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Optimization of the composition of hydrophobized cellular concrete according to its moisture-transporting and water-holding characteristics

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Abstract. The article is devoted to the study of the possibility of obtaining information about frost resistance and strength of concrete based on the analysis of its moisture-transporting and equilibrium water-holding properties. The dependences of the moisture diffusion coefficient, equilibrium moisture content at various values of relative humidity and porosity of autoclaved cellular concrete specimens on the concentration of water-repellent additive were studied. A comparison of the results of these measurements with the data on frost resistance, got by the direct method according to the current standards, have been made. It is shown, that moisture-transporting and water-holding characteristics can be used to assign concrete compositions optimal in frost resistance and strength. The mechanisms of the influence of water repellent on the frost resistance at its various concentrations are considered. It has been established that the composition of cellular concrete containing 2% water-repellent additive is optimal.

1. Introduction

The problems of energy efficiency, resource-saving, lowering operating costs and improving the comfort of housing remain their primary importance in the practice of construction. Increasing requirements for the heat-shielding properties of the external building envelope makes it necessary to use highly efficient heat-insulating and structural heat-insulating materials such as autoclaved cellular concrete for their installation. Products made from autoclaved cellular concrete are distinguished by fairly good strength, high heat-insulating ability and allow to get significant energy savings necessary for heating facilities while maintaining a healthy indoor climate [1-4].

To increase the competitiveness of cellular concrete in modern conditions, the urgent task is to further improve the physical and technical properties of these materials and create energy-saving technologies for their manufacture. Modern building standards impose several mandatory requirements on the wall fencing materials, among which one of the most important is high strength and frost resistance. For such porous building materials as cellular concrete, the values of these indicators vary depending on the moisture content in them: the strength and frost resistance of the material in a water-saturated state decreases, and the thermal conductivity increases [5]. It is possible to reduce the indicators of sorption moisture content and water absorption of cellular concrete to a certain optimal value by optimizing its structure.

The durability of concrete under alternating freezing-thawing is the main factor determining the possibility of its use in cold climates. It is directly related to the moisture properties of concrete, which depend on the value and nature of its porosity, the state of the surface of the solid phase. The lower the total porosity, the higher the frost resistance. At the same time, to increase it, small spherical pores, serving as reservoirs, into which excess water formed during freezing can migrate must evenly be distributed in concrete [6, 7].

One of the ways to optimize the structure of building materials is their hydrophobization using various organic compounds that can give the solid phase of the material water repellent properties. At the same time, the role of water repellent additives in increasing the frost resistance of concrete has not been adequately studied and is usually associated with the difficulty of water suctioning into concrete and its migration, and with an increase in the number of closed pores that are not filled with water when concrete is saturated [8, 9].

The magnitude and nature of the porosity of the material, which determines its frost resistance, can be established based on a study of its equilibrium water-holding and moisture-transporting properties, which reflect the kinetics of moisture migration in the material.

In this paper, the results of a study of the effect of hydrophobization on the frost resistance of cellular concrete are presented and the possibility of assigning an optimal concrete composition from this point of view according to its water-holding and moisture-transporting characteristics is shown.

2. Experimental

2.1. Autoclaved cellular concrete specimens' preparation

To prepare the concrete specimens, CEM I 42,5 N cement according to the Ukrainian standard DSTU B EN 197-1:2015, quicklime according to DSTU B V.2.7-90:2011 with active CaO + MgO content of 80%, quartz sand according to DSTU B V.2.7-32-95 with a quartz content of 85%, aluminum powder according to GOST 5494-95 and water according to GOST 23732 were used. For volume hydrophobization of cellular concrete, polydimethylsiloxane emulsion was used. To determine frost resistance and compressive strength, concrete specimens were made according to DSTU B V.2.7-45:2010 and DSTU B V.2.7-214:2009. The dimensions of the specimens were 100×100×100 mm.

