

**THE ROLE OF SODIUM NITRATE IN COUNTERACTING THE CARBONATION  
OF PLASTICIZED ALKALI-ACTIVATED SLAG CEMENT CONCRETE  
UNDER CYCLIC INFLUENCE OF SEA WATER**

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**Abstract.** Carbonation of alkali-activated slag cement concrete in marine structures under cyclic influence of sea water in actual operating conditions causes by increased risk connected with deterioration due to increased content of gel phase. Aggressive environment in case of drying under the influence of atmospheric CO<sub>2</sub> and wetting in sea water can be classified as combination of exposure classes XC4 and XS3. These risks increase in case of fresh concretes with high consistency due to the modern requirements.

The aim of this research was to determine the effectiveness of resistance to carbonation of plasticized alkali-activated slag cement concrete, exploited under cyclic influence of sea water, due to application of additive NaNO<sub>3</sub>.

The advanced crystallization of hydrates in alkali-activated slag cement based both on soda ash and sodium metasilicate was shown.

The state of plasticized alkali-activated slag cement concrete, which exposes to carbonation under cyclic influence of sea water in actual operating conditions, was estimated according proposed author's methodology. The performances properties were determined after 90 cycles of wetting in sea water and drying ( $t = 105 \pm 3$  °C) under the influence of atmospheric concentration of CO<sub>2</sub> ( $K \approx 0.04$  %). The deterioration of concrete was determined by falling of flexural strength. The major falling of flexural strength in case of sodium metasilicate (by 11.8 %) compared to soda ash (by 10.3 %), used as alkaline components, is due to higher volume of gel-like phases.

The effectiveness of additive NaNO<sub>3</sub> in plasticized alkali-activated slag cement concrete after 90 cycles of drying-wetting in sea water was confirmed by positive effect on one's properties. Advanced crystallization of gel-like phases caused the higher effectiveness of resistance to aggressive environment in the presence of sodium metasilicate compared to soda ash. The change of anion in alkaline component from carbonate to silicate ensured the lower open capillary porosity from 26.2 down to 11.9 % and the higher conditionally closed porosity from 22.9 up to 34.8 % in alkali-activated slag cement concrete while application of mentioned salt. Specified perfection of pore structure, caused by nature of alkali component, was accompanied by the higher density from 2.1 up to 2.7 %, the major compressive strength from 11.5 up to 15.0 % as well as flexural strength from 7.9 up to 11.2 % and the lower rate of carbonation from 25.0 down to 50.0 %.

**Keywords:** alkali-activated slag concrete, sodium nitrate, carbonization, sea water, pore structure.

**Introduction.** Increasing of durability of building constructions, especially under influence of aggressive media, is an actual worldwide direct of modern material science development. Durability of

materials on the hydraulic cements basis is determined as possibility to stand against atmospheric influence, chemical aggression, steering or any other process of destruction. Atmospheric influence and chemical aggression are often combined in the process of material degradation, known as carbonization. Beside this, in the real conditions of marina constructions exploitation (berths, piers, shore protection structures, dumps, e.t.c.) are under carbonization in the conditions of periodical influence of sea water. One of the ways of increasing of durability of reinforced concrete structures taking into account multi-year's experience of exploitation in aggressive medias is to use slag alkali activated cement. However, modern practice of construction industry need the use of high-slump concrete mixes, leading to the changes in the concrete structures, including slag alkali activated, and it is necessary to determine effective ways of increasing of durability of marine reinforced concrete structures.

**Analysis of the latest studies and papers.** Risk of degradation of slag alkali activated concrete under periodical influence of sea water is growing [1], that could be explained by high gel phase content [2]. Aggressive influence of media in the case of drying under influence of carbon acid from the air and saturation in the sea water is classified as influence class XC4 (influence of carbon dioxide together with periodically contact with water) and XS3 (periodically influence of sea water of air with sea water aerosol) according to [3].

Modern practice of construction science provides high demands to technologically-related properties of concrete mixes, namely to their consistence. To meet this demands makes it necessary to use high-consistence mixes, that leads to the negative changes in the concrete structure, providing increasing of porosity and, as a result, permeability [4]. The mentioned above changes are providing the risks of degradation of concrete structure.

