Розглянуто вплив найбільш поширеного у використанні кремній-органічного поверхневого гідрофобізатора ГКЖ-11К на фізико-механічні властивості та формування фазового складу цементного каменю з об'ємною гідрофобізацією. Важливість цього дослідження полягає в тому, що введення поверхневого гідрофобізатора ГКЖ-11К до складу цементного тіста призводить до утворення дисперсійних гідрофобних плівок різного рівня (розмірів). Ці плівки адсорбуються на поверхні зерен цементу, а також на поверхні гідратованих компонентів цементних часток, $Ca(OH)_2$, гідросульфоалюмінатів кальцію. Визначено, що дисперсні гідрофобні плівки частково блокують проникнення води вглиб поверхні цементних зерен та змінюють кінетику гідратації.

Це призводить до зниження ступеня гідратації та вмісту $Ca(OH)_2$ в цементному камені, внаслідок чого зменшується усадка в процесі тверділня. В той же час, дисперсні плівки, що адсорбовані на сусідніх гідратованих частках цементу у межах контактів останніх в процесі їх конденсації, можуть утворювати водневі та хімічні зв'язки. Ця взаємодія між плівками часток, незважаючи на зниження ступеню гідратації, призводить до зниження водопоглинання, підвищення міцності цементного каменю, що в даному випадку визначається також кількістю та площею контактів в одиниці об'єму цементного каменю. За рахунок перекриття пор гідрофобними ланцюжками знижується водопроникність цементного каменю по всьому його об'єму, що сприяє зростанню експлуатаційної надійності та довговічності конструкцій, особливо тонкостінних. Використання цієї домішки для об'ємної гідрофобізації може значно підвищити строки безремонтної експлуатації тонкостінних виробів та конструкцій, а також знизити собівартість за рахунок відсутності потреби у використанні матеріалів для захисту поверхні бетону

Ключові слова: гідрофобізуючі домішки, об'ємна гідрофобізація, усадка цементного каменю, водопоглинання, міцність при стиску

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INVESTIGATING THE INFLUENCE OF VOLUMETRIC HYDROPHOBIZATION ON THE FORMATION OF PHASE COMPOSITION OF CEMENT STONE AND ITS PHYSICAL-MECHANICAL PROPERTIES

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1. Introduction

The issue of durability of concrete structures and articles, particularly thin-walled, exposed to atmospheric influence, is always relevant. Durability of concrete is ensured by a series of measures – by the economically feasible optimization of concrete structure, by using plasticizing admixtures, wastes from enrichment in the mining industry, from iron ores [1, 2], and so on, including its hydrophobization, the selection of appropriate composition that provides for the minimal shrinkage at hardening. One way to enhance the operational efficiency of concrete that is exposed to such atmospheric effects over a long time as freezing, thawing, wetting and drying, as well to aggressive environments, is its volumetric hydrophobization. The largest degradation when concrete is exposed to the above factors is demonstrated by cement stone. In this regard, the studies were performed into the influence of volumetric hydrophobization of cement stone on its shrinkage, water absorption, and strength, which characterize its durability.

The most common are the hydrophobic admixtures for the surface hydrophobization of concrete structures already in operation; however, the processes of corrosion render such a protection non-durable: in 2–5 years, there is again a need for applying a protective layer. It is known that at surface hydrophobization a hydrophobic liquid penetrates the surface layer of concrete to 2–5 mm and condenses into films that are, over time, undergo hydrolysis, which leads to the losses of water repellent properties. The use of volumetric hydrophobization for thin-walled structures and buildings whose repair is associated with difficulties at its execution (green roofs, construction of tunnels, parking lots, road coatings) is relevant and appropriate. At first glance, volumetric hydrophobization is more expensive than the surface one but employing appropriate water repellent admixtures, the selection of concrete composition and the technology of structure fabrication could prove economically feasible compared with costs required to perform surface hydrophobization.

2. Literature review and problem statement

By analyzing the main trends addressed in the studies of concrete with volumetric hydrophobization [3], it was revealed that the most widely used admixtures are those based on silicone-organic compounds: GKZH-94 (136–157 m) – methylhydrosiloxane polymer, GKZH-11K – a solution of potassium or sodium methylsiliconate, product 119-215 – oligoethoxy-2-ethylhexoxyloxane [4] and water repellents based on silanes, which pass into silanols at hydrolysis. Papers [3–5] report studies into the physical and mechanical properties of concretes with organosilicon admixtures, but the authors failed to resolve issues related to investigating the impact exerted on the quantitative phase composition of cement stone new formations by volumetric hydrophobization. Silanols exhibit hydrophobic properties, but silanes are very costly. Researchers in [6], when comparing results from testing the admixture based on silanes for surface and volumetric hydrophobization, point out that it is more effective at surface application, while the volumetric hydrophobization reduces indicators of strength. Moreover, using it for lime-cement and pozzolana-lime mortars [7, 8] leads to a change in the physical properties and the kinetics of hydration. Paper [9] considers the influence of an admixture based on silane on the physical properties of cement with microparticles of silica, resulting in significant shrinkage at drying. The most widely used silanols are methyltriethoxysilane, methyltrimethoxysilane, isobutyltriethoxysilane, N-octyltriethoxysilane, which are applied to synthesize silicones, to make mineral fillers water repellent. Our analysis of the scientific literature has not revealed studies related to the influence of volumetric hydrophobization of GKZH-11K on the phase composition and properties of cement stone. Thus, the identified issue of the influence of volumetric hydrophobization on the formation of phase structure of cement stone and its properties and, as a consequence, improvement of durability, requires additional research.

