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DETERMINING THE CHARACTERISTICS OF CONCRETE IN A HISTORICAL BUILDING UNDER THE INFLUENCE OF CLIMATIC AND BIOLOGICAL FACTORS

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The object of this study was concrete samples of the cathedral and wall frescoes. The study solved the problem related to the destruction of concrete and wall frescoes under the long-term influence of biochemical and climatic factors.

Samples of concrete for research and wall murals were obtained from a historic listed building. Using microbiological studies and scanning electron microscopy, damage to wall murals and concrete by microscopic fungi was established: Aspergillus fumigatus, Penicillium brevicompactum, Aspergillus niger, Cladosporium sphaerospermum.

The study of concrete samples by the TPD-MS method showed the presence of an increased level of moisture and carbon compounds by 20 % in the test samples, compared to control. The sulfur content in all concrete samples was not significant. Determination of the mineral composition of concrete by X-ray diffraction showed the presence of Al_2O_3***, 36–44 %, which indicates a significant clay content. The presence of NiTi, 53 %, and*** $CoMg_7O_8$***, 46 %, in the concrete sample indicates the probable migration of the chemical elements of the paint pigments used to decorate the cathedral. The concrete control sample contained a significant amount of*** SiO_2***, up to 51 %, which is the main component of sand. A feature of the work is the determination of the corrosion effect on concrete under prolonged exposure to climatic and biological factors. The present study is distinguished by the use of non-destructive methods: microbiological studies, scanning electron microscopy, TPD-MS and X-ray diffraction to determine the destruction of concrete and wall frescoes of the building, which is a cultural heritage. The results of the study could be applied to the development and planning of restoration works for the restoration of buildings that have historical value***

Keywords: architectural monument, corrosion of concrete, destruction of wall frescoes, mineral composition of concrete

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1. Introduction

The Holy Resurrection Cathedral was built in 1702 in the 17th century. The architectural monument was built as a defensive structure, it has massive walls, narrow windows. The church is three-chambered and three-domed, built in the Ukrainian Baroque style. At the height of the second floor, the building becomes octagonal.

Concrete as the main building material began to be used for the construction of the pyramids in ancient Egypt. The material that is called "concrete" is fundamentally different in every structure that was built before the beginning of its industrial production. However, such natural components as limestone, sand, clay are very often found in most old concrete. It is interesting that despite the long period of operation, old buildings built from completely natural construction materials are still suitable for operation. However, given the influence of anthropogenic factors, natural disasters, and military operations, architectural monuments have suffered a lot in recent years.

Also, one of the enemies of natural concrete are microorganisms that are aggressive towards the material, use it as a substrate for growth and development, leading to its biogenic destruction as a result of metabolism. Microscopic fungi in the process of vital activity secrete substances – exotoxins, which completely change the chemical composition and, as a result, the structure of concrete. The most harmful effect is on the frescoes in the cathedral since after their destruction the possibility of their full restoration is lost. The problem is the preservation of frescoes as cultural and historical value, national heritage, and ethnicity. The use of scanning electron microscopy makes it possible to establish the depth of destruction in concrete. In addition, one can get visual confirmation of the presence of microscopic fungi in the building material. Microbiological research methods were used to identify microorganisms. Thermal stability of concrete samples and its chemical composition were determined by the method of temperature-programmed desorption mass spectrometry. With the help of the X-ray diffraction method, it is possible to determine the specificity of the mineral composition of the concrete of the historical building in comparison with the modern one.

It is important to preserve historical buildings through the use of new non-destructive diagnostic methods, which make it possible to predict the period of their operation and timely detection of damage in order to plan restoration works.

2. Literature review and problem statement

Old buildings, especially those with historical value [1, 2], require constant monitoring of their operational characteristics. For old buildings, it is necessary to use diagnostic methods that have a small destructive effect on buildings. The authors of paper [3], during the inspection of the water tower in Zabrze (Silesia Voivodeship, Poland) erected in 1909, established the presence of concrete carbonization as a result of sulfate corrosion. The issue of biogenic corrosion of the structure remained unexplored in the work. In addition, the depth of the corrosion process in concrete is not determined.

The development of new types of concrete requires compliance with the basic principles of design and mathematical forecasting, taking into account the operating conditions of the structure [4].

Emphasis is on the preservation of historical heritage in work [5] through the use of the latest technologies and tools. Special test monitoring systems allow timely detection of the degree of destruction of buildings during inspection and predict possible degradation of heritage buildings in the future. However, the use of exclusively mathematical prediction may not be enough to diagnose building damage, and it is better to rely on several methods at the same time to avoid calculation errors.