One of the main groups of methods of prevention of carbonization in alkali activated materials is based on perfection of the pore structure of concrete and reduction of carbon dioxide diffusion [5]. It is known that use of sodium nitrate salt ( $\text{NaNO}_3$ ) provides increasing of strength of the slag alkali activated cement by increasing of hydration rate of the slag [6]. There were shown the possibility to regulate setting times, and also perfecting of the pore structure by increasing of crystallinity of hydrated new formations (low basic calcium hydro silicates, calcium hydro alumina silicates) [7]. This leads to the compacting of structure, increasing of strength and reducing of shrinkage deformations of the artificial stone [8]. Moreover,  $\text{NaNO}_3$  is the one of the most popular inhibitors of corrosion of the steel reinforcement, providing effectiveness of use of this salt admixture in the reinforced constructions [9].

Summarizing of the mentioned data makes is possible to predict reduction of carbon dioxide diffusion in the structure of slag alkali activated concrete, obtain from high-flow concrete mixes, under periodical influence of sea water by using  $\text{NaNO}_3$  admixture.

**Aim and tasks.** The aim of the study is to determine effectiveness of prevention of carbonization of plasticized slag alkali activated concrete in the conditions of periodical influence of sea water by modification via  $\text{NaNO}_3$  admixture.

To obtain the aim the following tasks have to be solved:

- to study influence of sodium nitrate on structure formation and properties of slag alkali activated cement;
- to determine properties of slag alkali activated concrete, modified by sodium nitrate, under periodical influence of sea water and air.

**Materials and test methods.** As an alumina silicate component of slag alkali activated cement was taken granulated blast furnace slag (GBS) by «MMK named after Illich» LTD (Mariupol, Ukraine) (chemical composition  $\text{CaO}$  – 47.30 %;  $\text{SiO}_2$  – 39.00 %;  $\text{Al}_2\text{O}_3$  – 5.90 %;  $\text{Fe}_2\text{O}_3$  – 0.30 %;  $\text{MgO}$  – 5.82 %;  $\text{SO}_3$  – 1.50 %;  $\text{TiO}_2$  – 0.31 %) according to DSTU B V.2.7-302:2014, ground to the specific surface  $S_{\text{spec}} = 450 \text{ m}^2/\text{kg}$  (by Blain), modulus of basicity  $M_b = 1.11$ , glass content 84.0 %.

To speed up the ground process of alumina silicate components and in the name of long-term storage of the properties of the ground materials were used polyorganohydrosiloxane admixture (liquid 136-41), 0.1 % by the mass of GBS.

As an alkaline components of the cement were used soda ash ( $\text{Na}_2\text{CO}_3$ ) according to CAS 497-19-8 and sodium metasilicate pentahydrate ( $\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$ ) according to CAS 10213-79-3 in the dry state. To modify slag alkali activated cement was used sodium nitrate ( $\text{NaNO}_3$ ) according to CAS 7631-99-4.

Compositions of the slag alkali activated cement under study are given in the Table 1.

Table 1 – Compositions of slag alkali activated cements

Composition number	Composition, % by mass				Normal consistency, %	Strength at the age of 28 days, MPa	
	GGBS	Soda ash	Sodium metasilicate	NaNO <sub>3</sub>		Flexural	Compressive
CK	94.0	6.0	–	–	29.0	5.1	42.5
CH	92.0	6.0	–	2.0	29.5	5.3	45.5
MK	93.6	–	6.4	–	25.5	5.6	53.9
MH	91.8	–	6.2	2.0	26.0	6.1	58.7

As a plasticizing admixture was used sodium lignosulphonate (LST) according to CAS 8061-51-6, pH  $\geq 8.5$ , content – 0.8 % by the mass of the cement.

As a fine aggregate in the slag alkali activated concretes was taken standard quartz sand from Husariv deposits (Ukraine) (rests on the control sieves, %:  $\leq 0.5$  – less then 92,  $\leq 0.9$  – less then 1) according to DSTU B V.2.7-189:2009.

Concrete mixes were prepared in the Raimondi Iperbet mixer (Italy). Consistence of the fine grain concrete mix was tested by the cone dipping according to DSTU B V.2.7-239:2010.

