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COMPARISON OF MERCURY POROSIMETRY AND X-RAY MICROTOMOGRAPHY FOR POROSITY STUDY OF SANDSTONES

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ABSTRACT
Sandstones have been widely used as a building material since the medieval time all around the Europe. Porosity is one of the main factors affecting the resistance to weathering processes and consequently to the changes of mechanical and physical properties of these stones. Rock material is generally negatively influenced especially during the winter period when frost action takes place. Effect of salt crystals and ice formation depends on the character of pore space, including the pore size distribution. Mercury porosimetry is well known method which provides information about porosity and pore size distribution of samples, but as any other method, it has its own limitations. X-ray microtomography can be used as a complementary method enabling another "view" into the pore space. Main aim of this paper has been to provide the information about the use of these two mentioned methods and comparison of obtained results, within the study of sandstone weathering. The research was focused on two commonly used Czech Cretaceous sandstones - Hořice and Božanov. The stones were exposed to the accelerated durability test which is based on the meteorological data measured in Prague winters from 1998 to 2008. There were described the changes in the area of pores diameters > 5 μm. Use of mercury porosimetry together with X-ray microtomgraphy enabled more detailed understanding of the processes inside the stone structure.

KEYWORDS: sandstones, weathering, mercury porosimetry, X-ray microtomography

1. INTRODUCTION

Sandstones are consolidated clastic sedimentary rocks mainly formed of quartz grains with addition of feldspars and stone chips. The grains are joined by cement of different characters (e.g. calcareous, clayey, and ferruginous). The action of weathering processes leads to their deterioration and to the loss of their original physical and mechanical properties, i.e. to the changes of their durability. The weathering can lead to the loss of cement binder which can adversely influence strength characteristics. Intergranular spaces influence the porosity, presence and transport of the liquid phase inside the rock. Rock material is negatively influenced especially during the winter period when frost action takes place, what is typical for the climatic conditions in central Europe. The internal structure is changing as a result of water freezing and thawing and crystallization of salt in the pore space.

The freezing water in pores causes large pressures and leads to the degradation and disintegration of stone grains. Presence of water in the pore system affects the cohesion of grains if their state changes (Winkler, 1997; Thomachot and Jeannette, 2002). When water freezes, it increases in volume of approximately 9 % (Johannesson, 2010). This can cause large pressure and consequently changes in the pore structure. Hardness of frozen water crystals is 1.5 degrees of Mohs scale at 0 °C and 6 degrees at – 60 °C. The total porosity and pore radius increase depending on the number of freeze/thaw cycles (Winkler, 1997).

Crystallization of salts in pores can exacerbate the process of degradation as well. The origin of salt crystals forming from the solution in the pore space can cause also large pressure. According to Goodman (1989) the growth of salt crystals depending on temperature causes pressure of several tens to hundreds of MPa, which exceeds the tensile strength of most rocks. According to Pavlíkova et al. (2009) crystallization pressures of NaCl may reach up to 55 MPa. Besides crystallization pressures of forming salts, the process of hydration, contribute to the degradation as well. The volume of mineral phase increases during the hydration due to sorption of water and thus more pressure is exerted on the surrounding area. Hydration and dehydration processes take place in response to changes in temperature and relative humidity. The salt crystallizes first in the pores of larger diameter. When they are already filled thereafter then it crystallizes in small pores too (Putnis and Mauth, 2001). According to Goudie and Viles

(1997) rocks with high total porosity consisting of high amount of large pores and many small pores are highly susceptible to decay due to the effect of salt crystallization. Rocks with a high content of micropores are much more affected by salt weathering than rocks with a high content of pores of larger radius (Punuru et al., 1990), since the pores are connected.

The description of pore space, including determination of total porosity and pore size distribution, is important for the understanding of processes of deterioration. For these purposes the methods of mercury porosimetry and X-ray microtomography were used. The main aim of this study has been to demonstrate and compare the possibilities of selected methods for the purposes of detailed identification of the influence of the weathering processes on the changes in sandstone internal structure.