In papers [6, 7] it is stated that a thorough examination before the restoration of the building is an important stage. Because of an inaccurate and superficial diagnosis, repair work was carried out, which did not lead to the elimination of destruction but on the contrary, to its increase. The given examples of false diagnosis provide grounds for expanding the range of methods for building inspection. However, it is necessary to take into account the historical value of the site and use the most non-destructive methods.

Sometimes old buildings are demolished because of the danger of spontaneous destruction, especially in places with a large concentration of people. However, in most cases, valuable historical buildings [8] are regularly inspected, repaired, and protected. Old concrete structures require determination of their physical, chemical, and mechanical properties in order to properly evaluate the structure. Paper [9] investigated the change in the structure of concrete during seismic activity. The results showed significant damage to concrete structures, especially old ones.

Works [10, 11] tackled the problem of destruction of historical frescoes on the walls of buildings, which are cultural heritage in many countries. The main processes of destruction of frescoes are revealed, such as cracking, flaking, peeling, destruction, and crumbling. The researchers established that the cause of the destruction may be the influence of climatic conditions and biogenic corrosion. However, the study does not specify the microorganism that was isolated from the destroyed frescoes and does not suggest methods of their destruction.

Studies of damage to balconies, which have historical value, showed that the location of local thermal bridges coincides with the actual location of areas of concrete destruction in the walls [12]. Studies prove that the diagnosis of the destruction of concrete structures by several methods confirm the results of each other and are not mutually exclusive. In addition, due to the use of several methods, the chances of not making mistakes when diagnosing the operational condition of an old historical building increase.

Our analysis of the literature reveals that there is a problem of the destruction of historical facilities and their timely inspection. To predict the degradation of old buildings, there is a need to use the latest non-destructive diagnostic methods that provide the most accurate results possible. The simultaneous use of several research methods reduces the probability of false prediction of the degree of corrosion of concrete structures. Based on the results, it is possible to devise measures to reduce the destruction of the structure. Also, when studying a unique historical building, there is an opportunity to investigate the mineral and chemical composition of natural concrete and the corrosive effect of biogenic and non-biogenic origin.

3. The aim and objectives of the study

The purpose of our study was to determine the characteristics of the mineral and chemical composition of the concrete of the historical building and its destruction over time. This will make it possible to predict the evolution of destruction over time under the influence of climatic and biogenic factors. Based on the results of the experiments, it is possible to make a reasonable forecast of the operational period of the historical building (church) and plan restoration works.

To achieve the goal, the following tasks were solved:

– to investigate the presence of corrosion changes in concrete using electron microscopy and conduct thermal analysis of concrete samples using the TPD MS method;

– to determine the mineral composition of the concrete of the old building using X-ray diffraction.

4. The study materials and methods

4. 1. The object and hypothesis of the study

The idea of the research was that under the conditions of constant influence of climatic factors (changes in temperature, thawing and melting, humidity, carbon dioxide) structures are exposed to long-term effects of corrosion, which leads to changes in the structure, mineral composition, and heat resistance of concrete. Therefore, a set of non-destructive research methods was proposed for the current study: thermoprogrammed mass spectrometry, X-ray diffraction, and scanning electron microscopy.

The object of the research is wall frescoes and concrete of a historical building.

The subject of the study is the patterns of long-term interconnected physical and chemical destructive changes in concrete.

The main hypothesis of the study assumes that based on the determination of structural and mineral changes in concrete, it is possible to predict the life of the building and plan restoration works.

Samples of concrete for research (No. 1 and No. 3) and wall frescoes were obtained from the Holy Resurrection Cathedral in the city of Sumy, Ukraine (Fig. 1, *a, b*). Sample No. 2 is a control sample obtained from the bell tower of the cathedral, which was built in 1900, on the site of the old destroyed one. Concrete samples were tested in the electron microscopy laboratory at the Sumy National Agrarian University and in the laboratory of the radiation biophysics department at the Institute of Applied Physics of the National Academy of Sciences of Ukraine (Sumy, Ukraine).

a *b*

Fig. 1. Image of external damage to the facade of the Holy Resurrection Cathedral:
a – destruction of the basement part and wall of the belfry;
b – corrosion of church walls and columns

4. 2. Methodology of concrete sample research using TPD MS

Concrete samples were obtained from the plinth and walls of the cathedral with a size of 0.2–0.5 cm^2. Concrete samples weighing 5–10 mg were heated at temperatures from 40 to 900 °C in a thermally programmed mass spectrometry setup. At the same time, the mixture of gases was recorded using the mass spectrum, which was separated during heating. Gases were identified by molecular masses (m/z): H_2O – 18, CO – 28, CO_2 – 44, S – 32, SO_2 – 64 [13].