3. The aim and objectives of the study

The aim of this study is to investigate the influence of volumetric hydrophobization of cement stone using the surface water repellent GKZH-11K on its quantitative phase composition of new formations at hardening, as well as on its physical-mechanical properties. This would make it possible to enhance the physical and mechanical indicators of cement stone, which could improve the durability of concretes and mortars that are operated under the conditions of atmospheric effects.

To accomplish the aim, the following tasks have been set:

– to explore the shrinkage, water absorption, and compressive strength of cement stone without admixtures and with the admixture GKZH-11K;

– to conduct thermal, X-phase, and spectroscopic (infrared) studies of these samples;

– to suggest a protocol for the volumetric hydrophobization in contact zones that could elucidate the strengthening of cement stone following the introduction of GKZH-11K.

4. Materials and methods to study cement stone

The current research was based on the following materials:

– Portland cement from Zdolbuniv cement factory (Ukraine) CEMII/A-S32,5;

– Tap drinking water;

– GKZH-11K, mark B (Ukraine, Zaporizhzhia).

We accepted the percentage content of admixtures to equal 0.7 % of the mass of cement; it has demonstrated the best result during physical and mechanical tests on cement samples. We introduced the admixture with the last mixing water.

It is known that most modern water repellent admixtures demonstrate a plasticizing effect, which us why the W/C ratio in the manufacture of cement stone samples was taken to be 0.3.

We studied the processes of shrinkage on samples-beams with dimensions of 2×2×8 cm, using the comparator IZA-2 (No. 590303, USSR); compressive strength and water absorption – on samples-cubes with dimensions of 2×2×2 cm.

Influence of the admixture on the phase composition of cement stone was determined by methods of thermal and X-phase analysis, and its interaction with the components of cement stone – by the method of infrared spectroscopy.

The following equipment was used:

– Derivatograph MOM Q1200D System F. Paulik, J. Paulik, L. Erdey (Hungary). Testing parameters: max temperature T – 1000 °C, the rate of rise in temperature V – 10 °C/min., DTA – 1 mV, TG – 500. Batches of the tested material: 820 mg, 920 mg;

– X-ray diffractometer DRON-3.0 (Russia), an x-ray tube – BSV23, anode – copper, voltage – 40 kV, tape motion speed – 600 mm/h;

– IR spectrometer Termonicolet IR200, spectral range: 400–4000 cm^{-1}, the source of radiation – a halogen lamp.

5. Results of studying the impact of volumetric hydrophobization on the formation of phase structure of cement stone

5. 1. Results of studying the physical and mechanical properties of cement stone with the water repellent admixture GKZH-11K

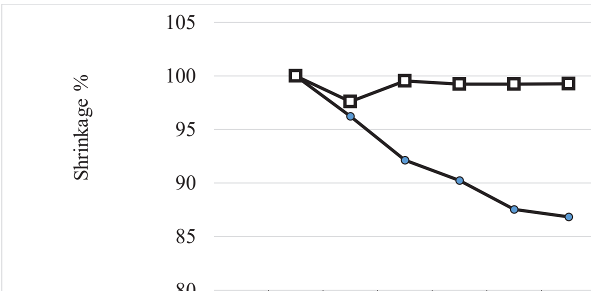
Results of research into the shrinkage of cement stone without admixtures, and with GKZH-11K in the amount of 0.7 %, are shown in Fig. 1, into strength – in Fig. 2, into water absorption – in Fig. 3.

Results from studying the strength at compression at the age of 28 days and 365 days of cement stone samples without admixtures and with the admixture GKZH-11K – 0.7 % are shown in Fig. 2.

