MATERIALS AND TECHNOLOGIES FOR THE CONSOLIDATION OF HISTORIC PLASTERS BY THE WETTING AND SURFACE PENETRATION METHOD

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ABSTRACT

The term of a historic plaster or surface coating, respectively, denotes a layer or a multilayer of materials firmly bonded to the substrate, whose structural and technical condition significantly affects not only the aesthetic effect, but mainly the service life of this surfacing. Due to the permanent exposure of plaster surfaces to the external environment (in the exterior as well as the interior), they belong to the group of the most exposed parts of historic and heritage buildings, often exhibiting a high degree of degradation and disintegration. The consolidation and rehabilitation of plasters with, among other things, a degraded binder component, damaged structure and insufficient adhesion of individual layers to each other and to the masonry substrate is a challenging task in the restoration of historic buildings. The paper presents the alternative of strengthening degraded surface layers by means of nanomaterials based on calcium hydroxide applied by the surface penetration method.

KEYWORDS

Plaster, Calcium hydroxide, Consolidation, Nanosuspension, Penetration

INTRODUCTION

The most common defects and failures of plasters are characterized mainly by changes in their structure, by overall disintegration, reduced cohesion of surface layers and their spalling from the substrate, the formation of bumps, occurrence of cracks and a change, or a loss of the colour of surface coatings. In the final phase of degradation, the plaster no longer fulfils its function and falls off. Depending on the manifestations of degradation, a suitable rehabilitation method can be chosen to observe the heritage conservation requirements, particularly in terms of preserving the value of the heritage building and its surface coating. The basic objective of the consolidation of surface coatings is the improvement of the mechanical properties of materials, strengthening of subsurface and surface layers and their return to a state close to the original properties of the treated material. Achieving this goal is one of the most challenging tasks in the restoration of historical heritage [1]. The principle of plaster renewal is based on adding the weathered and degraded binder to the structure of the treated material, or attaching the separated parts to the substrate (or to each other) and filling in the missing parts (cracks, cavities, etc.). The essential part of this process is, in particular, structural reinforcement, which is achieved with the help of appropriately selected consolidating agents based on the analysis of the treated material and the

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causes of its poor structural and technical condition. The role of the consolidating agent is to stabilize the damaged plaster system or replace the "leached" original binder material, ideally with a material of identical properties [2].

The improvement of adhesion between the surface coating and masonry, the strengthening of surface and subsurface layers, i.e. surface grouting, is currently most often performed by repeated impregnation (spraying, painting), by methods based on the capillary activity (wetting, sprinkling, capillary elevation) or microinjection of damaged parts [3] . In most cases, it is advisable to combine individual consolidation methods with each other to achieve the best results possible (e.g. microinjection and spraying, etc.). Among the commonly used consolidating agents, there are, above all, inorganic materials (silicic acid esters, aqueous SiO2 dispersions). Their use is limited by the moisture content in the plaster (in the case of organosilicon esters), or salts (in the case of a colloidal silica solution) [4]. In the last decades, the number of lime water applications, in particular, has increased, which, however, is associated with a series of problems [5]. In the case of using lime water, which has only a low lime component content in the injected agent (CaCO3 solubility in water is ca 1.6 g/l at a water temperature of 20°C), the application must be repeated in tens of cycles (120 to 180 cycles), which results in a significant long-term wetting of the structure and the activation of salts in the reinforced substrate.

In the last 20 years, the possibilities of using disperse and colloidal systems of not only organic consolidants, but also mineral systems with particle sizes in the nanoscale have been verified in the cultural heritage conservation. The use of nanomaterials with particle sizes of up to 1μm, whose properties are somewhere at the intersection between the properties at the level of molecules and matter [10, 11], is the subject of extensive scientific research due to their positive physical and mechanical properties such as high chemical performance, excellent plastic, consolidation and diffusion properties, cleaning capabilities and more. The tested nanomaterials mainly include nanodispersions based on hydroxides, carbonates, or sulphates [12, 13, 14].

In the case of hydroxide-based nanodispersions, there are products commercially available of mostly German and Italian origin (e.g. CaLoSiL®, or NanoRestore®) or SiO2-based colloids (e.g. Syton X30, Tosil).