2. SUBJECT AND METHODS

2.1. EXPERIMENTAL MATERIAL AND PROCEDURE

Two types of cretaceous sandstone of Hořice and Božanov, which have often been used in the Czech Republic as a building and sculpture material, were tested during this experiment. The cube samples (5 x 5 x 5 cm) were treated by an exposition in a climatic chamber. The treatment scheme was based on the meteorological data measured in Prague during winter periods in years 1999 - 2008. According to the statistical analysis, the treatment simulation program containing 56 freeze/thaw cycles in the temperature range of – 14 °C to 14 °C was developed. The weathering simulation program was divided into four stages, each consisting of 14 freeze/thaw cycles. The sandstone samples were soaked for 24 hours in distilled water and in a 2.5 % solution of NaCl before each stage. Each cycle lasted seven hours and the samples were maintained at minimal and maximal temperatures for two hours. To evaluate the level of deterioration process, the changes in the internal structure were determined before and after climatic treatment using the methods of Hg porosimetry and X-ray microtomography. The size of sandstone samples has to be changed to enable the maximum accuracy and reliability of measurement by both characterization methods (see chapters 2.2 and 2.3). The porosity was estimated in subsurface layer to the depth of 1 cm in both methods of porosity determination. This distance was chosen because the weathering processes cause the greatest changes first close to the surface (e.g. Winkler, 1997).

Fluid invading techniques as Hg porosimetry only investigate the open porosity whereas the X-ray microtomography shows the total porosity which is the sum of open porosity and closed porosity. The porosity of sample is defined as the ratio between the sample void volume and its external volume and is usually expressed as a percentage (%).

2.2. MERCURY POROSIMETRY

The method of mercury porosimetry is based on the phenomenon of capillary depression of mercury, where the wetting angle is > 90° and thus the mercury penetrates pores only by pressure applied. The volume of mercury intruded into the pores system is generally interpreted as a total pore volume within a sample measured, whereas the relationship between the actual pressure P and cylindrical pore radius r is given by Washburn equation (Drake, 1949):

$$P = -\frac{2\sigma\cos\varphi}{r} \quad (1)$$

where σ = surface tension of liquid and φ = wetting angle.

Dependence of the intruded volume of mercury on increasing pressure is expressed by the intrusion curve. The cumulative curve is obtained by converting the pressure P to the radius r according to the Washburn equation, from which pore radii distribution is calculated according to gradual derivation. The extrusion curve records the dependence of mercury volume on decreasing pressure. The shape and relative position of both curves are characteristic for a specific shape of the pores (Lowell et al., 2006). Maximum applied pressure determines the smallest measured radius and the largest radius corresponds to the initial pressure. According to the standard distribution of pores based on their radius as micropores (r < 1 nm), mesopores (r = 1 nm to 25 nm), macropores (r = 25 nm to 7500 nm) and coarse pores (r > 7500 nm) - mercury porosimetry can be used to identify a specific volume of meso-, macro-and coarse pores (IUPAC, 1973).

The total cumulative volume of pores, volumes of meso-, macro-, and coarse pores, and the total porosity were determined using coupled Pascal 140 + 240 porosimeters by Thermo Electron - Porotec. The Pascal 140 porosimeter was utilized for low-pressure measurements below 100 kPa. The Pascal 240 porosimeter works within the pressure range from 0.1 to 200 MPa. Using the pressure range 0.1 kPa to 200 MPa, pores with equivalent radii ranging from 3.7 nm to 58 μm can be detected. The samples were evacuated at 378 K for 2 h prior to analysis, and were then evacuated in the instrument until a stable pressure was reached. A contact angle of 140°and a value of 480.10^{-3} $N.m^{-1}$ for the Hg/air interfacial tension were used in the Washburn equation to determine the textural parameters. Each sandstone cube was resamples and two approximately 5 x 5 x 5 mm sized particles took from the depth 1 cm were measured. The coefficients of variation for measurement of cumulative volume were less than 7 %. The used values represent arithmetic average of the two measurements.

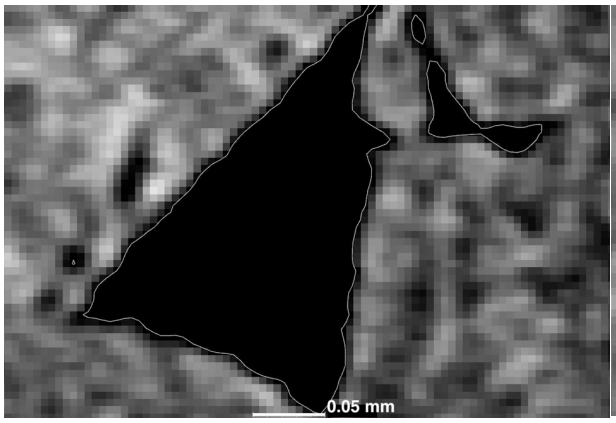

Fig. 1 Boundary determination (gray line) by automatic program algorithm VGStudio MAX 2.1 from unmodified data after 3D reconstruction.

