MONITORIZAREA COMPORTĂRII MATERIALELOR DE RESTAURARE APLICATE EXPERIMENTAL ÎN BISERICA RUPESTRĂ "CORBII DE PIATRĂ"

MONITORING OF THE BEHAVIOR OF RESTORATION MATERIALS EXPERIMENTALLY APPLIED IN RUPESTRAL CHURCH CORBII DE PIATRĂ

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Applied more than a decade ago, within an interdisciplinary project carried out at the cave church from Corbii de Piatră (14th century), Argeș County, the conservation-restoration materials created by CEPROCIM were intended for a future intervention to consolidate the support of the murals inside a unique medieval monument of exceptional value. Monitoring the behavior of new materials applied in situ was required in the case of a monument characterized by a severe microclimate, with considerable variations in temperature and humidity, caused by the condensation and infiltration, generating a large process of biodeterioration. The observations made in situ were corroborated with laboratory analyzes, using specific investigation techniques: X-ray diffraction, scanning electron microscopy coupled with X-ray dispersive energy and optical microscopy. In the absence of measures to improve microclimate conditions and the continues degradation processes (crystallization of salts, biological contamination) was found to maintain the functional qualities of new materials applied in situ.

Aplicate în urmă cu mai bine de un deceniu, în cadrul unui proiect interdisciplinar desfășurat la biserica rupestră de la Corbii de Piatră (sec. XIV), județul Argeș, materialele de conservare-restaurare create de CEPROCIM erau destinate unei viitoare intervenții de consolidare a suportului picturilor murale din interiorul unui monument medieval unic, de o excepțională valoare. Monitorizarea comportamentului noilor materiale aplicate in situ se impunea în cazul unui monument caracterizat printr-un microclimat sever, cu variații considerabile de temperatură și umiditate, provenită din condens și infiltrații, generând un proces amplu de biodeteriorare. Observațiile realizate in situ au fost coroborate cu analize de laborator, utilizând tehnici specifice de investigare: difracție de raze X, microscopie electronică cu baleaj cuplată cu energie dispersivă de raze X. În absența unor măsuri de ameliorare a condițiilor de microclimat și continuarea proceselor de degradare (cristalizarea sărurilor, contaminare biologică) s-a constatat menținerea calităților funcționale ale noilor materiale aplicate in situ.

Keywords: *microclimate monitoring, restoration material, rupestral church, degradations*

1. Introduction

Ecclesiastical construction carved in the sandstone rock specific to the site, the church from Corbii de Piatră, Argeș County, is unique in the Romanian medieval architecture. With the western façade rebuilt in 1887, following the collapse of a part of the rock, the church is composed of a single-nave space, closed at the altar with two symmetrical apses. A narthex attached late (1814) to the southern side of the building makes the passage to the outside. Also, at the beginning of the 19th century, in 1819, the wall of the iconostasis of the church was raised and painted in fresco. On this occasion, a single apse of the altar is created by destroying the wall that separates the two initial apses, which have now become pastophores. The latest research, based on iconographic and stylistic analysis, formulated the hypothesis of dating the church together with its murals in the first decades of the fourteenth century. The votive painting, preserved fragmentarily in the central niche of the southern wall of the nave, could be the first known mural representation of Basarab I. At the same time, the pictorial ensemble would be the first testimony of Byzantine painting on Romanian territory, appearing in the late 13th century and the beginning of the next century.

The church belongs to the Corbii de Piatră monastery which was reopened in 2003. The church is included in the new list of historical monuments with the number LMI 2015: AG-II-m-A-13710.01.

Previous studies [1,2] have shown that the combined action of temperature and relative humidity of the air, salts and humidity in the walls of the monument, as well as biological colonization has led to the occurrence of degradation over time in both the mural and the its support. Biological colonization occurred in optimal conditions of temperature and humidity, the phenomenon being favored by the presence of natural light that enters through the windows in the western wall. The

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biological colonization produced degradations in terms of appearance, the fresco being covered by cyanobacteria, actinomycetes, algae and bryophytes [3], especially on the northern wall, where the infiltration humidity is higher. It also contributed mechanically to accentuate the phenomenon of gap formation. The salts come from the soil, infiltration water and masonry components of the iconostasis [1, 4]. The presence of salts in the walls led, through the phenomenon of dissolution-crystallization in optimal conditions of temperature and humidity, to the appearance of epi-efflorescences and crypto-efflorescences. They produced both degradation in terms of image, by covering the architectural surfaces with veils or crusts of salts, and mechanical, by crystallizing the salts inside the component materials of the monument, with the production of gaps of different lengths and depths.

In order to find solutions for the conservation of this historical monument, within the mentioned project, interdisciplinary studies were carried out which included restoration specialists, microbiologists, geologists, as well as in the field of restoration materials. Based on research on the composition of frescoes and their state of conservation, a series of restoration materials were made, characterized in the laboratory and tested *in situ*. *In situ* experiments were performed on small unpainted surfaces on the iconostasis and on the northern wall of the altar. Shortly after application (approximately 1 year) it was found that they had a good behavior, showing physical-mechanical, chemical and aesthetic compatibility with the original. During the same period, a test of biological decontamination and superficial removal of salts was performed on a little surface on the north wall of the nave.

The aim of the research presented in this paper was to evaluate the behavior of these new restoration materials after about 11 years from their experimental application *in situ*, as well as to evaluate the efficiency of salt cleaning and biological decontamination. The paper presents the results of microclimate measurements, analyzes on the current state of conservation of the original fresco and the materials applied experimentally *in situ*, as well as the salt removal and decontamination test.

