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DEVELOPMENT OF CONCRETE COMPOSITIONS BASED ON ALUMINOCHROMITE CEMENTS

Korohodska A. M., Shabanova H. M., Logvinkov S. M.

INTRODUCTION

The intensification of industrial thermal processes in various branches of science and technology requires the creation of new structural materials that, in addition to exposure to high temperatures, can withstand the aggressive effects of melts, electrolyte solutions, and high pressure. Refractory materials that currently exist are mostly piece products and require additional seam fastening, which significantly reduces the service life. Replacing artificial refractories with a monolithic seamless concrete lining allows you to meet all the necessary requirements for the operational reliability of a thermal unit¹.

The main directions in the development of technological solutions for the production of refractory unshaped materials is the selection of the optimal qualitative and material composition of the filler, which ensures the formation of the necessary physical, mechanical and technical characteristics of the working layer of the lining when heated to the operating temperature².

1. Previously investigations

In the laboratory of special binders and composite materials of NTU «KhPI» compositions of calcium, strontium and barium aluminochromite cements were developed. According to the results of studies of physical and mechanical properties, it was found that the resulting cements are high-strength – up to 70 MPa, fast-setting – the beginning of setting is from 8 min up to 2 hours and 5 min, the end of setting – from 25 min up to 3 hours and 20 min; fast-hardening – compressive strength after 1 day of hardening up to 45 MPa; it's binders of both air and hydraulic hardening with a water-cement ratio of 0.18–0.22³. Thus, the synthesized cements, according to the totality of physical, mechanical and technical characteristics, can be recommended for the creation of refractory corrosion-resistant concretes.

¹ Vert T., Smith J.D. Refractory Material Selection for Steelmaking. Wiley & Sons, 2016. 390 p.

² Руденко Д. В. Фізико-хімічні основи технології модифікованого бетону для споруд спеціального призначення : монографія. Дніпро : Герда, 2018. 344 с.

³ Shabanova G. N., Korohodska A. N. Alkali-earth Element Aluminates and Chromites Cement Bonded Refractory Castables. *China's Refractories*. 2016. Vol. 25, No 1. Pp. 26-31.

2. Optimization of the particle size distribution of the aggregate

The main directions in the development of technological solutions for the production of refractory unshaped materials are the selection of the optimal qualitative and material composition of the filler, which ensures the formation of the necessary physical, mechanical and technical characteristics of the working layer of the lining when heated to the operating temperature.

Due to the impossibility of the coexistence of the main components of aluminochromite cements with Al_2O_3 , which does not allow the use of corundum in the composition of these unshaped materials, it is necessary to use periclase as an alternative refractory filler, which is characterized by high thermal and thermodynamic stability, as well as the ability to form aluminate and chromite spinel at high temperatures compounds that enhance the high temperature performance of developed materials.

To obtain concrete of high strength, density and uniformity, the selection of the optimal granulometric composition of the aggregate was carried out. Samples-cubes with a size of $5 \times 5 \times 5$ cm, were made by vibratory laying from a concrete mixture with a water-hard ratio of 0.08. Optimization of the quantitative ratio of adjacent aggregate fractions was carried out using the simplex-lattice method of experiment planning⁴.

To describe the dependence of the strength of concrete on the quantitative ratio of the aggregate fraction, an incomplete third-order polynomial was used. Based on the results of the experimental data obtained, the coefficients of the polynomial expressing the dependence of strength and porosity on the quantitative and granulometric ratio of the aggregate fractions were calculated.

