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DEGRADATION AND GEOMYCOLOGICAL ASPECTS REGARDING THE NATURAL STONE FROM BUILT OUTDOOR

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Abstract: The degradation processes of natural stone building are controlled by natural and anthropogenic factors. The environmental factors as humidity, temperature variations, wind, and air pollutants together with microbiological activity lead in time to stone decay. Mineralogic, petrographic and geomycological investigations were performed on the natural stone from Ursuline Monastery entrance portal, located in Oradea downtown, Romania. Optical transmitted light microscopy and X-ray diffraction were used in order to investigate the mineralogy and petrography of the samples collected from the investigated stone portal. Based on mineralogical and petrographic results, interpretations in connection with the effect of some atmospheric factors as humidity, air temperature, solar radiation, wind, air pollutants respectively, as well as with identified fungi (*Penicillium sp., Alternaria sp., Aspergillus sp., Cladosporium sp., Cryptococcus laurentii*), were made in order to evaluate de stone decay.

Key words: geomycology, X-ray diffraction, atmospheric factors, degradation of outdoor built heritage

1. INTRODUCTION

The interdisciplinary studies concerning the natural stones decay from buildings, pavements etc, are valuable for restore the built heritage, as well as for promoting the geoscience through cultural, didactic, tourist circuits (Ilieș et al., 2017a). In this respect detailed mineralogical and petrographic features, completed with information regarding area of origin of the stone, erosion, transport information etc. are very important. Stone processing techniques reflect the socio-economic context of that period as well as the reasons for choosing this natural material (Panizza & Piacente, 2003; Ilieș et al., 2017b; Biacsi & Kalmar, 2014).

The present study is focused on heritage building stone gate from Ursuline Monastery, in Oradea town (Romania). The complex of heritage buildings, currently a historic monument, built 1771-1774, is located in the Oradea downtown. The house bought by the abbot István Szénczy to establish a girls' school was transformed into a convent; it began the construction in baroque style of the church *"Saint Ana"* (Chifor, 2018). Later on (1858) the convent was extended with a new wing and the portal to the entrance in the convent, located next to the church, were renovated in *Neo-Gothic* style (Chifor, 2018). At the beginning of the 20th century, the old rectangular windows from the ground floor, facing main pedestrian street, Calea Republicii,

Figure 1. Location of the studied natural stone portal, Oradea downtown

a. Location of the heritage building, Calea Republicii pedestrial street, Oradea downtown

b. The analysed portal made by natural stone

c. Detalied view with degradation processes of natural stone of the portal

Oradea downtown, were replaced by decorative shop windows, paved with natural stone (Fig. 1 a, b, c) in the attempt to open a shop in the future (according the designing plans of the architect Ferenc Sztarill), function maintained till nowadays.

The heterogeneous subsystems as atmosphere, hydrosphere, biosphere and lithosphere are open and are characterised by energy exchanges with various inputs and outputs. Microorganisms are critically important biotic agents contributing to physical, mechanical and geochemical changes of the lithic substrate (e.g. rock coatings generated by fungi**)** (Ilieș et al, 2018a). Microorganisms, through their organic geoactive metabolites (Gadd, 2017a), can affect and induces many transformations at minerals level through their filamentous, branching, favoured by the texture of the rock, mineralogical-chemical composition, state of preservation (high vulnerability is registered in the areas with high porosity, cracks, fractures, etc). Processes are limited by the penetration depth into the solid substrate of the rock which has a certain hardness and resistance to physical gradients, gases, solutions, organisms.

2. PREVIOUS RESEARCH

The natural stone decay of the outdoor historic buildings and monuments and the microbial impact have been well documented and presented over the years by researchers, such as: Warscheid et al., 1991; Saiz-Jimenez, 1994; Gaylarde et al., 2003; Zammit et al., 2008; Cutler & Viles, 2010; Ilieș et al., 2019. Geomycology studies contributed to understanding the biological impact on the alteration of natural rocks and have analysed the biogeochemical roles of microorganisms as main agents of complex geological changes (Gadd, 2007; 2017a and b). To study in situ the activity of fungi on rocks it is very complex and difficult; some results were published by various researchers such as: Groudev & Groudeva, 1986; Koestler & Vedral, 1991; Braams, 1992; Ehrlich, 1998; Sterflinger, 2010; Gaylarde et al., 2003; Huniadi et al., 2019. The interactions between the substrate made of natural stone and microorganisms are multiple and very complex, leading to changes of physical, mechanical and chemical nature, as deterioration or sometimes, by the contrary leading even to the prolongation of life of the surface through the protective coatings and biofilms which can be generated by the microorganisms (Wolf & Krumbein, 1996; Salvadori et al., 2016). The different types of alterations were evaluated, catalogued and also mapped. Microbial decay of natural stone monuments in different climates were analysed: e.g. limestone and sandstone monuments by Ghany et al., 2019; Palla et al., 2002; Shakya et al., 2020; limestone and granites from building in urban environments, by Schiavon, 2002. Biodeterioration phenomena of different old monuments and cathedrals investigated by: e.g. Trovão et al., 2019 in Portugal; Gómez-Alarcón et al., 1995 in Spain; Videla & Herrera, 2004 in Latin America. In Romania such research on cultural heritage were highlighted by Schröder et al., 2019, Ion et al., 2019, Olteanu, 2015, Paușan et al., 2017, Ilieș et al., 2018b, Alba & Boengiu, 2020 etc.

