



# Microstructure modification of cement mortars: Effect on capillarity and frost-resistance



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## HIGHLIGHTS

- We presented tests on capillarity, frost-resistance and micro-structural parameters.
- Four cement mortars with modified microstructure were tested.
- Superplasticizer, silica fume and an air-entraining agent led to different porosity.
- Modifications resulted in changes in capillarity and frost-resistance of the mortars.
- It has significant influence on durability of composites of this kind.

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## ABSTRACT

The paper presents results of tests on capillarity, frost-resistance and micro-structural parameters of four cement mortars. Basic cement mortar, which was the reference recipe I, was modified “option by option”, becoming gradually recipes II, III and IV. In recipe II, a superplasticizer was used, causing a noticeable reduction of the  $w/c$  ratio. In recipe III, silica fume was applied. Whereas recipe IV contained an air-entraining agent. Firstly, an experiment on capillary rise in the tested mortars was carried out. The tests for all mortars I, II, III and IV lasted two months and the resulting data on capillary rise were used to determine sorption coefficients. Frost-resistance of each mortar was evaluated on the basis of their mechanical strength after 80 cycles of freezing–thawing of four sample series type I, II, III and IV. Structural parameters were measured using the Mercury Intrusion Porosimetry (MIP). According to the porosimetry test results, it was concluded that application of the selected admixtures and additives distinctly influences pore structures of the mortars. The capillarity experiment indicated that modifications made to the internal structure of the mortars resulted in a different course of the water uptake process. Application of a superplasticizer, silica fume and an air-entraining agent in tested mortars led to decrease of the water sorption coefficient characterizing the rate of capillary suction in the first few hours of contact between water and the material. Another effect of the use of air-entraining agent was revealed in a subsequent phase of the experiment in a significant water absorption. The freeze–thaw test showed that all the undertaken material modifications resulted in an absolute frost-resistance of the mortars II, III, IV after an established number of freeze–thaw cycles, whereas in the reference mortar I more than 60% decrease of compressive strength was noted.

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## 1. Introduction

The characteristic property of capillary-porous materials, including cement matrix composites, is their ability to absorb and transport moisture both in their liquid and gas phases. The primary phenomenon responsible for transport of liquid water in porous materials is capillary rising, which in practice contributes to the structural components exposed to moisture, for example, precipitation, or upon contact with ground water or flood water.

Due to the volume increase of the freezing water which can lead to arising of cracks, as well as a possibility of introducing into the material deleterious substances such as chloride and sulphate ions by the penetrating water, the parameters describing the capillarity largely determine durability of the material [1–5].

A material-wise factor determining the velocity and nature of migration of moisture is the porous structure of the body – the volume of pores, their shape, size, and continuity. In practice, the basic properties determined at quantitative assessment of material pore structure are: total porosity, effective porosity, specific surface area. Total porosity is defined as volume of pores included in volume unit of material or as volume of pores in mass unit of material and described by formula:

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$$p_o = \frac{V_p}{m}, \quad (1)$$

where  $p_o$  is the total porosity [ $\text{m}^3/\text{g}$ ],  $V_p$  is the volume of pores [ $\text{m}^3$ ],  $m$  is the mass of material sample [g].

The flow of liquids and gases appears only in open pores and therefore another quantity is often used to describe structure of materials:

$$p_e = \frac{V_{po}}{V_o}, \quad (2)$$

where  $p_e$  is the effective porosity [–] or [%],  $V_{po}$  the volume of open pores [ $\text{m}^3$ ],  $V_o$  is the volume of material [ $\text{m}^3$ ].

Specific surface area of material  $S$  is defined as total internal surface of pores in relation to a mass unit of the body. The quantity is of a relative character since it is determined assuming a spherical shape of the pores:

$$S = \frac{4\pi \sum_i r_i^2}{m}, \quad (3)$$

where  $S$  is the specific surface area [ $\text{m}^2/\text{g}$ ],  $r$  is the radius of a spherical pore [m],  $m$  is the mass of material [g].

Considering the fact that macro-, mezzo-, and micro-pores determine different material properties, participation of particular pores in overall porosity of the material is essential in practice. For description of pore size distribution, integral curve is applied as well as differential curve whose extremes enable to determine diameters of dominating pores.

Microstructural parameters of building materials can be most conventionally denoted by Mercury Intrusion Porosimetry (MIP) which consists in forcing mercury into pores at increasing pressure, assuming it does not moisten the material researched. Basing on the amount of liquid forced in at a given pressure, volume of pores of a given diameter is determined, which enables to define the distribution of pore size. Effective porosity is determined basing on the overall volume of mercury forced into the sample.

The relationship between the type of material, the liquid, the radius of the capillary and the value of the capillary suction  $p_k$  is described by the law of Laplace:

$$p_k = -\frac{2\sigma \cos \theta}{r}, \quad (4)$$

where  $\sigma$  is the surface tension of the liquid [N/m],  $\theta$  is the angle of contact [deg],  $r$  is the radius of the capillary [m].