The lime nanosuspension is composed of calcium hydroxide nanoparticles dispersed in an alcoholic medium, where individual sorts differ from each other in concentrations and the type of alcohol. The curing of this material results, in a simplified way, in carbonation where calcium hydroxide reacts with atmospheric carbon dioxide to produce calcium carbonate, like in fresh plaster [15]. Due to the deposition of calcium carbonate in the degraded material, the bonds are re-strengthened and the material reinforced.

Nanosuspensions can be diluted to a concentration of 5 to 50 g/litre, where the alcohol will evaporate completely after the application. The degree of penetration is significantly affected by the particle size, where in the case of lime nanodispersions we can achieve penetration rates to such depths where common lime suspensions (prepared from slaked lime) do not penetrate, thanks to the low particle size with a large specific surface area [16, 17]. The penetration depth can be affected either by adding a small amount of other solvents such as acetone or pentane, or by choosing a suitable application technique. The positive properties of these materials include excellent water vapour permeability, the absence of chemical changes or shiny effects and the absence of the formation of surface hydrophilic films.

MATERIALS AND METHODS

The strengthening and stabilization method of historic plasters by overall surface pressure grouting (penetration) and wetting is based on an additional mechanical supply of Ca2+-based nanomaterials to the structure of materials and the activation of unreacted Ca2+-based substances contained in the structure of grouted materials, which increases their strength, cohesion or adhesion to the substrate.

Before performing the overall surface pressure grouting (penetration) and wetting of historic plasters, the edges of cracks or cavity walls must be cleaned of dirt, dust, oils, grease and other particles that reduce adhesion, and then they must be filled with adequate lime mortar and levelled on the surface.

In overall surface pressure grouting, the grouting solution is "pushed" into the pore (injectable) plaster system (surface coating) so that the required gradual saturation of the pores of the respective layers occurs. The main advantage of this method is its high efficiency and uniformity in terms of strengthening the plaster and increasing its adhesion to the substrate.

During wetting (mechanical application), the grouting solution penetrates through the capillary phenomena into the pore system and thus into the plaster structure. The grout propagation speed and the penetration depth depend on the grout properties, the number of wetting repetitions, the type of pore system [18, 19], but also on the moisture and salt content in the grouted material.

Grouting mixtures and agents for the wetting and surface penetration method

The choice of consolidating agents must fully respect the requirements for application to historic or heritage masonry and its surface coating. For overall surface pressure grouting (penetration) and wetting, calcium hydroxide-based grouting agents were prepared, which have a high penetration and distribution (uniform) ability, material and chemical compatibility and adequate consolidation ability (to avoid local over-reinforcement of the treated substrate compared to untreated material - the strength of the treated material should not increase by more than 10 - 20%).

The grouting agents based on calcium hydroxide nanoparticles were developed within the NAKI II DG16P02M055 research project in the researchers' workplace cooperation with the Centre of Polymer Systems (Tomas Bata University in Zlin):

- **Ca4O (V5)** The test portion of calcium oxide CaO was dispersed in ethanol and distilled water was added. The reaction was complete after 4 hours. The suspension was successively made up to a volume of 2 litres. The $Ca(OH)_2$ concentration was Ca 7 g/l. The prepared nanosuspension contains a lot of sliced hexagonal $Ca(OH)_2$ crystals with a different size distribution. According to DLS, 2 peaks are visible – one with a maximum of 4813 nm, which is probably caused by large agglomerates of $Ca(OH)_2$ particles, and then the peak with a maximum of 1667 nm, which, in our case, corresponds to the size of (separated) $Ca(OH)_2$ particles in the solution. Consolidation, sealing of concrete and brick, stone and mixed masonry (Figure 1a);

- **CaMg1 (V7)** Test portions of $Ca(OAc)_2.H_2O$ and $Mg(OAc)_2.4H_2O$ were mixed and dispersed in isopropyl alcohol. Water was added to the suspension and precursors were dissolved. The NaOH (28.0 g in 700 ml H_2O) solution was added to the solution of Ca and Mg precursors and a white suspension was formed. The reaction was left in the mg mixer for 24 hours and then the white hydroxide precipitate was separated by centrifugation and washed with a mixture of water and isopropyl alcohol. After washing, the product was centrifuged and dispersed in 2 l of isopropyl alcohol. The resulting $Ca(OH)_2+Mg(OH)_2$ concentration is ca 7 g/l. Elemental (EDX) analysis confirmed the content of magnesium (8.5 wt%), calcium (37.8 wt%) and oxygen (48.4 wt%). Consolidation, sealing of concrete and brick, stone and mixed masonry (Figure 1b);