3. MATERIALS AND METHODS

Seven samples were collected based on macroscopic features taking in account the physical aspects of degradation and color of the stone. Samples were prepared for transmitted light microscopy and X-ray investigations. Thin sections made on the stone samples were studied using a polarized light Nikon Optiphot T2-Pol microscope with one polarize or under crossed polarizers and microscopic images were taken. X Ray diffraction (XRD) investigations were performed using a DRON-3.0 diffractometer with Cu Kα radiation (λ= 1.541874 Å) (Zhao et al., 2019). The working parameters were U = 35 kV; I = 20 mA. The

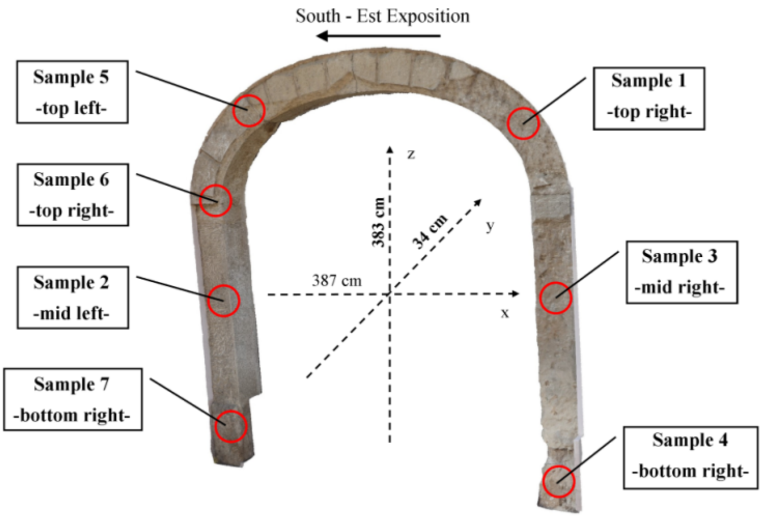

Figure 2. The locations of samples on the analysed ornamental stone porta

data were collected between 5° and 60° 2ϴ and a PDF2 database was used for mineral phases identification. Interpretation of X-ray data and calculation of interplanar spaces were made using the EVA software. Optical investigations were performed in the Department of Geology at ``Babeş – Bolyai`` University, while X – ray diffraction was performed in the Laboratory of Geological Institute, Almaty, Kazakhstan.

In order to have a better overview of the degradation process of the studied samples, atmospheric factors as precipitation, relative air humidity, air temperature, solar radiation, wind speed and air pollutants were analysed (Masschaele et al., 2004; Pop et. al., 2019). The data was obtained from the Environmental Protection Agency in Bihor County over a period of 13 years (2008-2020).

4. RESULTS AND DISCUSSION

The decay transformations can affect the structure and stability of natural ornamental stone. Important transformations are represented by chemical processes (e.g. mineral dissolution) as well as deterioration due to different organism activity. During weathering, the crystalline structure of the primary minerals is destroyed, and new mineral phases are formed. The newly formed minerals are developed as crusts, films etc., (Gadd, 2017a), usually difficult to be investigated in situ.

Under the microscope, the analysed samples represent natural rocks of sedimentary origin represented by a sandstone with carbonate cement. The rock consists of figurative elements with a participation of about 75% (mineral and lithic fragments, bioaccumulated organic fragments and ooids) embedded in a carbonate cement (approx. 25-30%). The rock has a mechanical, of bioaccumulation and chemical precipitation structure. The texture is psamitic with the dimensions of the clasts between 0.063 mm and 2.00 mm. From a compositional point of view, the rock consists of allogenic components represented by mineral fragments (quartz and microclines; Fig. 3-4 and 5), and lithic fragments (quartzite and micaschists) embedded in a sparitic cement (Fig. 3-6). The rock also contains micritic oolids or composite ones (core of the mineral grain surrounded by micritic calcite; Figs. 3 and 4). Organism as red algae (Fig. 3 and 4), foraminiferides (milliolides and rotalides; Fig. 6), fragments of unidentifiable molluscs (bivalves or gastropods), fragments of echinoderms are also present. The cement is sparitic (Fig. 3-6). The petrographic features of rocks are typical of a bio-extraclastic grainstone with carbonate cement.

Detailed investigations using X-ray diffraction point out the presence of quartz as the main component accompanied by calcite and K-feldspars, muscovite (probably from the micaschists) and albite as subordinate phases.