4. 3. Methodology of scanning electron microscopy of concrete samples

The study of the microscopic structure of concrete samples was carried out by the method of scanning electron microscopy using the device (REP 106 i) (VAT SELMI, Sumy, Ukraine) under the mode of secondary electrons at the range of electron-optical magnification from 200 to 5,000 times. The samples were previously covered with a layer of silver to provide electrical conductivity [14].

4. 4. Methodology of researching the mineral composition of concrete

The mineral composition of the samples was studied using a DRON 4-07 X-ray diffractometer. Shooting was performed in the range from 200 to 800, where 2θ is the Bragg angle. For research, the samples were previously ground to a powder state of 15–20 µm and placed in a fixative. It was used for X-ray focusing tests according to Bragg-Brentano [15].

4. 5. Method of conducting microbiological research

Samples for microbiological research were taken from wall frescoes in the cathedral. The frescoes were washed and pieces of paint that had peeled off the walls were collected and placed in sterile containers. Microscopic fungi were grown in Petri dishes on Capek-Dox medium. Exposure of samples in a thermostat lasted 10 days at a temperature of 22 °C. Colonies were then identified [16].

5. Results of the study of concrete samples of the historical building

5. 1. Results of research into the presence of corrosion changes in concrete

Long-term exposure to climatic factors such as humidity, temperature changes, and carbon dioxide have a negative effect on concrete. Carbonization of concrete results in microscopic cracks and splits that move deep into the building.

This study is aimed at determining the structural properties, chemical and mineral composition of old concrete (321 years old), which was used for the construction of the Holy Resurrection Cathedral in the city of Sumy, Ukraine.

The historical wall frescoes are also subject to corrosion due to the high humidity (condensation) that occurs during the cold period of the year as the church has no heating. In addition, there are favorable conditions for the growth of microscopic fungi, which were found on the walls with paintings (Fig. 2, *a*, *b*).

Microbiological studies were conducted to confirm micromycete damage to frescoes and concrete, which established the presence of fungal colonies (Fig. 3).

a *b*

Fig. 2. Image of wall frescoes affected by microscopic fungi: *a* – entrance to the church; *b* – the main hall of the church

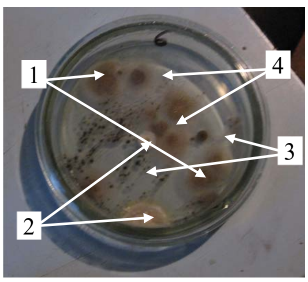

Fig. 3. Colony images of microscopic fungal isolates obtained from wall murals identified as: 1 – *Aspergillus fumigatus*, 2 – *Penicillium brevicompactum*, 3 – *Aspergillus niger*, 4 – *Cladosporium sphaerospermum*

The growth of microscopic fungi was also found in the samples (Fig. 4); consequently, corrosion of concrete is a result of the metabolism of microorganisms.

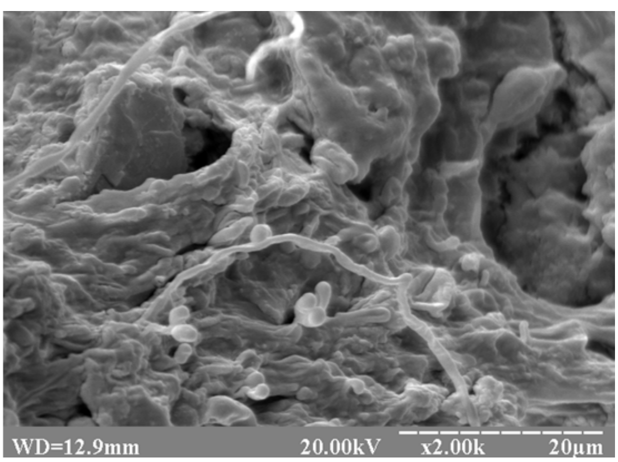

Fig. 4. Raster electron microscopic image of concrete samples with the presence of microscopic fungal growth

Structural changes in concrete under the influence of long-term climatic changes and biological corrosion were also investigated by the TPD MS method. Concrete samples were heated to a temperature of 1000 °C (Fig. 5).