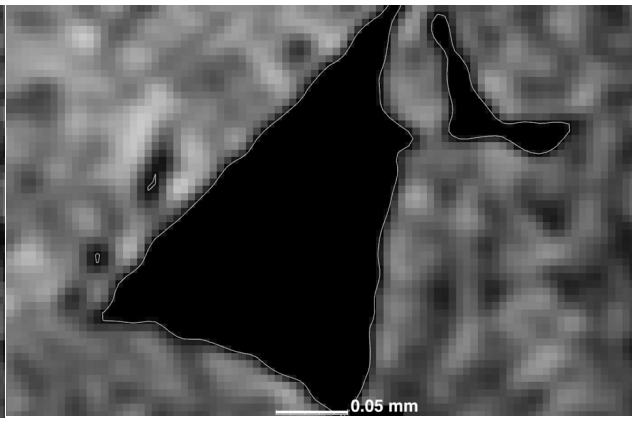

Fig. 2 Manual determination with the optimal settings of boundary (gray line).

2.3. X-RAY MICROTOMOGRAPHY

The method of X-ray computed microtomography (hereafter microCT) belongs to non-destructive tests method (except sample preparation), which was used to obtain the information about open and closed porosity as well. MicroCT is a characterization method based on the investigation of internal sample structure by image analysis (e.g. Moreira et al., 2010; Appoloni et al., 2007). MicroCT is composed of two basic steps - acquisition of projections and volume reconstruction. In the phase of the acquisition the object is turned around the rotation axis at a small selected angle and so called X-ray projections are measured. The absorption of X-ray beams as they pass through a material is a logarithmic function of the absorptivity of the material, and the distance through which the light must travel (so called Beer's law). The absorption can be than calculated according to following formula (Landis and Keane, 2010):

$$\tau = \ln\left(\frac{I_0}{I}\right) \tag{2}$$

where τ = total X-ray absorption along the ray path, I_0 = intensity in front of the studied object and I = intensity behind the studied object.

Projections are X-ray attenuation images, which are created on two-dimensional detector after the beam transmission through the object. To accomplish the measurement, the sample can be rotated at 180º or 360°, which was in our case. During this one turn the selected number of projections is captured. The three dimensional image of the object is reconstructed after obtaining all projections, when two-dimensional detector is used as in our case (Hain et al., 2011). This method enables to create 3-D video and therefore the detailed visualization of pore space is possible.

Small cylinder's samples with the diameter 1 cm and the height 1.5 cm were prepared from untreated and treated sandstone's cubes. Samples were measured by microCT phoenix|x-ray nanoton180 with 5 Mpx 2D detector at 90 kV, 100 μA with timing 2 s and 2880 projections. Voxel size was 5 μm (Kovářová et al., 2011). This experimental arrangement enables to evaluate pores with diameter d > 5 μm.

The porosity consisting of pores with a diameter d > 5 μm (coarse pores and portion of macropores) was calculated as the arithmetic average of five values, which were determined by various ways of setting grain/pore boundary after 3D reconstruction: i) by automatic program algorithm VGStudio MAX 2.1 from unmodified data (Fig. 1); ii) by automatic program algorithm VGStudio MAX 2.1 after convolution of an image by Gaussian filter to remove noise from untreated data; iii) manually with the optimal settings of boundary after convolution of an image by Gaussian filter (Fig. 2); iv) manually using the extreme position minimizing the total porosity after convolution of an image by Gaussian filter; v) manually using the extreme position maximizing the total porosity after convolution of an image by Gaussian filter.

3. RESULTS

3.1. MERCURY POROSIMETRY

As showed the results of mercury porosimetry (see Table 1), the porosity of Božanov and Hořice sandstone decreased in both cases of treatment - freeze/thaw cycles with distilled water and with NaCl solution. The change was more significant after the treatment with NaCl in both sandstones.

The results in Božanov sandstone showed increase in the portion of pores d > 5 μm calculated according to their specific volume in both cases of treatment - by 6.14 % after freeze/thaw cycles with distilled water and by 7.43 % with NaCl. The porosity of this portion of pores is 12.69 % by untreated samples and decreased after the both ways of treatment - by 8.30 % after freeze/thaw cycles with distilled water and by 11.60 % with NaCl.

Table 1 The influence of weathering simulation program on the porosity change according to Hg porosimetry.