Mineralogical transformation, in the presence of water with dissolved CO_2 and atmospheric oxygen and CO_2, affect mainly the calcite and pyrite. The atmosferic CO_2 could dissolve and later reprecipitate calcite, similar to the processes which take place in both natural and experimental systems (Langmuir, 1977; Anderson, 2002):

$$H_2O + CO_2 \rightarrow H^+ + HCO_3^-$$

$$H^+ + CaCO_3 \rightarrow Ca^{2+} \text{ and } HCO_3^-$$

$$Ca^{2+} + 2(HCO_3^-) \rightarrow CaCO_3 + H_2O$$

Removing of calcite by dissolution from the cement of the rock lead to physical disaggregation of the affected rock and formation of a residual sand, consisting of mineral and lithic grains.

The presence of pyrite leads to formation of sulfuric acid and finaly deposition of iron hydroxides as ferrihydrite (Har et al., 2019):

$$2FeS_2 + 7O_2 + 2H_2O \rightarrow 2\ Fe^{2+}SO_4 + 2H_2SO_4$$

$$2Fe_2(SO_4)_2 + 2H_2SO_4 + O_2 \rightarrow 2Fe_2^{3+}(SO_4)_3 + 2H_2O$$

$$Fe_2^{3+}(SO_4)_3 + 6H_2O \rightarrow 2\ Fe(OH)_3\ + 3H_2SO_4$$

The deterioration of stone and minerals (even intact ones) can be also induced by fungi. It is generally defined, by different authors, as biogeophysical and biogeochemical processes (Gadd, 2007, 2017 a, b). Savković et al., 2016, tested with good results anti-*Aspergillus* activity on

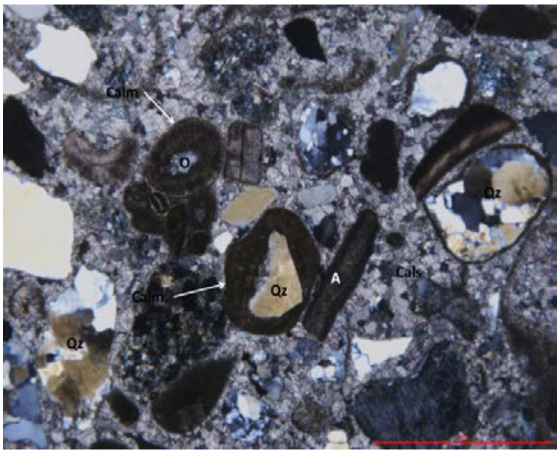

Figure 3. Microscopic cross-sectional image at crossed polarizers of sandstone with carbonate cement with quartz elements (Qz), composite ooids (O) with quartz core and micritic calcite crown (Calm) and red algae (A) embedded in sparitic cement (Cals)

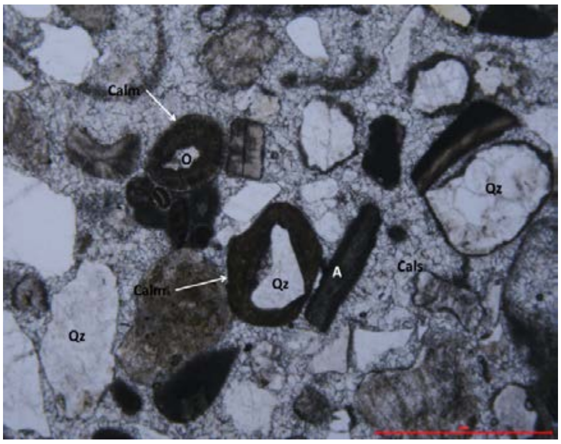

Figure 4. One polarizer microscopic image of sandstone with carbonate cement with quartz elements (Qz), composite ooids (O) with quartz core and micritic calcite crown (Calm) and red algae (A) embedded in sparitic cement (Cals)

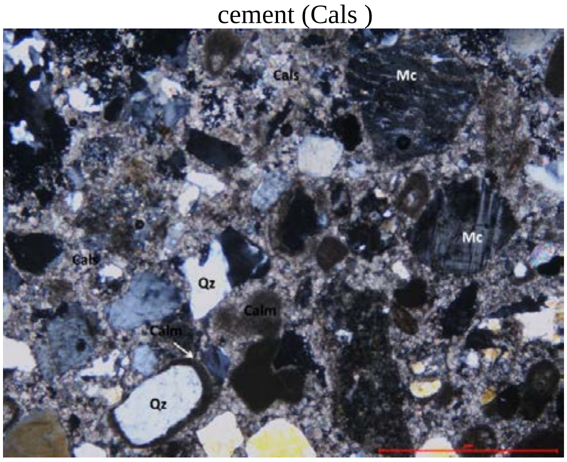

Figure 5. Microscopic image at crossed polarizers of sandstone with carbonate cement. Qz - quartz / quartzite, Mc - microcline, Cals - sparitic calcite