Capillary force is balanced by the forces of friction, gravity and inertia. Neglecting the inertial force due to the low capillary flow velocity, an equation is obtained from the equilibrium condition [6]:

$$\begin{aligned} \frac{dx}{dt} &= \frac{1}{8\pi\eta x} \left( \frac{2\sigma \cos \theta}{r} \pi r^2 - \pi r^2 \rho g x \cos \beta \right) \\ &= \frac{r^2 \rho g}{8\eta} \left( \frac{2\sigma \cos \theta}{r \rho g x} - \cos \beta \right), \end{aligned} \quad (5)$$

where  $\rho$  is the water density [ $\text{kg}/\text{m}^3$ ],  $\eta$  is the coefficient of dynamic viscosity of water [ $\text{N s}/\text{m}^2$ ],  $g$  is the gravitational acceleration [ $\text{m}/\text{s}^2$ ],  $\beta$  is the angle of the capillary toward vertical [deg],  $x$  is the length of flow path [m],  $t$  is the duration time of the process of capillary rise [s].

In the case of horizontal flow ( $\beta = 90^\circ$ ) after transformations and integration, the following equation is obtained:

$$x = \sqrt{\frac{\sigma \cdot r \cos \theta}{2\eta}} \sqrt{t}. \quad (6)$$

It is a function, confirmed in many experiments, of a linear relationship between the water front position  $x$  in the capillary and the square root of time  $\sqrt{t}$ . Therefore, to describe the rate of this phenomenon the water penetration coefficient  $B$  is often used, as defined in [7] with the formula:

$$x = B\sqrt{t}, \quad (7)$$

and the water sorption coefficient  $A$ , defined in [7] with the formula:

$$m_s = A\sqrt{t}. \quad (8)$$

where  $B$  is the water penetration coefficient [ $\text{m}/\text{s}^{1/2}$ ],  $A$  is the water sorption coefficient [ $\text{kg}/(\text{m}^2\text{s}^{1/2})$ ],  $x$  is the penetration depth of the water front during sorption from a water surface [m],  $t$  is the time [s],  $m_s$  is the the mass per area of adsorbed moisture from a water surface. Therefore:

$$m_s = \frac{\Delta m_t}{F}, \quad (9)$$

where  $\Delta m_t$  is the change in the mass of the material sample during the time  $t$  [kg],  $F$  is the sample surface area exposed to water [ $\text{m}^2$ ].

Observation of the rising water front conducted to determine the rate of penetration  $B$  is often quite problematic [8], owing to the fact of the diversified spread of the liquid in the material and the difficulties with a clear definition of the damp area. To improve the measurement of the location of the water front line, the method of neutron radiography is used in the study of water capillary transport [3,9–14]. The method is based on the differences of attenuations of thermal neutrons by hydrogen which is the main water component and by the elements of which cement matrix is composed. Thermal neutrons are strongly scattered by hydrogen, while cement matrix is “transparent”. Therefore, in the produced images (radiographs) of the irradiated sample a contrast between dry and saturated zone can be observed.

Much more conclusive results on the parameters describing the capillary rise are obtained by determining the coefficient  $A$ . The measurements are usually carried out with the traditional gravimetric method, which consists in recording the changes in the mass of the samples remaining in contact with water. Usually it is done by weighing the samples at regular intervals of time. Due to the fact that such an arrangement of the experiment interrupts the continuity of the process, in recent years more and more measurements are carried out automatically, i.e. [15–18], by continuous monitoring of the mass of the sample suspended on a cord of the scales. However, this strongly limits the number of samples tested in the experiment. Therefore, other techniques, free from these shortcomings, are being tested [19–21]. For instance, in this work [8] a cap method was applied. Water for the tested samples is applied in a continuous manner, so this solution permits testing of multiple samples simultaneously, and also evaluating the impact of such additional factors as the direction of flow or the pressure exerted by flowing water. Results compared in [8], referring to the coefficients of capillary water absorption, as obtained in studies conducted with the traditional gravimetric technique and the cap method, reflected a significant overlap between the two methods.

Coefficients  $A$  and  $B$  are often determined on the basis of measurements carried out in different time ranges, i.e. [2,11,13,17,22]. In [23] it was found that estimation of capillary diffusivity should be conducted on the basis of tests carried out at longer intervals of time. Otherwise, the effect of small pores that gain importance in capillary transport after the phase of intense absorption is not taken into account. The reason of the slow movement of liquid in narrow capillaries are large friction resistances. Data on the time of saturation of capillaries, depending on their cross-section can be found in [6].

Level of water saturation of material and its microstructural parameters influence considerably durability in lower temperatures. Describing the process in a simplified way, the destruction of material by frost is caused by freezing of liquid in material pores which leads to a stress resulting from bigger volume of ice

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