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Analysis of Research Procedures for Testing Capillary Absorption Properties of Rock Materials with Respect to Standard Requirements

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Abstract. Currently, there are over twenty European quality standards related to the methods of testing natural stone. These quality standards describe the determinations of density, porosity, frost resistance or abrasiveness. In the paper, the authors focus on the tests of capillary absorbability of rock materials. The tests described in the paper present what the diversification of test results looks like, using all possible variants of the research procedure which are indicated by the quality standard in question. Samples of different dimensions and shapes were compared. The authors have also proposed an alternative methodology for testing capillary adsorption, involving the isolation of the side wall of a sample in order to enable the highest possible precision in representing the actual conditions under which rock elements work. The representation of the actual conditions under which the given material will be working in a laboratory is a very important factor, indicating the fact that the conducted tests have their reflection in reality. By getting acquainted with the quality standards in question and analysing the research procedures, it may be concluded that rocks are considered to be a homogeneous material. In reality, that is not the case. Rocks are the most diverse group of materials. Unlike ceramic materials or concrete products, for which we are capable of designing a mixture, which would fulfil our expectations without much trouble, rocks are an unpredictable material which was created thousands and millions of years ago, and on which at this moment we have no influence whatsoever. In this article, we are dealing with highly diverse porosity, microstructure or even anisotropy. All these factors affect the test results. Due to this, when conducting the tests of rock materials, the samples should be selected in a manner which would make them representative for the given portion of the rock. It is a very important process, which should be executed with awareness and care, in order to make sure that the results obtained by us accurately reflect the actual parameters of the material. To this end, it is necessary to possess proper knowledge about rocks, the whole deposit and the genesis of its creation.

1. The determination of absorbability according to the quality standard

Until the beginning of the 21st century, quality standards did not determine methods for testing the absorbability of rock materials. However, considering that this is one of the more important factors defining the usefulness of rocks, quality standard PN-EN 1925:2001 [1] has been developed and introduced, being an official translation of European quality standard EN 1925:1999. The abovementioned quality standard allows the measurement of absorbability using cubical samples with 50 mm or 70 mm edges, as well as cylinders with heights and diameters of 50 ± 5 mm or 70 ± 5 mm. The drying of samples should proceed in a temperature of $70^{\circ}C \pm 5^{\circ}C$ to a constant mass (differences

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submerged to a level of 3 ± 1 mm in a sealable tray. The measurements are performed until the last measurement is higher, by no more than 1% of the mass of absorbed water in relation to the previous one.

The purpose of the present paper was to check, what the results of capillary absorbability of rock materials look like in different variants allowed by the quality standard. The same samples were used each time in the tests, adopting an assumption about the absence of any changes occurring in the microstructure of the material due to drying. Changes resulting from the diversity of stone materials are described in papers [2, 3].

2. Materials and methodology

2.1. Materials

Samples for the tests were prepared in accordance with the table below.

	Cylinder with a diameter and	Cylinder with a diameter and	Cube with an edge of 50mm	Cube with an edge of 70mm
	height of 50mm	height of 70mm		
Quantity	39	8	9	10
Sum		66		

Table 1. The number and shape of samples used for the tests

The samples were drilled out of larger rock blocks collected directly in mines, and cut to their correct sizes. Figure 1 presents a picture of rock blocks from which samples were drilled out for the

tests, while Figure 2 presents samples already drilled and cut to the right dimensions.



Figure 1. Rock blocks used in the tests



Figure 2. The prepared cylindrical samples

The table below presents a list of rocks, which were used in the tests and their short petrographic descriptions, along with basic physical, chemical and mechanical properties declared by their manufacturers [4].

Rock code	General petrographic description	Physical, chemical and mechanical properties
A	Gneiss. Metamorphic rock. Fine- grained rock with a foliated texture	density: 2.68 g/cm ³ bulk density: 2.61 – 2.64 g/ cm ³ porosity: 2.7 % absorbability by weight: 0.49 %
В	Basalt. Tertiary rock, compact	bulk density: 2.94 g/ cm ³ absorbability by weight: WA24=0.6% frost resistance: F1
С	Jurassic limestones (Upper Oxfordian)	density: 2.73 g/ cm ³ bulk density: 2.53 – 2.70 g/ cm ³ porosity: 0.3 – 4.8 % absorbability by weight: 0.14 – 2.11 %
D	Jurassic limestone	density: 2.70 – 2.72 g/ cm ³ bulk density: 2.55 – 2.70 g/ cm ³ porosity: 3.7 – 4.4 % absorbability by weight: 0.6-3.8 %
E	Devonian limestones and dolostones	density: 2.87 g/ cm ³ bulk density: 2.51 – 2.81 g/ cm ³ porosity: 2.0 % absorbability by weight: 0.5 – 3.8 %
F	Basalt	bulk density: $2.73 - 2.88 \text{ g/ cm}^3$ absorbability by weight: $1.3 - 1.5 \%$
G	Basalt. Tertiary rock, compact	density: 2.73 g/ cm ³ bulk density: 2.53 – 2.70 g/ cm ³ porosity: 0.3 – 4.8 % absorbability by weight: 0.14 – 2.11 %

Table 2. The list of rocks used in the tests

The samples were prepared for the tests under uniform, standardised conditions.

2.2. Methodology

The quality standard [1] allows the use of samples of two shapes, each of which may take on two defined sizes. In addition, the quality standard does not define the impact of factors resulting from conducting the tests on various anisotropic planes of the material. It only informs that at least six samples should be selected for each water ascent direction in relation to the direction of the anisotropic planes. Therefore, can results developed based on the quality standard for samples of various geometries and the arrangement of anisotropic surfaces be treated in the same manner and compared to each other directly?