- **CaMg2 (V8)** Test portions of Ca and Mg were dispersed in iPrOH and water was added. The reaction was complete after 6 hours. The suspension was made up to a volume of 2 litres (by adding 0.5 l of iPrOH). The $Ca(OH)_2+Mg(OH)_2$ concentration is 6 g/l. Elemental (EDX) analysis

confirmed the content of magnesium (7.3 wt%), calcium (43.4 wt%) and oxygen (44.5 wt%) (Figure 1c);

- **Ca4O-Si (V9)** The test portion of CaO was dispersed in EtOH and water was added. Then, APTES (aminopropyltriethoxy silane) was added. The suspension thickened and EtOH was added. The total volume of the suspension is thus 2.8 litres, and the $Ca(OH)_2$ concentration is approximately 5 g/l. According to DLS, 2 peaks are visible - the first, more intense peak with a maximum of 792 nm can be assigned to $Ca(OH)_2$ particles. The peak with a maximum of 184 nm corresponds to alkoxysilane or silica structures formed after the addition of APTES to the reaction mixture. Elemental (EDX) analysis confirmed the content of silicon (11.5 wt%), calcium (39.3 wt%) and oxygen (33.7 wt%) (Figure 1d).

Fig. 1 - Ca(OH)2 particle size in nanolime suspensions was measured by the dynamic light scattering (DLS) method. The Malvern Zetasizer ZS device was used for the measurement: a) CaMg1; b) CaMg1; c) CaMg2; d) Ca4O-Si.

Verification of consolidation of historic plasters

The verification of injectability and selection of a suitable grouting mixture are based on experimental studies performed during the project research in 2018 and 2019, which included the verification of grouting technologies of historic masonry and its surface coating, masonry elements and binders with similar or different pore systems for selected materials (Figure 2).

Verification of consolidation of historic plasters by the wetting and surface penetration method

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The technological principle was verified by consolidation tests of historic plasters reinforced by overall surface pressure grouting - penetration and wetting, performed in-situ on historic lime, partially degraded plasters [20]. Prior to grouting, degraded plaster specimens were sampled and laboratory moisture and porosimetry measurements performed.

The grouts were (a) repeatedly uniformly pushed into historic plasters using a highly absorbent and compressible material applied with a gradually increasing compressive force induced by the front plate; (b) repeatedly applied with a brush (wetting method), (Figure 3).

In the case of both methods, the consolidating agent was applied in 3 to 5 cycles with a comparable resulting volume during the experimental verification. In both cases, the grout was applied in such a way to avoid its surface run-off (or minimize the run-off). Each subsequent application cycle was performed before the previously applied grout was completely dry.
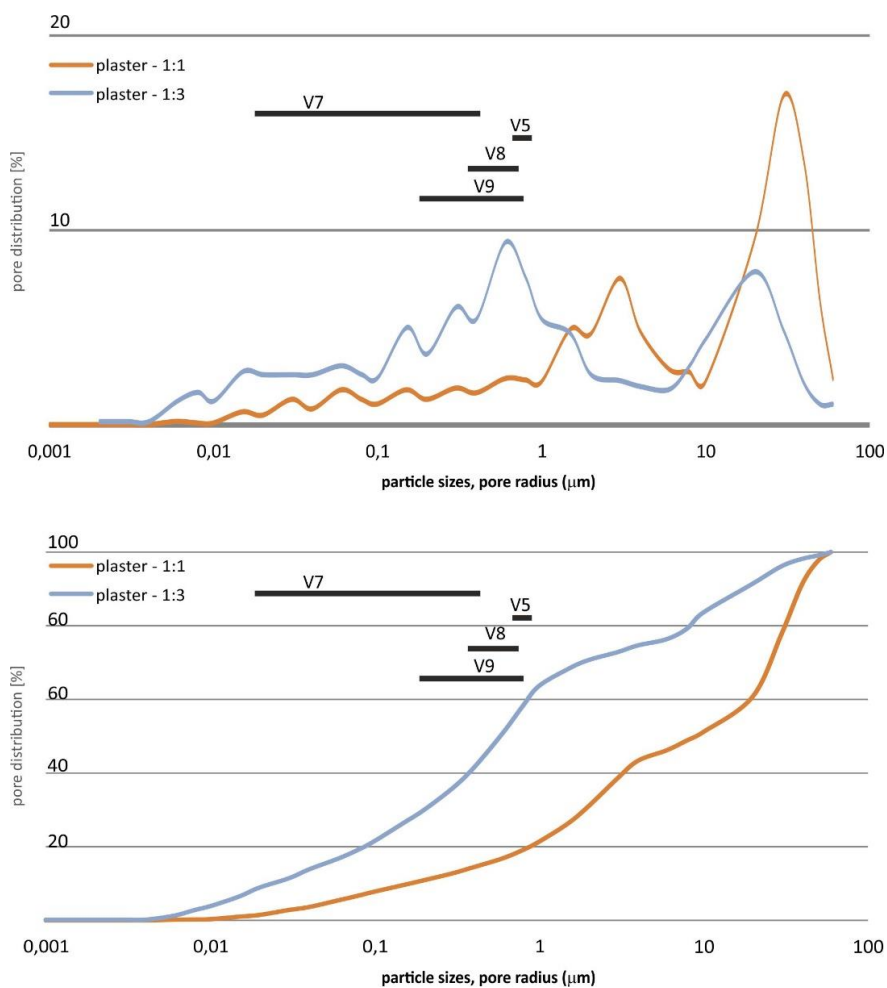