Results from studying water absorption at the age of 28 and 365 days for the samples of cement stone without admixtures and with the admixture GKZH-11K – 0.7 % are shown in Fig. 3

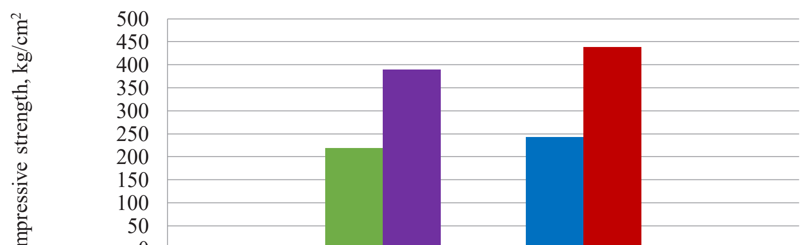
The research results shown in Fig. 1–3 indicate that the smallest shrinkage deformations are demonstrated by the samples of cement stone with the admixture GKZH-11K;

indicators of water absorption – by the samples of cement stone with GKZH-11K – 5.7 % at the age of 28 days, 5.8 % – at age of 365 days. A slight increase in the water absorption by samples with GKZH-11K over time is due to the partial hydrolysis of CH_3. The largest increase in strength was demonstrated by samples with the admixture GKZH-11K – from 243 kg/cm^2 at the age of 28 days to 438.05 kg/cm^2 at the age of 365 days.



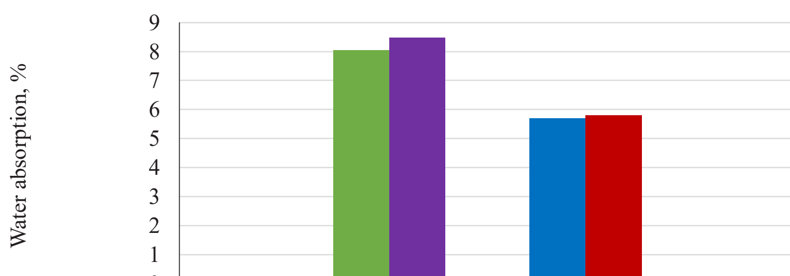
	1 days	3 days	7 days	14 days	21 days	28 days
sample without admixture	100	96,24	92,13	90,25	87,56	86,86
sample with GKZH-11K 0,7 %	100	97,62	99,54	99,24	99,24	99,28

Fig. 1. Shrinkage of samples without admixtures and with GKZH-11K in the age of 1, 3, 7, 14, 21, 28 days



	Cement stone without admixture	Cement stone with GKZH-11K, 0.7 %
0,70 %, 28 days	0	243,02
0,7 %, 365 days		438,05
0 %, 28 days	218,26	
0 %, 365 days	390,05	

Fig. 2. Compressive strength at the age of 28 and 365 days, kg/cm^2



	Cement stone without admixture	Cement stone with GKZH-11K
0,70 %, 28 days	0	5,7
0,7 %, 365 days	0	5,8
0 %, 28 days	8,045	
0%, 365 days	8,47	

Fig. 3. Water absorption by samples of cement stone at the age of 28 days, %

The admixture GKZH-11K refers to group III of water repellants that reduce water absorption by 1.4–1.9 times.

5. 2. Results from thermal, X-phase, spectroscopic studies of cement stone samples

The results of DTA for cement stone samples without admi-xtures and with the admixture GKZH-11K are shown in Fig. 4, 5, X-phase – in Fig. 6, 7, infrared spectroscopy – in Fig. 28 ,29.

Results from X-phase study of cement stone samples with the admixture GKZH-11K and without admixtures are shown in Fig. 6, 7.