Properties of slag alkali activated concrete were tested on specimens 40×40×160 mm after 28 days of hardening in normal conditions, after following 90 cycles of periodically saturation and drying or after 90 days of storage in water for control specimens. Strength characteristics were determined according to DSTU B V.2.7-187:2009.

Characteristics of the pore structures of slag alkali activated concrete were tested by the methods, specified in DSTU B V.2.7-170:2008.

Carbonation depth was tested by the height of the layer with the lower intensity of coloring comparing to non-carbonized layer of the specimen after spraying 0.1 % solution of phenolphthalein in ethyl alcohol immediately after their cracking.

To determine carbonation of the specimens of plasticized slag alkali activated concrete, which surface is periodically in contact with the sea water, the authors methodic was used. Specimens after 28 days of hardening in normal conditions were periodically saturated (3 hours of total saturation in the sea water) and dried (21 hours of storing in conditions of atmospheric concentration CO<sub>2</sub> ~ 0.04 % and temperature 105 °C). In this case aggressive influence of the media on concrete is described by the combination of classes of influence XC4 and XS3 and it is modelling the real conditions of the concrete exploitation. After 90 cycles of periodically saturation in the sea water and drying were determined exploitation properties of the slag alkali activated concretes (strength, pore structure, density of the structure, carbonation depth). To compare the obtain results there were tested properties of the slag alkali activated concrete, which after hardening during 28 days in normal conditions ( $t = 20 \pm 2$  °C, RH= 95  $\pm$  5%) was hardening during following 90 days in the water.

Studies on influence of NaNO<sub>3</sub> salt on structure formation of the slag alkali activated cement were done using methods of physical-chemical analysis: thermal analysis (DTA) on derivatograph system R. Paulik, I. Pualik, L. Erday by MOM company (Budapest); zoned analysis on SEM microscope with micro analyzation tool PEMMA 102-02.

**Test results.** *Influence of sodium nitrate on structure formation of the slag alkali activated cement.* Using DTA test method in the hydration products of the slag alkali activated cement of control composition on soda ash basis at the age of 28 days (Figure 1, a, curve 1) were found formation of calcium hydro silicates type CSH(B). Presence of low-basic calcium hydro silicates if proved by the endo effects at  $t=185$  and 520°C (dehydration) and exoeffect at  $t=840$  C (recrystallization into the vollastonite).

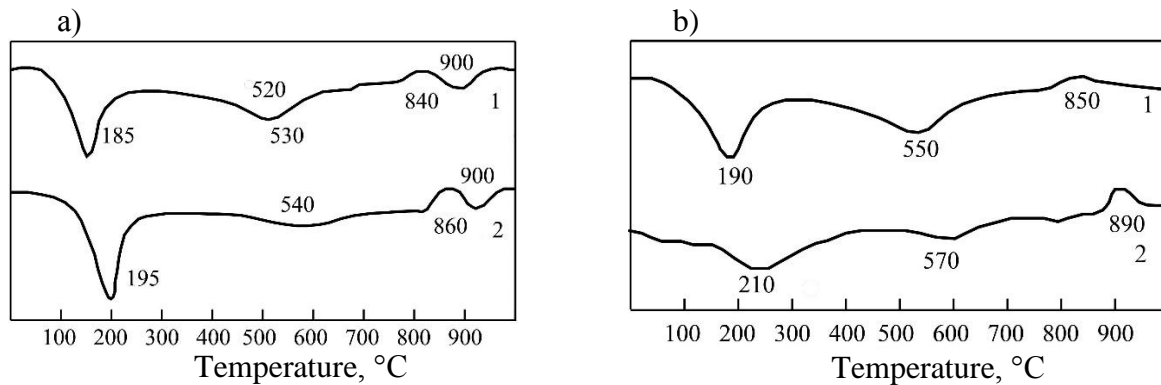


Fig. 1. DTA curves of slag alkali activated cement compositions using soda ash (a) and sodium metasilicate (b) after 28 days of hardening in the normal conditions:

1 – control compositions (CK, MK); 2 – compositions, modified by sodium nitrate (CH, MH)

Beside of hydro silicates, if is also present formation of calcite  $\text{CaCO}_3$ , proving by the endo effect at  $t = 900^\circ\text{C}$  (dissociation). The mentioned above phase composition of hydration products is a typical for slag alkali activated cement [10].