The conducted tests also checked whether the isolation of the side wall of a stone sample affects the course and the result of capillary absorbability compared to unisolated samples. The thesis whether the result of capillary absorption would change by decreasing the surface area participating in the ascent has been checked. Isolating the side wall of a sample was supposed to reflect the actual conditions which are present in stone materials. This issue will be discussed more deeply in the following paragraphs.

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3. Comparative results

3.1. Cubes vs cylinders

On figures below we compare selected samples to show, how was the differentiation between them in capillary absorbability.



Figure 3a. Selected samples and their capillary absorbability plots - limestone



Figure 3b. Selected samples and their capillary absorbability plots - limestone

Next figures present results for basalt rock.



Figure 4a. Selected samples and their capillary absorbability plots - basalt



Figure 4b. Selected samples and their capillary absorbability plots - limestone

3.2. Samples with an isolated side wall

When studying stone materials, it seems purposeful to use isolated side walls of samples. This results from the fact that small samples were cut out of large rock elements. Therefore, it has been assumed that isolation would simulate the continuity of a larger stone element, due to which the produced results would more precisely reflect the actual capillary absorbability of an element, as was the case with the tests of isolated concrete samples according to PN-EN 12504 [5]. The isolation of the side wall was intended to increase the internal pressure of the capillaries. Due to higher pressure, water in the capillaries would ascend higher and as a result the mass of water in the sample would increase. A chart of the described assumption is presented below:



Figure 5. A chart presenting the described concept (own research)

A sample plot of the observed differences in absorbability for isolated samples and samples without isolation is presented below.



Figure 6. Differences in absorbability for isolated samples and samples without isolation - limestone







Figure 8. Differences in absorbability for isolated samples and samples without isolation - basalt



Figure 9. Differences in absorbability for isolated samples and samples without isolation - gneiss

4. Summary and conclusions

4.1. Cubes vs cylinders

When comparing the results of capillary saturation for cubical and cylindrical samples, it can be concluded that cylindrical samples result in the capillary ascent of a larger amount of water compared to cubical samples. For samples D shown in Figure 3a and Figure 3b, the course of capillary saturation lines over time is very similar for both the cylindrical and the cubical sample. These rocks represent the group of Jurassic limestones. The rocks B and F are magmatic rocks. In the case of rock B there is

a clearly visible discrepancy between the measurement of capillary absorbability for two types of samples - the cylinder and the cube. In this case, the cubical sample absorbed almost two times less water than the cylindrical sample of the same rock. Rock F is distinguished from the others in that it was the cubical sample that absorbed more water. Discrepancies observed in the case of magmatic rock samples may result from various arrangements of the planes of anisotropy.



Figure 10. The arrangement of planes and their impact on capillary absorbability (own research)

This principle seems very important when choosing cubical samples for tests. As a result of testing cubical samples in any nonstandardised manner, they will cause discrepancies in test results. All plots of capillary absorbability for cubical samples run in a much more stable manner compared to cylindrical samples.

4.2. Samples with an isolated side wall

While analysing the plots of isolated and unisolated samples, it can be seen, that they have similar plot line shapes and contain similar changes in values. This looks as if the plot of the capillary absorbability of isolated samples was a shift in the function of an unisolated sample plot. On the other hand, the assumption that samples with an isolated side wall will cause the ascension of more water compared to samples without isolation has not been confirmed. All the results of conducted tests indicate the fact that it is exactly the opposite. It should be explained that in this case anisotropic diversity had no impact on test results. Precisely the same samples were used in here. At first, capillary absorbability without isolation was tested, and subsequently the samples were dried, isolated and again saturated in a capillary manner in the same position.

When drilling a sample, open pores of various sizes were created on the wall. In an unisolated sample these pores participated in the ascension of water directly from the tank, due to which there was a pressure drop in the remaining capillaries. An explanatory drawing of the described phenomenon is presented below.



Figure 11. A drawing presenting open side pores, which have been filled with water not by means of capillary ascent

5. Conclusions

The presented analysis did not confirm the assumption that while affected by an increased pressure, cylindrical samples with an isolated side wall were supposed to cause the ascension of more water than unisolated samples. As can be seen on plots on Figures 6 - 9, the isolation of samples caused a shift in the line of capillary ascent. The samples with an isolated side wall absorbed an average of 25% less water than samples without isolation. During the time provided for the performance of the test, none of the isolated rock samples exceeded the level of saturation achieved by a sample without an isolated side wall. This analysis excluded the impact of the arrangement of the planes of anisotropy. Both the tests for samples without isolation and with isolation were performed under identical conditions on the same samples.

By performing successful isolation of the side walls of rock samples, the capillaries and side pores were closed, due to which higher capillary pressure was sustained within a sample. Supposedly, if at a certain stage of the tests both samples were cut, water in samples with an isolated side wall would be present higher.

In the case of rocks whose plots of capillary ascent of samples with and without isolation exhibited the highest similarity, we may assume that the number of open side pores was low. Moreover, the pores themselves were of small sizes, due to which the mass of water contained within them was not significant. As a result, plots of ascension, which were very similar to each other, were obtained. In the case of samples where plots were very similar in terms of shape and resembled a shift of one plot relative to another, they probably differed only due to the filling of open side pores with water. The path of the penetration of water via a system of capillaries for both samples remained similar and unchanged in the case of isolated samples.

There is also a third probable pattern of the capillary saturation of isolated and unisolated samples. It is one where the impact of side wall isolation on the test result is the highest. This can take place in a case when the number of open side pores is considerable. Their closure via the isolation of the side wall may cause water to find another pattern of penetrating inside the samples. This would result in highly discrepant plots for capillary saturation between samples with or without isolation.

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