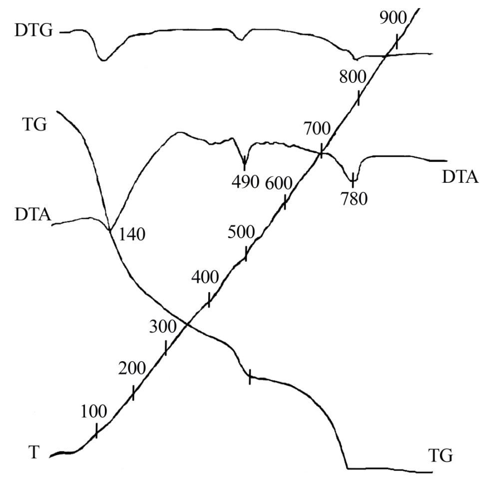


Fig. 4. Derivatogram of sample CEMII/A-S32,5 without admixtures

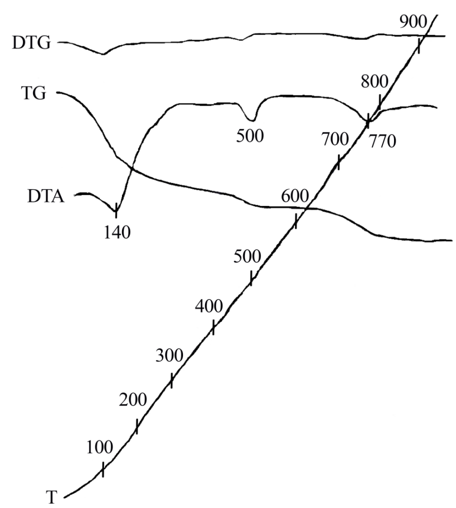


Fig. 5. Derivatogram of sample CEMII/A-S32,5 with the admixture GKZH-11K – 0.7 %

When comparing data on DTA curves, one can see that the samples of cement stone without admixtures and with the admixture GKZH-11K contain C-S-H (II) in the gel-like form (endo effects at 140 °C), $Ca(OH)_2$ – (endo effects at 490 °C, 500 °C), $CaCO_3$ – (endo effects at 770 °C, 780 °C).

At the same time, the DTA curves for the samples without admixtures and with the admixture

GKZH-11K demonstrate a wide endo effect in the temperature range of 350–470 °C, which testifies to the presence in them of compounds $3CaOAl_2O_36H_2O$; $3CaOH_2O_3CaSO_412H_2O$ and, probably, $3CaOFe_2O_3CaSO_412H_2O$ or a solid solution of the latter compounds.

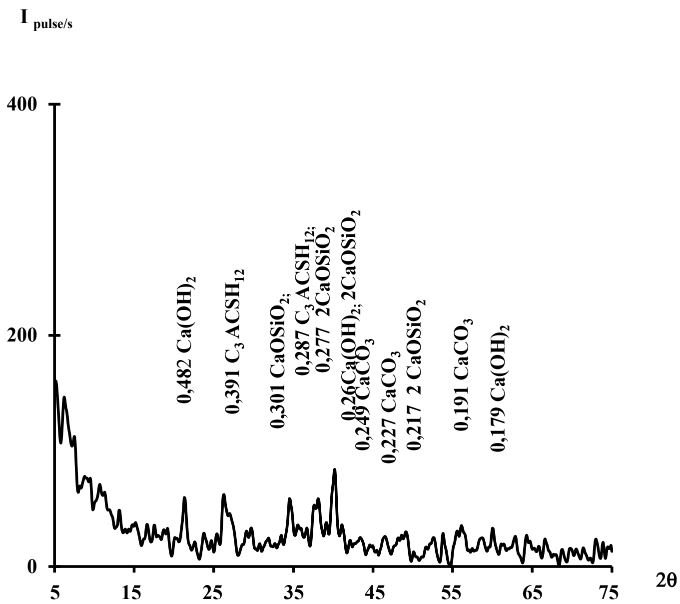


Fig. 6. Diffractogram of sample CEMII/A-S32,5 with the admixture GKZH -11K

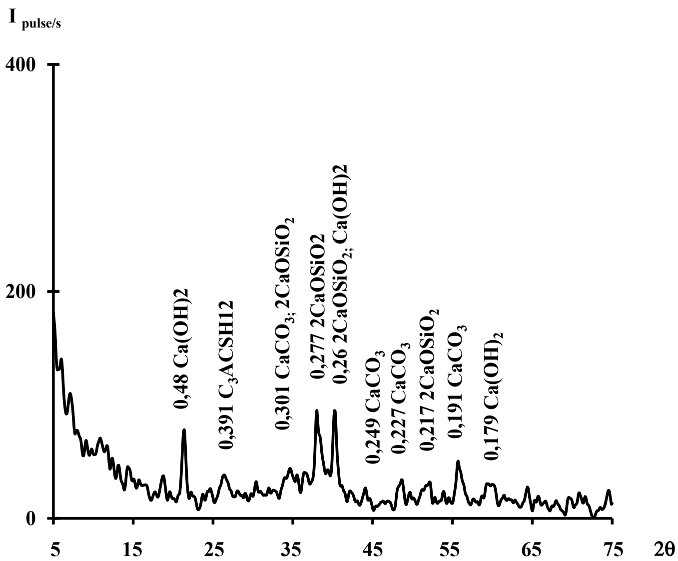


Fig. 7. Diffractogram of control sample CEMII/A-S32,5 without admixtures

The content of $Ca(OH)_2$ and $CaCO_3$, $2CaOSiO_2{\times}nH_2O$ in the cement stone samples was determined by a loss in mass based on TG curves in the respective intervals of temperatures; it is given in Table 1.

Diffractograms of the cement stone samples indicate that the samples both without admixtures and with the admixture GKZH-11K are mostly dominated by:

– $Ca(OH)_2$ – identified according to inter-plane distances (*d*=0.48–0.482; 0.26; 0.179 Nm);

– $CaCO_3$ (*d*=3.01; 0.249; 0.227; 0.191 Nm);

– Clinker fund (non-hydrated part of cement grains) in the form of $2CaOSiO_2$ (*d*=0.301; 0.287; 0.26; 0.217 Nm).