Research has established the release of the maximum amount of moisture from sample No. 3 at a heating temperature of up to 130 °C with an intensity of 0.3. Sample No. 1 lost moisture with an intensity of 0.25 at a temperature of 40 °C, indicating the brittleness of the concrete sample. In concrete sample No. 2 (control), the intensity of H_2O evaporation was almost equal to 0.

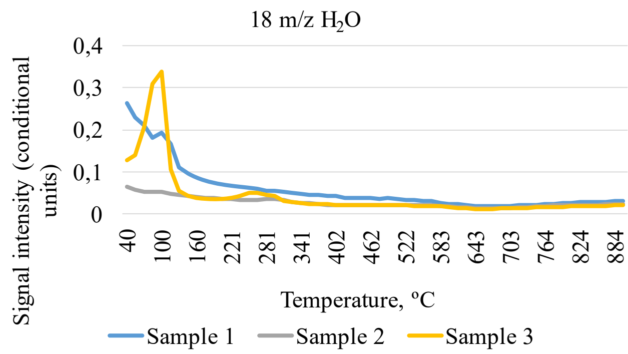

Fig. 5. Intensity of release of H_2O (m/z=18) from concrete samples of a historical building

The integrity of the structure of the samples was also shown when determining the intensity of carbon monoxide emission (Fig. 6).

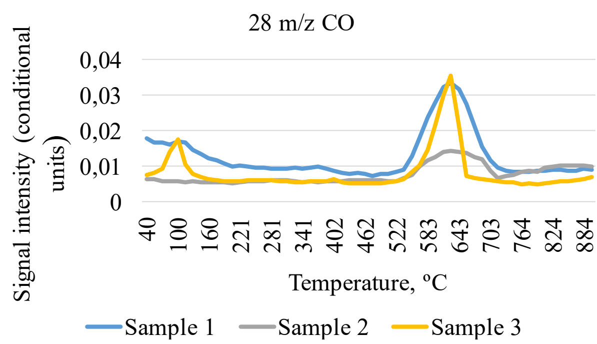

Fig. 6. Intensity of emission of CO (m/z=28) from concrete samples of a historical building

The intensity of CO emission from samples No. 1 and 3 was the same within 0.03 at a temperature of 643 °C. At the same time, the intensity of carbon monoxide emission was 0.015.

A similar result was also observed when determining carbon dioxide in concrete samples (Fig. 7).

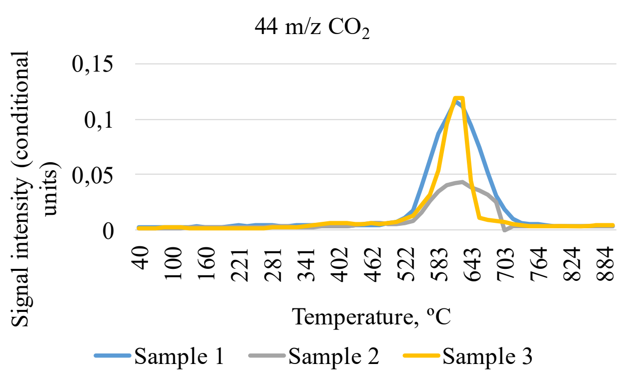

Fig. 7. Intensity of release of CO_2 (m/z=44) from concrete samples of a historical building

The intensity of CO_2 emission from test samples No. 1 and No. 3 was 0.15 at a heating temperature of 643 °C. The results of the emission of carbon dioxide from control sample No. 2 show an intensity of 0.03 at t=643 °C.

Sulfur content in concrete samples was also determined using the thermoprogrammed mass spectrometry method (Fig. 8).

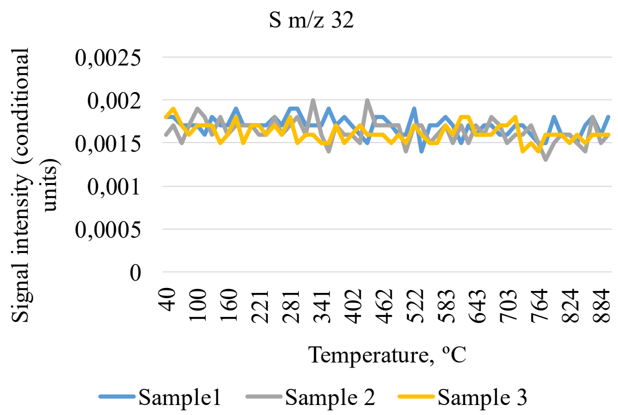

Fig. 8. Intensity of release of S (m/z=32) from concrete samples of a historical building

Our results of the thermogram show that in the test and control samples of concrete, the content of S was not significant during the entire period of heating from 40 to 884 °C with a constant intensity of 0.0015–0.002. The result obtained with the thermogram of SO_2 release from concrete samples is completely repeated (Fig. 9).