In the case of modifying of slag alkali activated cement by  $\text{NaNO}_3$  salt it is present increasing of crystallinity rate of low-basis hydro silicates because of catalytic function of the admixture, that if proved by shifting of effects to the zone of higher temperature (Figure 1, a, curve 2).

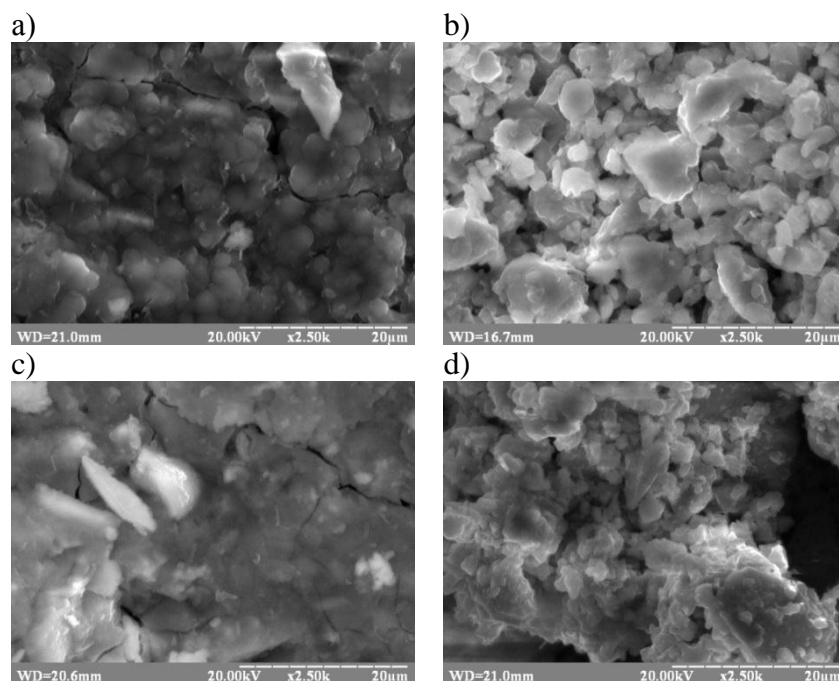


Fig. 2. SEM images of the slag alkali activated cement crack surface after 28 days of hardening: a, c – control compositions using soda ash and sodium metasilicate (compositions «CK», «MK»), b, d – using soda ash and sodium metasilicate, modified by  $\text{NaNO}_3$  admixture (compositions «CH», «MH»)

Results of the SEM analysis are in correspondence with the high rate of crystallinity of hydrated slag alkali activated cement structure, modified by  $\text{NaNO}_3$  (Figure 2, b), comparing to the non-modified analogue (Figure 2, a).

The same regularity in influence of  $\text{NaNO}_3$  admixture on structure formation properties of the slag alkali activated cement if observed in case of using sodium metasilicate as an alkaline component (Figure 1, b, Figure 2, c, d).

*Influence of sodium nitrate of the properties of the slag alkali activated cement. Consistence of the concrete mix met the requirements of P12. W/C ration for slag alkali activated concrete using soda ash and sodium metasilicate were correspondently: for control compositions – 0.39 and 0.37, for modified by  $\text{NaNO}_3$  – 0.40 and 0.37.*

**Strength.** Strength properties of the control composition using soda ash after 28 days of hardening in normal conditions were respectively flexural strength 5.8 MPa (Figure 3, a) and compressive strength 41.2 MPa (Figure 3, b).

