	509
A5	<LOD	67152	415	88	72	116
A6	<LOD	103288	526	623	7858	2398
A7	6053	284508	2803	46	85	126
A8	6794	24735	2507	6716	415	16774
A9	13471	36928	9866	693	1652	1909
A10	13800	27267	8935	580	1811	1878
A11	6185	33593	2749	5716	413	15083
A12	5033	70071	3985	5402	371	892
A13	4165	55964	3036	7297	2650	790
A14	6782	58486	2939	35384	458	1463
A15	6767	51694	2395	17262	422	775
A16	11336	28417	3718	16961	2065	89008
A17	10014	28417	3333	15529	1864	78467
A18	6247	23551	2935	11130	33744	37566
A19	23923	105158	3936	10130	86995	24093
A20	11128	31509	1525	28959	2584	48889
A21	6473	32098	1029	17521	2677	37376
A22	10938	37833	5365	4448	42141	6785
A23	7899	126253	3321	10587	24368	20741
A24	11326	53588	2083	18868	1758	100611

Table 1
XRF DATA FOR *ST. NICHOLAS* WOODEN ICON

A high concentration of mercury was observed in the A1, A2, A3, A6, A18, A19, A22, A23 points, which suggests the presence of a red mercury-based pigment, most probably vermilion. The A4 point corresponds to a ground area made from gypsum. The presence of iron in A7 point suggests the use of an iron based red pigment (red ochre). A13 and A14 points seem to represent a mixture of red, yellow and mercury red. A15 is an orpiment similar to A17, A20 and A21. A24 seems to be a mixture of orpiment and lead-red.

Some areas (A8, A11, A16 and A 24), which appear to be dark, are due to the modification of white lead in the presence of sulfur compounds (e.g orpiment) [30].

FTIR analysis

Canvas investigations

The canvas was applied over the junction of the two wooden pieces which form the icon support. Over this a ground layer was applied and covered with a final layer of white lead. FTIR spectrum of canvas compared to hemps and lines ones is presented in figure 2[31].

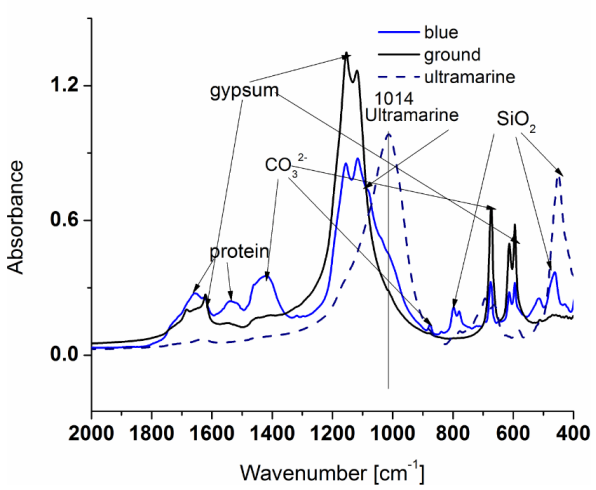


Fig. 2. FTIR spectrum of icon canvas as compared to hemp and lime standards spectra

Taking into account the similarities of the FTIR spectra in the 3000-2800 and 1800 to 1300 cm^{-1} spectral ranges, it was established that hemp is the employed canvas material. One can define, in agreement with literature [31] some indexes that were employed to establish the degradation status of the canvas in comparison with a standard one as follows: crystallinity indexes $TCI=A_{1335}/A_{2920}$ and $LOI=A_{1430}/A_{897}$, degrading monitor $D=A_{1335}/A_{1318}$ and amorphous/crystalline ratio $A/C=A_{900}/A_{1280}$. The table 2 presents these parameters for icon canvas as compared to hemp standard.

Table 2

CONSERVATION STATUS PARAMETERS FOR ICON CANVAS

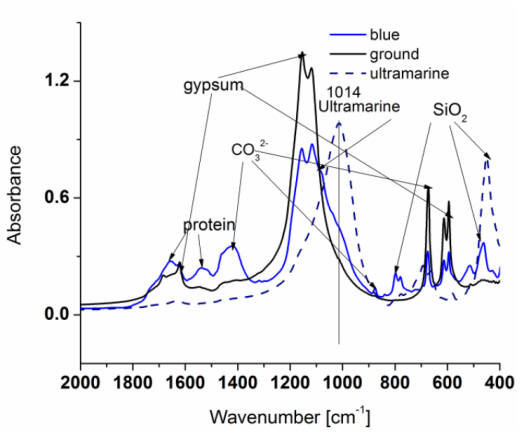
Sample	*TCI*	*LOI*	*D*	*A/C*
Hemp standard	*0.929*	*1.302*	*0.976*	*1.026*
Icon	*1.106*	*1.841*	*0.964*	*0.836*

The crystallinity of the canvas is higher than for hemp standard. One can see some degradation (D) of the canvas and also a decrease of the amorphous/crystalline (A/C) state.

