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Research paper

Influence of environmental factors on physical and mechanical characteristics of the opoka-rocks

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Abstract: This paper presents laboratory analyses of the influence of an acidic environment, salinity and temperature change are able to exert on the geomechanical properties of the opoka-rocks. This rock material, deriving from four sites in East–Central Poland, was found to be variously resistant to factors like the destructive action of water-soluble salts and the effects of an acidic environment, on account of the actually-diverse nature of the rocks in question. Ultimately, the work offered a basis for a distinction to be drawn between the light opoka-rocks present at the Annopol and Kazimierz Wielki sites, and the heavy opoka-rocks from Bochotnica and Krasnobród, in terms of both textural and physical-mechanical features. The heavy opoka-rocks from Krasnobród proved least resistant to an acidic environment, which left strength reduced significantly. This kind of rock also experiences both an increase in porosity and absorbability and a decrease in weight. Furthermore, the influence of an acidic environment on aesthetic features of the examined rocks was in all cases negative, salts formed a patina on surfaces that obscured original structural and textural features. None of the tested types of rock presented resistance to the crystallization pressure such salt is able to exert.

Keywords: crystallization pressure, acidic or saline environments, geomechanical properties, transitional rocks

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1. Introduction

Stone has played its part in human existence from almost the very beginning. Flints and other solid rocks were first made use of in primitive toolmaking in the Pliocene (about 3–2.5 million years ago). Human development, and in particular the exchanging of a nomadic lifestyle for a sedentary one, raised the importance of stone as a building material greatly. Eleven thousand years ago, human beings began to erect the first stone structures, while over 9000 years back clayey raw materials began to be used in the manufacture of ceramic dishes and bricks. Rapid development of what came to be known as monumental Romanesque architecture caused a demand for high-strength stone, though raw and simple architectural forms continued to prevail. Stone walls sometimes reached thicknesses close to 1 m, and building stone was usually taken with little discrimination from nearby areas. Only with the turn of the 18th and 19th centuries did the beginning of an era of dynamic development of industry and technology see the place of natural stone begin to be taken gradually by modern materials such as concrete, cellular concrete, ceramic hollow bricks or silicate bricks.

However, even in the 21st century, stone raw materials have not lose their significance when it comes to building. Private investors are in fact increasingly interested in natural stone, appreciated on account of its unique texture, aesthetic value and ecological character. However, the proper selection of stone as a material in construction and decoration demands a knowledge of physical and mechanical properties, as well as possible surface impacts exerted by environmental factors. High-intensity capillary action, absorbability, salinity and variable pH in the environment all have impacts impairing strength parameters, as well as potentially compromising aesthetic value significantly, when and as the process of deterioration is also being accelerated. Damage to items of architectural heritage built from stone is most often caused by factors such as variable temperature conditions, decomposition or transformation of the primary mineral components of the rock, crystallization pressure exerted by salts dissolved in water, and the action of microorganisms.

In addition, a contaminated atmosphere accelerates destructive processes through surface adsorption of tar particles, dusts and acidic chemicals. These only increase the presence in rocks of products of chemical corrosion, *inter alia* salts that are soluble in water [19,32]. The influence of chemical and physical factors on building materials was studied in many aspects, laboratory tests, and included various types of rocks [5, 8, 13, 15, 16, 33]. In the analysed literature, there are no publications in the field of research on the group of transition rocks characterized as opoka-rock. These rocks are often used as a construction material and as an additive to cement. Due to the transitional nature of siliceous and carbonate rocks, these materials differ in terms of geomechanical properties.

1.1. The Characteristics of the opoka-rocks

The object of the research is the opoka-rock that is included among the transitional rocks between silica rocks and those of a carbonate character. Analyses show secondary mineralisation processes at work in these rocks, and leading to significant differentiation of their phase and chemical composition, and also therefore their physical and mechanical features. On a petrographic basis, it was possible to assign the opoka-rocks studied as the so-called heavy or decalcified (light) categories – see Table 1 [3].