Fig. 2 - Comparison of pore radii in plaster (distribution and integral curves) and particle sizes of grouting agents based on nanolime (legend: plaster 1:1 and 1:3 – slaked lime-sand mixing ratio)

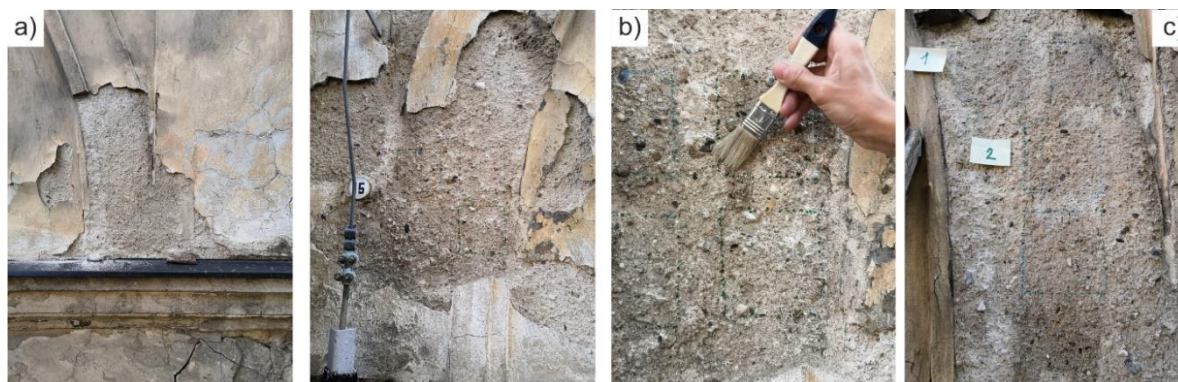

Fig. - 3 Documentation of a degraded historic plaster used for the verification of the degree of consolidation by means of prepared nanomaterials: a) selected points for consolidation and sampling; b) consolidation of plasters by wetting, c) structure with consolidated surfaces

Scotch Tape tear-off test

To verify the effectiveness of the consolidating agent or the consolidation method used, in-situ "Scotch Tape tests" were conducted [21, 22]. The tear-off test was performed on the surface of plasters (i.e. individual tested fields) before and after grouting. As part of the laboratory verification, the total porosity, distribution and integral curves describing the pore system were identified after the consolidation of plasters.

The in-situ measurement of the cohesion of surface and shallow subsurface layers (lime plaster on mixed masonry) was performed after 28 days of maturing. The sampling procedure for the Scotch Tape tear-off test was as follows: a marked adhesive tape 20 mm wide with a high adhesion capacity and softness was pressed against the tested surface and after approximately 60 seconds it was peeled off perpendicular to the surface and successively put in the transport case (Figure 4). The sampling from each spot was repeated 10 times to reduce the degree of bias in the results due to uneven material moisture contents, weathering, surface roughness and the application method used (degree of pushing and peel-off speed and angle). Each strip with torn-off material was weighed and the weighted average was calculated after weighing.

The sampling was performed at a temperature of 20.5 ° C and a relative humidity of 49%.