Pigment investigation

The white layer applied over the ground has the following composition: $PbCO_3$ (1419 and 874 cm^{-1} bands, lead white, OH groups in the 3700-3200 cm^{-1} range), one compound with S-H group (2515 cm^{-1}), (probably a degraded egg yolk product) (1798, 1640 cm^{-1}), Si-O (1033 cm^{-1}), traces of gypsum (doublet at~615 and 573 cm^{-1}). The composition of ground (gypsum) employed as color *diluent* is defined by the absorption bands at 3544, 3406, 1621, 1154, 1119, 615 cm^{-1}. The composition of red painting material: HgS+ gypsum as color *diluent*, based on XRF data, gypsum as color *diluent* (3544, 3406, 1621, 1154, 1119, 615 cm^{-1} FTIR bands). The composition of blue painting material, see Fig.6a is: ultramarine, gypsum, lead carbonate. Red pigment – Composition of red pigment (Fig. 3b): HgS (from XRF data) + gypsum (3544, 3406, 1621, 1154, 1119, 615 cm^{-1} bands). The composition of yellow painting material (fig. 3c): As_2S_3 (orpiment from XRF data) + gypsum as color *diluent* (3544, 3406, 1621, 1154, 1119, 615 cm^{-1} bands).

Based on the comparison between specific FTIR absorption bands and XRF data, one can establish the possible painting materials employed in the case of *St. Nicholas* wooden icon: white layer: $PbCO_3$, S-H (protein degrading products), Si-O compounds; ground - gypsum; canvas - probably hemp, amorphous/crystalline ratio decreased in time; red pigment - HgS+ gypsum; blue pigment- ultramarine; yellow pigment - As_2S_3 (orpiment)+ gypsum.



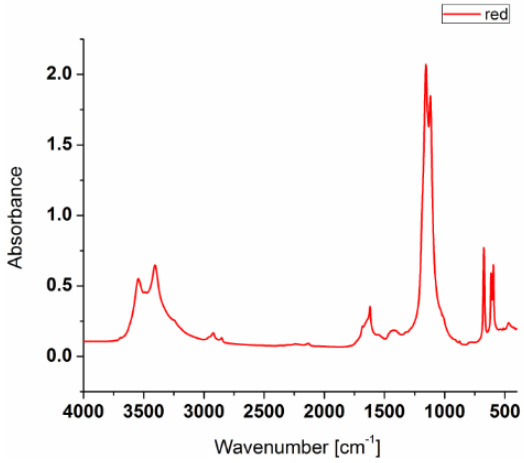


Fig. 3a) FTIR spectrum of blue painting material (4000-400 cm^{-1} spectral domain); b) FTIR spectrum of red painting material (4000-400 cm^{-1} spectral domain)

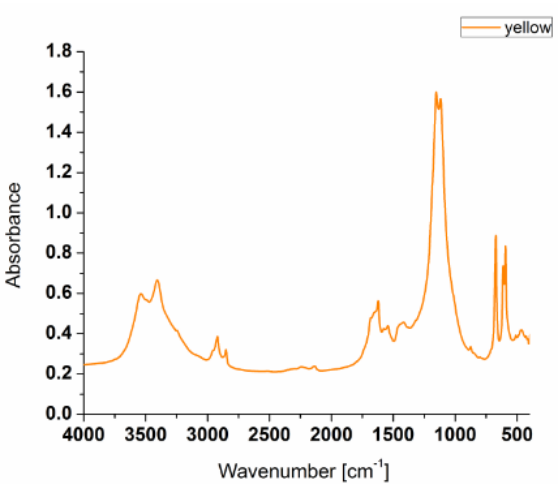


Fig. 3c) FTIR spectrum of yellow painting material (4000-400 cm^{-1} spectral domain)

Characterization of Jesus Christ Imperial icon

X-Ray analysis

X-ray photograph of the investigated iconis presented in figure 4.