The regression equations are as follows: - for calcium aluminochromite cement $Y_{\sigma} = 43.9x_1 + 45.0x_2 + 51.3x_3 + 21.4x_{12} + 10.4x_{13} - 3.8x_{23} + 69.0x_{123}$ $Y_p = 15.7x_1 + 4.2x_2 + 9.9x_3 + 3.8x_{12} + 8.0x_{13} - 7.0x_{23} - 51.3x_{123}$ - for strontium aluminochromite cement $Y_{\sigma} = 44.4x_1 + 47.2x_2 + 48.8x_3 - 1.2x_{12} - 2.8x_{13} + 7.6x_{23} + 78.3x_{123}$ $Y_p = 15.3x_1 + 14.0x_2 + 12.2x_3 - 5.4x_{12} - 7.6x_{13} - 8.6x_{23} - 2.7x_{123}$ - for barium aluminochromite cement $Y_{\sigma} = 51.2x_1 + 53.5x_2 + 56.1x_3 + 8.2x_{12} + 12.2x_{13} + 10.0x_{23} + 19.5x_{123}$ $Y_p = 15.7x_1 + 14.4x_2 + 12.4x_3 - 4.6x_{12} - 9.0x_{13} - 10.0x_{23} - 20.1x_{123}$

where x_1 , x_2 , x_3 – aggregate fractions with grains size (2.5 – 1.25) mm, (1.25 – 0.63) mm and (> 0,63) mm, respectively.

⁴ Konitufe C., Elinwa A. U., Duna S. Simplex Lattice Method: A Predictive Tool for Concrete Materials. *American Journal of Engineering Research*. 2017. Vol. 6. Pp. 19-27.

The adequacy of the equation was checked using the Student's criterion and setting up additional control experiments. The regression equation was calculated using a computer with a step of variation of 10 wt. %. Based on the results of the calculations and mathematical processing of the results of the experiment, simplex diagrams «composition – strength» and «composition – porosity» and projections of lines of equal level for refractory concretes based on aluminochromite cements are constructed, shown in Fig. 1–6.

From the presented results it can be seen that the course of lines of an equal level of simplex diagrams for both strength and porosity for all types of aluminochromite cements is similar. The data obtained show the adequacy of the selection of the optimal particle size distribution of the aggregate. From the results of the calculation it can be seen that in order to obtain concrete of high strength, density and uniformity, a three-fraction mixture of aggregate with the following quantitative size ratio is required grains: fraction (2.5 - 1.25) mm - 0 - 40 wt. %, (1.25 - 0.63) mm - 20 - 50 wt. %, > 0,63 mm - 20 - 70 wt. %.



Fig. 1. Diagram «composition – strength, MPa» for concrete based on calcium aluminochromite cement



Fig. 2. Diagram «composition – strength, MPa» for concrete based on strontium aluminochromite cement



Fig. 3. Diagram «composition – strength, MPa» for concrete based on barium aluminochromite cement



Fig. 4. Diagram «composition – porosity, %» for concrete based on calcium aluminochromite cement



Fig. 5. Diagram «composition – porosity, %» for concrete based on strontium aluminochromite cement



Fig. 6. Diagram «composition – porosity, %» for concrete based on barium aluminochromite cement

With this ratio of adjacent aggregate fractions, strength values of up to 50 MPa and porosity of up to 12% are achieved^{5,6}.

Subsequent studies of concretes were carried out taking into account the optimal particle size distribution of the aggregate.

3. Investigation of physico-mechanical and technical properties of refractory concretes based on aluminochromite cements

In order to determine the optimal quantitative ratio of cement: aggregate in concrete, an experiment was carried out on concrete samples measuring $5\times5\times5$ cm, made by vibration compaction at W/S (water-solid ratio) = 0.08. In Table 1 presents the results of physical and mechanical tests of concrete, depending on the ratio of cement and aggregate in it.

From the above results it can be seen that with an increase in the cement content in the composition of the concrete mixture, the strength of the hardened concrete increases, and the porosity, on the contrary, decreases. This suggests that the introduced cement is actively involved not only in the formation of concrete strength, but also leads to a decrease in porosity⁷.

⁵ Korogodskaya A. Refractory concretes resistant to melted slag action. *18 International Baustofftagung*, 12-15 September, 2009. Tagungsbericht. Band 2. Weimar, Bundesrepublik, Deutschland, 2012. Ss. 696-702.

⁶ Шабанова Г. М., Корогодська А. М. Вогнетривкий бетон на основі шпінельвмісного цементу. *Будівельні матеріали, вироби та санітарна техніка*. Київ: Знання, 2012. Вип. 43. С. 144-149.

⁷ Kurdowski W. Cement and Concrete Chemistry. Heidelberg (DE): Springer, 2014. 700 p.