The amount of separated material is indicative and only serves to compare the values before and after consolidation. The measurement results show some variance in the measured values and thus also the resulting characteristics, which is probably caused by some deviations in plaster properties at individual sampling points, by local moisture contents of the material, inhomogeneity of the plaster, roughness or the degree of surface degradation and the execution method, in particular the pushing and tear-off speed and angle.

The results of Scotch Tape tear-off tests are presented in Table 1.

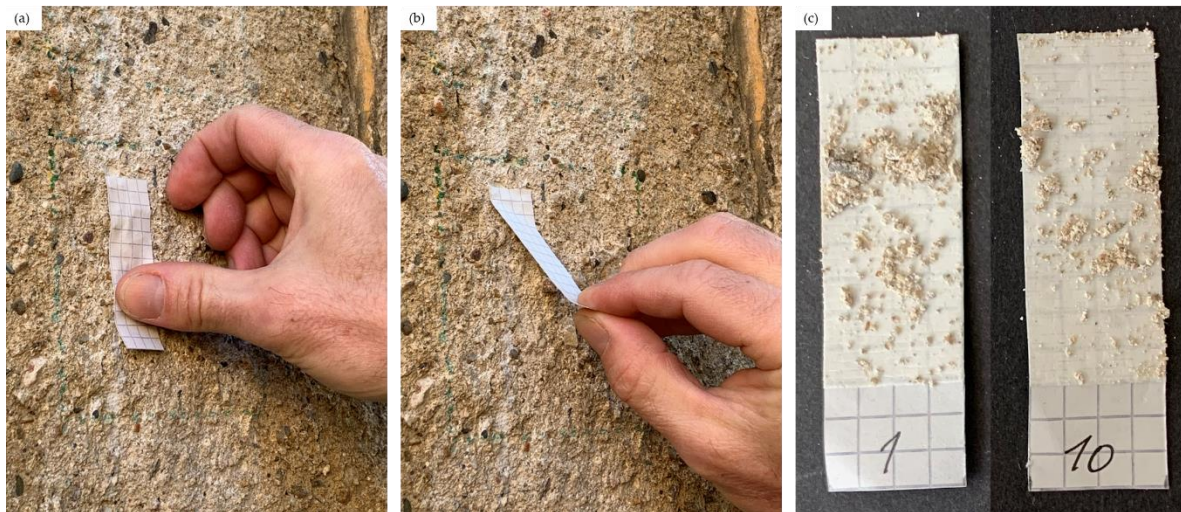--

Fig. 4 - Scotch Tape test performed 28 days after the application of grouts: a) adhesive tape pressing against the tested surface; b) test tape peel-off by smooth movement; c) comparison of the first and the last tear-off tape of a specimen consolidated by pressure penetration of Ca4O-Si nanosuspension

Tab. 1 - Results of the Scotch Tape test in relation to the grouting technology used

specimen marking	grouting agent	grouting technology	Scotch Tape test mean (g)	Scotch Tape test 1st, 5th, 10th peel-off (g)
No. 1	Ca4O-Si	wetting	0. 4354	0. 4648
				0. 4005
				0. 3366
No. 2	Ca4O	wetting	0. 3164	0. 6467
				0. 3840
				0. 2724
No. 3	CaMg1	wetting	0. 4442	0. 3268
				0. 2780
				0. 3239
No. 4	CaMg2	wetting	0. 4358	0. 3368
				0. 2870
				0. 3241
No. 5	Ca4O-Si	pressure penetration	0. 3534	0. 4902
				0. 3188
				0. 4428
No. 6	Ca4O	pressure penetration	0. 3159	0. 4477
				0. 4872
				0. 6152
No. 7	CaMg1	pressure penetration	0. 4435	0. 5059
				0. 3886
				0. 4043
No. 8	CaMg2	pressure penetration	0. 5685	0. 9696
				0. 5299
				0. 4858
No. 9 REF	-	-	0. 4653	0. 6025
				0. 6182
				0. 3003

Determining changes in porosity

The pore distribution was determined by high-pressure mercury porosimetry on sampled plaster specimens. The measurements were performed on plaster specimens with a size of cca 5 mm (cca 1 cm^3), and two independent measurements were always made on each specimen in a set of Pascal 140 + 240 porosimeters manufactured by Thermo Electron - Porotec. The Pascal 140 porosimeter serves as a filling device and low-pressure measurements up to a pressure of 100 kPa are performed in it. The 240 porosimeter operates in a pressure range of 0.1 to 200 MPa. Before the analysis, the specimens were evacuated at a temperature of 90 °C and then they were evacuated to a constant pressure of less than 0.01 kPa in the device. The evaluation was performed in instrument software vers.1.05, using the surface stress value $\sigma = 480 \cdot 10\text{-}3$ N.m^{-1} and the contact angle value $\varphi = 140$o.