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Pook name	Chemical component [wt.%]			
Rock name	SiO ₂	CaO		
Calcium opoka-rock ("heavy" opoka)**	5-25	53.2–42		
Calcareous opoka-rock**	25-50	42–28		
Decalcified opoka-rock "light" opoka**	50-75	28-14		
Silificated opoka-rock*	40.2–66	16-30.5		

Table 1. Classification of opoka-rocks depending on the percentage of silica and carbonates [3*, 30*]

Outside Poland, rock of this kind is to be found in Germany, France, the Czech Republic, Slovakia and Russia. In Germany, it is in Saxony (in the Dresden area) and in Westphalia that it is possible to find opoka-rocks from the lower parts of the Upper Cretaceous i.e. the Turonian and Cenomanian. The rich deposits of these rocks extend into the Czech Republic from Kadania, east of Prague through to the Moravian region. Such rocks can also be found in Lower Tertiary deposits in the Volga Valley, in the eastern parts of the Urals and in Upper Cretaceous sedimentary deposits of the eastern European part of Russia through to Cretaceous deposits in the Paris Basin.

In Poland, such deposits occur in the centre and east of the country, over extensive areas of the Lublin Upland, as well as in the Nida and Mogilno-Łódź Troughs. In terms of lithostratigraphic profile, these sediments have been found from the Turonian through to the Maastrichtian. In addition, these rocks are often found in the upper parts of the Mesozoic bedrock complex, in the so-called contact zone with Neogene deposits [10].

This paper describes analyses of stone building materials in terms of the impact of acidic or saline environments and low temperature on the opoka-rocks. These are not solely issues of cognitive relevance, given the practical significance, as obtained results were expected to point to directions for the proper use of the rocks studied, as well as appropriate methods of conserving them. Indeed, while the nature of the stone in question has long been studied, effective methods by which to protect it from harmful environmental effects have not gained presentation thus far. And, as this stone is also used in European countries other than Poland, knowledge acquired in the course of the research detailed here was expected to be of more than just local significance.

2. Research material and methodology

The research material came from the east-central part of Poland, and specifically from the Kazimierz Dolny, Annopol, Bochotnica and Krasnobród areas. Throughout the field and laboratory work there were a total of 334 rock samples. Thus, physical and mechanical properties established in line with standards were:

- open and total porosity [24],
- compressive strength in the air-dry state [25],
- density of solid particles and bulk density [24],
- absorbability of stone material [26] and capillary absorbability [27],

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- resistance to salt crystallization [28],
- determination of frost resistance [29].

In order to determine the structural and textural features of the rocks an FEI Quanta 200FEG scanning microscope (SEM) equipped with an X-ray spectrometer (EDX Genesis) and a backscattered electron detector (BSE) were used.

While the statistical calculation of uniaxial compressive strength test results was carried out in accordance with the recommendations set out in Annex C of standard [25]. The factor for estimating the k_s quantiles was assumed on the basis of Table C.1 [25], (intermediate values were interpolated).

3. Results

Geomechanical studies of the opoka-rocks from Annopol, Kazimierz Dolny, Bochotnica and Krasnobród showed differences between the determined physical and mechanical parameters and the linear relationship between them.

The bulk density of rocks from Bochotnica as well as from Krasnobród exceeded 1.8 mg/m³ (Table 2). In line with Standard [24], this provided for a categorisation of the rocks as heavy opoka-rocks. Qualification for the so-called "light" opoka-rocks in turn takes place where bulk density is below this value. Light rocks were those sampled at Kazimierz Dolny and Annopol.

Origin de [mş	Dullt	Bulk density $[mg/m^3]$ Density of solid particles 	Absorbability		Porosity [%]	
	density [mg/m ³]		Atmospheric [%] n = 12	Capillary [kg/m ²] n = 12	Open <i>n</i> = 12	Total $n = 2$
Annopol	1.46 n = 27	2.64	23.9	17.1	33.9	45.7
Kazimierz Dolny	1.46 n = 33	2.49	24.5	15.3	31.8	44.2
Bochotnica	2.02 n = 12	2.65	7.2	6.7	20.2	26.7
Krasnobród	2.32 n = 18	2.77	4.8	3.1	11.0	16.3

Table 2. Physical properties of the opoka-rocks (average value

n – number of samples

All the tested opoka-rock samples showed relatively high porosity. The light opoka-rocks had an average value for open porosity of up to 30% and closed porosity up to 50%. The heavy opoka-rocks from Krasnobród had open porosity values less than one-third as great (Table 2). Rock surface analysis in 3D using a Hirox-RH-2000-EN digital microscope confirms the highly porous nature of the rocks studied, with pore shape ranging from oval to irregular, albeit with interconnected networks not present. The porosity coefficient determined for part of the rock surface was of almost 10% [2].

