Experimental diagnostics on potential degradation of a selected building construction

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Abstract

The aim of the study was to evaluate the historical building of the Old Town Hall of Košice city in terms of degradation processes of building materials. Regular maintenance, especially of historic buildings, is essential in terms of preserving the historic architectural heritage. Research was focused on selected parts of the basement of the historical construction with a special regard to the effect of humidity, salinity and carbonation of masonry structures. Samples were collected in the building basement and subsequently analysed in the laboratory. Chemical analysis of materials was performed by X-ray fluorescence analysis and the soluble salts were investigated by colorimetry. In addition, the content of sulphates was confirmed by infrared spectroscopy. Humidity of individual samples ranged from very low to very high, the highest values were measured around the perimeter of the room (positioning to the outside terrain). The carbonation ranged from grade III to IV, sulphates were detected in practically all samples. The results of study can help to design corrective measures.

Key words: diagnostics, humidity, salinity, analytical methods, assessment of technical conditions

1 Introduction

The diagnostic assessments for all types of constructions is very important. Each building is subject to degradation of individual elements of the structure due to the impact of physical, chemical, biological, but also mechanical factors over the time. At a certain age of the building, defects may appear, so it is necessary to preventively evaluate their condition [1]. Diagnosis of parts of building structures consists of sampling from selected defects locations after visual inspection. With regard to the preservation of the architectural heritage, it is necessary to avoid destructive techniques in solving the problem of determining the condition and design of the protection of historic buildings and to give priority to the contactless method of structural assessment. The non-destructive method in the evaluation of the historic building are widely used by experts, e.g. those dealing with preventive diagnostics in the case of the Italian church of Santa Maria ad Cryptas (from the 13th century) in the area of L'Aquila. Their method consisted of using IR (InfraRed) thermography to evaluate the

agreement between earthquake damage and previous thermography results. They also wanted to confirm the effectiveness of thermography for long-term research [2]. Researchers from the National Technical University of Athens focused in detail on non-destructive technical methods of cultural heritage protection. Among the methods of non-destructive assessment of buildings, they proposed Digital Image Processing (DIP). It is a software that converts an image (photograph) into a digital image, which allows you to classify but also extract properties or recognize patterns on the architectural surface, both at the macro and micro level. Ground Penetrating Radar (GPR) is a non-destructive technique that uses radar pulses to image the subsurface structure under investigation. It uses electromagnetic radiation (100 MHz to 2.6 GHz, which is usually preferred in the case of a historic building). The GPR transmitter transmits electromagnetic energy to the ground. When energy encounters a subsurface object or a boundary between materials with different properties, the radiation may be reflected, refracted or to disperse back to the surface. Ultrasound testing is also an example of a non-destructive method. This detects surface or subsurface defects or discontinuities in materials. High-frequency sound waves are introduced into the surface to be examined. These pass through the material, with a certain loss of energy (attenuation). The waves are then reflected at any interface they encounter as they pass through the material. The reflected beam, which is captured on the surface, is then analyzed to accurately determine the presence and location of the faults or discontinuities sought in the material. It is most often used to detect cracks in structures [3]. A 3D scanner is also used to assess the condition of buildings, which can be used to the actual condition of the assessed building. The result is technical drawings and the overall model of the building, which can be used as a basis in other - design procedures of reconstruction.

If needed, the samples are collected from the detected damaged places. After taking adequate samples (either by destructive or non-destructive method according to the needs and severity of the defects), the next procedure is performed in the laboratory. Based on basic evidence reactions, information about degradation processes are obtained from the extracts of the taken samples. Classical methods are used to evaluate the parameters such as e.g. gravimetric determination of humidity or modern analytical methods, mostly spectral methods for qualitative identification of elements or functional groups, or quantitative determination of selected pollutants.