To conduct studies of the water-holding and moisture-transporting properties of cellular concrete, an individual series of experimental specimens 50×50×50 mm in size were made. Three specimens of each series, together with specially selected steel plates for damping positive buoyancy, were used to measure the diffusion coefficient of water. The three remaining specimens of each series, after crushing in pressing equipment, were fractionated using sieves, leaving a fraction with an average size of 4 mm to study the equilibrium water-holding properties.

2.2. Determination of cellular concrete properties

2.2.1. Determination of compressive strength and frost resistance. Examinations of compressive strength and frost resistance were conducted according to the above Ukrainian standards. According to DSTU B V.2.7-45:2010, the frost resistance of concrete is equal to a certain number of freezing–thawing cycles of water-saturated specimens, at which concrete compressive strength is reduced by no more than 15%, and specimen mass loss does not exceed 5%. Frost resistance determination method involves cyclic freezing of the specimens in the air with a temperature of −18 ± 2°C for at least 4 h and then defrosting under the water at room temperature for at least 4 h. Each step of freezing and defrosting was a single cycle of the research. For research, percentage mass and compressive strength losses were determined compared to the samples without the freezing–defrosting procedure after 25th cycles, and then after each 5th cycles.

2.2.2. Determination of water-holding and moisture-transporting characteristics. The measurements of the equilibrium water-holding characteristics of the experimental cellular concrete specimens were carried out by the known method for the determination of adsorption isobars [10]. For this purpose, ground concrete specimens (fraction 3...5 mm) at a temperature t_1, were blown with air saturated with water vapour at a lower temperature t_2. The relative humidity φ at which the specimens were located was calculated by the formula:

$$\varphi = \frac{p_\mathrm{s}(t_2)}{p_\mathrm{s}(t_1)}, \tag{1}$$

in which $p_\mathrm{s}(t_1)$, $p_\mathrm{s}(t_2)$ – saturated water vapour pressure at temperatures t_1 and t_2, respectively. The equilibrium moisture contents of the specimens U_φ were calculated by their masses measured by the weight method:

$$U_\varphi = \frac{m_\varphi - m_0}{m_0}, \tag{2}$$

where m_φ – the mass of specimen in equilibrium with air having relative humidity φ, m_0 – dry mass. For this study, not a complete adsorption isobar was determined, but only the moisture content at three fixed values of relative humidity φ: 0.3, 0.6, and 0.95.

The moisture diffusion coefficient D of the studied cellular concrete specimens was determined by the kinetics of capillary impregnation [11] of specimens completely immersed in water. In this method, the maximum moisture content U_m of the samples is automatically determined as the relative amount of moisture absorbed by the porous material upon its contact with water in relation to the dry mass m_0 of the material:

$$U_\mathrm{m} = \frac{m_\mathrm{s} - m_0}{m_0}, \tag{3}$$

where m_s – the mass of the water-saturated specimen. The maximum moisture content allows to determine the open porosity P_O of dispersed materials – the ratio of the absorbed moisture volume $V_{\mathrm{H_2O}}$ to the volume of the specimen V_0:

$$P_\mathrm{O} = \frac{V_{\mathrm{H_2O}}}{V_0} = \frac{(m_\mathrm{s} - m_0)\cdot\rho_0}{\rho_{\mathrm{H_2O}}\cdot m_0} = \frac{\rho_0}{\rho_{\mathrm{H_2O}}}\cdot\frac{m_\mathrm{s} - m_0}{m_0} = \frac{\rho_0}{\rho_{\mathrm{H_2O}}}\cdot U_\mathrm{m}, \tag{4}$$

where ρ_0, $\rho_{\mathrm{H_2O}}$ – the density of the dry specimen and water, respectively.

The value of the closed porosity P_c can be calculated from experimental data. Its value is equal to the difference between the total P and the open porosity P_O: $P_\mathrm{c} = P - P_\mathrm{O}$. The total porosity P is determined by the average density of the specimens ρ_0 and the average density of the solid phase ρ_1 of the mixture raw components:

$$P = 1 - \frac{\rho_0}{\rho_1}. \tag{5}$$

By the component composition of the mixture, for ρ_1 we find $\rho_1 \approx 2600$ kg·m^{-3}, which gives almost the same total porosity for all series of specimens $P = 80\%$.