Table 1

Content of $Ca(OH)_2$, $CaCO_3$, $2CaOSiO_2{\times}nH_2O$ in the samples of cement stone

No. of entry	Sample titles	Content, %		
		$Ca(OH)_2$	$CaCO_3$	$2CaOSiO_2{\times}{\times}nH_2O$
1	Control (no admixtures)	9.31	9.56	28.67
2	With the admixture GKZH-11K	4.38	6.88	16.01

Halo in the diffractograms of all samples in the intervals of angles 5–15° and 30–40°, as well as diffraction maxima *d*=0.9–0.95, indicate that the samples contain gel-like hydrosilicates C-S-H (II) with the ratio of Ca/Si from 1.5–2. The sample without admixtures and with GKZH-11K also include $3CaOAl_2O_3CaSO_412H2O$ (*d*=0.391; 0.287 Nm).

The samples with GKZH-11K include less $Ca(OH)_2$ and $CaCO_3$, indicating partial blocking of water access into the surface of the hydrated cement dispersions due to coating with dispersive water repellent films and contribution of $Ca(OH)_2$ to the formation of the water repellent dispersible films themselves.

Data from thermogravimetry and Table 1 indicate that the volumetric hydrophobization of the cement stone GKZH-11K reduces the content of $Ca(OH)_2$, $CaCO_3$, $2CaOSiO_2{\times}nH_2O$.

Results from IRS infrared spectroscopy of the samples of cement stone with the admixture GKZH-11K and without admixtures are shown in Fig. 13, 14.

The infrared spectrum analysis of sample with GKZH-11K has revealed the emergence of two expanded bands with peaks at frequencies of 2,973.7 and 2,894.7 cm^{-1}, which relate to the asymmetric and symmetric valence fluctuations CH of methyl groups. The expansion of absorption bands and the displacement of their peaks were compared to data given in [10]; they confirm the occurrence of an intermolecular interaction in the condensed hydrophobic film at the surface of the hydrated cement dispersions.

Comparison of the infrared spectra of sample without an admixture and with the admixture GKZH-11K indicates that their spectra include bands centered on frequencies 3,628.1 cm^{-1} and 3,640.4 cm^{-1}, which relate to the valence fluctuations of OH groups; 3,373.2 cm^{-1}, 3,373.9 cm^{-1} which relate to the valence fluctuations of OH groups of adsorbed water [11]; 1,633.1 cm^{-1}, 1,645.3 cm^{-1} – to the deforming fluctuations of adsorbed water; 1,416.6 cm^{-1}, 1,404.2 cm^{-1}, 947.75 cm^{-1}, 874.6 cm^{-1}, 855 cm^{-1} – which relate to the valence fluctuations of Si–O bonds in calcium silicates. An increase in the intensity of the split absorption bands with a maximum of 519.9 cm^{-1} in the samples with GKZH-11K is associated to the additional formation of bonds Si–O–Ca, in the newly created water repellent films.

The emergence of an absorption band with a maximum of 1,251.3 cm^{-1} in the samples with GKZH-11K is due to the fluctuations in the $Si–CH_3$ bond, while two centers at frequencies 1,098 cm^{-1} and 1,095 cm^{-1} along the expanded band testify to the twisting oscillations of the methylene group, probably chemisorbed at a condensed tetrahedra in calcium silicate, which is associated with the displacement of a peak in silica from 1,115.1 cm^{-1} to 1,098 cm^{-1}.