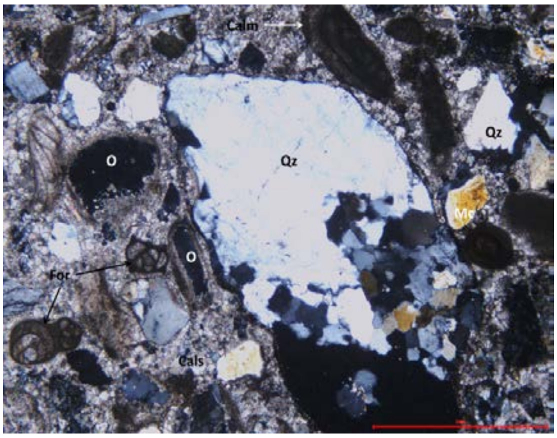

Figure 6. Microscopic image at crossed polarizers of sandstone with carbonate cement. Qz - quartz / quartzite, O- composite ooide, For - foraminiferide, Cals - sparitic calcite

Table 1. Results of semiquantitative X-ray phase analysis of crystalline phases of the samples.

Compound name	**Formula**	**S-Q (%)**						
		Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7
quartz	SiO_2	56	57.4	45.8	50	43.5	58.4	26.9
calcite	$Ca\ (CO_3)$	24.6	26.3	35.5	31.8	46.3	24	50.1
mica	$KAI_2(AISi_3O_{10})\ (OH)_2$	6.6	2.3	N/A	N/A	N/A	N/A	4.6
feldspar(albite)	$Na\ ((AISi_3O_8)$	6.5	6.1	12.5	3.7	4.9	3.3	5.7
K- feldspar	$KAISI_3O_8$	6.4	7.9	6.3	5.9	5.3	6.7	12.7
pyrite	FeS_2	N/A	N/A	N/A	8.5	N/A	7.6	N/A

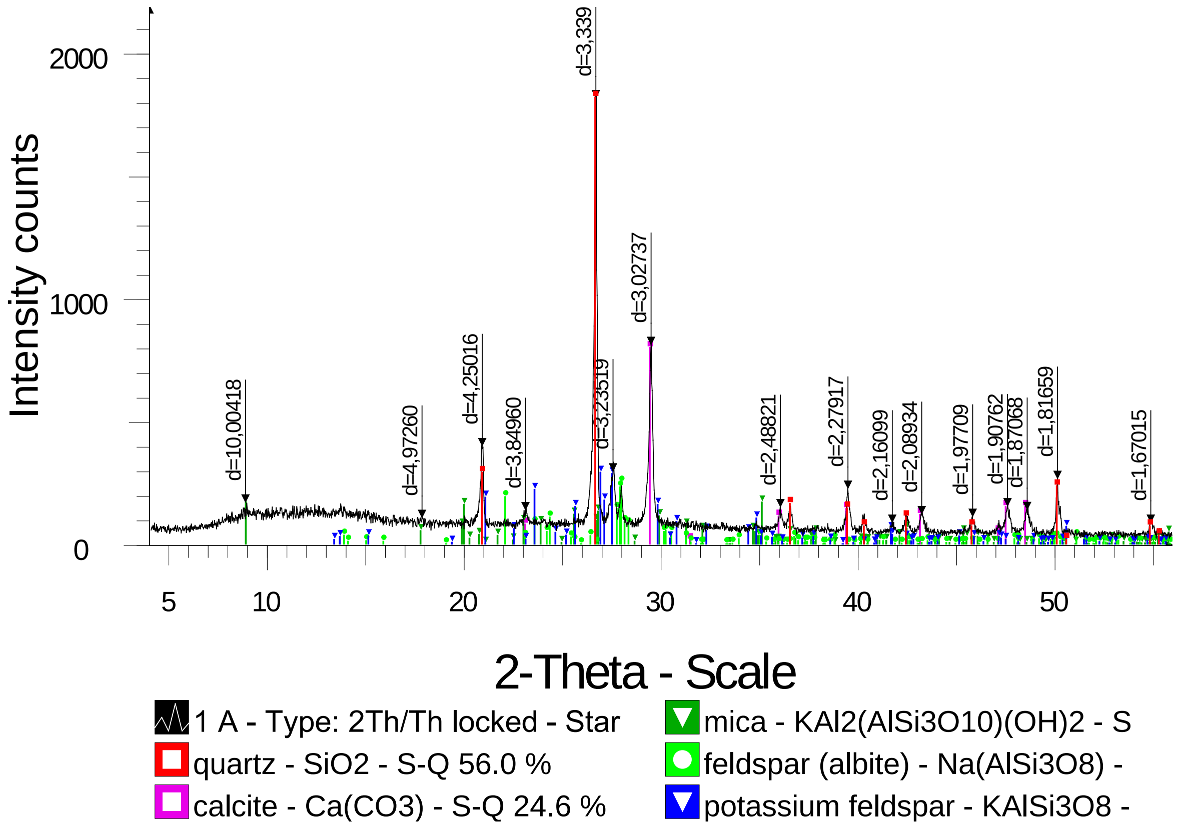

Figure 7. X-Ray diffraction pattern of sample 1 with typical line for quartz, calcite, mica (muscovite), albite and K-feldspar (microcline)