Modern optical analytical methods of spectrometry such as X-ray diffraction (XRD) or X-ray fluorescence spectrometry (XRF) or colorimetry are used to obtain a more accurate concept of the chemical content in the sample. Fourier-transform infrared spectroscopy is a technique used to obtain the infrared spectrum of a solid, liquid or gas. At the same time, the FTIR spectrometer collects data with high spectral resolution over a wide spectral range. This provides a significant advantage opposite a dispersive spectrometer that measures intensity in a narrow range of wavelengths at once. The output of the FTIR method is the infrared spectrum, which is a graphical representation of the functional energy dependence. The energy dependence on wavelength is logarithmic, therefore the wave number is used which is defined as the inverse value of the wavelength, and thus the energy dependence on wavelength will be a linear function [4].

After determining the factual state of the examined material, the measured parameters are evaluated according to the relevant standards and guidelines. If it is confirmed that the condition does not comply with the required regulations, it is necessary to modify the damaged structure in the form of remediation.

In the past, the buildings were founded and protected against the ingress of water, based on the material and technical possibilities during construction at the time. Humidity problems are one of the most common defects, especially in older buildings [1]. The main causes of wet masonry can include: detrition or absence of masonry waterproofing; damage to sanitary-technical installation pipes; neglect of indoor ventilation; the building surroundings are insufficiently drained; unexpected changes around the building (effects of vegetation, shading, etc.); harmful interventions on the building (asphalt paint is applied to the masonry, which causes capillary water to rise into the zone above the paint); change of vapor-permeable floors to no vapor-permeable floors (from wooden, stone to concrete with asphalt / cardboard insulation); large vapor-tight areas around the building (asphalt carpet on the road, on the sidewalk, in the yard, around the building) etc.

Defects in building elements are related not only to the amount of water present, but also to its quality, i.e. its purity. Water as a transport medium migrates into the pores of the materials, thus allowing the transport or concentration of salts in these elements. The accumulation of salts subsequently leads to the destruction of building structures. Salt sources can be divided into several groups: salts that are primarily contained in one of the materials used in the construction (poor quality sand, lime, bricks, etc.); salts transported by the rise of moisture from the surrounding terrain or the penetration of moisture from other parts of the building; salts formed by chemical corrosion of materials used on the construction site by the influence of the atmosphere (e.g. formation of sulphates by the reaction of carbonates with sulphur oxide - the main component of acid rain); salts formed from biological sources (e.g. by conversion of urea to nitrates); salts formed as a result of remediation measures (e.g. sodium water glass is formed to a sodium as a by-product) [5]; salts as part of building materials or salts formed during the hardening of these materials; salts formed by contact with the environment, especially with the atmosphere or flowing water; salts contained in the material or salts contained in the material as a result of processing [4]. It should be noted that some materials may contain salts before it was built into the structure. E.g. cement may show traces of gypsum and alkalis. Bricks may contain salts depending on the quality of the clay used in their production [5]. The impact of salts in masonry also depends on the solubility of the salts in water. The higher the solubility is, the more harmful are the salt effects in the masonry. These are in particular the effects of calcium, magnesium and sodium salts in the form of chlorides, sulphates and nitrates [6].

This paper is aimed at the investigation of the deterioration state of historical building of the Old Town Hall in Košice city by applying modern analytical methods. Special attention was paid to investigation of humidity, salinity and carbonation of materials used.

2 Material and Methods

2.1 Studied building

The experimental research was devoted to a selected building of the Košice city Old Town Hall, in particular to the basement of the buildings. Firstly, the screening of the deterioration state in whole basement was performed by visual inspection and collecting and analysing the representative samples (Figure 1). Consequently, the more attention was paid to the room,

where the archive was (Figure 2).

Figure 1: Ground plan of the examined part of the basement with marked screening points

Figure 2: Ground plan of a part of the basement with marked sampling points

2.2 Methodology

Surveys of buildings in general, not only of historical buildings, do not have a precisely defined sequence of steps according to guidelines and standards. Every historical work is unique, it is not possible to create a universal methodology, and therefore it is necessary to approach each object within the survey individually. However, these main common steps were carried out in our study:

- Visual inspection
- Preliminary investigation and identification of major defects
- Selection of sampling points
- Detailed survey, in situ measurements, including sampling
- Processing of samples in the laboratory
- Evaluation of results
- Additional survey and, if necessary, additional samples
- Conclusion of the assessment and possible proposal for remediation measures [7].