Parameter	Porosity		Portion of pores d > 5 μm (%)		Portion of pores d > 5 μm	
Sample	Božanov	Hořice	Božanov	Hořice	Božanov	Hořice
untreated samples	16.59	23.42	12.69	14.89	76.49	63.60
F/T cycles + H_2O	14.33	21.46	11.64	9.60	81.19	44.75
F/T cycles + NaCl solution	13.65	19.37	11.22	8.59	82.17	44.32

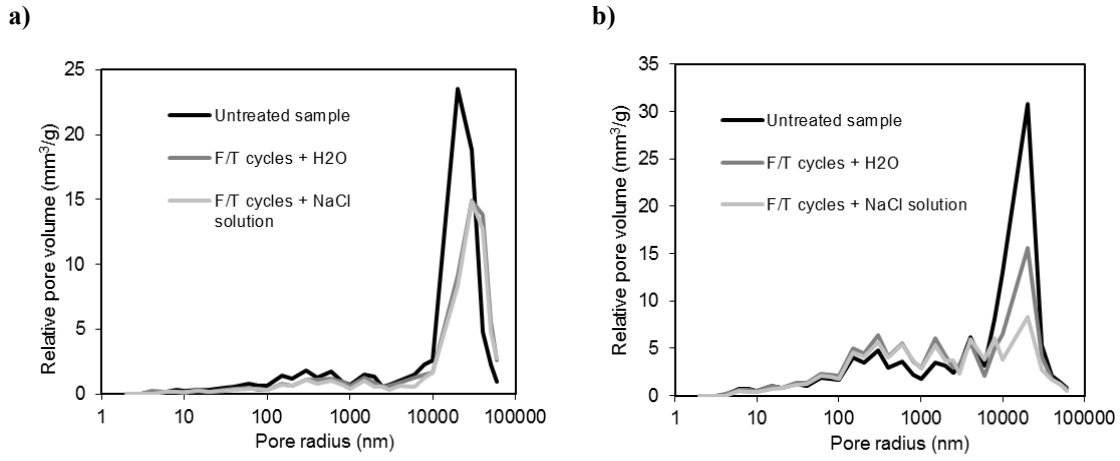

Fig. 3 Distribution of pores a) Božanov sample, b) Hořice sample.

Table 2 Changes of pore size distribution after weathering simulation.

		Total cumulative volume (mm^3/g)	Specific volume (mm^3/g)			
			Mesopores	Macropores		Coarse pores
				d < 5 μm	d > 5 μm	
untreated samples	Božanov	75.20	2.12	15.55	5.46	52.07
	Hořice	116.58	5.56	36.89	20.39	53.74
F/T cycles + H_2O	Božanov	63.20	2.02	9.86	4.03	47.29
	Hořice	104.05	6.79	50.71	16.72	29.85
F/T cycles + NaCl solution	Božanov	59.68	1.58	9.06	3.48	45.56
	Hořice	92.74	5.29	46.26	19.69	21.50

The Hořice sandstone has generally another pore size distribution. The portion of pores d > 5 μm is lower than in the Božanov sandstone and significantly decreased after both treatments - by 29.64 % after freeze/thaw cycles with distilled water and by 30.31 % with NaCl. The porosity of this portion of pores decreased after the both treatments - by 35.50 % after freeze/thaw cycles with distilled water and by 42.28 % with NaCl. This decrease is more significant than in the Božanov sandstone as well. The distribution of pores according to their radius of Božanov and Hořice sandstone is presented in Figure 3 and Table 2. The specific volume of pores was determined before and after the treatment in climatic chamber.

The total cumulative volume decreased in both sandstones after both ways of climatic treatment. The results show by Božanov sandstone, that the pore