2.Materials and methods

Microclimate measurements were made using a HOBO data logger U12-012 temperature and relative humidity sensor, with a temperature measurement range from -20^{o}C to + 70^{o}C and relative humidity from 5% to 95%. The humidity of the walls was determined with a Burg Wachter Dry PS 7400 type humidometer with the measuring range for solid materials (construction materials) from 0.2 to 3% (with the ranges: “good” = 0.2-0.3%, “at the limit” = 0.4-0.9% and “too high humidity” = >1%). The measurements were carried out along the walls of the nave, altar and iconostasis, at intervals of 2 m and at heights of 1.0, 1.5 and 2.0 m. Samples were taken from the northern wall of the altar and on the iconostasis, from places with veils or crusts of salt or biologically contaminated, to assess the current state of conservation of the original fresco. Samples were also taken from the mortars applied experimentally *in situ*, as well as from the northern wall of the nave, from the area decontaminated and superficially cleaned of salts in 2009, to evaluate the efficiency of the materials and methods used (Table 1). During the sampling of the restoration mortars, their consistency and their adhesion to the rock wall and to the brick masonry of the iconostasis were observed.

Table1

Place of sampling / *Locul de prelevare a probelor*

Sample code	Sampling location
S1	Iconostasis, north, towards the nave, left arm St. Philip, efflorescences in the form of veils
S2	Iconostasis, north, to the nave, angel wing, efflorescence in the form of compact veils
S3	Altar, North, foot of the first arch, efflorescences in the form of a hard crust
S4	Altar, North, median arch, efflorescence in the form of a hard crust
S5	Altar, North, anaphora vault, efflorescences in the form of a hard crust
S6	Altar, North, anaphora table, hard crusty efflorescences
S7	Naos, Northwestern niche, decontaminated area, salts on the fresco, hard crust
S8	Naos, Northwestern niche, decontaminated area,from the gap
ST	Altar, north wall, next to the area with experimentally applied restoration mortars
C1C ÷ C6C	Iconostasis, to the north, altar interior, restoration mortars
C3Z, C4Z, C5Z	stone wall, altar, North, restoration mortars

Table2

Content of soluble salts in restoration mortars / *Conținutul de săruri solubile în mortarele de restaurare*

Soluble compounds (%)	Mortar type					
	C1	**C2**	**C3**	**C4**	**C5**	**C6**
Na_2O	0.0000	0.0000	0.0073	0.0073	0.0000	0.0000
SO_3	0.0000	0.0000	0.0657	0.0657	0.0000	0.0000
Cl^-	0.0111	0.0124	0.0183	0.0183	0.0248	0.0230
Total	0.0111	0.0124	0.0913	0.0913	0.0248	0.0230

Restoration mortars applied *in situ* were mortars based on hydrated lime (codes C1 ... C6) whose physical-mechanical and chemical characteristics make them compatible with the original fresco, having a low apparent density (1.49 ... 1.70 g/cm^3), moderate compressive strength at 56 days (0.5 ... 3.1 MPa) and low content of soluble salts, expressed as SO_3, Cl^- and Na_2O respectively (Table 2), soluble salts with potassium was not found in the mortars [5].

The samples were characterized by X-ray diffraction, optical microscopy and scanning electron microscopy coupled with energy dispersive X-ray.

X-ray diffraction analysis (XRD) was used to identify the degradation compounds potentially formed in the fresco and restoration mortars. The XRD analysis was conducted using a Shimadzu XRD 6000 Diffractometer with Ni-filtered CuKα radiation (λ = 1.5406 Å). Diffraction patterns were taken at 0.05° intervals from 5° to 80°. The analysis of the lime plaster was performed on samples ground to pass through a 63 µm sieve.

Scanning electron microscopy coupled with energy dispersive X-ray analysis (SEM) was carried out to highlight the morphology and to identify possible degradation and biodegradation products. The analysis was performed using an Inspect F Quanta analyzer with 1.2 nm resolution (FEI-Philips, Netherlands), equipped with an energy dispersive X-ray (EDX) spectrometer with a resolution of 133 eV at MnK. The analysis was performed using high vacuum and an accelerating voltage of 30 kV. The samples were coated with a thin layer of gold.

Samples had been also analyzed under optical microscope Nikon AZ100.

3.Results and discussions

3.1. Microclimate

Inside the monument, between November 2019 and June 2020, the minimum air temperatures varied between 4.14^{o}C and 10.76^{o}C, and the maximum between 7.94^{o}C and 20.93^{o}C. The records of relative humidity showed an extremely humid microclimate, with lows between 46.75% and 73.69% and highs between 88.93% and over 95%. The values of the records from this period were slightly higher than those from 2008-2010, when the restoration mortars were applied, the minimum temperature being 2.46^{o}C, and the minimum relative humidity of 35.30% [6]).

The humidity of the walls is influenced by the humidity of infiltration, condensation and capillarity [4, 6]. For these reasons, the values of humidity measurements fluctuate along the walls and in height. No record is within the range indicated as "good" (0.2-0.3%) in the meter instructions. There are some records in the nave and altar that are towards the upper end (0.7-0.9%) of the "limit" range, the rest are in the "too high humidity" range, ranging from 1.1% to 2.7% and often exceeding the detection limit of the device. The presence of moisture in the walls is an important factor of degradation, it favors the migration of salts and biological contamination.

3.2. Observations during the sampling of restoration mortars

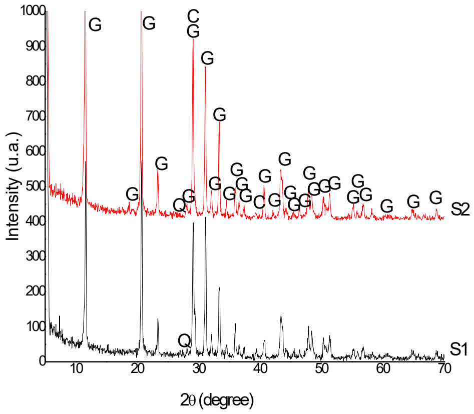
All restoration materials have a good adhesion to the rock wall of the altar and to the masonry of the iconostasis. During sampling, the rupture occurred in the restoration mortar. The exception was the C3 mortar on the iconostasis which came off at the mortar restoration-wall iconostasis interface, but this could be due to some deficiencies in its application on the wall. Restoration materials are compact without being hard. No cracks were observed on any of the applied mortars.