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The absorbability of the opoka-rocks from Kazimierz (24.5%) and Annopol (23.9%) is inversely proportional to bulk density. However, the heavy opoka-rocks from Krasnobród had a figure for absorbability that was only one-fifth as high (at 4.8%) (Table 2).

Strength tests carried out for rocks in air-dry state supported classifications as of low or medium strength [25]. The average values presented in Table 3 were calculated from 12 samples, while the maximum range of values noted for this parameter was from 14.6 MPa (in the case of the light opoka-rock from Kazimierz Dolny) to 39.1 MPa (in the case of heavy opoka-rock from Krasnobród).

The strength tests were additionally carried out after cyclical saturation with a water pH < 3 and freezing and thawing in the presence of a salt solution (Table 4). Test studies in this case involved rock material previously found to differ most, hence the selection of light opoka-rocks from Kazimierz Dolny as well as heavy opoka-rocks from Krasnobród.

Rock name	Strength in the air-dry state [MPa] average value	Standard deviation	Expected lower value [MPa]	Coefficient of variation v
Light opoka-rock/Annopol	15.7	5.0	7.8	0.32
Light opoka-rock/Kazimierz Dolny	14.6	4.1	8.0	0.28
Heavy opoka-rock/Bochotnica	18.7	3.7	12.4	0.20
Heavy opoka-rock/Krasnobród	39.1	8.4	24.0	0.22

Table 3. Results of compressive strength in the air-dry state of the studied opoka-rocks

Table 4. Results of compressive strength tests in changing environmental conditions of light and heavy opoka-rock

Rock name	Type of sample $n = 6$	Compressive strength [MPa], Average value	Standard deviation	Expected lower value [MPa]	Coefficient of variation v
Light opoka-rock/ Kazimierz Dolny	In the air-dry state	14.6	4.1	8.0	0.28
	After water saturation pH < 3	11.6	5.8	3.0	0.50
	After the test of frost resistance in salt	15.9	3.1	10.2	0.20
Heavy opoka-rock/ Krasnobród	In the air-dry state	39.1	8.4	24.0	0.22
	After water saturation pH < 3	27.3	8.9	10.2	0.33
	After the test of frost resistance in salt	50.4	21.2	15.6	0.42

n – number of samples



3.1. Research on cyclic saturation in acidic solution

The test for cyclic interaction of citric acid after 5 days of study revealed significant differences between rocks in terms of macroscopic appearance and changes of properties (Fig. 1). A white raid appeared on the surface of the opoka-rocks, which completely concealed their original textural nature. The reaction of calcium carbonate which builds a rock with citric acid sees calcium citrate to be formed (Fig. 1b). The discussed reaction was much more intense in the opoka-rocks from Krasnobród, given that it is richer in CaO than the rocks from Kazimierz Dolny (Fig. 1a). Activity in an acidic environment results in a decrease in compressive strength and an increase of absorbability of the opoka-rocks, in particular the heavy opoka-rocks from Krasnobród (Table 4). Treating with acidic solution caused a decline in the mass of samples of a few percent, while increasing their absorbability. In addition, absorption of water on the surface of a heavy opoka-rock specimen from Krasnobród was almost doubled (Table 5).



Fig. 1. Appearance of the opoka-rocks following the acid test: a) light opoka-rock on the left and heavy opoka-rock on the right after soaking in acidic solution, b) heavy opoka-rock – salt precipitation, c) light opoka-rock – color change on the surface

Rock name	Type of sample, n = 6	Atmospheric absorbability [%], Average value	Capillary absorbability [kg/m ²] Average value	Change of mass
	Natural	24.5	15.27	n.d
Light opoka-rock/ Kazimierz Dolny	After water saturation pH < 3		19.01	-1.9%
	After the test of frost resistance in salt	25.5	19.10	-0.2%
Heavy opoka-rock/ Krasnobród	Natural	4.8	3.11	n.d
	After water saturation pH < 3	8.1	5.20	-4.1%
	After the test of frost resistance in salt	3.9	4.51	-0.8%