Heating of concrete samples No. 1, No. 2, and No. 3 at temperatures from 40 to 884 °C shows the intensity of sulfur dioxide release in the range of 0.0015–0.002.

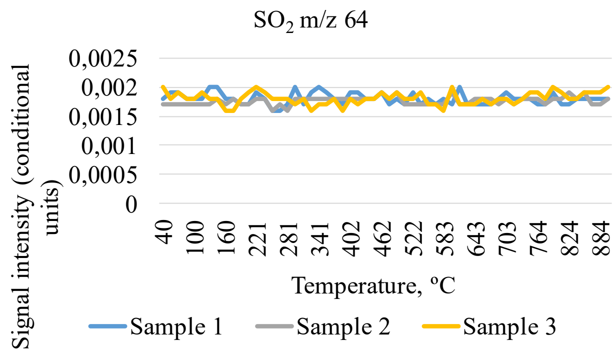

Fig. 9. Intensity of SO_2 release (m/z=64) from concrete samples of a historical building

5. 2. Results of investigating the mineral composition of concrete using X-ray diffraction

X-ray diffraction method was used to determine the mineral composition of the concrete of the historical building (Fig. 10).

The resulting diagram shows the presence of 36–44 % Al_2O_3 in the sample. This is shown by the peaks in Fig. 10. Aluminum oxide is one of the main components of clay rocks, which were used as a building material hundreds of years ago. The X-ray diffractogram of the control sample of concrete No. 2 has two intense peaks (Fig. 11).

Control sample No. 2 was dominated by silicon dioxide (SiO_2) with a relative amount of 39 to 51 %. In modern concrete, a significant share is sand, the main chemical element of which is SiO_2. The X-ray diffractogram of concrete test sample No. 3 differed from the previous ones in terms of composition (Fig. 12).