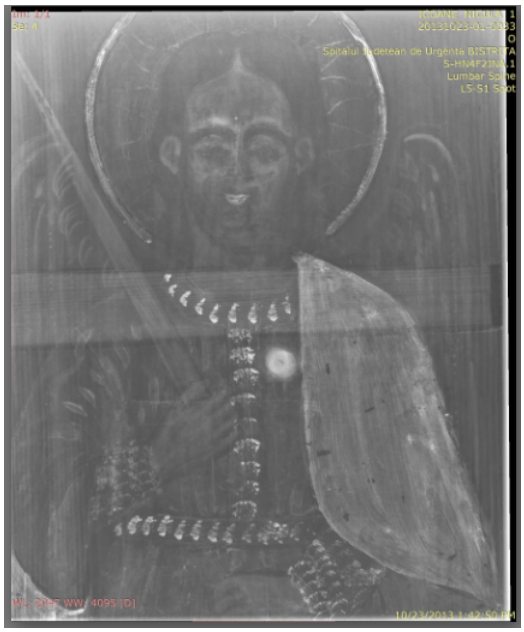


Fig. 4. X-ray photograph of the investigated icon

The icon *St Nicholas w*as over paintedover an older icon, entitled St. Arch. Michael. Note the use of lead white in areas of high X-ray absorption(white areas).

X-Ray Fluorescence data for the icon is presented in table 3.

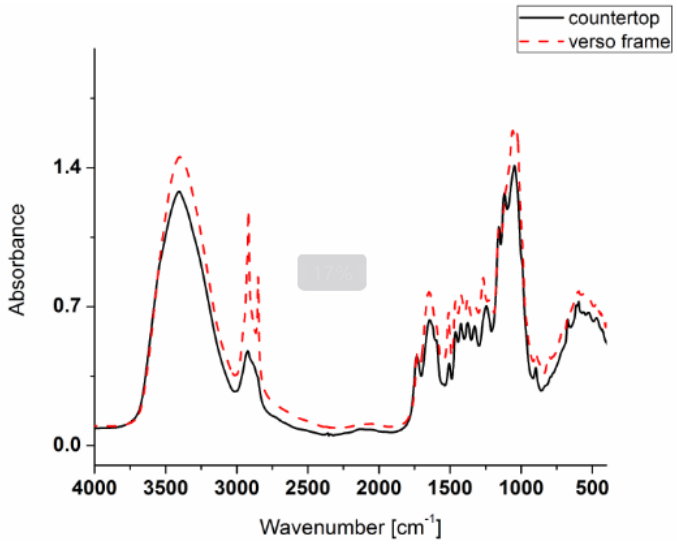


Fig. 5. FTIR spectra of back wood-frame- (fir wood) and of back side (countertop) wood, 4000-400 cm^{-1} spectral range

Based on XRF analysis, one can propose the following possible composition: B1 to B4 samples could be assigned to the following pigments: B2 - Prussian Blue-Lithopone, B3- orpiment; B4-lithopone, along with lead-white, which is commonly used as painting extender. Samples B5 to B9 do not contain lead white, but bronze (B5, B6) and red tones of iron red(B7),mercury red (B8) and againiron red – border (B9).

FTIR investigation of the painting materials

FTIR spectra of back-frame and back-side (countertop) woods are presented in figure 5.

In order to establish the wooden species one can employ two cellulose/lignin ratios defined as [9].

(1) Cellulose/Lignin= A_{1738}/A_{1503}

(2) C/L= A_{1738}/A_{1375}

The results of these calculations are presented in table 4:

By studying the ratios presented in table 4 it is possible to establish that the back-side wood is ash wood.

FTIR spectra of different painting materials are presented in figure 6.

Based on XRF data analysis and FTIR spectra, one can identify the employed pigments: Pb_3O_4+HgS (Pb and Hg from XRF data) + $CaSO_4$ (ground) (Ca from XRF data) +$BaSO_4$ (Ba from XRF data) (employed as diluent for red); Prussian blue (Fe from XRF data and CN stretching at~2088 cm^{-1} in the FTIR spectra) + $CaSO_4$ (+gypsum as diluent) (Ca, Ba and S from XRF data) for black; bronze as yellow pigment (Cu, Zn, Fe elements from XRF analysis); $CaSO_4$ (diluent, 3407, 1621, 1118 and 1083 cm^{-1}), aliphatic (2924 and 2852 cm^{-1})[32].