Table 5. Comparison of absorbability of the studied opoka-rocks with changing environmental conditions

n – number of samples; n.d. – not determined, the samples disintegrated



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3.2. Studies on cyclical salt interaction

The next stage of the research entailed cyclical tests of the opoka-rocks which immersed in a solution of NaCl. Some samples withstood 15 cycles in such a solution, only to be destroyed during the final immersion in water. The light opoka- rock from Annopol and Kazimierz Dolny was in turn destroyed after just 5–7 cycles, while the heavy opoka-rock from Bochnica was destroyed with the number of 8 cycles. The greatest resistance to salt crystallization was found in the samples from Krasnobród. The number of cycles after which such samples were destroyed was in the 14–15 range. However, none of the tested rocks displayed genuine resistance to crystallization pressure imposed by the salt.

On the basis of the investigations into freezing in the salt solution, an increase in compressive strength was to be noted for both the light and heavy opoka-rock. Some of the heavy opoka-rock samples attained a compressive strength exceeding 60 MPa (Table 4). Observations with the use of SEM / EDX showed changes in the structural and textural characteristics of the opoka-rocks. Especially in the light opoka-rock from Kazimierz Dolny. In these rocks, the voids and gaps have been filled with salt (Fig. 2).



Fig. 2. Salts crystallizing in the light opoka-rocks space from Kazimierz Dolny. SEM/EDX

4. Discussion

The results of the geomechanical tests carried out on opoka-rocks from east-central Poland allowed to observe a certain linear relationship between the determined parameters. For example a greater porosity is for example associated with lower bulk density. Obtained results of this kind are seem to be comparable with the data in the literature (Fig. 3).

The tested opoka-rocks are also characterised by a very high absorbability as a reflection of the considerable aforementioned porosity. Water absorbability is likewise inversely proportional to the bulk density of the rock being tested (Fig. 4).

Different properties as porosity and bulk density exert their influence where the muchdifferentiated water absorption capacities of surfaces are concerned. Together with bulk density, rocks have capillary absorbability values varying across a wide range from 3–17 kg/m². Despite

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Fig. 4. Relationship between absorbability and bulk density of the opoka-rocks

being characterised by this high level of capillary action, the light opoka-rock from Annopol and Kazimierz does not differ significantly from ceramic construction products. Fig. 5 shows the weight of absorbed water per 1 m² of surface of the studied natural stone after 24 hours. Literature data on other building materials were also used for comparative purposes [11]. The concrete in this combination is characterized by the lowest absorbability, and represents the closest competition of natural stone. Pore diameter in the range 0.1 mm – 10⁻⁴ mm combines with the oblong shape of pores to influence the high observed values for capillary absorbability in the tested opoka-rocks. Structural and textural features here often reflect the presence of bioclasts forming the tubular skeleton of the studied rocks studied. The presence of sponge needles was observed in the opoka-rocks from the Lublin region. Those needles form tubular pores conducive to high absorbability and capillary action. Water absorption may also be enhanced by the presence of clay minerals from the smectite group, as mineralogical studies have demonstrated [4].

The work on capillary absorbability in this opoka-rock showed that the weight of absorbed water increases very slowly after 24 hours (Fig. 6).



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1 – Concrete with density 2300 kg/m³, 2 – The Krasnobród opoka-rock, 3 – The Wola Komborska sandstone,
4 – The Bochotnica opoka-rock, 5 – Calcareous-ceramic mortar, 6 – Fine grained sandstone, 7 – The Kazimierz opoka-rock, 8 – The Annopol opoka-rock, 9 – Ceramic brick

Fig. 5. Capillary absorbability of the studied natural stones and selected building materials after 24 hours



Fig. 6. Capillary absorbability of the studied opoka-rocks in time

Equally, a stone wall exposed to even short-term (two-hour) rainfall already reaches almost half of its maximum humidity. All of the tested natural stone proved to be unresistant to the crystallization pressure imposed by salt. The heavy opoka-rock from Krasnobród was the most resistant. Despite the partial loss of components, the mass of all samples increased with each cycle, due to the accumulation of sodium sulphate crystals. Research by Tsui et al. [22] and Benavente et al. [9], points to there being two stable phases formed from sodium sulphate, relating to mirabilite and tenardite. Impregnation of the sample with the sodium sulphate solution results in crystallization of the synthetic mirabilite, as the most stable phase in the air-dry state [6,7,14]. In case of induced dehydration, salt present undergoes structural change as it turns into tenardite.