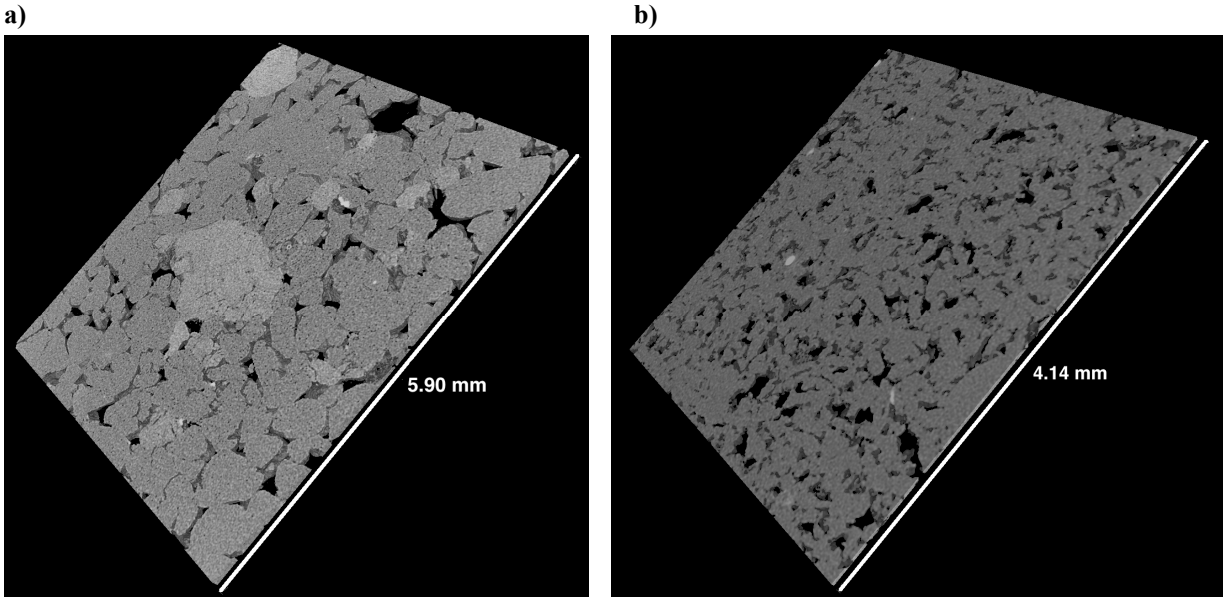

Fig. 4 The 3-D reconstruction of untreated Božanov (a) and Hořice (b) sandstones.

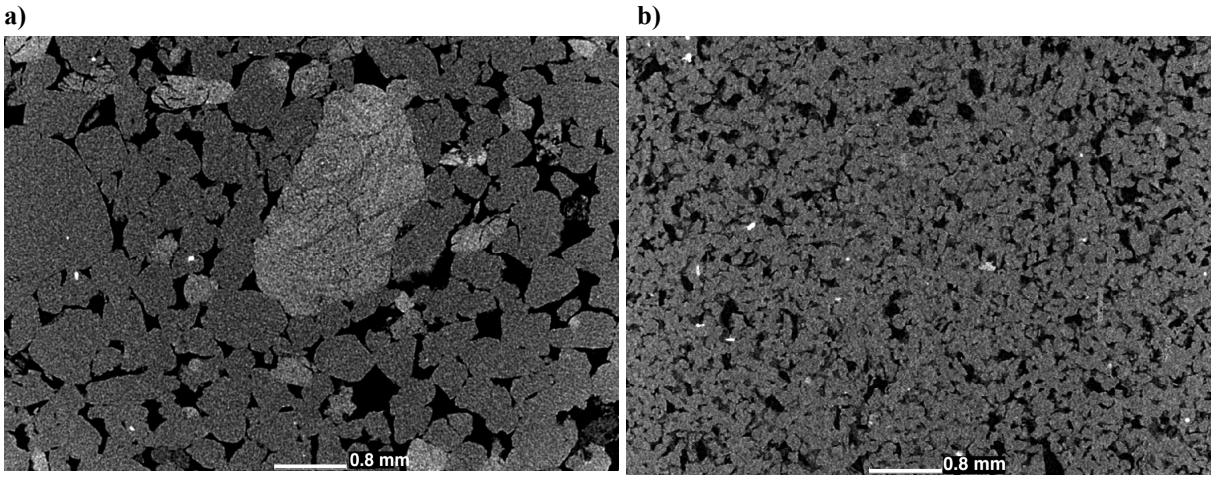

Fig. 5 The cross-section of untreated Božanov (a) and Hořice (b) sandstones.

specific volume of each size range decreased and the changes after the treatment with NaCl were more significant. The biggest changes were determined in the specific volume of macropores. On the other hand, pore specific volume of mesopores and macropores in Hořice sandstone increased after the treatment with H_2O whereas the specific volume of mesopores decreased after the treatment with NaCl. The specific volume of coarse pores and macropores d > 5 μm decreased in both ways of treatment.

3.2. X-RAY MICROTOMOGRAPHY

The amount, arrangement and shape of macropores and coarse pores (in black) are clearly apparent in the following tomography images (Figs. 4 and 5). The intergranular space is characterized by presence of heterogeneous pores by both types of sandstones. Hořice sandstone is more fine-grained and homogeneous in grain size than Božanov sandstone. The 3-D reconstruction of untreated Božanov and Hořice sandstone is shown in Figure 4 and the cross-section of the same samples are shown in Figure 5. The difference in the pore space is shown in Figure 6.