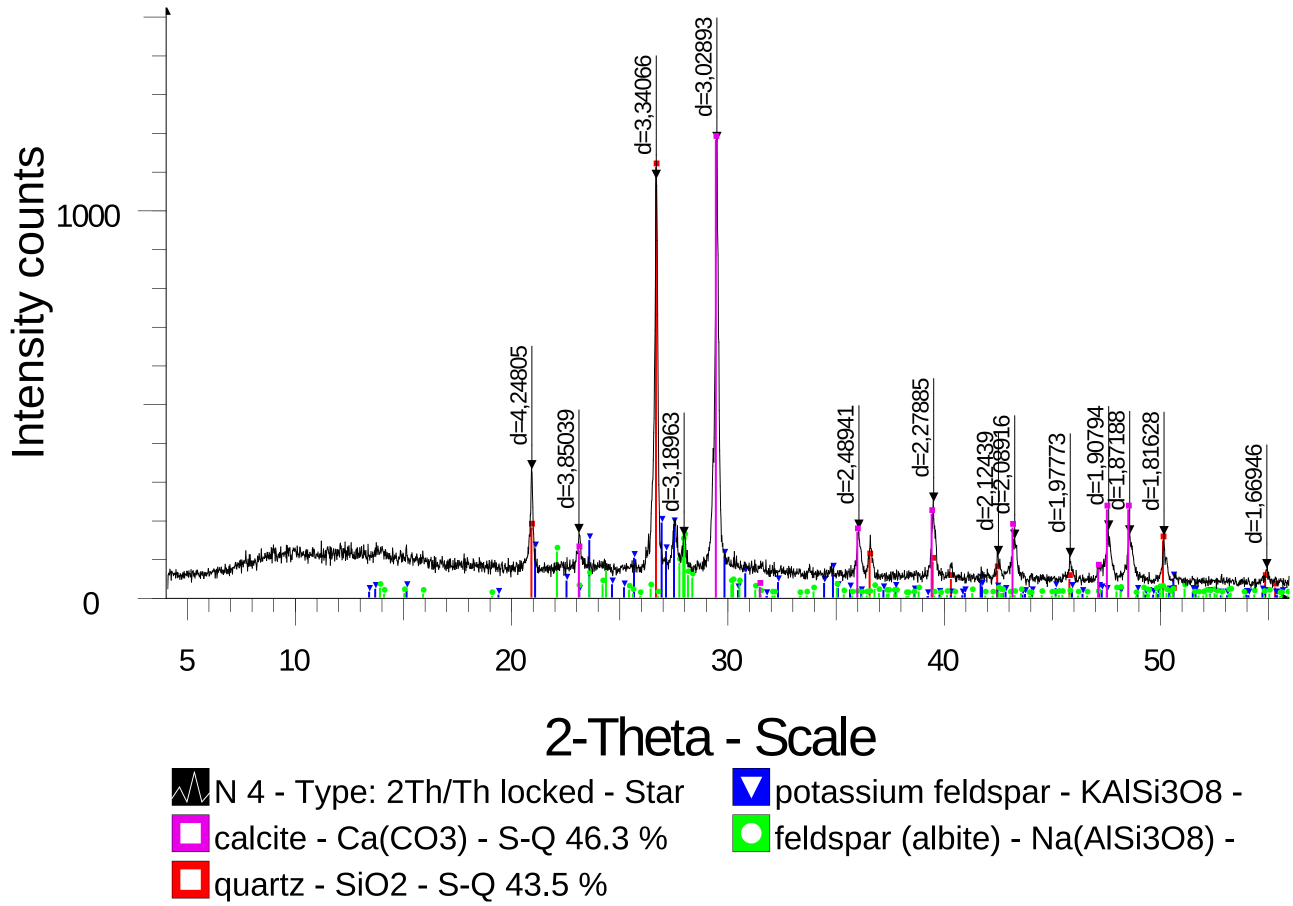

Figure 8. X-Ray diffraction pattern of sample 5 with typical line for calcite, quartz, k-feldspar and albite