After visual inspection, the sampling process was performed in two stages. In both cases, sampling was focused on the basement of the building. In the first phase, the entire basement was screened by sampling various types of materials such as brick, plaster, wood, and masonry and paint coat. The screening of the basement was indicated in the ground plan (Figure 1). 21 samples were obtained, taken by drilling from a depth of up to 10 - 30 mm or by scraping at the same height level - 0.5 m. The drilling method was performed at low speed to not to lose moisture. The samples were closed in PE bags with accurate marking and processed in the laboratory within about 2 hours after sampling.

Based on the results of the screening, in the second phase, the sampling was focused on the archive room. Archive room was used for archiving important documents; therefore, it was a priority to diagnose, analyse and propose corrective measures. Based on the sampling points shown in the ground plan (Figure 2), samples of eleven plasters and mortars were analysed.

The attention was paid to the humidity, salinity and carbonation state of the samples.

2.2.1 Humidity analysis

The most common failures of buildings are closely related to humidity. Humidity assessment is therefore part of every construction survey. Standard ČSN P 73 0610: 2000 Waterproofing of buildings, Rehabilitation of wet masonry, The basic provision [8] characterizes the moisture values in structures (Table 1), which are decisive for the design of remediation measures. Mass moisture was determined gravimetrically according to Equation 1:

$$W_{hm} = \frac{m_v - m_s}{m_s} * 100 [\%] \quad (1)$$

where:

- m_v - weight of wet material [g]
- m_s - weight of dry weight [g]
- W_{hm} - mass moisture [%]

Table 2: Evaluation of masonry moisture according to ČSN P 73 0610 [8]

Humidity content w_{hm} [%]	Degree of humidity
$w \leq 3$	humidity very low
$3 \leq w < 5$	humidity low
$5 \leq w < 7.5$	humidity increased
$7.5 \leq w < 10$	humidity high
$10 \leq w$	humidity very high

2.2.2 Salinity analysis

As a representative of salinity, sulfates have been under examination. Sulfate is an anorganic anion that contains sulfur, general formula is (SO_4^{2-}) . Sulfate salt solutions are corrosive. Sulphate corrosion can be considered as one of the most common form of deterioration in a masonry structure. This is linked with the process of voluminous sulfate products, such as gypsum, ettringite or thaumasite formation. Example of the gypsum formation in the concretes is given in Eq. 3 and 4.

Gypsum ($CaSO_4 \cdot 2H_2O$) is involved in the volume changes of structures and at a certain stage of moistening it is partially leached [7]. Under certain conditions, recrystallization can occur only to the formation of ettringite ($3CaO \cdot Al_2O_3 \cdot CaSO_4 \cdot 32H_2O$) [5].

Table 3: The most important water-soluble sulphate salts occurring in building materials [5]

Sulphates	Solubility [g/l]
$CaSO_4 \cdot 2H_2O$	2.4
$MgSO_4 \cdot 7H_2O$	1172
$Na_2SO_4 \cdot 10H_2O$	583
Na_2SO_4	481
K_2SO_4	111
$3CaO \cdot Al_2O_3 \cdot CaSO_4 \cdot 32H_2O$	-

Table 4: Main reactions of sulphate formation in building elements [5]

Reactions	Increasing the volume %	Multiple increase in solubility
$CaCO_3 + H_2SO_4 + 2H_2O \rightarrow CaSO_4 \cdot H_2O + CO_2 + H_2O$	about 100	160
$MgCO_3 + H_2SO_4 + 7 H_2O \rightarrow MgSO_4 \cdot 7H_2O + CO_2 + H_2O$	about 430	7 500