The research makes it clear that natural stone of more limited open porosity displays greater resistance. According to data in the literature [17], it is usual that the more limited the bulk density of the studied opoka-rock, the greater the amount of silica present in its mineral composition. In spite of silica binding the components of the light opoka-rock strongly, the high degree of open porosity denotes a limitation of this high resistance. The heavier opoka-rock is of lower porosity, but does not contain such a significant amount of silica. In addition, these opoka-rocks contain clay minerals [12], and reduce resistance to the pressure accompanying



crystallizing salt. Considerable stress imposed as walls dry is due to the crystallization of salts in building materials.

When the obtained research results are compared with data in the literature on stress modelling in a concrete wall and a brick wall of thickness 25 cm as a result of salt crystallization [21], absorbability of the light opoka-rock is seen to be more similar to brick. The evaporation of water on the surface of the wall and attendant growth of salt crystals causes stretching stresses even exceeding the 4.5 MPa of a brick wall 25 cm thick. Such high stresses may exceed the stretching strength of bricks and opoka-rock which leads to damage and scratches on, with the result that walls (often historic in character) sustain damage and scratching where insulation protecting against penetration by harmful salts is lacking.

Strength tests revealed that the light opoka-rocks are of significantly greater strength in relation to their bulk density, on account of the presence of siliceous sponge needles that reinforce stone structure and limit its formation of microcracks. According to Klemm [23], strength does indeed increase with silica content, while it is decreasing in relation to the content of carbonates. Figure. 7 presents the relationship between the strength (Rc) and bulk density (ρ) of the studied opoka-rocks. This graph was also supplemented with literature data on other carbonate rocks present in the studied area (Fig. 7).



Fig. 7. Relationship between compressive strength and bulk density of rocks (own elaboration and based on [3, 12, 30]

In line with the huge impact on rock strength exerted by humidity, the research suggests that a content of clayey minerals has an additionally disadvantageous effect on the strength parameter.

The light opoka-rocks have their strength reduced by more than a half, and data in the literature [12] associate this with a considerable content of minerals from the smectite group. They swell greatly in the presence of water, which penetrates into pockets. Rock compactness is reduced in this way, and strength likewise – a fact that is recognised as mitigating against the use of the opoka-rock in hydrotechnical construction. The unfavourable effect of an acidic environment (and especially acid rain) on carbonate rocks has obviously gained repeat mention in the literature [1, 18, 20]. Harmful substances occurring in the atmosphere are most often formed in industrial districts, and acid solutions arise when water contained in the air join with the sulphur dioxide formed as a result of the burning of solid fuels, or else oxides of nitrogen (from petrol combustion), or hydrogen chloride or dust (from industrial plants), or other compounds. In particular strong sulfuric and nitric acids are characteristic for their destructive effects on limestone and other carbonate rocks. In an acidic environment, calcium carbonate is decomposed into salts that are very soluble in water. Some of these are washed away by rain, while the remainder pass into stone and continue a reaction process with it. Sulphate corrosion products may be deposited on the surface of the rock, ensuring its sealing. Sulphates can also react with metal carbonates in the presence of small amounts of water that are unable to dissolve the formed salt. In such cases, the newly-formed compound, which occupies several times more volume than calcium carbonate, exerts a very strong internal pressure capable of leading to the destruction of the stone [31].

The acid treatment of carbonate contained in rock leads to an increase in pore sizes. As a result of the decomposition of rock-forming material, some pores that were originally closed become now open. The components of the opoka-rocks that are the most sensitive to corrosion are the carbonate bioclasts [12]. Pore enlargement acts to reduce both the mass and strength of rocks, while increasing their absorbability. The latter can be increased further by means of new compounds, e.g. calcium nitrate and calcium chlorate – both of which have strong hygroscopic properties. This was also confirmed by studies carried out on the opoka-rock originating at Janowiec and in fact taken from its 16th-century castle walls. The work in questions points to absorbability that is 24% greater than in the case of quarried stone [1].