3.3. X-ray diffraction analysis

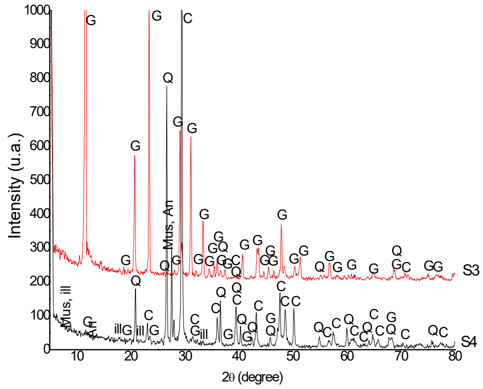
Samples taken from the original fresco on the iconostasis and on the north wall of the altar

XRD analysis revealed the presence of gypsum in most of the samples taken from the original fresco on the iconostasis and the altar. The gypsum in these samples is brought by the infiltration waters, coming from the Cascadei brook that passes through clay-silt rocks containing gypsum [4, 7]. The gypsum from these infiltration waters, in optimal conditions of temperature and humidity, crystallized either in the pores of the sandstone wall or in the pores of the fresco mortar or on the surface of the mural painting, causing their degradation. [4, 8].

The mineralogical analysis of the S1 and S2 samples taken from the areas with efflorescences from the upper part of the iconostasis, highlighted the almost exclusive presence of gypsum, as a product of degradation of the mural support (Fig. 1a). In the case of these samples, in addition to the infiltration waters, the gypsum can also come from the brick from which the iconostasis is built [9]. Traces of calcite and quartz come from the support of the mural. Samples S3 and S4 taken from the altar, from the arches from the north, the areas with crusts, contain gypsum, but in different proportions (Fig. 1b). In S3 sample, gypsum is predominant, calcite and quartz being in traces. Sample S4 taken from the median arch contains mainly the minerals of the fresco: mainly calcite and quartz, together with anorthite, illite, muscovite; the gypsum is in traces. Sample S5 taken from the vault of northern anaphora contains the minerals of the fresco (calcite, quartz, muscovite and anorthite); no gypsum was identified in this sample (Fig. 2a). In contrast, sample S6 taken from the crusts on the table of the northern anaphora in the altar contains mainly gypsum and traces of calcite and quartz (Fig. 2b). The variations observed in the gypsum content

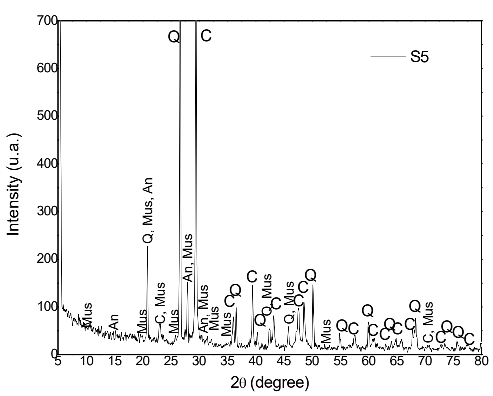


a



b

Fig. 1 - X-ray diagrams of samples S1 and S2 - veils of efflorescences from the upper northern part of the iconostasis (a) and of samples S3 and S4 - crusts on the northern arches of the altar (b): G – gypsum, C – calcite; Q – quartz; ill – illite; An – anorthite; Mus – muscovite / *Difractogramele probelor S1 și S2 - voaluri de eflorescențe în partea nordică superioară a iconostasului (a), și ale probelor S3 și S4 - cruste pe arcele nordice ale altarului (b): G – gips, C – calcit, ill – ilit, An – anortit, Mus – muscovite.*



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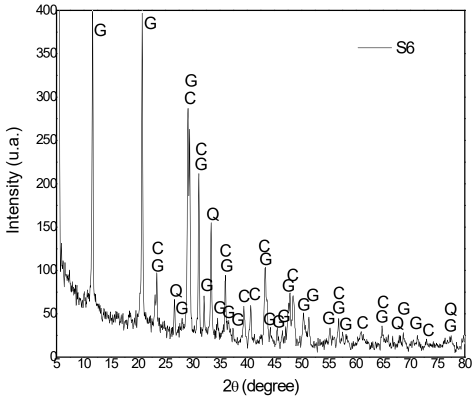


b

Fig. 2.- X-ray diagrams of the S5 sample from the altar, the vault of the northern anaphora, crusts (a) and the S6 sample from the altar, the mass of the northern anaphora, crusts (b): Q – quartz; C – calcite; An – anorthite; Mus – muscovite; G – gypsum / *Difractogramele probei S5 din altar, bolta nordică a proscomidiei, cruste (a) și probei S6 din altar, masa proscomidiei nordice, cruste (b): Q – cuarț, C – calcit, An – anortit, Mus – muscovit, G – gips.*

of samples S3-S6 are due to the existing crack system in the walls that allows the entry of infiltration water differently [7].

The X-ray diffractogram made on the S7 sample, taken from the crust on the decontaminated fresco and from which the salts were superficially removed, from the nave, the northern wall, shows the predominant presence of gypsum from infiltration waters, as well as traces of calcite and quartz from the support of the mural (Fig. 3).