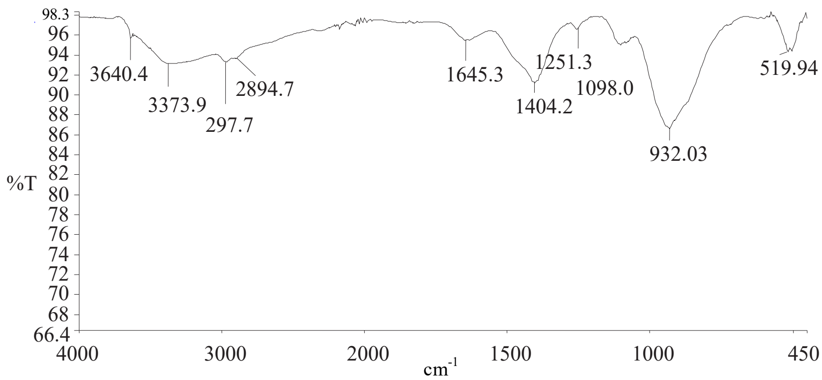


Fig. 8. IRS of cement stone sample with the admixture GKZH-11K

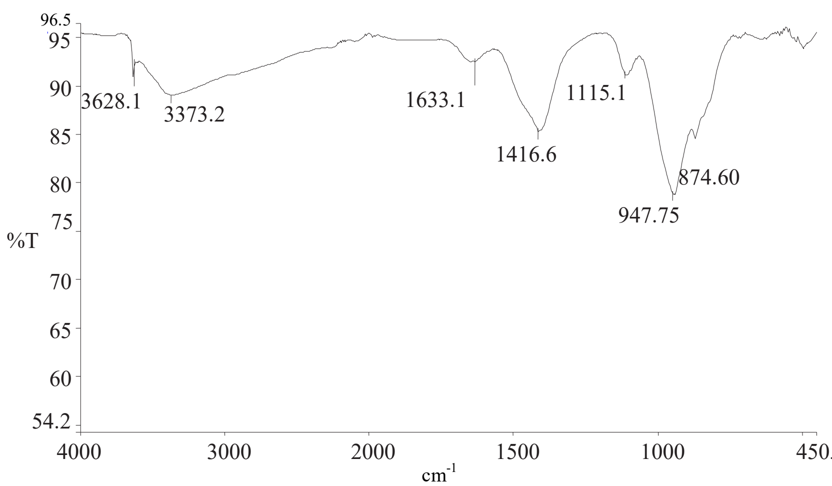


Fig. 9. IRS of cement stone sample without admixtures

5. 3. Theoretical substantiation of the mechanism of interaction between hydrophobic compounds and components of cement slurry

The surface hydrophobization of GKZH-11K is known, which results, following the condensation process involving monomers $CH_3[Si(OH)_2OK]$ after applying on the surface of the material, in the formation of a more or less dispersive hydrophobic film. Methyl groups of CH_3 at a material's surface reduce the surface tension of water and perform water repellent functions. When using GKZH-11K for volumetric hydrophobization, taking into account moisture content of the internal environment of cement stone, a hydrophobic film in the contact zone between the hydrated parts of cement can be represented by the following scheme, similar to the case for surface hydrophobization (Fig. 10).

According to Fig. 10, using GKZH-11K for volumetric hydrophobization should lead to a reduction in the interaction at contact zones with the formation, at them, of hydrogen bonds and the action of van der Waals forces that would ultimately reduce the strength of cement stone. However, results from studying strength for the cement stone modified with GKZH-11K, shown in Fig. 12, indicate that the strength grows despite the reduction in the degree of hydration and the content of $Ca(OH)_2$, $CaCO_3$, $2CaOSiO_2\ nH_2O$ (Table 1). To elucidate this phenomenon, it is advisable to suggest the following schemes for the formation of dispersed hydrophobic films in cement stone considering the mosaicism of surface of the cement particles. The notion of mosaicism refers to the presence, at the surface of the hydrated cement particles, of both the regions of $Ca(OH)_2$ and Si–OH.

When GKZH-11K is mixed with water, the oligomer of potassium methylsiliconate breaks down into monomeric molecules of $CH_3Si(OH)_2OK$, their dimers and trimmers [12]. In the preparation of a cement slurry, the admixture and its monomers, dimers and trimmers $CH_3Si(OH)_2OK$ adsorb on the dispersion of cement. In line with [13], as a result of the larger polarity, water molecules, monomers, dimers and trimmers adsorb at the surface of cement dispersion; in this case, monomers enter the exchange reactions with $Ca(OH)_2$, present at the surface of cement particles, according to reactions shown in Fig. 11.

Following the reaction of replacement $Ca(OH)_2$ with potassium oxide, shown in Fig. 11, *a*, the scheme takes the form given in Fig. 11, *b*, and, after full condensation, the form shown in Fig. 11, *c*.

As a result of the represented reactions of condensation with surface $Ca(OH)_2$, Fig. 12, *a* shows a scheme to form a contact zone of hydrophobic films that explains the formation of hydrogen bonds*, which are responsible for the enhanced strength of cement stone.

During the adsorption of GKZH-11K at the surface of the hydrated cement particles from –Si–OH – the scheme of hydrophobic films formation is shown in Fig. 12, *b*.

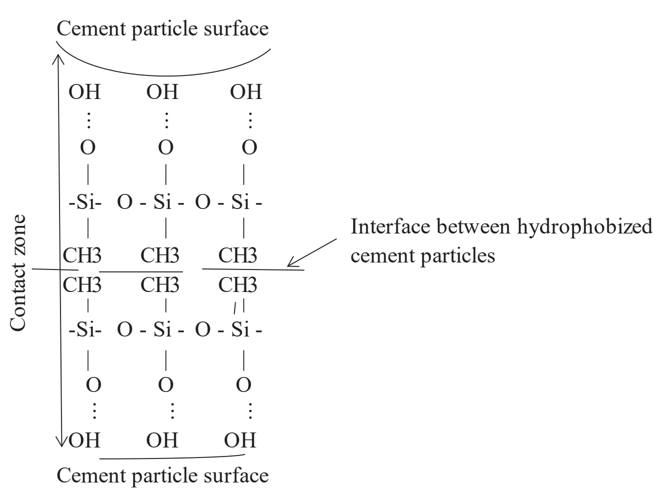


Fig. 10. Scheme of contact of hydrophobic films between hydrated cement particles