Table 1

Type	Cement: aggregate	Compressive s ag	Porosity, %					
of cement	ratio	7 days	28 days					
CACr-cement	1:3	43,6	54,6	11,0				
	1:4	40,2	52,3	11,6				
	1:5	37,2	45,3	13,8				
SrACr- cement	1:3	40,3	50,8	10,8				
	1:4	38,0	49,5	11,2				
	1:5	30,4	41,2	14,2				
BACr-cement	1:3	56,5	59,3	10,3				
	1:4	55,0	56,4	10,9				
	1:5	41,8	49,3	13,2				

Dependence of the strength and porosity of concrete on the ratio cement: aggregate

The optimal ratio that provides the necessary strength and economy of cement is the ratio of cement : aggregate 1: 4.

To obtain concrete with high operational suitability, an analysis of the dependence of the mechanical strength of concrete on molding methods was carried out. For this purpose, samples of $5 \times 5 \times 5$ cm in size were formed on the basis of aluminochrome cements of optimal composition with periclase filler of a given fractional composition. The results of the research are presented in Table 2.

Table 2

The influence of	technological factors on the strength of concrete
	(ratio cement: $aggregate = 1:4$)

Type	Molding method	W/S	Compressive strength, MPa,			
of comont			aged, day			
of cement			1 3 7	7	28	
	Manual tamping		8,0	23,0	36,2	46,3
CACr- cement	Vibration compaction	0,12	9,0	26,4	40,2	52,3
	Pressing		10,5	27,7	45,6	54,6
SrACr- cement	Manual tamping		5,0	19,2	30,3	41,0
	Vibration compaction	0,08	7,5	23,6	38,0	49,5
	Pressing		8,2	26,2	40,5	50,7
BACr- cement	Manual tamping		6,5	23,7	45,6	50,3
	Vibration compaction	0,06	11,4	28,2	55,0	56,4
	Pressing		14,5	28,7	55,5	58,8

As can be seen from the presented results, the optimal method of molding is pressing. However, this method is suitable for the manufacture of piece products, for the manufacture of large-sized products, vibration compaction of the material should be used.

Since it is impractical to use only one type of aggregate to obtain refractory unshaped materials, it is proposed to use natural chromite concentrate as an alternative, and studies on the use of corundum have been carried out for comparison, since low-melting eutectic is formed only when using barium cement. As an alternative, aggregates are also used artificially obtained monochromites of alkaline earth elements. Aggregates were used fractional composition, presented in sect. 2. Physical and mechanical properties of concretes based on aluminochromite cements with various aggregates are presented in Table 3.

From the results obtained, it can be seen that the most optimal aggregate for concrete on all types of cements is periclase. When corundum is used as a filler, there is a significant decrease in strength. All other things being equal, artificial aggregates are more preferable to natural ones, since no changes occur during hardening with aggregate⁸.

From the point of view of fire resistance, periclase having the highest melting point are also the most optimal. It should be noted that for barium aluminochromite cement, the use of corundum and barium chromite as aggregates is unacceptable, since the refractoriness of the unshaped material is reduced by an average of 500°C.

Determination of the temperature of the beginning of softening of concretes based on aluminochromite cements with periclase aggregate was carried out on a certified test facility in accordance with ISO 528:1983 «Refractory products. Determination of pyrometric cone equivalent (refractoriness)»⁹. The deformation of the test samples was measured by a differential method using a differential measuring device made of single-crystal corundum. During the test, a load a corresponding to the pressure (0.20 ± 0.01) N/mm² is applied to each of the samples.

The rate of increase in the heating temperature of the test samples was: not more than 10° C/min – at temperatures up to 300° C; 4.5° C/min – at a temperature of more than 300° C.

The results obtained show that, as for conventional periclase refractories, the temperature of the onset of deformation under the load of the developed unshaped materials is in the range of $1500 - 1550^{\circ}$ C. Since this characteristic of the refractory material depends on porosity, it can be argued that the selection of the

 $^{^{8}}$ Handbook of Composites / Ed. by S. T. Peters. Tornbridge (England): Springer Science, 2013. 1069 p.