When a pressure interval of 0.1 kPa to 200 MPa is used, pores with radii from 3.7 nm to 58 μm can be indicated, which represents the identification of meso, macro and coarse pores due to the standard pore distribution according to their radius into micropores (r <1 nm), mesopores (r = 1 - 25 nm), macropores (r = 25 - 7 500nm) and coarse pores (r> 7 500 nm).

In terms of a change in total porosity, no significant changes in the total porosity of the samples were found in the majority of tested specimens. In specimens No. 2, 4 and 7, the total porosity identified was by ca 10% higher compared to the total porosity of the reference specimen. In specimen No. 6, there was a decrease in total porosity by ca 20% compared to the total porosity of the reference specimen. The results of changes in total porosity are presented in Tables 2 and 3 and in Figure 5.

Tab. 2 - Change in total porosity in relation to the grouting technology used

specimen marking	grouting agent	grouting technology	total porosity (%)
No. 1	Ca4O-Si	wetting	29.415
No. 2	Ca4O	wetting	31.91
No. 3	CaMg1	wetting	29.55
No. 4	CaMg2	wetting	32.785
No. 5	Ca4O-Si	pressure penetration	29.945
No. 6	Ca4O	pressure penetration	23.875
No. 7	CaMg1	pressure penetration	32.85
No. 8	CaMg2	pressure penetration	29.385
No. 9 REF	-	-	29.875

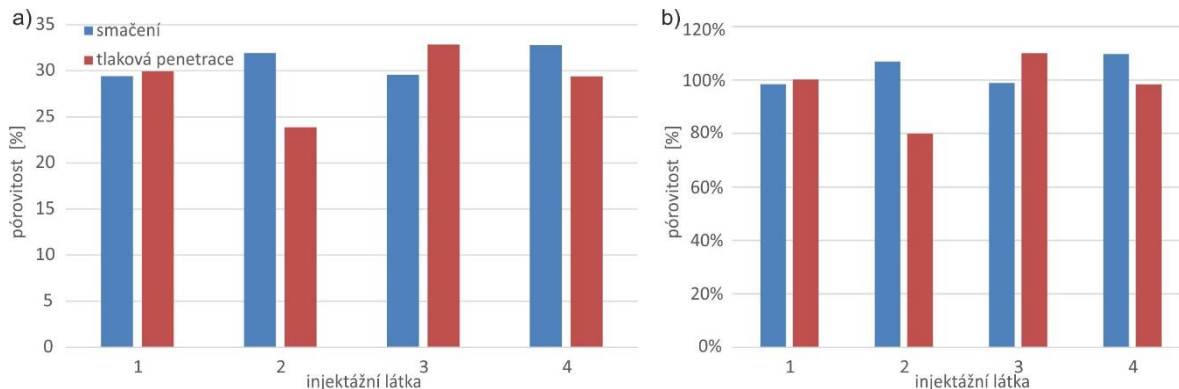

Fig. 5 - Total porosity of plaster (a) related to the reference specimen (b) in relation to grouting agents and grouting technology

In terms of changes in the pore distribution, the effect of grouting was manifested mainly in the domain of macropores and coarse pores compared to the pore distribution in the reference specimen. With the exception of specimen No. 7, the proportion of pores with a radius of 5000 to 100000 nm decreased by ca 6 to 32% compared to the reference specimen. The change in the proportion of pores with a radius of 700 to 5000 nm was more significant in the case of pressure penetration where there was a decrease in the proportion of these pores by up to 15% compared to the reference specimen. In the case of the wetting technology, the decrease in these pores was by ca 5% compared to the reference specimen. When both grouting technologies were used, the proportion of pores with a radius of 10 to 700 nm increased (by ca 5 to 15%) or decreased by ca 5% compared to the reference sample. The changes in the pore system distribution are shown in Figures 6, 7 and in Table 3.