3. Results and discussion

The obtained values of frost resistance F, compressive strength R and average density ρ_0 of the studied cellular concrete specimens at various concentrations c of water repellent are presented in table 1 and figure 1. Established water-holding and moisture-transporting characteristics are shown in table 2, figure 2 and figure 3.

Table 1. Frost resistance, compressive strength and average density of cellular concrete at various concentrations of water repellent.

c (%)	*F* (cycles)	*R* (MPa)	ρ_0 (kg·m^{-3})
0	25	2.2	535
1	30	2.4	530
2	50	3.5	540
3	35	3.2	537
4	40	2.9	531
6	75	2.6	522

As follows from table 1 and figure 1, the frost resistance of cellular concrete specimens increases in the entire studied range of water repellent concentrations, except for the range of 2...3%, where it falls. Moreover, at c > 2...3%, the strength of the samples decreases markedly.

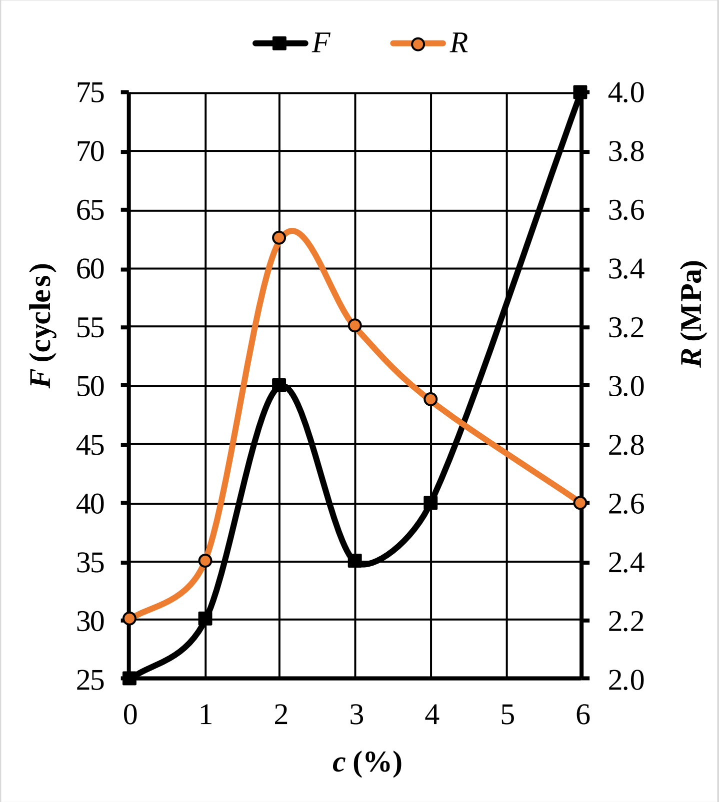

Figure 1. Dependences of frost resistance and compressive strength of cellular concrete on the concentration of water repellent.

Following the existing ideas (e.g., [12, 13]), the destruction of concrete under the influence of frost is caused not only by an increase in the volume of water during freezing. The hydraulic pressure of water when it is squeezed out from the freezing front, the crystallization pressure of ice during its aggregation, the arising osmotic pressure are the main destructive factors that determine concrete damage. The manifestation of each of these factors depends on the conditions of freezing, water saturation of concrete, its structure, the state of the surface of the solid phase and other parameters.

When a water repellent additive is introduced into the concrete, it is adsorbed in the form of monomolecular layers on the walls of large pores and capillaries, which are formed during the setting and expansion of cellular concrete mass. This leads to an increase in the number of closed pores that are not filled with water (closed porosity P_c), which can act as reserve ones and increase the frost resistance of concrete. An increase in the number of fully or partially closed pores with a change in the water repellent concentration between 0...2% is indicated by a decrease in the mass transfer coefficient and open porosity P_O of the specimens in this range (figure 3). A slight increase in porosity in the 0...1% area is associated with air entrainment during introducing a water repellent additive.