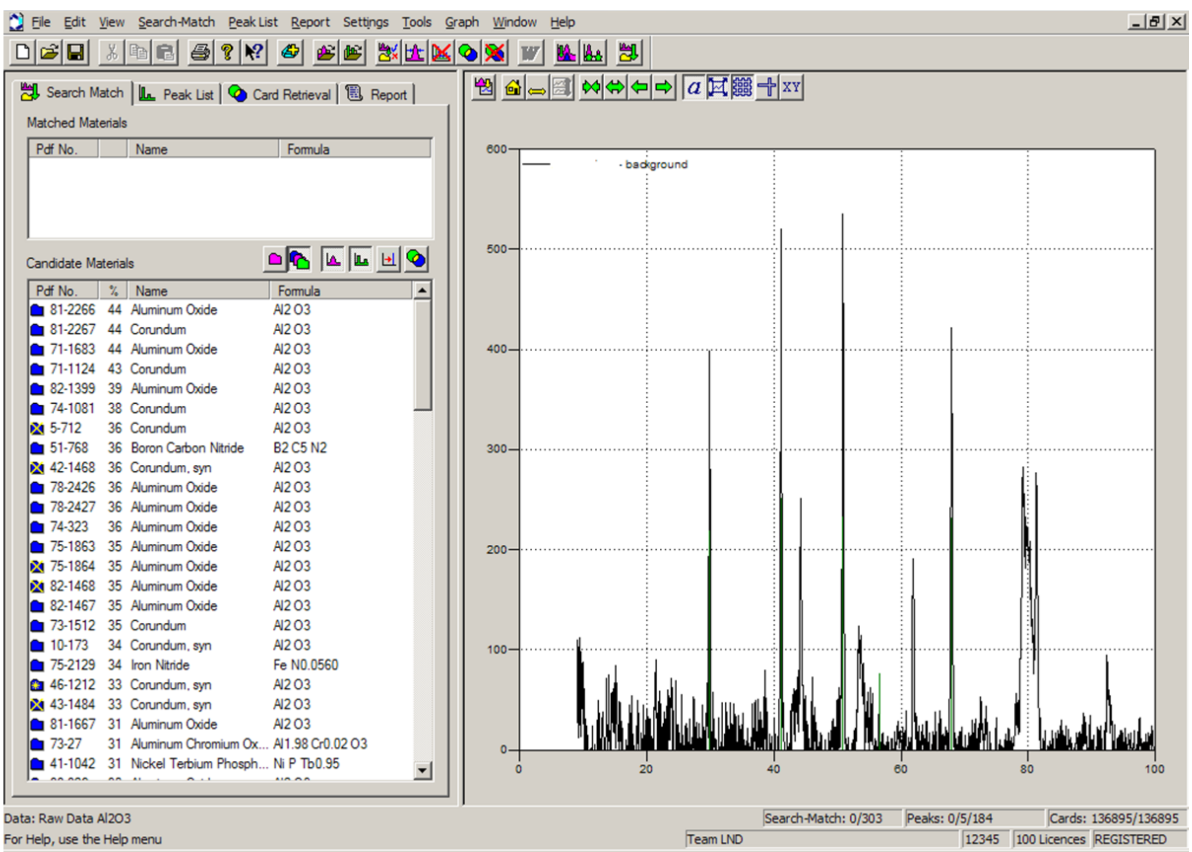

Fig. 10. Results of examination of concrete sample No. 1 using an X-ray diffractometer

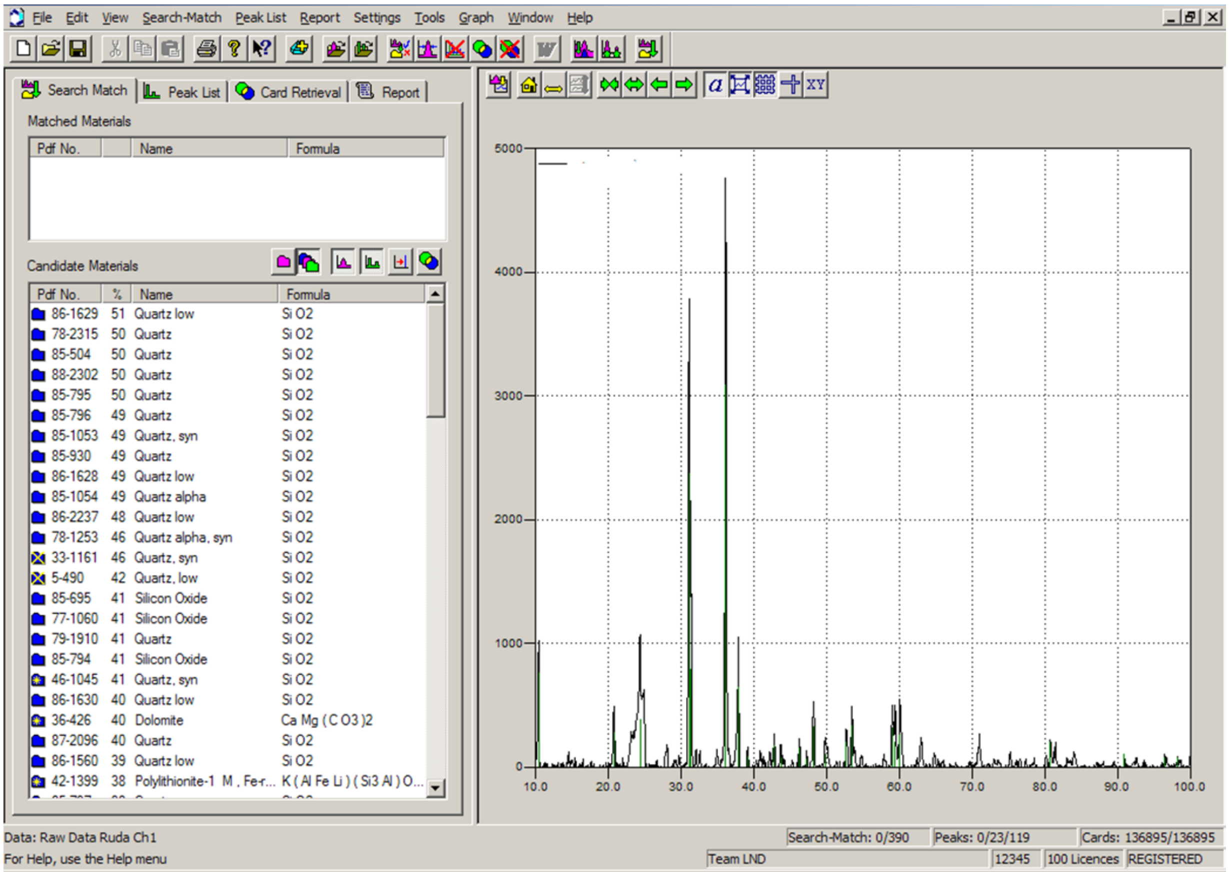

Fig. 11. Results of examination of concrete sample No. 2 (control) using an X-ray diffractometer