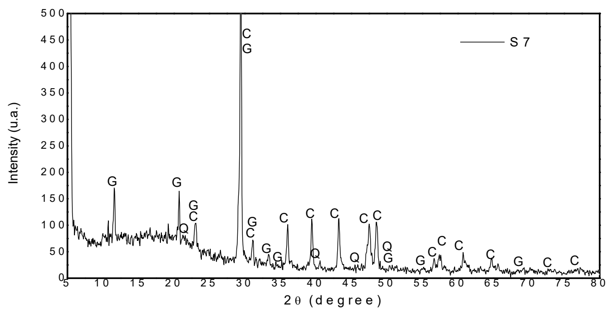
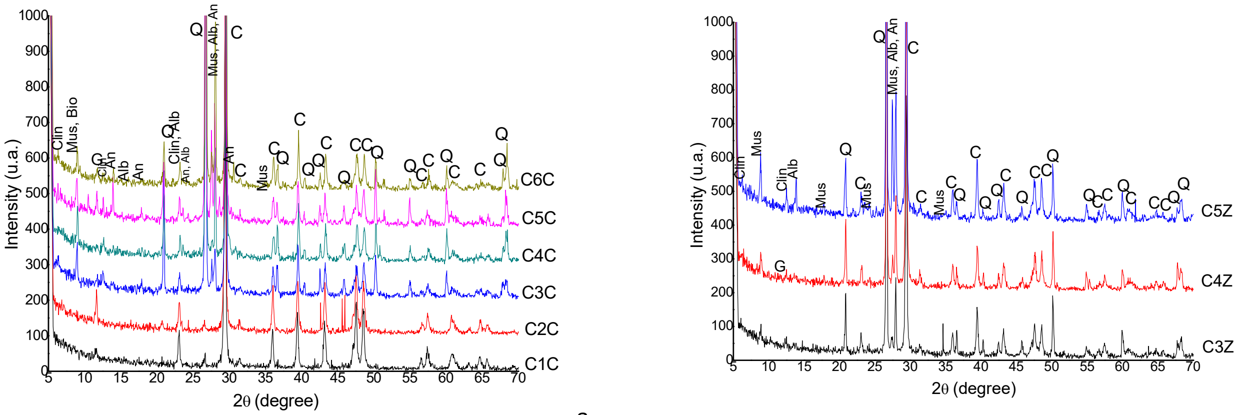


Fig. 3 -X-ray diagram of the S7 sample from the nave, decontaminated area, crusts: – gypsum; Q – quartz; C – calcite / *Difractograma probei S7 din naos, zona decontaminată, cruste: G – gips, Q – cuarț, C – calcit.*

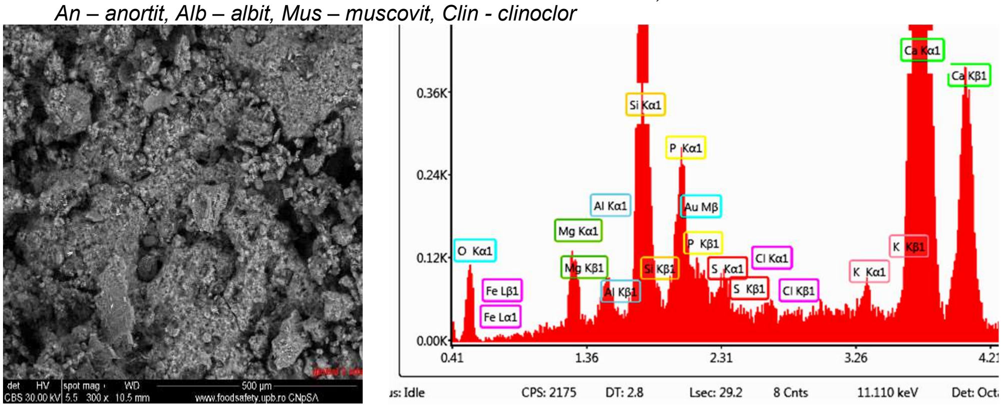
Samples taken from experimentally applied restoration mortars

In the case of samples taken from restoration mortars applied to the iconostasis, the intensity of the gypsum-specific lines in the X-ray diffractograms is lower than in the case of samples taken from the original fresco. This suggests that gypsum is found in smaller quantities in restoration mortars (Fig. 4a). In the restoration mortar samples on the wall, the gypsum was identified only in the C4Z sample, and only in traces. The other minerals identified are those of restoration mortars: calcite, quartz, anorthite, albite, muscovite, clinochlore (Fig. 4b).



a b

Fig. 4 - Diffractograms of samples taken from restoration mortars applied to the iconostasis (a) and to the northern wall of the altar (b): C – calcite; Q – quartz; G – gypsum; An – anorthite; Alb – albite; Mus – muscovite; Clin – clinochlore / *Difractogramele probelor prelevate din mortarele de restaurare aplicate pe iconostas (a) și pe peretele nordic al altarului (b): C – calcit, Q – cuarț, G – gips, An – anortit, Alb – albit, Mus – muscovit, Clin - clinoclor*



Element	Cantitate (%)
C K	20.1
O K	19.68
Mg K	1.29
Al K	0,2
Si K	8.64
P K	3.0
S K	0.42
Cl K	0.01
K K	0.23
Ca K	45.64
Fe K	0.78

a b c

Fig. 5 - Sample taken from the altar, near the area of experimental application of mortars: SEM image (a); elemental analysis spectrum (b) and quantitative elemental analysis (c) / *Probă prelevată din altar, aproape de zona mortarelor aplicate experimental: imagine SEM (a); spectrul de analiză elementală (b) și analiza elementală cantitativă (c).*

3.4.Scanning electron microscopy coupled with energy dispersive X-ray analysis

Sample taken near the area of experimental application of restoration mortars

The sample ST consists mainly of the calcium and silicon elements, as well as magnesium, aluminum, potassium, iron which are found in the minerals of the original fresco and the sandstone rock. The sulfur element was also identified in small quantities, coming from the infiltration waters with gypsum content (Fig. 5). The sample also contains the element phosphorus, which may be generated by biological contamination, the sample being covered by numerous branched hyphae (Fig. 6). Microbial colonization was very low, being visible only under electron microscope.