Table 2. Water-holding and moisture-transporting characteristics of cellular concrete at various concentrations of water repellent.

c (%)	U_φ (%) at φ			$D\cdot10^8$ (m^2/sec)	U_m (%)	P_O (%)	P_c (%)
	0.3	0.6	0.95				
0	0.22	2.6	4.5	48.0	81	43	37
1	0.23	1.9	3.7	7.4	92	48	32
2	0.22	1.4	1.9	1.0	66	36	44
3	0.24	2.1	2.7	24.0	102	54	26
4	0.23	2.5	4.2	0.9	84	44	36
6	0.22	2.3	5.1	0.1	66	34	46

From table 2 it can be seen that those series of specimens where the closed porosity is maximum have the highest frost resistance. This is a series with a concentration of water repellent additives of 2% and 6%.

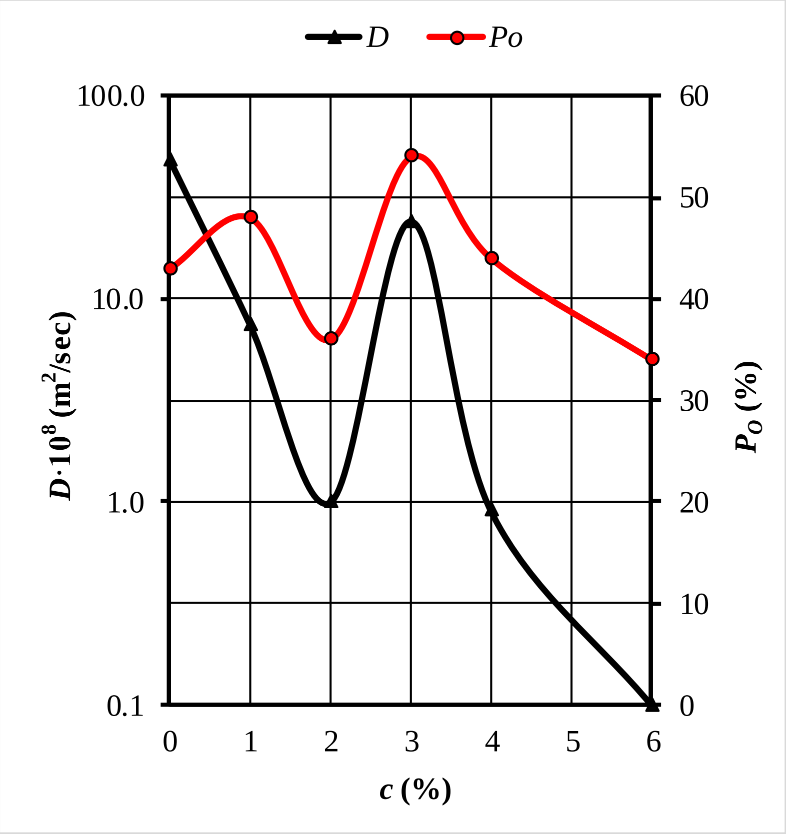

Figure 2. Dependences of the coefficient of moisture diffusion and open porosity of cellular concrete on the concentration of water repellent.

As was shown in [14], the use of polydimethylsiloxane emulsion additives in the composition of cellular concrete mixtures in an amount of up to 2% promotes the formation of closed pores with a smaller diameter and a smaller thickness of the inter-pore partitions as compared to the composition without additives. This should lead [15] to a decrease in hydraulic pressure and, therefore, also contribute to an increase in the frost resistance of concrete in this concentration range of the water repellent.