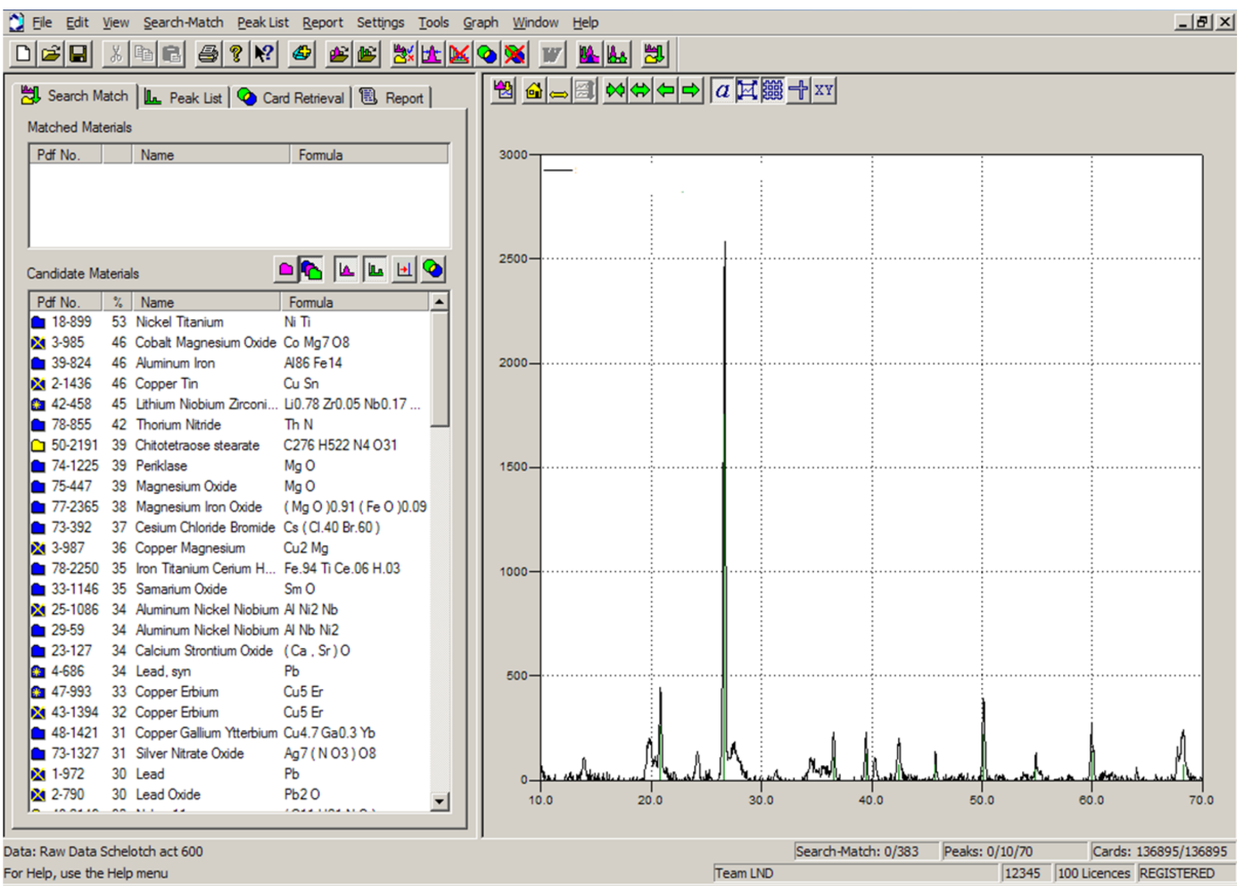

Fig. 12. Results of examination of concrete sample No. 3 using an X-ray diffractometer

Based on the results of the mineral composition of the sample, a relatively high amount of NiTi (nickel titanium) was established – 53 %, $CoMg_7O_8$ (cobalt magnesium oxide) – 46 %. The walls of the church were painted more than once over the years, so its components got into the concrete.