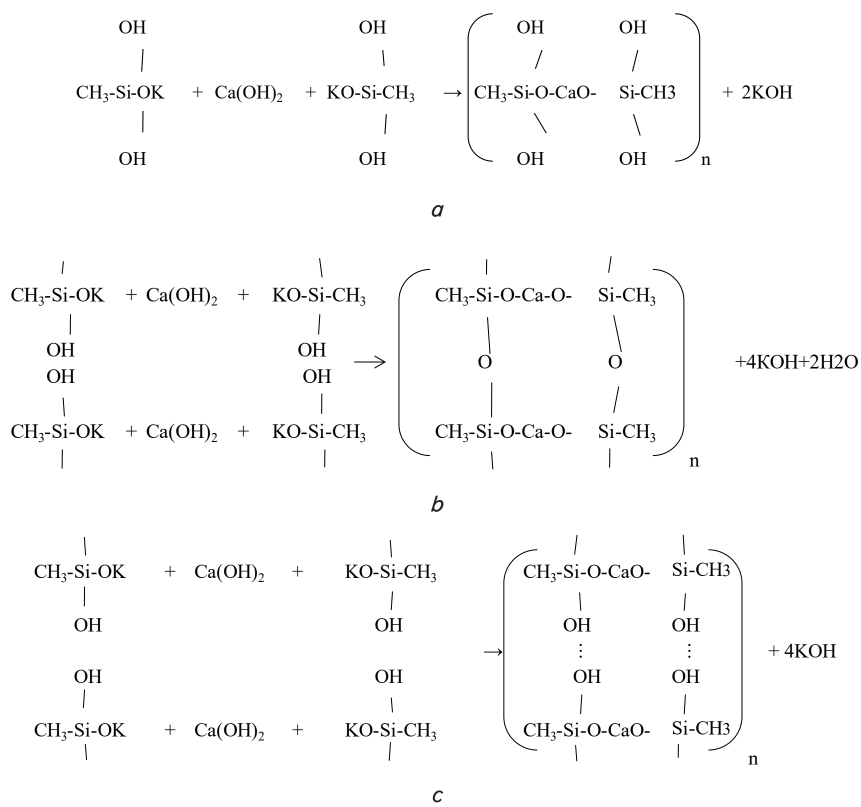


Fig. 11. Types of reactions of substitution and condensation: *a* — reaction od replacement with potassium oxide $Ca(OH)_2$; *b* — intermediate condensation reaction; *c* – full-condensation reaction