Table 2. Interplanar spacing and mineral composition of samples

Mineral	Sample													
	1		2		3		4		5		6		7	
	d, Å	*I %*	*d, Å*	*I %*	*d, Å*	*I %*	*d, Å*	*I %*	*d, Å*	*I %*	*d, Å*	*I %*	*d, Å*	*I %*
Mica	10.00418 4.9726	9.5 6.0	9.97064	3.2		N/A							9.93843 1.99177	9.0 4.1
Quartz	1.81659 1.67015 1.97709 2.12597	14.7 4.9 6.2 7.1	4.2564 2.45596	21.8 100	4.25288 3.33899	25.4 100	4.25883 3.34492	17.2 100	4.24805 3.34066	28.1 91.7	4.25631 3.34178	16.6 100	4.24901 3.33837	16.6 52.8
Calcite	3.8496 3.02737 2.48821 2.08934 1.90762 1.87068	7.7 44.7 8.4 6.8 8.6 7.8	3.85212 3.03133 2.83603 2.48937 1.92365 1.90854 2.90116 1.81749	5.7 45.8 3.7 6.9 4.6 7 5.5 9.7	3.84593 3.02995 2.48833 2.08979 1.92439 1.8728	11 76 10.5 12 5.9 16.4 12.9	3.84701 3.03016 2.8376 2.49098 2.09121 1.92366 1.90917 1.87286	7.4 62.2 5.7 8.5 7.1 5.5 7.2 8.5	3.85039 3.02893 2.48941 2.08916 1.9233 1.90794 187188	14.2 100 15.1 12.9 6.9 15 13.9	3.84824 3.03112 2.83704 2.49022 2.09091 1.92417 1.90937 1.87279	5.8 40.6 3.1 4.9 6.2 2.9 7.2 5.4	3.84458 3.02867 248831 2.0895 1.92212 1.90764 1.8706	11 100 9.8 12.5 6.4 13.1 13.4
K-feldspar	3.23519 2.16099	16.4 4.9	2.15678 3.24263	2.7 19.5	3.23896	19.1	3.78229 3.24278 2.16284	7.1 17.3 3.3	3.23903	17.2	3.24146 2.56862 2.16364 1.7757	16.4 2.8 2.4 3.8	3.2396 2.15682	35.2 4.8
Feldspar (albite)	3.18881	13	3.19072	12.6	3.18922	32.2	3.19238	10.1	3.18963	13.5	3.19022	7	3.18932	15.6
Quartz – calcite	2.297917		2.28015	12	2.27934	18.2	2.28124	13.1	2.27885	21	2.28017	10.6	N/A	
K-feldspar-calcite	1.92402	5.3					N/A							
Pyrite					N/A						2.70444	2.7	N/A	

different natural stone substrates of *Origanum vulgare* L. essential oil. The geomycological investigations on the mentioned decorative stone, were done within a larger observations in September-October 2020, showing its colonisation by microorganisms: *Penicillium sp., Alternaria sp., Aspergillus sp., Cladosporium sp., Cryptococcus laurentii*; the mentioned research highlights the inhibitory action of the tested essential oils: *Lavandula angustifolia, Citrus limon* and *Mentha piperita* on it, for a tested period of 24h, 48h, 72h, 7 days, 14 days and 30 days since the application.

Stone-decaying as the result of chemical changes can be induced by interaction of identified *fungi* (which can generate acids, such: acetic, citric oxalic, glucuronic, fumaric) with the mineral matter (Warscheid et al., 1991; Hirsch et al., 1995; Braams, 1992; Gaylarde et al., 2003; la Rosa-Garcia et al., 1993; Sterflinger, 2010), increased by pollutants and their penetrant hyphae which contribute to develop the existent fissures and cracks.

As consequences can be emphasised the total or partial decomposition, demineralization of the lithosubstrate, solubilizing feldspar, muscovite and phyllosilicates, found in the investigated samples (see above mentioned results detected by using semiquantitative X-ray analysis (Table 1, Fig. 7)). In the case of the inorganic substrate represented by the natural stone, microorganisms (autotrophs and heterotrophs) colonize it because of their capability for CO_2 use during photosynthesis processes, with the consequent precipitation of $CaCO_3$, that dissolves stone and increases the external pH level (Sorlini, 1984; Griffin et al., 1991; Zanardini et al., 2000; Gaylarde et al. 2003; Olteanu, 2015).

In the oxidative environment fungal activity could accelerate the dissolution of iron from iron sulphides (e.g. pyrite present on sample 3 –see table 1 and 2; sample 5, Fig. 8) by species of *Aspergillus* and *Penicillium.* Several fungi can contribute to the development of the oxidation process through production of hydroxycarboxylic acid metabolites, such as: citrate, lactate, malate or gluconate (Gadd, 2017a, b). The oxidation of iron and manganese is influenced by fungi strains isolated from rock (which are identified on the studied ornamental stone); mentioned species in this sense are: *Penicillium sp., Alternaria sp., Fusarium sp., Cladosporium sp.* (Grote & Krumbein, 1992; Sterflinger, 2010; Ilies et al., 2018 a, b).

Carbonates from the stone, represented by calcite, being the main component of the cement of the rock, can be also affected by microorganisms due to acid local environment generated by them and deposited as secondary calcite (Adamo & Violante, 2000; Burford et al., 2003a; Olteanu, 2015).

Regarding *the silicates*, which are very well represented in the studied ornamental stone (Table 2, Fig. 7, 8) through K-feldspar and albite, the microorganisms (e.g., fungi) can play an important role in the dissolution process (generate important nutrients (e.g., K, P and Fe) and have the possibility to form secondary clay minerals (Banfield et al., 1999; Adamo & Violante, 2000; Fomina & Skorochod, 2020). In fungal reaction with silicates, "*fungal extracellular polysaccharides can become mixed with calcium, potassium, iron, clay minerals and nanocrystalline aluminous iron oxyhydroxides*" (Gadd, 2017b). Presented Mica-group minerals can be impregnated by fungal hyphae, being favoured by cleavage planes and can suffer complex transformations.