Using the data of Hg porosimetry and microCT, it is possible to calculate the residual porosity of pores d > 5 μm. The residual porosity is consisting of closed pores. The residual porosity is calculated according to the following formula:

$$\Phi_R = \Phi_{CT} - \Phi_{Hg} \tag{3}$$

where Φ_{CT} = total porosity determined using X-ray microtomography and Φ_{Hg} = porosity of pores d > 5 μm determined using Hg porosimetry.

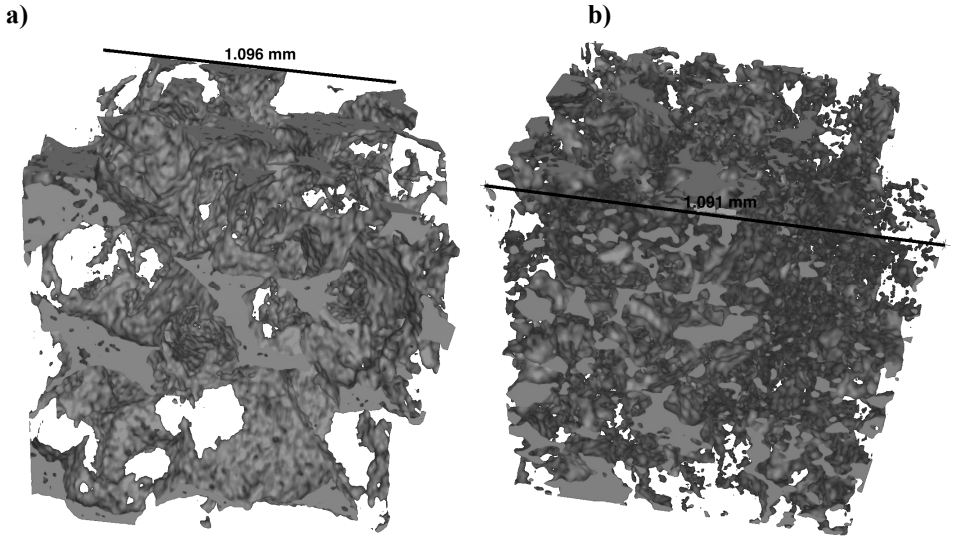

Fig. 6 The 3-D reconstruction of pore space (in gray) of Božanov (a) and Hořice (b) sandstone after the treatment with NaCl.

Table 3 The influence of weathering simulation program on the total and residual porosity changes using microCT.

Parameter	Total porosity of pores d > 5 μm (%)		Residual porosity of pores d > 5 μm (%)	
Sample	Božanov	Hořice	Božanov	Hořice
untreated samples	16.00	22.00	3.31	7.11
F/T cycles + H_2O	14.75	15.29	3.11	5.69
F/T cycles + NaCl solution	16.78	17.83	5.56	9.24

The total porosity was obtained by both types of sandstone samples using the microCT. The results are presented in the following Table 3.

The total porosity of pores d > 5 μm in Božanov sandstone decreased by 7.81 %, after freeze/thaw cycles with distilled water but increased by 4.85 % after freeze/thaw cycles with NaCl. The residual porosity decreased by 6.04 % in the first treatment and increased by 67.99 % in the second treatment.

Results of Hořice sandstone show the decrease of total porosity of pores d > 5 μm by 30.49 %, after freeze/thaw cycles with distilled water and by 18.95 % after freeze/thaw cycles with NaCl. The residual porosity decreased by 19.97 % after the treatment with distilled water and increased by 29.96 % after the treatment with NaCl solution.

4. DISCUSSION

The ostensibly non-comparable results are caused due to limitations of both methods. Cnudde et al. (2004) demonstrates that mercury porosimetry generally gives higher total porosity values than the microCT. This phenomenon could be caused due to the different resolution and noise of microCT equipment and used software, which allowed the detection of pores d > 10 μm. In our case the detection limit was 5 μm and therefore our results are more accurate. It is not possible to consider the results of both techniques without their recalculating for the same spatial resolution. The other source of incomparability and depreciation, if the same spatial resolution is considered, can be caused due to the character of studied rock material. If the studied sample contains such a material, which weakly absorbs X-ray beams, then the overall results given by microCT may be distorted. The character of internal structure can affect the results of microCT as well. If the sandstone sample contains high amount of large grains and small amount of large pores (it means pores with diameter larger than the detection limit of microCT), the results may be distorted too. In the case of both methods the size of rock samples is unfortunately still incomparable. For the purposes of Hg porosimetry the samples are several folds smaller and therefore the fault in results may be caused due to inhomogeneity within the rock sample.