Samples taken from the surface cleaned of salts and decontaminated

The electron microscopy analysis of the S7 sample taken from the fresco revealed the presence of the sulfur element in both points analyzed with EDX (Fig. 7), which may be an indication that the sample taken from the crust on the surface of the fresco contains gypsum. Zone 2 has a lamellar morphology (Fig. 7 a) and is richer in sulfur

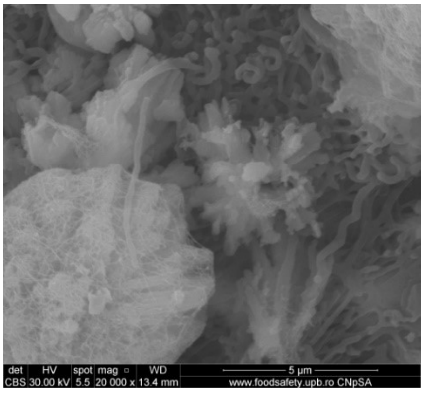
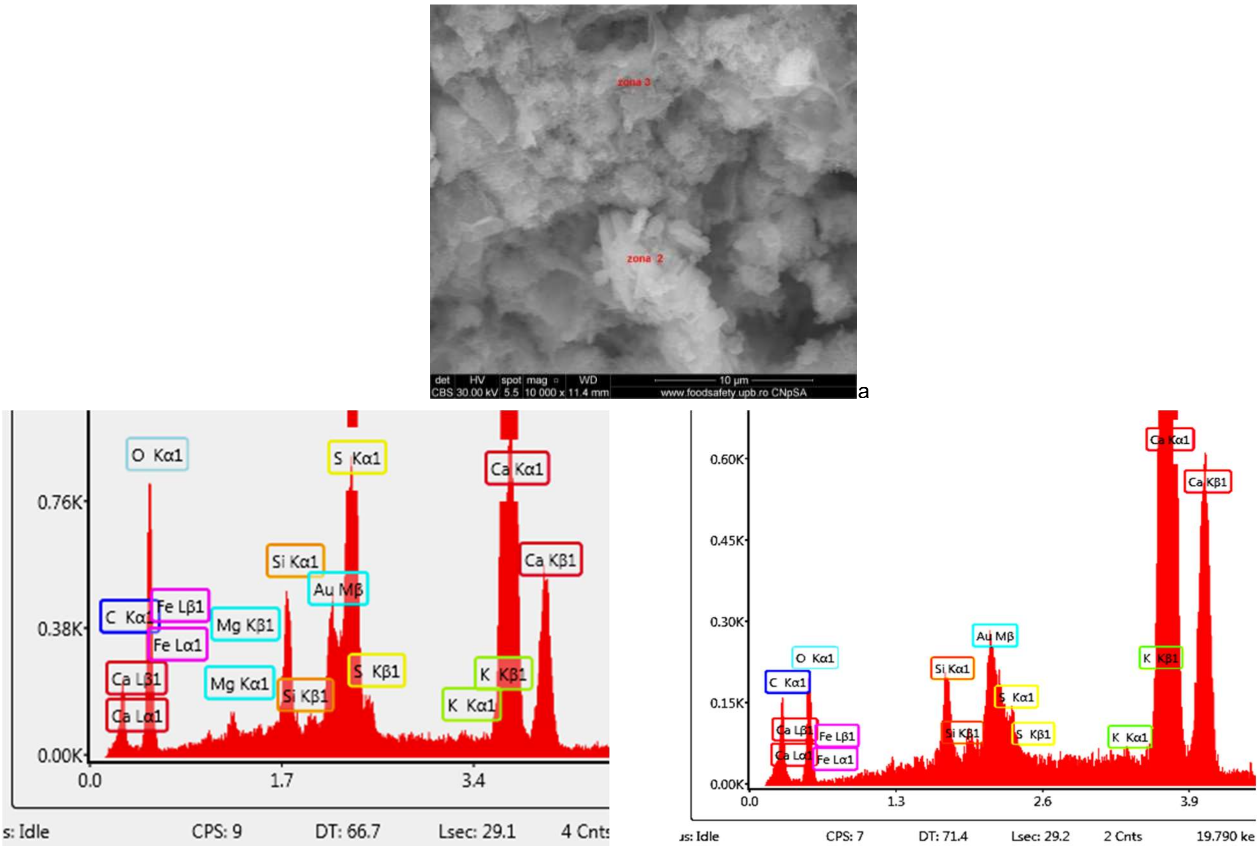


Fig. 6 - SEM image of the ST sample covered by branched hyphae / *Imagine SEM a probei ST acoperită cu hife ramificate.*

(Fig. 7 b) than zone 3 in which it is in traces (Fig. 7 c). The presence of calcium is due to the binder (lime), and the silicon, iron, magnesium, potassium elements belong to the mortar aggregate. This result correlates well with the diffractometric analysis, which highlighted the existence of gypsum, in addition to calcium carbonate from binder, quartz and other minerals of the aggregate. Although the surface of the fresco was cleaned in 2009 of salts, the fact that the water containing sulfate and calcium ions continues to infiltrate,