$\text{FeCO}_3 + \text{H}_2\text{SO}_4 + 7\text{H}_2\text{O} \rightarrow \text{FeSO}_4 \cdot 7\text{H}_2\text{O} + \text{CO}_2 + \text{H}_2\text{O}$	about 480	16 000
$\text{Al}_2\text{O}_3 + 3\text{H}_2\text{SO}_4 + 15\text{H}_2\text{O} \rightarrow \text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$	about 1400	27 000

The concentrations of sulphates in the samples were analysed in two ways: by leaching the samples and consequently determination of the solved ions by colorimetry as well by calculation based on the results from the X-ray fluorescence analysis, which provided the total sulphur content. Subsequently the determined concentrations of sulphates were converted to the percentage of sulphates in the original dry sample. Salinity caused by sulphates was assessed applying the WTA guidelines' criteria (Tables 5 and 6), [10]. Although, these WTA criteria are primarily considered for masonry materials, for purpose of the study, the criteria were used to evaluate the sulphate content also for all brick materials.

Table 7: Criteria for salinity assessment according to WTA Guidelines 4-5-99: Assessment of masonry, diagnostics of masonry [9]

Salt type	Concentration expressed as % of the weight of the dry sample			
Sulphates	<0.5	0.5 – 2.0	2.0 – 5.0	>5.0
Salinity level	Low	Increased	High	Very high

Table 8: Criteria for evaluating the impact of salt ions according to WTA 2-9-04 / D Remedial plaster systems [10]

Salt type	Concentration expressed as % of the weight of the dry sample		
Sulphates	<0.5	0.5 – 1.5	>1.5
Salinity level	Low impact	Middle impact	High impact

2.2.3 Alkaline reaction and carbonation analysis

The carbonation analysis in the samples was determined based on the alkaline reaction and determination of pH. The samples were crushed, the aggregate grains from the analysed sample were separated and each particular sample was homogenized. To determine the pH, approximately 1 gram of sample was poured into flasks and leached in 100 ml of deionized water. After at least 24 hours, a part of the leachate from the flasks was measured for pH determination. Based on the obtained pH values, the degree of carbonation was estimated (Table 6).

Table 9: Evaluation of carbonation based on the determined pH value [7]

Carbonation	Degree I.	Degree II.	Degree III.	Degree IV.
pH	>10.8	>9.6	8.3-9.6	<8.3

Carbonation takes place not only in conventional concretes but also in other silicate materials such as plasters, masonry mortars or prefabricated samples. The impact of carbonation in silicate materials can reduce the durability of the material and the reliability of the structure [7].

2.2.4 Analytical methods

X-ray fluorescence analysis (XRF) was performed to find the elemental chemical composition of the samples and to obtain the overall content of sulphur. The measurements were carried out on powdered samples by using AMETEC equipment with silicon detector.

Fourier-transform infrared equipment (FTIR) Bruker was used to confirm a presence of the sulphate functional groups in the samples. The measurement was performed in range $400 - 4000 \text{ cm}^{-1}$ using ATR unit.

Colorimeter DR2800 Hach Lange was used to analyse the soluble salts – sulphates in the leachates.

3 Results and discussion

3.1 Visual inspection

Figure 3 and Figure 4 illustrate the results of visual inspection in the basement of the historical building of the Old Town City Hall. As it can be seen in the pictures, serious deterioration of materials was observed, represented by mainly biodegradation of walls, floors and ceilings, which were covered by significant mould on the surface (Figure 4) or by leaked walls and places with large patches of dissolved salts (Figure 3).

Figure 3: Results of visual inspection in archive (walls and ceilings)

Figure 4: Results of visual inspection in storage (walls and ceilings)

3.2 Screening of the basement

Results of the screening are summarised in the Table 7, where the values of mass, pH, carbonation, salinity and humidity of the samples were determined.