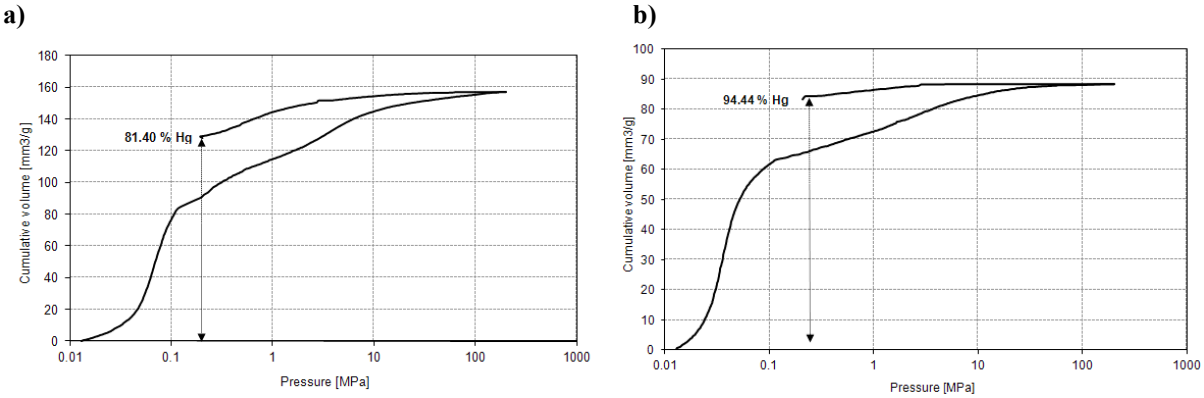

Fig. 7 Mercury intrusion/extrusion cycle a) Božanov - untreated sample, b) Hořice – untreated sample (dashed line – the trapped amount of mercury in the sample at pressure 0.1 MPa).

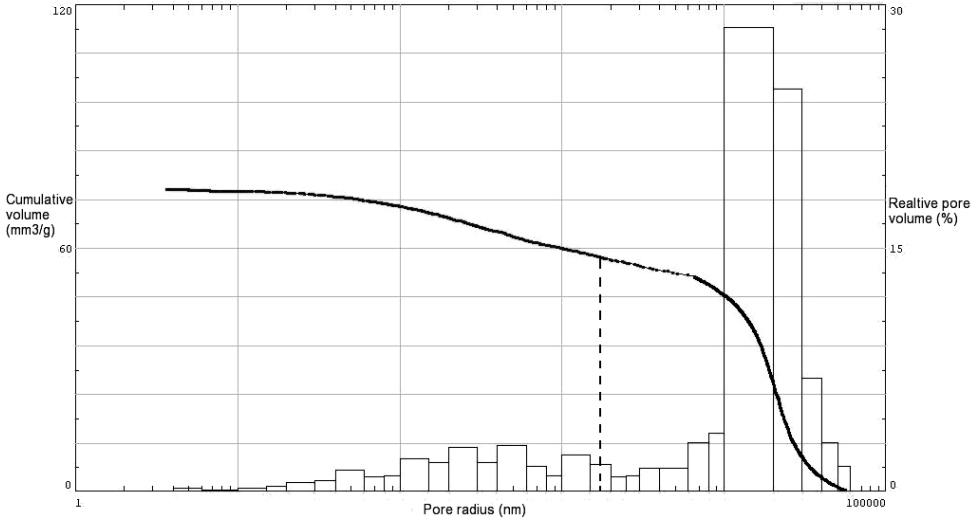

Fig. 8 Pore size distribution – untreated sample Božanov (dashed line – boundary of pores d > 5 μm).

The different values of percentage of pores d > 5 μm obtained by Hg porosimetry (see Table 1) compared with the results of microCT (see Table 3) are mainly caused due to the characteristic shape and connection of the pores in the sandstone. While the total porosity according to Hg porosimetry represents correct value determined on the basis of the measuring of total open pore volume, the determination of porosity in the pore area with d > 5 μm is dependent on the evaluation of pore distribution by their size.