mg/kg	K	Ca	Mn	Fe	Cu	Zn	As	Ag	Ba	Hg	Pb
B1	<LOD	140976	93	613	<LOD	467	6553	<LOD	75	360	31902
B2	23830	39382	321	11940	127	19635	5158	<LOD	2644	285	28821
B3	<LOD	92873	109	1437	87	106	4460	<LOD	<LOD	327	19898
B4	6768	9706	1893	42413	<LOD	175011	<LOD	<LOD	9699	<LOD	37939
B5	<LOD	17024	4285	84019	77913	71468	<LOD	363	1079	928	236
B6	<LOD	78519	1362	24832	61256	16325	52	649	448	<LOD	475
B7	9298	77201	641	46867	477	6933	1020	<LOD	1459	1079	7271
B8	3189	12950	153	1146	<LOD	19802	208	<LOD	19097	16726	1016
B9	<LOD	37218	547	23086	256	267	288	515	1718	108	538

Table 3
XRF DATA FOR *JESUS CHRIST IMPERIAL* WOODEN ICON

Table 4

CELLULOSE/LIGNIN RATIOS CALCULATED FOR DIFFERENT WOOD SPECIES

Wood species	(1)	(2)
Lime	1.39	0.85
Ash wood	0.99	0.77
Sycamore maple	1.06	0.81
Fir	0.69	0.59
Back side wood	1.08	0.73

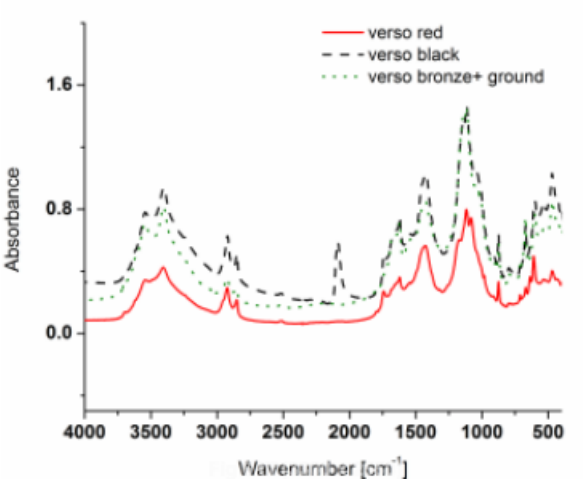


Fig. 6. FTIR spectra of verso red, of verso black and of verso bronze + ground: gypsum (diluent, 3407, 1621, 1118 and 1083 cm^{-1}), $PbCO_3$ (ground, 1423 and 874 cm^{-1}), traces of silicate (~1033 and 797 cm^{-1}), traces of aliphatic (2924 and 2852 cm^{-1})

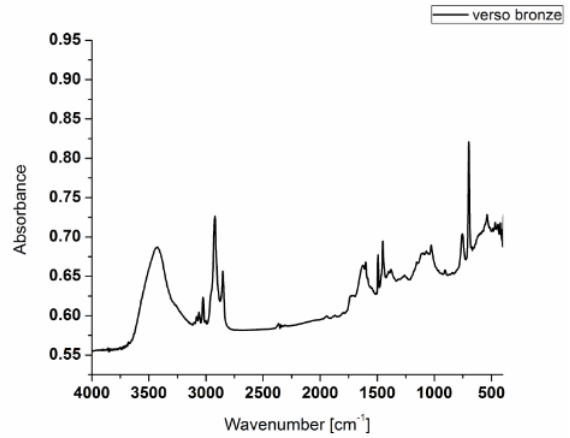


Fig. 7. FTIR spectrum of-verso bronze (Cu+Zn+Fe). A detailed part (3838-2393 cm^{-1} spectral range) of the FTIR spectrum- can reveal the added compounds to verso bronze: aromatic and aliphatic compounds traces, probably natural resins.

Conclusions

The icon presented in this paper is painted on both sides, and beside that, an older icon has been previously painted on one side, which is evidenced by X-Ray radiography. Both *St. Nicholas* wooden iconand *Jesus Christ* wooden icon painting materialshave been investigated using spectroscopic techniques. The presence of the specific pigments (Prussian blue, lithopone) and the style employed by the painter shows that *St. Nicholas* icon was made in the XVII^{lth} century.

Acknowledgments: This work was supported by a grant of the Romanian Minister of Research and Innovation, CCCDI - UEFISCDI, project number PN-III-P1-1.2-PCCDI-2017-0812 / 53PCCDI, within PNCDI III.

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Manuscript received: 17.07.218