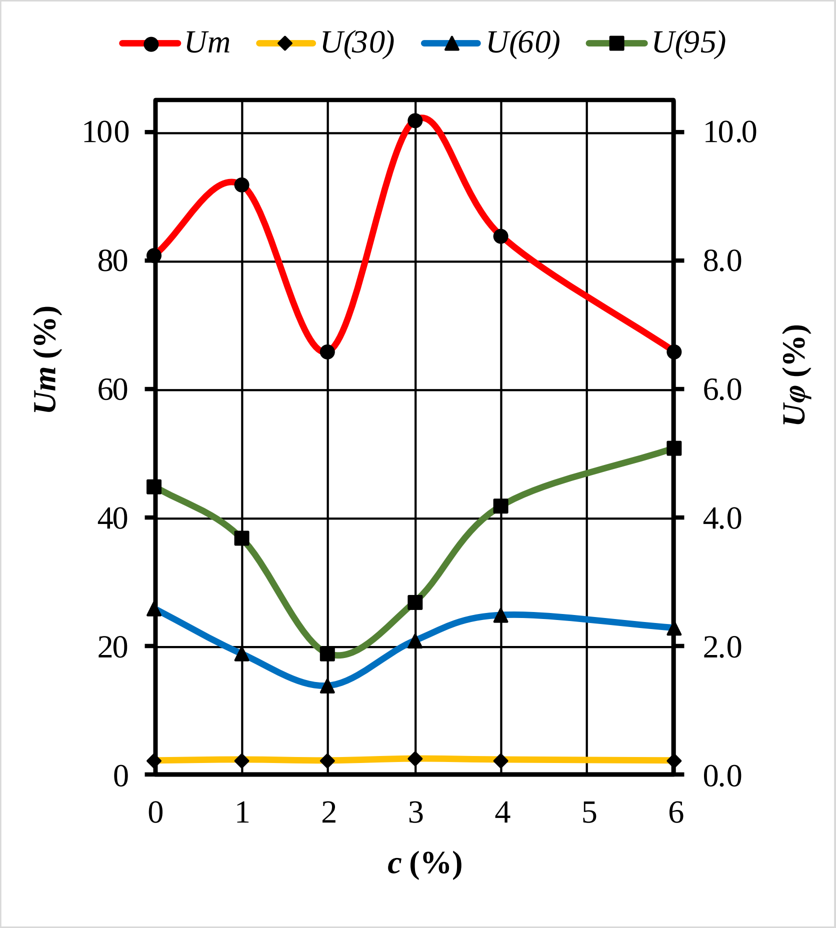

Figure 3. Dependences of the moisture content of cellular concrete at various values of relative humidity on the concentration of water repellent.

Another mechanism for increasing the frost resistance of cellular concrete during its hydrophobization can be a significant weakening of osmosis [6], which is responsible for the formation of ice lenses in the mouths of capillaries. Clusters of water molecules involved in osmosis can form at $t < 0°C$ only in the near-wall water layers, the structure of which is changed in the presence of powerful unsaturated fields of the solid phase surface. According to sorption measurements, the near-wall film thickness decreases with an increase in the concentration of water repellent from 0 to 2%, as indicated by a decrease in the equilibrium moisture content of the specimens (figure 2). The decrease in adsorption occurring at $\varphi = 0.6...0.95$ by the capillary condensation mechanism during hydrophobization is explained by a decrease in the affinity between water and the surface of the solid phase which leads to a decrease in the capillary potential. A decrease in ice formation in the mouths of capillaries leads to a weakening of such destructive factors as the hydrostatic pressure of unfrozen water and the crystallization pressure of the ice. As a result, frost resistance grows.

It should be noted that, as can be seen from figure 2, hydrophobization practically does not change the moisture content of concrete samples at $\varphi = 0.3$. According to [10], the value of U_φ at $\varphi = 0.3$ characterizes the capacity of a monolayer of water molecules on the solid phase surface. Its immutability shows that hydrophobization does not affect the formation of the microstructure of autoclaved cellular concrete. The addition of a water repellent affects only the meso- and macro-structure of concrete, because of which the contact angle of wetting liquid with the solid phase surfaces changes. This effect should increase with an increasing concentration of water repellent.