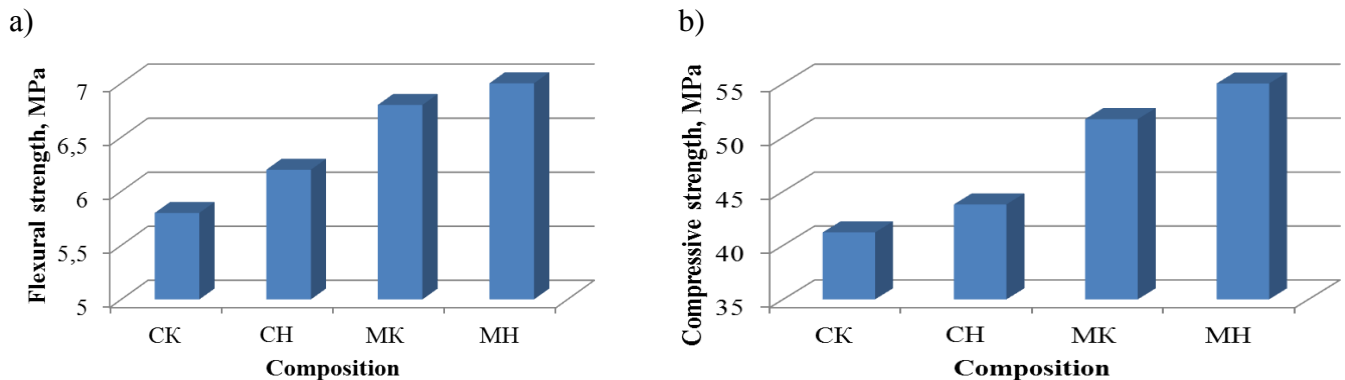


Fig. 3. Mechanical properties of slag alkali activated concrete after 28 days of hardening:  
a – flexural strength; b – compressive strength

Slag alkali activated concrete of the control composition basing on the soda ash after 90 cycles of saturation in the sea water and drying is characterized by flexural strength 5.2 MPa (Figure 4, a) and compressive strength 39.2 MPa (Figure. 4, b). Thus a way, influence of aggressive media leads to decreasing of strength properties of plasticized slag alkali activated concrete using soda ash corresponding to those at the age of 28 days: flexural strength – by 10.3 %, compressive strength –4.9 %.

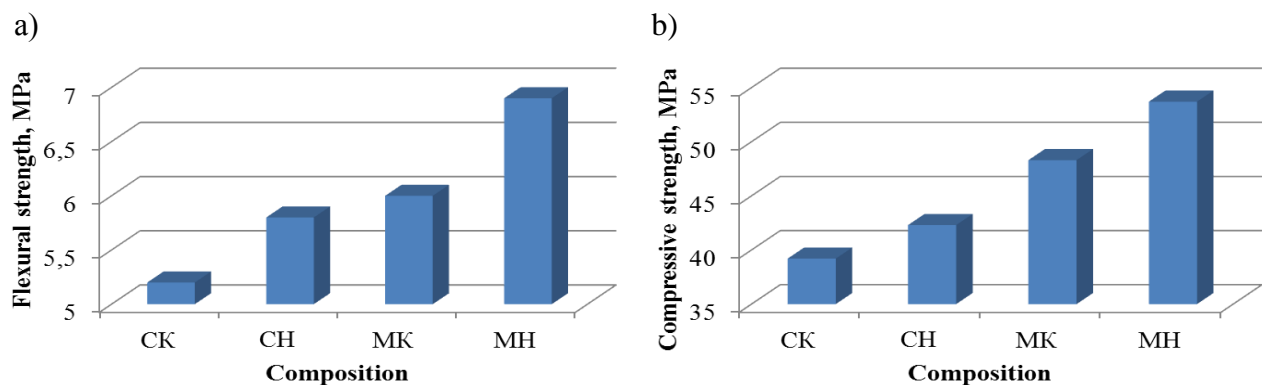


Fig. 4. Mechanical properties of slag alkali activated concrete after 90 cycles of saturating in the sea water and drying:  
a – flexural strength; b – compressive strength

The same regularity of strength changes if observing for the concrete on the sodium metasilicate basis. Comparing to the strength at the age of 28 days such concrete is characterized by flexural strength drop from 6.8 MPa to 6.0 MPa (11.8 %) (Figure 4, a) and compressive strength from 51.7 to 48.3 MPa (6.6 %) (Figure 4, b) after 90 cycles of saturation in the sea water and drying. Significant strength drops of the alkali activated concrete, mainly flexural strength, is provided by high content of gel phase and low speed of hydration product crystallization [11].