It is also important to establish the interrelations with the environmental factors: temperature, humidity, sun exposure, climatic events, which assure potential nutrient sources etc (Sterflinger, 2010).

Rock damage can also be caused by ***atmospheric factors***. Therefore, moisture can cause rock degradation by infiltration and the generation of mechanical and chemical stress (Tombach, 1982); solar radiation and temperature changes lead to the formation of cracks and increased rock porosity and wind, by conducting solid and liquid particles to the surface of the rock, causes local wear (Moncmanová, 2007). Potential causes of rock damage are also air pollutants, of which sulphur dioxide (SO_2) and nitrogen dioxide (NO_2) are shown to have severe destructive effects (Tombach, 1982).

In the studied case, in term of precipitation, the highest average amount was recorded in June (86.1 mm). February represents the main minimum value for the amount of precipitation, the multiannual average value being 30.9 mm, while the minimum value has dropped to 0.3 mm (Fig. 9).

Regarding the air relative humidity (Fig 10), it is noticed that the highest average monthly value was recorded in January (98%) and the minimum in March (13%). The multiannual average value for relative humidity was 66.2%, recording the highest monthly value in December (78.67%), and the lowest value in April (55.54%).

The exposure of the building plated with natural stone (Saiz-Jimenez et al., 1990; Gadd, 2017b) is very important (south – east in this study case); biofilms generated by colonies of microorganisms can create a special environment and can contribute to the removal and immobilization of pollutants, especially in the case of northern facades, exposed to precipitation.

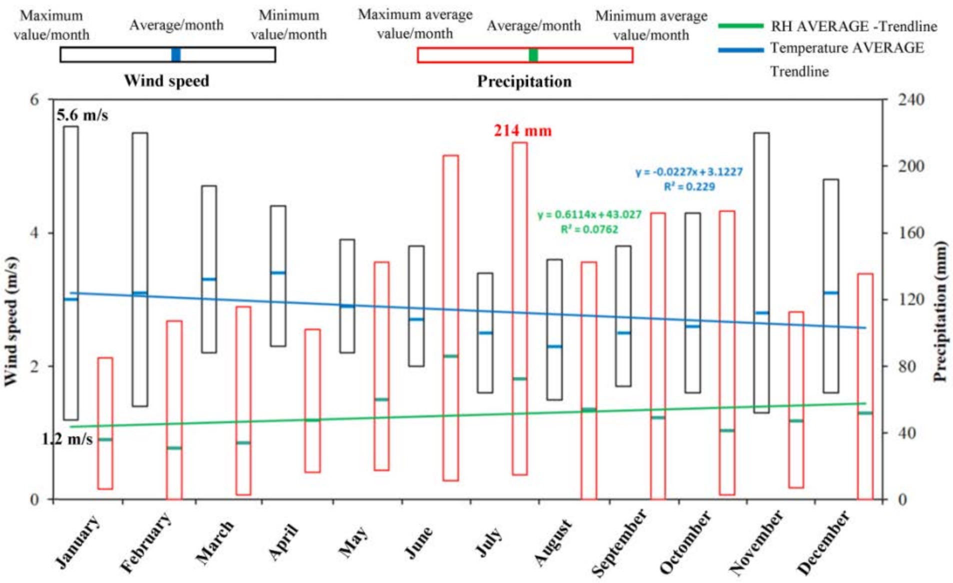

Figure 9. Multiannual monthly variations in average, maximum and minimum wind speed and changes in average, maximum and minimum monthly multiannual precipitation in Oradea city, Romania (2008-2020) (data sources: APM Bihor)

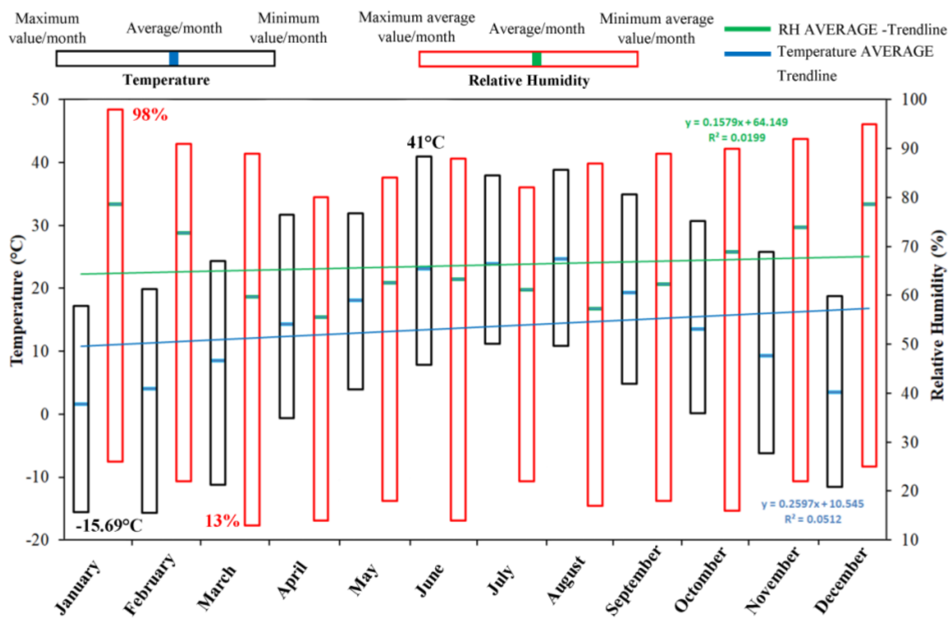

Figure 10. Multiannual monthly average values and absolute maximum and minimum air temperature values and variation in the multiannual monthly average, maximum and minimum values of relative humidity in Oradea city (2008-2020) (data sources: APM Bihor)