Tab. 3 - Change in the pore distribution and total porosity

specimen marking	grouting agent	total porosity	pore radius [%]			pore radius [%]		
			10-700 nm	700-5000 nm	5000-100000 nm	10-700 nm	700-5000 nm	5000-100000 nm
		[%]	[%]	[%]	[%]	[%]	[%]	[%]
No. 1	Ca4O-Si	98.5	51.4	51.3	15.5	109.0	94.6	94.4
No 2	Ca4O	106.8	46.8	51.2	15.5	99.3	94.5	94.4
No. 3	CaMg1	98.9	52.6	54.4	10.9	111.5	100.4	66.7
No. 4	CaMg2	109.7	43.9	57.9	11.9	93.1	106.7	72.5
No. 5	Ca4O-Si	100.2	46.2	56.1	13.5	97.9	103.6	82.2
No. 6	Ca4O	79.9	49.6	54.4	12.1	105.3	100.4	73.7
No. 7	CaMg1	110.0	45.2	46.0	21.9	95.8	84.9	133.9
No. 8	CaMg2	98.4	54.3	49.5	12.8	115.2	91.3	78.4
No. 9 REF	-	100.0	47.2	54.2	16.4	100.0	100.0	100.0

Note: The total sum of the proportion of pore radii can be greater than 100 % due to overlapping intervals of pore radii.

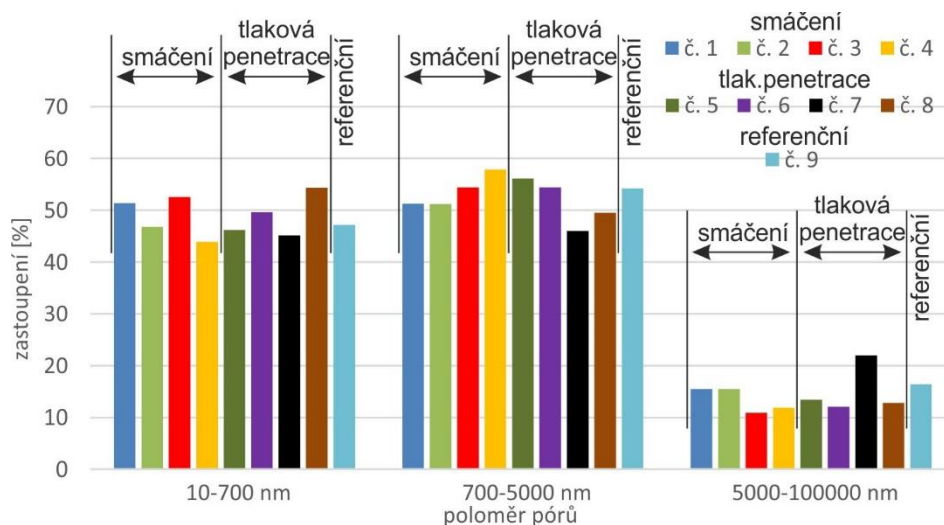

Fig. 6 - Changes in the pore distribution in selected intervals of pore radii

While comparing the effect of the particle size of grouting agents on changes in the pore distribution of historic plaster specimens, based on the analysis of the results of mercury porosimetry, we can conclude that there was a change – drop by 5 to 33% – in the proportion of pores with a radius of 5000 to 100000 nm in the majority of grouts. The changes in the proportion of pores with smaller radii were mostly manifested in the CaMg1 grouting agent applied by the pressure penetration technology. In all cases of a drop in the proportion of pores with a radius of 5000 to 100000 nm, there was an increase in the proportion of pores with radii of 10 to 700 nm and 700 to 5000 nm.

CONCLUSION

The visual evaluation of the colour of test surfaces after the application of nanususpensions indicates the deposition of lime particles and the undesirable formation of a whitish shade of the plaster. A significant deposition occurred mainly in the case of the Ca4O-Si and Ca4O nanosuspensions when applied by wetting. When applied by pressure penetration, the whitish surface deposits are minimal.

The possibility of removing the colour shade by the application of H2O (distilled water) was not part of the in-situ performed test in order not to affect the penetration depth and the degree of consolidation by the nanosuspension.

The highest degree of strengthening was achieved by the pressure penetration application in the case of CAMg2 nanosuspensions (by ca 50%), then Ca4O-Si (by ca 35%) and CaMg1 (by ca 20%). When applied by wetting (by painting) there was a partial strengthening of the shallow surface layer, but the overall degree of consolidation is insufficient.