Table 10: Results of the screening samples according to Figure 1

Sample	Material	Mass of sample [g]	pH	Carbonation degree	Salinity degree	Humidity w_{hm} [%]	Humidity degree
1.	Plaster	0.814	8.2	IV	very high	2.34	very low
2.	Plaster	0.702	8.9	III	very high	6.51	increased
3.	Plaster	0.480	8.7	III	very high	7.26	increased
4.	Plaster	0.879	7.9	IV	very high	1.14	very low
5.	Plaster	0.802	7.7	IV	very high	6.73	increased
6.	Plaster	0.820	8.4	III	very high	9.98	high
7.	Plaster	0.597	8.3	III	very high	5.19	increased
8.	Plaster	0.579	8.2	IV	very high	2.98	very low
9.	Plaster	0.823	8.0	IV	very high	5.92	increased
11	Plaster	0.296	8.5	III	very high	27.01	very high
12.	Mortar	0.687	8.4	III	very high	3.19	low
13.	Brick	0.812	8.5	III	very high	2.88	very low
14.	Mortar	0.619	9.2	III	very high	22.29	very high
15.	Paintcoat	0.642	8.2	IV	very high	5.62	increased
16	Mortar	0.451	9.1	III	very high	17.33	very high

17.	Brick	0.748	9.1	III	very high	10.43	very high
18.	Plaster	0.881	9.7	II	very high	6.14	increased
19.	Brick	0.901	8.5	III	very high	1.07	very low
20.	Mortar	0.421	7.9	IV	very high	29.60	very high
21.	Brick	0.875	8.8	III	very high	6.82	increased

According to ČSN P 73 0610, the humidity of individual samples ranged from very low to very high, the highest values were measured around the perimeter of the room (positioning to the outside terrain). Regarding moisture, 16 of the samples evaluated showed increased humidity, high to very high humidity in 10 cases. The degree of carbonation ranged from grade III to IV, salts were detected in practically all samples (Table 7). The presence of sulphates was observed in each sample. The increased content of sulphates in the plasters can be justified mainly by the fact that gypsum could have been used for their realization. Samples obtained the very high humidity degree were also subjected to investigation by IR spectrometry, the examples of the IR spectra are shown in Figure 5 and Figure 6. Based on the position of the main peaks, very similar spectra can be observed for all analysed samples.

Figure 5: IR spectra of plaster sample

Figure 6: IR spectra of mortar sample

Besides the obvious functional groups present in mortars spectra such as silicate and carbonate, the FTIR measurements confirmed the sulphate and phosphate contents in the samples.

3.3 Results from the archive room

In the archive room, the investigation was exceptionally focused on the places which were visually identified to be damaged. In total, eleven samples from 6 sampling points in the room according to Figure 2 have been collected. The sampling points were located on both internal and external walls at various height levels (0.3 m, 0.5 m, and 1.8 m from the floor). After a semi-quantitative test, where a white precipitate of BaSO_4 formed after reacting the salt samples with barium chloride, selected samples were subsequently subjected to XRF spectrometry to determine the exact concentration of the elements. Summarisation of all results from the analysis of the materials in the archive room is given in Table 8.

Table 11: Results of wall materials from the archive room

Sample	Material	m [g]	Alkaline reaction		Sulphates		Humidity		Sampling point location
			pH	Carbon atation	SO ₄ ²⁻ [%]	Salinity degree	w _{hm} [%]	Humidity degree	Height/wall type
22.	Brick	1.0659	8.49	III.	0.09	Low	0.21	Very low	0.3 m interior wall
23.	Plaster	1.0592	8.13	IV.	3.78	High	1.2	Very low	0.3 m interior wall
24.	Plaster	1.0752	8.17	IV.	1.21	Increased	0.75	Very low	0.3 m interior wall
25.	Plaster	1.0426	8.2	IV.	0.29	Low	0.48	Very low	0.3 m interior wall
26.	Brick	1.0693	8.27	IV.	3.74	Very high	0.68	Very low	0.3 m interior wall
27.	Brick	1.312	8.32	III.	0.38	Very high	0.33	Very low	0.3 m interior wall
28.	Plaster	1.0195	8.24	IV.	0	Low	0.65	Very low	0.5 m exterior wall
29.	Plaster	1.0447	8.8	IV.	5.74	High	1.54	Very low	0.5 m exterior wall
30.	Plaster	0.5175	8.11	IV.	0.10	Low	0.35	Very low	0.5 m exterior wall
31.	Plaster (bloom)	0.5032	7.82	IV.	59.14	Very high	39.08	Very high	1.8 m exterior wall
32.	Plaster (bloom)	0.2586	7.83	IV.	55.3	Very high	35.10	Very high	1.8 m exterior wall
33.	Plaster (bloom)	0.1732	7.86	IV.	72.1	Very high	26.72	Very high	1.8 m exterior wall