The mercury porosimetry works with model pore shapes, and the determination of distribution of pores according to Washburn was derived for cylindrical pores, which is characterized by a reversible process of the intrusion and extrusion of mercury. If a certain amount of mercury remains captured in the sample after a total reduction of pressure, the extrusion curve lies markedly over the intrusion one, within the whole pressure range. This indicates the so-called structural hysteresis, which is typical for ink-bottle pores. The ink-bottle pores cause, that the true pore size distribution measurement is distorted due to detection of the smaller diameter of the throat entrance of pore and thus the whole volume of mercury behaves as if it was pore with the size of throats diameter (e.g. Cnudde et al., 2004; Fitzner, 1988). The greater the ratio of pores size (respectively intergranular spaces) to the size of entrance throats, the more mercury remains captured in the sample (Wardlaw and McKellar, 1981). This phenomenon is apparent in the following graphs (Fig. 7) where the porosimetric curves show the amount of residual mercury in the samples a) coarse grained Božanov sandstone - untreated sample and b) fine-grained Hořice sandstone - untreated sample. It is obvious that in the coarse-grained sandstone remains more mercury captured than in the fine-grained sandstone after pressure release.

An example of pore size distribution, with marked border of 5 μm, in its entirety measured by Hg porosimetry is shown in Figure 8. It is evident that the pores of d < 5 μm can also create entrance throats

into larger cavities. In this case, the volume of pores d > 5 μm would be underestimated. The use of microCT enables to measure the total porosity including the closed pores and also the pores, which could be incorrectly included among pores with smaller diameters due to this underestimation, i.e. due to the "ink bottle" effect.

Use of the microCT clarified the non-typical change of porosity (measured by Hg porosimetry) with increasing number of freeze/thaw cycles. According to Fitzner (1988) the total porosity and pore radius increase depending on the number of freeze/thaw cycles and the pore size distribution changes toward coarser pore size, especially the porosity increases in pore radius categories d > 10 μm. The total porosity and the specific volume of pores d > 5 μm measured by Hg porosimetry decreased after both ways of treatment of both types of sandstone in our experiment (see Table 1 and 2). Thomachot and Jeannette (2005) point out that the different pore structure by two types of sandstone can cause the different response to frost action. In their experiment the Hg total porosity increased after freeze/thaw cycles by used types of sandstones and the volume of coarse pores increased too. Our results are contrary to previous claims. The explanation of our non-typical changes is the change of the residual porosity. For example, in the case of treatment with NaCl, the residual porosity increases. Closing of the pore entrance throats, induced by the salt crystallization pressure, can cause this increase. On the other hand, the residual porosity decreased after the treatment with H_2O. This can be accompanied by the increase of residual porosity under 5 μm, what we are not able to detect using the microCT, or the used treatment scheme causes opposite changes in porosity, which implies the important role of selected treatment scheme. Our experiment confirmed the different evolution of porosity by both types of sandstone depending on different initial properties of pore structure.

The microCT enabled to determine the residual porosity and confirmed that the changes in the internal structure depending on cyclic freezing caused the origin or destruction of closed pores. Generally, the results of the microCT show that only the use of Hg porosimetry does not give satisfactory information. Unfortunatelly, in this case, the microCT does not provide the information about pores with the diameter smaller than 5 μm but we can assume that similar changes of internal structure also occur in the remaining pore size ranges, which may explain the atypical changes of total porosity measured by Hg porosimetry.

Supporting physico-chemical analysis as X-ray diffraction and DTA analysis did not show the presence of any newly formed phases in pore space so it is obvious that the changes of porosity and of pore space properties are the result of frost and salt action.

5. CONCLUSIONS

To sum up, the mercury porosimetry is a well known method allowing the monitoring of changes of porosity and pore size distribution after the weathering simulation tests. This method is limited by some factors - "ink-bottle" effect and the possibility to determine only effective porosity which depends on the kind of pycnometric medium. These factors can distort the results and can lead to the distorted conclusions. The use of microCT for determination of pore space properties is relatively new but its development goes ahead. This method is also useful for monitoring of developed changes although it only provides the information about portion of macropores and coarse pores. The 3D reconstruction, and thus the possibility to look into the internal structure, has certainly a great future. The microCT was first used for description and determination of pore space by two widely used Czech sandstones during this experiment. The obtained results provide a brand new more complex "view" into the internal structure of these stones.

Both methods are limited by the sample size and consequently by the explanatory power. The mercury porosimetry is a destructive method thanks to the capture of mercury inside of stone samples whereas the microCT is in that point of view a non-destructive method, so the samples are reusable at any conditions. In conclusion, the microCT seems to be useful as a complementary method in many research areas of rock material characterization, not only in the branch of stone weathering.

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