The existence of near-wall water layers with a changed structure can also determine the mechanism of destruction, which consists in the appearance of additional tensile stresses during the formation of ice in the capillaries. This can lead to the breaking of hydrogen bonds between the near-ice and near-wall films. The hydrophobization of the surface, causing a decrease in the near-wall film and the specified tensile stresses, should contribute to an increase in frost resistance.

With an increasing concentration of water repellent between 2...3%, the frost resistance of cellular concrete decreases. To explain this, the authors proposed a mechanism based on the wedging of micropores and recesses on the surface of large pores with water repellent molecules.

The effect of disjoining pressure [16, 17] is known, which consists in increasing the potential energy of molecules in a thin film between two interphase surfaces. If the film thickness is less than or equal to two radiuses of action of intermolecular forces, then the excess energy of the molecules in it is not fully compensated. Molecules of the film draw molecules with lower energy from the environment, which leads to the appearance of disjoining pressure. In cellular concrete, wedging can be carried out by water repellent molecules in recesses on the surface of large pores. Water repellent molecules interact with water molecules, leading to the irreversibility of the wedging. The interaction of water repellent molecules and water in micropores can cause an increase in their size.

Thus, the wedging effect should determine an increase in the effective radius of the pores involved in moisture transfer, and an increase in the number of active adsorption centres on the surface of the solid phase of cellular concrete. This leads to an increase in equilibrium moisture content, moisture diffusion coefficient and open porosity of the specimens at a concentration of water repellent 2...3%. An increase in the moisture-transporting and water-holding characteristics of the material indicates a decrease in the relative number of closed pores and growth in the thickness of the near-wall layers of water. As a result, the factors discussed above that contribute to frost destruction of concrete are strengthened, and frost resistance decreases.

With a water repellent content of over 3%, the frost resistance of cellular concrete specimens is determined by the competing effects of increasing adsorption and decreasing porosity and mass transfer coefficient, which prevails. The decrease in moisture transfer characteristics is apparently because at such concentrations the hydrophobic effect becomes so significant that the penetration of water into the mouth of the pores is difficult.

A decrease in adsorption at a relative humidity of 0.6 and a water repellent concentration of over 5% can be explained as follows. With a decrease in vapour pressure during capillary condensation, following the Kelvin equation, only pores of small radius are filled with water. And at high concentrations of water repellent, such pores can be clogged by its molecules.

Starting with a concentration of water repellent of 3% frost resistance of cellular concrete increases. However, the amount of water repellent additive that can be incorporated into concrete is limited by a decrease in its strength because of increased air entrainment into the concrete mixture, and worsening hydration and hydrothermal synthesis conditions. Moreover, in the process of autoclave processing, recrystallization and enlargement of new formations occur [14], which causes a violation of the structure.

4. Conclusions

The dependences of the moisture diffusion coefficient, equilibrium moisture content at various values of relative air humidity and porosity of autoclaved cellular concrete specimens on the concentration of water-repellent additives were studied. A comparison of the results of these measurements with the data on frost resistance, got by the direct method according to the current standards, have been made. It is shown, that moisture-transporting and water-holding characteristics can be used to assign concrete compositions that are optimal in frost resistance and strength. The mechanisms of the influence of hydrophobization on the frost resistance of cellular concrete are considered.

It was shown that at additive concentrations of 0...2% the increase in frost resistance is because of a growth in the number of closed reserve pores of small diameter, a decrease in tensile stresses in the capillaries and ice formation in their mouths. The drop in frost resistance in the concentration range of 2...3% is associated with an increase in open porosity due to the wedging effect of the water repellent molecules in the recesses on the surface of large pores. At additive concentrations above 3%, frost resistance increases because of a significant hydrophobic effect that impedes the penetration of water into the pore mouths. At the same time, the strength of cellular concrete samples decreases due to increased air entrainment into the concrete mixture and deterioration of hydration and hydrothermal synthesis conditions. It was found that the concrete composition containing 2% of water repellent additive is optimal in frost resistance and strength.

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