As seen in Table 8, significant carbonation degree was observed in all analysed samples (degree III and IV). All samples (except the No. 28 sample) contained sulphates in a wide range from low to very high degrees of salinity. Surprisingly, low humidity was detected for almost all samples even for the samples collected at the lower height (30 cm from the floor). Samples No. 31-33, representing crystal flakes from the gypsum plaster (Figure 3, Figure 7) exhibited sulphates in high percentage 59.14% for sample 31, 55.3% for sample 31 and 72.1% for sample 33. The results of the salt analysis are illustrated in Figure 7.

No significant differences were found regarding the measured parameters and the materials analysed. The results in Table 8 did not prove the influence of humidity, type of material, carbonation or height of sampling on the salinity results. However, the salinity results seem to be higher for the exterior walls when compared to the interiors ones. From the obtained results, it is not possible to state any relationship between the damage of the material and the

height of where it was located.

Anyway, the research was carried out during the reconstruction of the Old Town city Hall building and its results were helpful in replacing materials with new ones and proposing corrective measures.

Figure 7: Determined sulphate concentrations for sampling points at 1.8 m

4 Conclusion

The presented work focused on the diagnostic evaluation of the historic building in terms of humidity, carbonation, and salinity with a deeper focus on selected parts of the basement. The visual inspection created a picture of the condition of the building of the Old Town Hall of Košice. The diagnostic of the building showed a significant degradation of the material and structures due to the action of moisture and salinity. The measured values confirmed the need for corrective measures and application novel materials and technologies in reconstruction process.

Acknowledgements

This work has been supported by VEGA 1/0648/17 and 2/0142/19.

References

- [1] Balík, M., Starý, J. 2003. *Sklepy opravy a rekonstrukce*. Praha: GRADA Publishing, a.s. ISBN 80-247-0221-5
- [2] Moropoulou, A., Labropoulos, K.C., Delegou, E.T. 2013. Non-destructive techniques as a tool for the protection of built cultural heritage. *Construction and Building Materials*, 48, 1222-1239. DOI 10.1016/j.conbuildmat.2013.03.044
- [3] Paoletti, D., Ambrosini, D., Sfarra, S. 2013. Preventive thermographic diagnosis of historical buildings for consolidation. *Journal of Cultural Heritage*, 14, 116–121. DOI 10.1016/j.culher.2012.05.005
- [4] Bálintová, M. *Environmentálna analýza materiálov – Úvod do moderných analytických metód*.
- [5] Balík, M. a kol. 2005. *Odvhlčování staveb*. Praha: GRADA Publishing, a.s. ISBN 80-247-

0765-9

- [6] Ďurica, P. 2012. *Poruchy budov diagnostika a sanácia*. Žilina: Edis. ISBN 978-80-554-0561-2
- [7] Terpáková, E. 2017. *Diagnostika stavebných konštrukcií*. Košice: SvF TUKE.
- [8] ČSN P 73 0610: 2000. Hydroizolace staveb - Sanace vlhkého zdiva - Základní ustanovení
- [9] WTA Smernice 4-5-99. 2009. Posudzovanie muriva. diagnostika muriva
- [10] WTA 2-9-04/D: 2009. Sanační omítkové system