5. Conclusions

Research on the opoka-rocks originating in the east-central part of Poland, reveals the degree to which these have diverse physical and mechanical parameters. Heavy opoka-rocks are found to be least resistant to an acidic environment and salinity. They also experience changes of greatest intensity, i.e. a reduction in porosity and absorbability. All of the examined rocks also showed a lack of resistance to salt crystallization.

The results presented here may prove of practical significance in signaling the proper use that may be made of the studied rocks, as well as the way those present in existing buildings may be preserved. The light opoka-rocks are shown to have the more favourable features where use in housing construction is concerned. They are characterised by favourable thermal and strength properties, and offer greater resistance to the negative impact of an acidic environment and salinity. In heavily-polluted parts of an urban agglomeration, salts crystallize faster and solutions become more intensely acid, often to the total detriment of the stone [20, 32]. This makes it important that there be background knowledge on the opoka-rocks, as regards their origin, age of building and nature of structural and textural features. Such information is very relevant to the effectiveness of conservation treatments, such as desalination, reinforcement or hydrophobisation of stone [34].



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Wpływ czynników środowiskowych na właściwości fizyczne i mechaniczne opok

Słowa kluczowe: ciśnienie krystlizacyjne, środowisko kwaśne, sole, właściwości geomechaniczne, skały przejściowe

Streszczenie:

W ramach przeprowadzonych badań nad opokami pochodzącymi z miejscowości: Kazimierz Dolny, Annopol, Bochotnica i Krasnystaw stwierdzono, że należa one do grupy skał przejściowych pomiedzy węglanowami a krzemionkowymi. W efekcie badań stwierdzono opoki lekkie oraz ciężkie, różniące się gęstością objętościową. W miejscowościach Annopol i Kazimierz Dolny rozpoznano opoki lekkie, natomiast w Bochotnicy i Krasnobrodzie, opoki ciężkie. Przedmiotowe skały charakteryzują się dużym współczynnikiem zmienności w badaniach wytrzymałościowych, dlatego zaleca się wykonywanie badan opok na większej liczbie próbek. Wysoka porowatość opok wpływa na ich dobre właściwości termoizolacyjne, jednak cecha ta oddziałuje na wzrost nasiąkliwości badanych skał. W wyniku oddziaływania wody radykalnie spada ich wytrzymałość na ściskanie. W opokach lekkich zanotowano trzykrotny spadek tego parametru od wartości pierwotnej. Wszystkie typy skał, wykazały brak odporności na krystalizację soli. Wykazano, że obecność kryształów soli w porach i pustkach skalnych powodowała wzrost wytrzymałości na ściskanie. W przypadku opoki lekkiej z Kazimierza Dolnego zaobserwowano wzrost wytrzymałości o 8.9%, a opoki ciężkiej z Krasnobrodu o 28,9%. Sól krystalizująca w warstwach przypowierzchniowych ma właściwości higroskopijne, co wpłynęło na zwiększenie nasiąkliwości powierzchniowej. Zaobserwowano, że w pewnych przypadkach, kiedy pory kamienia mają duże rozmiary lub gdy roztwór soli nie wypełnił ich w dostatecznym stopniu, krystalizacja soli nie powoduje wzrostu ciśnienia, w wyniku czego nie dochodzi do obniżenia wytrzymałości. Badania oddziaływania kwaśnych roztworów na opoki,



wykazały wpływ na redukcję wytrzymałości. Największy spadek wytrzymałości na ściskanie wykazała ciężka opoka z Karsnobrodu. Wynikało to z największej zawartości węglanu wapnia wchodzącego w reakcje z kwasami. Ponadto, rozpuszczanie się składników skały w kwaśnym roztworze zwiększa jej porowatość, co skutkuje zwiększeniem nasiąkliwości. W wyniku reakcji węglanu wapnia z kwasem na powierzchni kamienia, tworzą się sole o właściwościach higroskopijnych, które mogą znacznie zwiększyć powierzchniową absorpcję wody. Na skutek zanurzania w kwasie i rozpuszczania się składników skały, zaobserwowano utratę ich masy. Największy spadek wynoszący 4,1% wykazała ciężka opoka z Krasnobrodu. Oddziaływanie kwaśnego środowiska powoduje pogorszenie ich walorów estetycznych. Sole krystalizujące na ich powierzchniach tworzą warstwę patyny, która zaciera pierwotne cechy strukturalnoteksturalne opok.

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