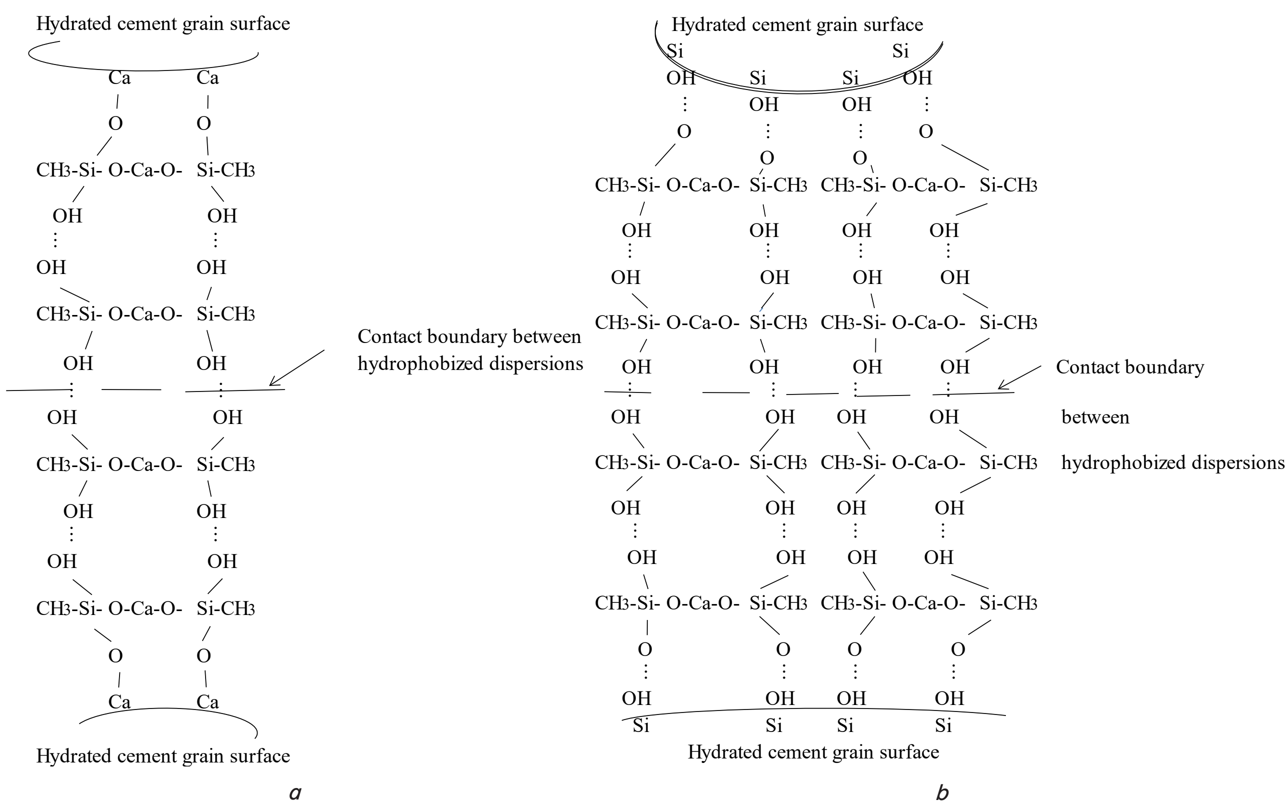


Fig. 12. Schemes of dispersive hydrophobic films: *a* — scheme of dispersive hydrophobic films formed at the surface $Ca(OH)_2$; *b* — scheme of dispersive hydrophobic films formed at the surface Si–OH

Note: ⋮ *– schematic representation of hydrogen bonds. We have proposed schemes of contact zones at which, between the hydrophobic films of neighboring cement particles, hydrogen bonds may form, which lead to the additional strengthening of cement stone*

6. Discussion of results of studying the formation of phase composition and its influence on the properties of cement stone

The results obtained are explained by that the volumetric hydrophobization, due to the dehydration of the system cement-water, leads to condensation into dispersive films of varying magnitude of chain or linear structures, which probably could be twisted into a spiral. This process in cement stone is significantly different from film-formation at surface hydrophobization.

Dispersive films chemisorb at the surface of hydrated dispersions of gel of calcium hydrosilicates, hydrosulphoaluminates, the hydrated CAF phases due to exchange reactions at the surface with $Ca(OH)_2$ and the formation of a hydrogen bond, thereby reducing the degree of cement stone hydration. This occurs due to that the dispersive hydrophobic films reduce the access of water into the surface of the particles.

Owing to such features as the formation of hydrogen bonds at contact zones, their number and area per unit volume of cement stone, the increase in the strength indicators is ensured.

The merit of the current study is the fact that despite the reduction in the basic substances of $Ca(OH)_2$, $CaCO_3$, $2CaOSiO_2 \times nH_2O$ in the cement stone with GKZH-11K, the hydrogen bonds are formed at contact zones (Fig. 12, *a, b*) in comparison with a known pattern, shown in Fig. 10, where the interaction is not observed at contact zones.

It is known that at surface hydrophobization of GKZH-11K, there occurs, over time, the process of hydrolysis of CH3-groups, which reduces the hydrophobic properties of concrete and promotes the penetration of moisture and, consequently, corrosion of concrete. At volumetric hydrophobization, the hydrophobic groups have a chain structure, which makes it possible to prevent the penetration of water throughout the entire thickness of concrete articles over a long time, thereby improving durability.

One should point to the limitations of the current study, which is based on the fact that volumetric hydrophobization applies only to thin-walled structures.

The drawback of the study is that we did not determine the degree of increase in durability compared with surface hydrophobization.

This work could be advanced by studying concrete structures with volumetric hydrophobization under conditions of atmospheric effects.

7. Conclusions

1. It was established that the introduction of an admixture in the amount of 0.7 % by weight of cement reduces water absorption by 1.43 times, shrinkage phenomena, and improves strength by 1.82 times.

2. Introducing the admixture GKZH-11K to the composition of cement slurry reduces the degree of hydration of cement stone. We have determined the extent of influence exerted by the hydrophobic admixture GKZH-11K on the quantitative phase composition of new formations in cement stone. The content of the admixture in the amount of 0.7 % by weight of cement reduces the amount of $Ca(OH)_2$ by 2.1 times, $CaCO_3$ – by 1.4 times, as well as $2CaOSiO_2 \times nH_2O$ by 1.8 times.

3. We have suggested a mechanism for enhancing the strength of cement stone with a decrease in the degree of hydration due to the formation of additional hydrogen bonds at contact zones between the hydrophobic films. The dependence of a decrease in water absorption due to volumetric hydrophobization has been established.

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