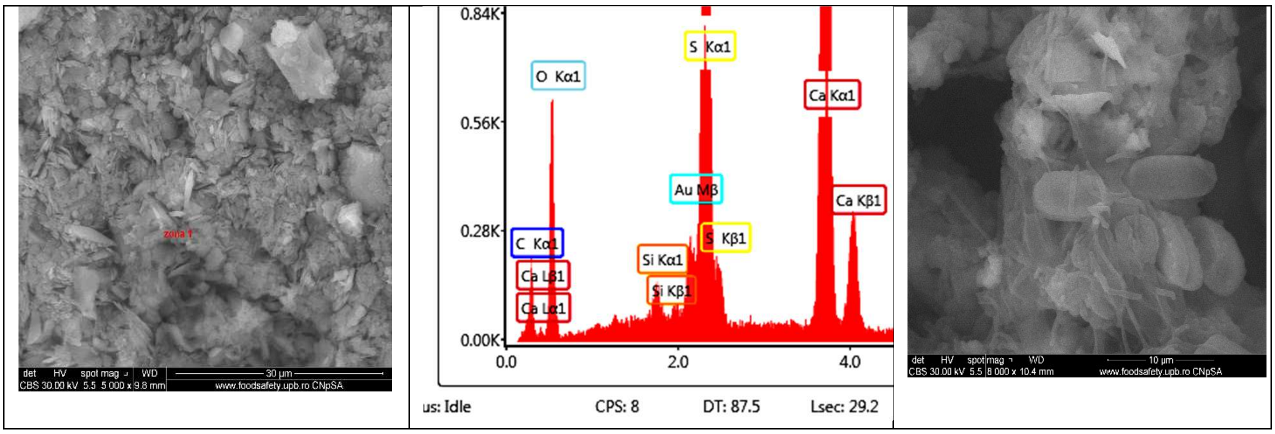


b c

Fig. 7 -S7 sample taken from the edge of the fresco decontaminated and cleaned of salts, from the nave, the western niche of the northern wall: SEM image (a); elemental analysis spectra of areas 2 (b) and 3 (c) / *Proba S7 prelevată de pe muchia frescei decontaminate și curățată de săruri, din naos, nișa vestică a peretelui nordic: imagine SEM (a); spectrele de analiză elementală a zonelor 2 (b) și 3 (c).*

from the Cascadei brook, the reappearance of gypsum crusts took place. No traces of biological contamination were observed on this sample.

In the electron microscopy image of the S8 sample, taken from the gap (Fig. 8 a), it is observed that the surface of the sample is covered with crystals in the form of lamellae. The elemental analysis performed on a group of such crystals highlighted mainly the elements sulfur and calcium (Fig. 8 b), which indicates the presence of gypsum in the sample. The surface of the sample is heavily contaminated biologically. In Figure 8 c actinomycetes and algae are observed.



a b c

Fig. 8 - S8 sample taken from the nave, the western niche of the northern wall, the gap in the surface cleaned of salts and decontaminated: SEM image showing gypsum crystals (a); elemental analysis spectrum of zone 1 (b) and actinomycetes (c) / *Proba S8 prelevată din lacuna de pe suprafața decontaminată și curățată de săruri, din naos, nișa vestică a peretelului nordic: imagine SEM în care se observă cristalele de gips (a); spectrul de analiză elementală al zonei 1 (b) și actinomicete (c).*

Samples taken from restoration mortars applied experimentally on the iconostasis

In all mortars experimentally applied on the iconostasis, the EDX spectra highlighted the sulfur element. It was observed on the entire surface of the investigated samples, being in large proportions, according to the quantitative elemental analysis (5.18 ... 24.78%). The presence of calcium beside sulfur, as well as the scaly or lamellar morphology suggests that there is gypsum in those areas (Fig. 9).