Modifying by  $\text{NaNO}_3$  admixture leads to increasing of strength properties of the plasticized concrete. In case of soda ash use flexural strength of concrete is rising from 5.8 MPa to 6.2 MPa

(6.9 %), compressive strength – from 41.2 MPa to 43.8 MPa (6.3 %). The same regularity is observed for the case of modifying of concrete on sodium metasilicate basis: flexural strength is rising from 6.8 to 7.2 MPa (5.9 %) (Figure 3, a), compressive strength – from 51.7 to 55.1 MPa (8.5 %) (Figure 3, b). Increasing of strength properties of alkali activated concrete modified by sodium nitrate admixture is the result of increasing of crystallinity grade of microstructure. The given data is in correlation with the results of studies on influence of  $\text{NaNO}_3$  on properties of slag alkali activated concrete [7, 8].

Modifying of slag alkali activated concrete by  $\text{NaNO}_3$  admixture leads to reducing of negative influence of saturation in the sea water and drying. Thus, use  $\text{NaNO}_3$  provides increasing of flexural strength from 5.2 to 5.8 MPa (11.5 %) and compressive strength from 39.2 to 42.3 MPa (7.9 %) of slag alkali activated concrete after 90 cycles of saturation and drying. Using of such salt admixture in the concrete mix using sodium metasilicate leads to the increasing of flexural strength from 6.0 to 6.9 MPa (15.0 %) and compressive strength from 48.3 to 53.7 MPa (11.2 %).

Increasing of strength characteristics of plasticized slag alkali activated concrete under modification by  $\text{NaNO}_3$  admixture in the condition of periodically influence of sea water is caused by compacting of microstructure, resulting in increasing of hydration new formations crystallinity grade.

*Pore structure.* It was set, that modifying of concrete on soda ash bases with the fixed total porosity value leads to reducing of capillary pores volume for 11.9 % and increasing of volume of closed pores for 34.8 % after 90 cycles of saturation in the sea water and drying (Table. 2). Perfecting of the pore structure is also observed in the case of modifying of slag alkali activated concrete using sodium metasilicate: capillary pores volume is reducing for 22.9 %. In that case salt admixture provides reducing of total porosity value from 9.6 to 8.8 %.

Table 2 – Characteristics of the pore structure of slag alkali activated concrete after 90 cycles of saturating in the sea water and drying

Composition number	Mean density of the dry concrete, $\text{kg/m}^3$	Volume of the open capillary pores, %	Volume of the closed pores, %	Total pores volume, %
CK	1950	6.7	2.3	9.0
CH	1990	5.9	3.1	9.0
MK	1985	6.1	3.5	9.6
MH	2040	4.5	4.3	8.8

Changes in the pore structure leads to the compaction of concrete structure using soda ash and sodium metasilicate by 2.1 and 2.7 % respectively. Formation of the tensor structure using sodium metasilicate could be explained by higher hydro silicate gel volume, crystallization of which is increasing in case of  $\text{NaNO}_3$  presence.

*Carbonation depth.* Average carbonation depth of slag alkali activated concrete specimens of the control composition after 90 cycles of saturation and drying is 2 mm regardless of alkaline component type. Specimens of alkali activated concrete, modified  $\text{NaNO}_3$ , are characterizing by carbonation depth 1.5 mm using soda ash and 1.0 mm using sodium metasilicate.

### Conclusions:

1. Determined effectiveness of carbonation of plasticized slag alkali activated concrete prevention in the conditions of periodically influence of sea water by using of modifying admixture represented by  $\text{NaNO}_3$ .
2. Increasing of crystallinity of hydrated new formations under introducing of  $\text{NaNO}_3$  admixture leads to the changes of pore structure, resulting in formation of more tensile structure, rising of strength and reducing of carbonation depth.
3. In the conditions of media, represented by combination of XC4 and XS3 classes of influence, it was found intensive degradation of plasticized slag alkali activated concrete in case of use of sodium metasilicate as an alkaline component of the cement as a result of high gel phase content comparing to the case of use of soda ash, proving by increasing of flexural strength drop

from 10.3 to 11.8 % after 90 cycles of saturation in the sea water and drying.