Based on the analysis of changes in total porosity and pore distribution, it can be stated that the grout did not fully penetrate into the deeper pore structure of the plaster, and remained only in the surface layers. In the applied grouting technologies (wetting and surface penetration), the grouts mostly fill in the pores with a radius greater than 700nm. When pressure penetration was used, there was a more significant reduction in the proportion of pores with a radius of 5000 - 100000 nm compared to the wetting technology. Due to the changes in total porosity and pore distribution, the pressure penetration technology is more effective. Among the grouting agents

used, the most effective is Ca4O injected by pressure penetration due to changes in total porosity and pore distribution.
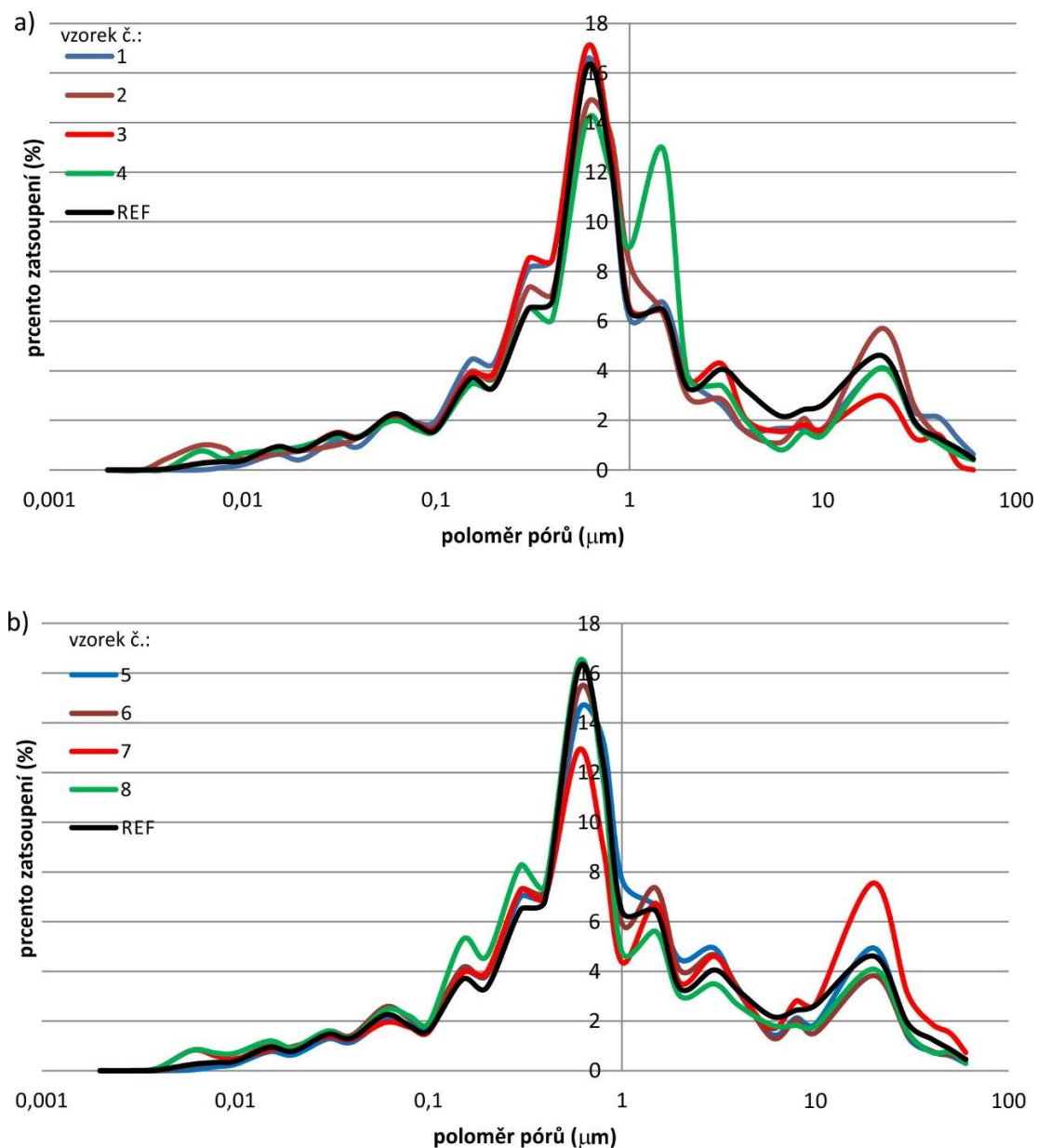

Fig. 7 - Distribution curves of plaster specimens grouted by wetting (a) and pressure penetration technology (b)

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