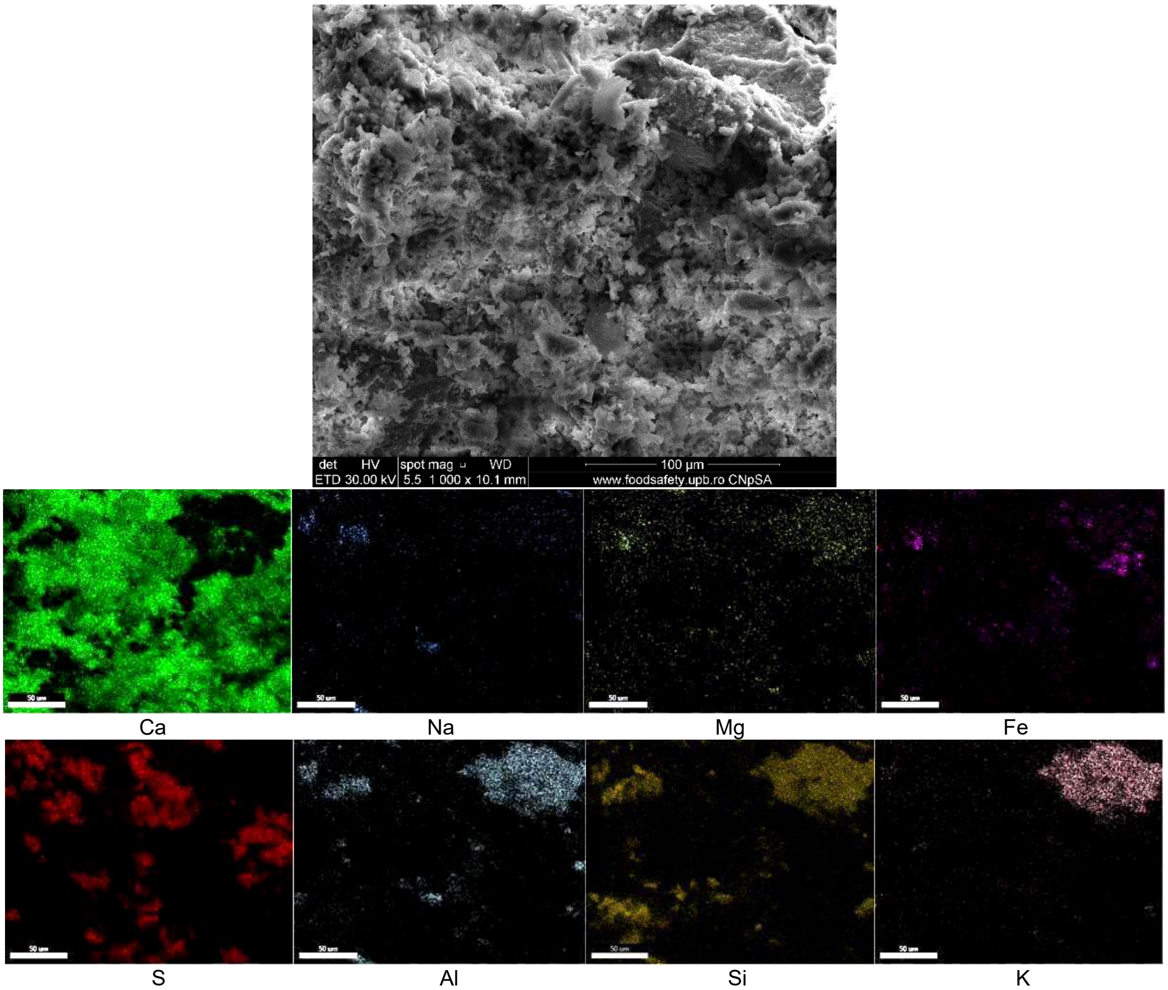


Fig. 9. - SEM image of the C3C sample and positioning of the elements contained in the sample / *Imagine SEM a probei C3C și poziționarea elementelor conținute.*

The samples are contaminated with actinomycetes, which are not visible under optical microscope but frequently visible in electron microscopic images (Fig. 10). Carotenoid pigment-producing bacteria have been identified in some areas with efflorescence under an optical microscope.

Samples taken from restoration mortars applied experimentally on the northern rock wall, altar

On the surface of the analyzed samples there is a fine crust in which the calcium element is predominant (Fig. 11). Possibly, due to the infiltration moisture, a slight solubilization of the calcium carbonate in the mortars took place, which, in optimal conditions of temperature and humidity and in the presence of carbon dioxide in the air, recrystallized on the surface of the mortars [10].

Sulfur was highlighted only on the crust surface of the C3Z sample, in proportion of 1.61% (Fig. 12).

Microbial colonization is not visible by direct observations or under the light microscope, but has been identified under the electron microscope.

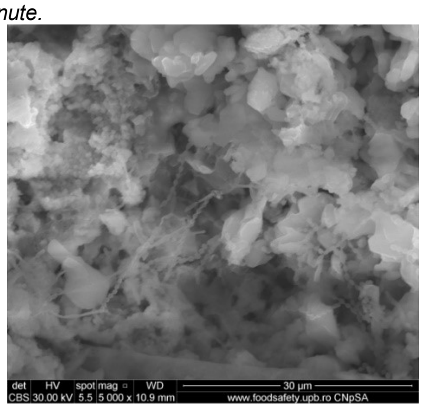


Fig.10 - SEM image of the C4C sample: actinomycetes included in the biofilm / *Imagine SEM a probei C4C: actinomicete incluse in biofilm.*

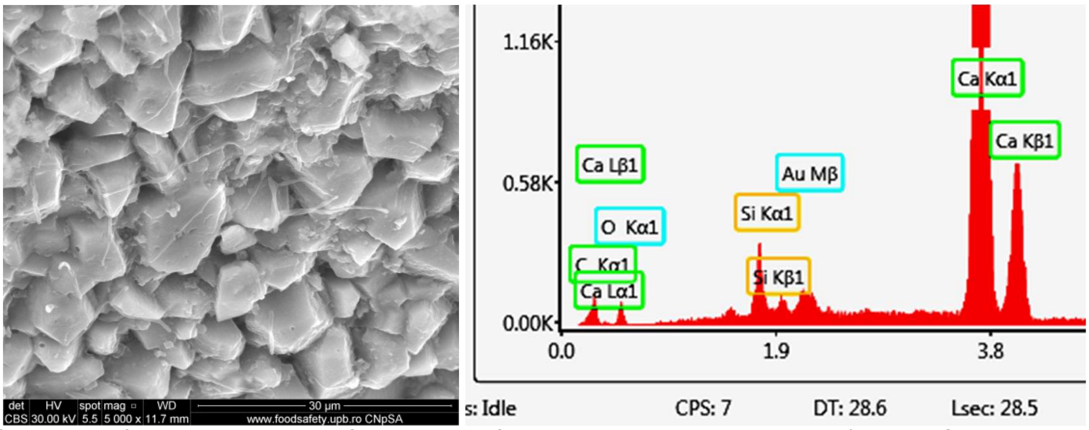


Fig. 11 - SEM image of calcite crystals on the C5Z sample surface and related elemental spectrum / *Imagine SEM a cristalelor de calcit de pe suprafața probei C5Z și spectrul elemental afferent.*

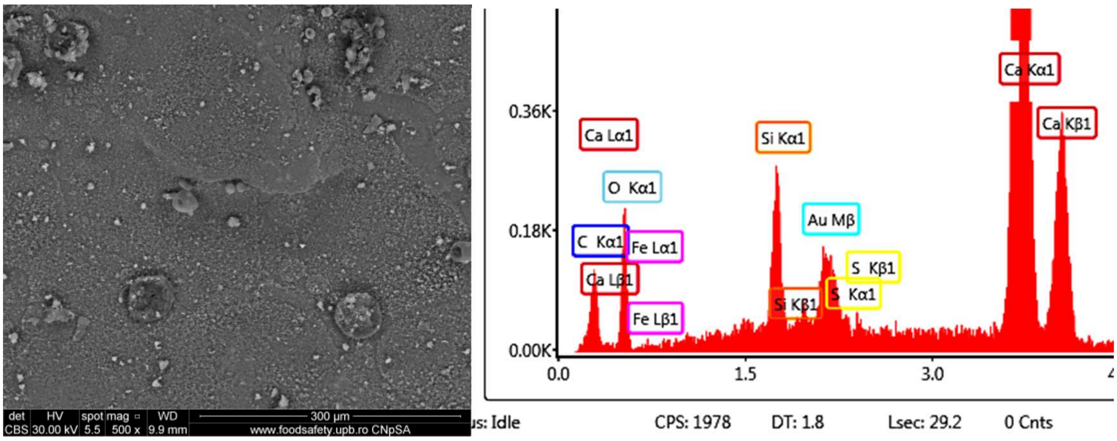


Fig. 12. SEM image of the C3Z sample and the elemental spectrum / *Imagine SEM a probei C3Z și spectrul elemental.*