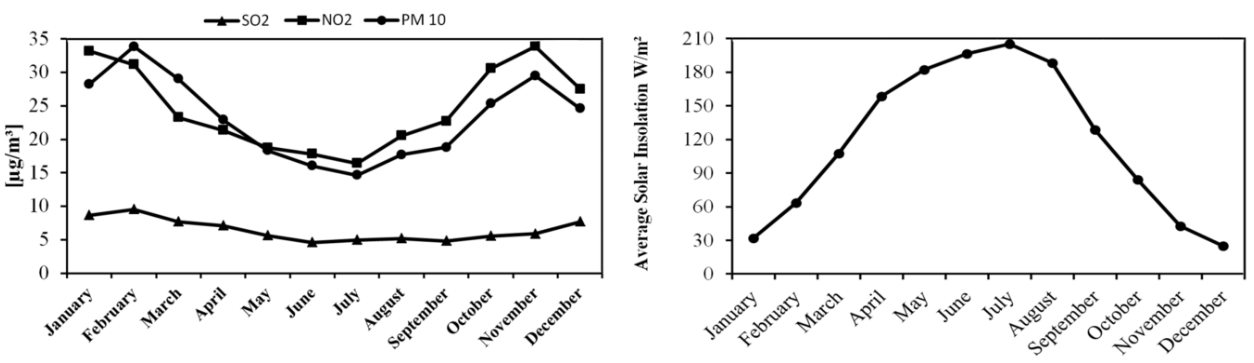

Figure 11. Variation of multiannual monthly average values of SO_2 (µg/m³), NO_2 (µg/m³), PM_{10} (µg/m³) (left) and solar radiation (right), in Oradea city, Romania (2008-2020) (data sources: APM Bihor)

Solar radiation recorded an average multiannual value of 117.8 W/m^2. It presents an annual upward trend, starting in January, when it registers an average value of 39.98 W/m^2, until July when it reaches the maximum value of 205.17 W/m^2, after which its value decreases (Fig 11 right).

The result of the analysis of wind speed data shows that the average value in the analysed interval was 2.85 m/s. The highest values are recorded in the cold months and exceed 3 m/s.

Air pollutants (especially in urban areas) can also significantly contribute, especially with nutrients, to microorganisms (Griffin et al., 1991; Hirsch et al., 1994; Perry et al., 2005; Sablier & Garrigues, 2014). Moroni & Pitzurra (2008) state that fungi on the lithosubstrate of the outdoor buildings use in their metabolisms carbon and nitrogen species from natural (biomass) and anthropogenic (atmospheric pollution) sources (Wolf & Krumbein, 1996), sulphate and metal compounds (Garraway & Evans, 1984; Burgstaller & Schinner, 1993; Burford et al., 2003b). A higher sensitivity is mentioned in the case of carbonate rocks, according to the experiments made and published by Moroni & Pitzurra (2008), in which it was shown that survival of fungi on the contaminated substrata are remnant even after the elimination of the pollutants.

An essential part in the deterioration of rocks is represented also by the suspended particles that are deposited on surfaces, which form a suitable environment for the development of fungi (Uring et al., 2020). The analysis of figure 11 points out that the maximum monthly average value for the concentration of sulphur dioxide (SO_2) was registered in November (6.5 µg/m³), and for the concentration of nitrogen dioxide (NO_2) in July (214 µg/m³). At the same time, PM_{10} particulate matter presents the highest monthly average value in February (33.85 µg/m³).

5. CONCLUSIONS

The study may contribute to mineralogical, petrographic, geomorphological and environmental analyses for identifications of degradation processes which affect the natural stone as components for old heritage buildings. The results will be useful for reducing the stress factors on one hand and for finding scientific-based solutions in conservation and restoration of heritage building in the large towns. The stone decay of the bio-extraclastic grainstone from the portal of the Ursuline Monastery from Oradea city took place under the influence of mineralogy and petrology of the raw material and environmental (natural and anthropogenic) factors.

Identification of the row material offers the possibility of bringing natural and ornamental building materials/stones from the same place, as initial, with the same physical-chemical, mechanical or similar properties. The study also helps to transfer the knowledge about the growing impact of the environmental factors on historical buildings and monuments to the public (tourists, students etc.), representing a possible stop in an urban geotrail (tourist, didactic etc.) in Oradea city. The research article was produced with equal scientific involvement of all authors.

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