6. Discussion of results of studying the corrosion changes and mineral composition of concrete

When examining the wall frescoes of the cathedral, it was established that their biological corrosion was caused by microscopic fungi (Fig. 2, *a*, *b*). Study [17] confirms that microscopic fungi are one of the main reasons for the destruction of frescoes. Microbiological studies (Fig. 3) were carried out for the presence of micromycetes: *Aspergillus fumigatus*, *Penicillium brevicompactum*, *Aspergillus niger*, *Cladosporium sphaerospermum*, as well as scanning electron microscopy (Fig. 4). In [18, 19], the effects of biological corrosion on concrete structures of a chemical enterprise are considered.

Examination of concrete samples by the TPD MS method showed the presence of a large amount of moisture (Fig. 5) compared to the control. Concrete has been exposed to anthropogenic influence and climatic factors for a long time (more than 300 years). Therefore, as a result, microcracks and cracks appeared in which moisture accumulates. The control sample is obtained from a building that is much younger, so the concrete is of higher quality and less destroyed. Similar results were reported by scientists in [20] when investigating the causes of corrosion of livestock buildings. In addition, the intensity of the emission of carbon monoxide and carbon dioxide during heating of the experimental samples was 20 **%** higher compared to the control sample (Fig. 6, 7). The result is explained by the fact that clay was used as the main building material for the construction of the cathedral. This is confirmed by the results of X-ray diffraction (Fig. 10). Scientists [21] considered the shift of brickwork as a result of the use of different components of cement masonry.

Younger concrete No. 2 of the control sample does not contain a significant volume of carbons but a large proportion of silicates (Fig. 11). Thermograms (Fig. 8, 9) showed the presence of an insignificant content of sulfur and sulfur dioxide in the experimental and control samples of concrete. Studies [22, 23] also raised the issue of the peculiarities of the restoration of old buildings.

The X-ray diffractogram of concrete test sample No. 3 showed the presence of a significant amount of NiTi (nickel titanium) – 53 % and $CoMg_7O_8$ (cobalt magnesium oxide) – 46 % (Fig. 12). Under the influence of climate change (freezing, thawing, oxidation, weathering), the concrete was corroded and the chemical elements of the color pigments of the paints, which were used to decorate the building, migrated. The concrete sample was saturated with NiTi and $CoMg_7O_8$.

The advantages of this study compared to similar known ones are that in order to determine the long-term destructive effect of climatic factors on the historical building, a combination of non-destructive research methods (TPD MS, X-ray diffraction, and scanning electron microscopy) was used, which made it possible to determine changes in the structural and mineral composition of concrete .

Our experiment could be an example of long-term chemical and biological corrosion of concrete, which is caused by the influence of climatic factors and anthropogenic influence.

The limitation of the study is that we investigated the influence of climatic and biogenic factors on a unique historical building, so it is quite difficult to reproduce the corrosion conditions in the laboratory or to investigate all aspects.

The disadvantage of research is that it is difficult to find a similar object for examination because each cultural heritage site is unique.

The area of further research is the development and planning of restoration works for the restoration of the historical building.

7. Conclusions

1. Corrosion of wall frescoes and concrete caused by micromycetes (*Aspergillus fumigatus*, *Penicillium brevicompactum*, *Aspergillus niger*, *Cladosporium sphaerospermum*) was established by microbiological and microscopic studies. The study of concrete samples by the TPD-MS method showed the presence of an increased level of moisture and carbons by 20 % in the test samples, compared to the control. The sulfur content in all concrete samples was not significant.

2. The study of the mineral composition of concrete using the X-ray diffraction method showed the presence of Al_2O_3, 36–44 %, which indicates a significant clay content. The presence of NiTi, 53 %, and $CoMg_7O_8$, 46 %, in the concrete indicates the migration of the chemical elements of the paint pigments used to decorate the cathedral. The concrete control sample contained a significant amount of SiO_2, up to 51 %, which is the main component of sand. Due to the application of a complex of non-destructive research methods, a long-term corrosive effect on the historical building was proven, namely the destruction of concrete and wall frescoes by microscopic fungi and the change of the microstructure. In addition, the presence of an increased content of clay and pigments in the old concrete was established.

Conflicts of interest

The authors declare that they have no conflicts of interest in relation to the current study, including financial, personal, authorship, or any other, that could affect the study and the results reported in this paper.

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Data availability

All data are available in the main text of the manuscript.

Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies when creating the current work.

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