4. Increasing of gel phases crystallization rate leads to the higher effectiveness of resistance to the aggressive media in the presence of sodium metasilicate comparing to the soda ash and it is proved by reducing of closed pores volume from 26.2 to 11.9 % and increasing volume of closed pores from 22.9 to 34.8 % in the plasticized slag alkali activated concrete, modified by  $\text{NaNO}_3$  admixture after 90 cycles of saturation in the sea water and drying. Perfecting of the pore structure under change of anion part of the alkaline component from carbonate to the metasilicate leads to increasing of the density from 2.1 to 2.7 %, increasing of flexural strength from 11.5 to 15.0 % and compressive strength from 7.9 to 11.2 %, and also reducing of carbonation speed from 50 to 25 %.

**Acknowledgement.** Authors would like to thank for the financial support of the study, providing under the budget project № 1020U001010, and also according to the joint project of Scientific Community of Lithuanian and Ministry of science and Education of Ukraine «Solutions to reduce aggressive ions  $\text{SO}_4^{2-}$  and  $\text{Cl}^-$  transport in Portland cement concrete with steel reinforcement in hydraulic structures», project code S-LU-22-7.

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**РОЛЬ НІТРАТУ НАТРІЮ В ПРОТИДІЇ КАРБОНІЗАЦІЇ  
ПЛАСТИФІКОВАНОГО ШЛАКОЛУЖНОГО БЕТОНУ  
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**Анотація.** Карбонізація шлаколужного бетону морських споруд при періодичному впливі морської води в реальних умовах експлуатації обумовлює підвищений ризик деградації внаслідок високого вмісту гелевої фази. Агресивність середовища в разі висушування в умовах дії атмосферної вуглекислоти і зволоження в морській воді описується поєднанням класів впливу XC3 і XS4. Використання бетону з високорухомих сумішей згідно вимогам сучасної будівельної практики підсилює ризик руйнування морських конструкцій.

Метою роботи є визначення ефективності протидії карбонізації пластифікованого шлаколужного бетону в умовах періодичного впливу морської води шляхом модифікації добавкою натрію  $\text{NaNO}_3$ .

За допомогою методів фізико-хімічного аналізу показано підвищений ступінь закристалізованості гідратних новоутворень шлаколужного цементу як на соді кальцинованій, так і метасилікату натрію.

Оцінка стану пластифікованого шлаколужного бетону, що зазнає карбонізації в умовах періодичного впливу морської води, здійснювали згідно запропонованої авторами методики. Властивості бетону визначали після 90 циклів зволоження в морській воді і висушування при атмосферній концентрації  $\text{CO}_2 \sim 0,04 \%$  та температурі  $105^\circ\text{C} \pm 3^\circ\text{C}$ . Показана деградація бетону, яка підтверджується падінням міцності на згин. Більше падіння міцності на згин при використанні метасилікату натрію як лужного компонента (на 11,8 %) порівняно з содою кальцинованою (на 10,3 %) обумовлено більшим вмістом гелевої фази.

Ефективність добавки  $\text{NaNO}_3$  в пластифікованому шлаколужному бетоні після 90 циклів зволоження в морській воді і висушування підтверджена позитивним впливом на властивості. Підвищення ступеня закристалізованості гелевої фази обумовлює більшу ефективність протидії агресивному середовищу в присутності метасилікату натрію порівняно з содою кальцинованою. Зміна аніонної складової лужного компонента з карбонатної на силікатну призводить до зменшення об'єму відкритих капілярних пор з 26,2 до 11,9 % і підвищення об'єму умовно закритих пор з 22,9 до 34,8 % в шлаколужному бетоні, модифікованому добавкою нітрату натрію. Вказане поліпшення порової структури, обумовлене природою лужного компонента, супроводжується збільшенням щільності з 2,1 до 2,7 %, міцності на згин з 11,5 до 15,0 і міцності на стиск з 7,9 до 11,2 %. При цьому спостерігається сповільнення швидкості карбонізації з 25 до 50 %.

**Ключові слова:** шлаколушний бетон, нітрат натрію, карбонізація, морська вода, порова структура.

Стаття надійшла до редакції 12.05.2022