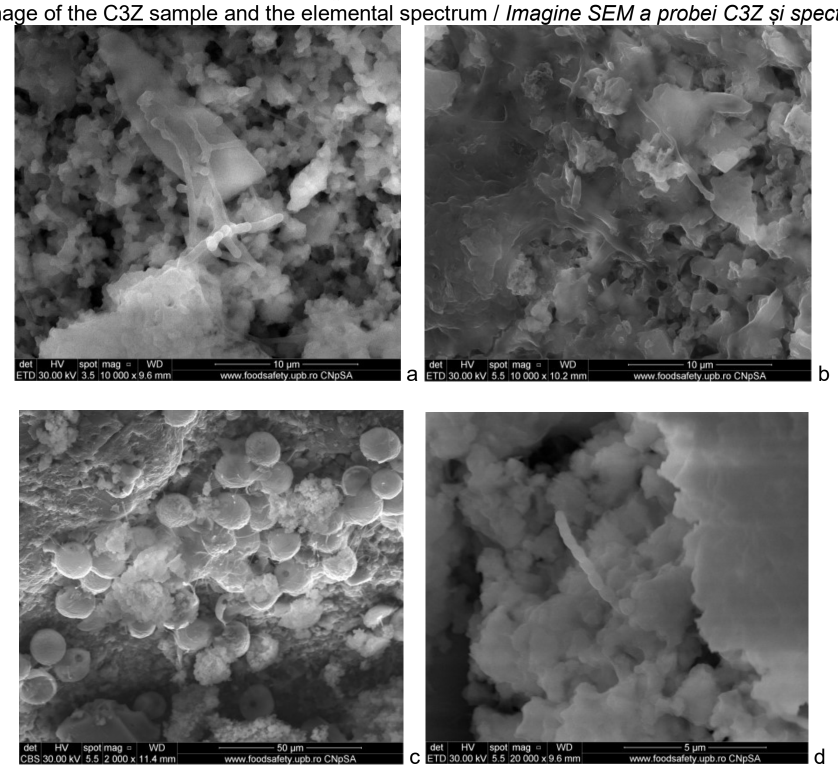


Fig. 13.- Biological contamination of restoration mortars applied on the Nordic wall of the altar: a - non-sporulated mycelium formed by weakly branched hyphae; b - biofilm consisting of single-celled algae to which hyphae adhere; c - coci; d - bacilli arranged in chains / *Contaminare biologică a mortarelor de restaurare aplicate pe peretele nordic al altarului: a – miceliu nesporulat format de hife slab ramificate; b - biofilm format din alge unicelulare la care aderă hifele; c - coci; d - bacili dispuși în lanțuri.*

Has been put in evidence bacilli arranged in chains, cocci, non-sporulated mycelium formed by weakly branched hyphae, biofilm formed by green algae adhering to hyphae (Fig 13).

Measurements of the microclimate and of the humidity of the sandstone walls and of the masonry of the iconostasis, as well as the results of the analyzes performed on the samples taken, show that the degradation process is active. The presence of efflorescence and biological contamination was further observed in the various points analyzed, and in the area cleaned of salts and decontaminated; although slowly, there was crystallization of gypsum from permanent infiltrations with sulfate and the reappearance of biological degradation.

4.Conclusions

In this paper was evaluated the behavior of restoration materials applied experimentally, in 2009, in the cave church Corbii de Piatră. The efficiency of surface cleaning of salts and decontamination of a small surface with original frescoes and gaps on the northern wall of the nave was analyzed. The current state of preservation of the original frescoes has been assessed. Measurements of the microclimate and humidity of the church walls were performed. The following were found:

- positive air temperatures were recorded, even during winter, the minimum being 4.14°C;
- inside the church there is an extremely humid microclimate, with minimums between 46.75% and 73.69% and maximums between 88.93% and over 95%;
- the records of the humidity of the walls of the nave and altar, as well as of the wall of the iconostasis, determined up to a height of 2 m, are in few places "at the limit" (up to 0.9%), the rest being in the field of "too high humidity" between 1.1% and 2.7%, often above the detection limit of the device (3%);
- most of the samples taken from the original fresco contain gypsum brought by the infiltration waters, coming from the Cascadei stream, which passes through clay-silt rocks containing gypsum; in the altar the samples have a lower gypsum content and are variable depending on the network of cracks in the rock that allows the infiltration of water with gypsum content; in the samples on the iconostasis the plaster also comes from its brick masonry;
- the samples taken from the surface cleaned of salts and decontaminated (from the fresco and from the gap) contain gypsum, also coming from the infiltration waters;
- samples taken from restoration mortars applied to the iconostasis contain gypsum, as a result of infiltration water, capillarity, as well as the constituent elements of the iconostasis;
- from a microbiological point of view:
    - o the sample (ST) taken from the area of application of the restoration mortars from the altar has a very low biological contamination, being visible only under electron microscope;
    - o the S8 sample taken from the gap of the decontaminated area is contaminated by actinomycetes visible only under SEM;
    - o samples taken from the iconostasis are contaminated only by carotenoid pigment-producing bacteria;
    - o samples taken from the rock wall: microbial colonization is not visible by direct observations or under the light microscope but has been identified under the electron microscope.

In the absence of measures to remove infiltration moisture and salts, as well as due to the microclimate specific to caves, the degradation process continues, and restoration mortars applied in situ on an experimental basis generally have salt crystals on their surface and a low contamination with microorganisms coming from atmosphere.

Despite the ongoing degradation processes, the mortars have maintained their quality of consolidating the intervention areas.

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