 $^{^9}$ ISO 528:1983 (1983) Refractory products. Determination of pyrometric cone equivalent (refractoriness). ANSI Intern. Doc.

quantitative particle size distribution of the aggregate is carried out adequately, providing effective values of the porosity of the products.

Туре	Aggregate type	Compressive strength, MPa, aged, day				Porosi	Refract
of cement		1 day	3 days	7 days	28 days	ty, %	°C
	Periclase	9,0	26,4	40,2	52,3	11,6	2000
CACr- cement	Chromite	8,0	25,2	30,6	40,5	12,0	2000
	Corundum	7,8	24,1	25,8	36,5	12,0	1950
	Calcium chromite	8,5	26,8	38,3	45,0	11,8	2000
	Periclase	7,5	23,6	38,0	49,5	11,2	1900
SrACr-	Chromite	7,0	20,4	32,2	41,1	11,8	1800
cement	Corundum	6,5	22,3	25,4	30,5	12,0	1850
	Strontium chromite	7,2	21,8	30,2	42,6	11,6	1900
BACr- cement	Periclase	11,4	28,2	55,0	56,4	10,9	1800
	Chromite	10,7	24,1	43,6	48,2	11,0	1700
	Corundum	6,1	15,2	17,8	22,3	12,8	1570
	Chromite barium	11,4	20,8	31,2	49,4	10,8	1400

Dependence of the strength characteristics of concrete on the type of aggregate (ratio cement: aggregate = 1 : 4)

The nature and course of the deformation curves of unshaped materials indicate that they consist of crystals that do not form an intergrowth and have a less refractory crystalline bond that melts at a temperature much lower than the melting point of the refractory aggregate, the crystals of the main phase of which dissolve little in the resulting melt. The amount of the liquid phase at the temperature of determination of refractoriness in this case is 5-8 wt. %.

The obtained data are confirmed by the results of microscopic analysis (Fig. 7).

As can be seen from the presented results, before firing, all concrete samples have a dense, almost non-porous structure. In the total mass, colorless grains of the correct shape are distinguished, belonging to the coarse fraction of the aggregate. The bulk is represented by small aggregate grains and hydration products of aluminochromite cement. After firing, it can be seen that only the binder part, represented by hydrated cement and fine aggregate, undergoes a change. There is a residual interaction, leading, especially in barium concrete, to the formation of a melt.

Table 3

Results of X-ray analysis of concretes after firing identify peaks related to the main phases of both the aggregate and the hydraulic component (Fig. 8 - 10).

For CACr-concrete (Fig. 8), the peaks $CaCr_2O_4$ (d = 0.553; 0.363; 0.276; 0.253; 0.23; 0.186; 0.149 nm) and $CaAl_2O_4$ (d = 0.467; 0.317; 0.297; 0.296; 0.253; 0.252; 0.239; 0.219; 0.188 nm) are the basis of cement, as well as MgO (d = 0.243; 0.210; 0.149; 0.127; 0.121 nm) aggregate. On the X-ray diagram, slight peaks of aluminum-magnesia spinel MgAl₂O₄ (d = 0.446; 0.286; 0.244; 0.202; 0.1 56; 0.1429 nm), which is formed by the interaction of periclase with aluminum hydroxide of hydrated cement stone.

SrACr-concrete (Fig. 9) is characterized by the peaks of $SrCr_2O_4$ (d = 0.582; 0.386; 0.366; 0.291; 0.274; 0.255; 0.233; 0.192; 0.147 nm), SrAl₂O₄ (d = 0.444; 0.441; 0.391; 0.314; 0.305; 0.298; 0.257; 0.255; 0.210; 0.193 nm) and $Sr_3Al_2O_6$ (d = 0.280; 0.289; 0.198; 0.162; 0.140 nm), which are the basis of cement and MgO (d = 0.243; 0.210; 0.149; 0.127; 0.121 nm) aggregate. There are no traces of interaction between cement and aggregate.



b)

Fig. 7. Microfotographs of concretes based on aluminochromite cements with periclase (magnification×22): a) before firing;
b) after firing at a temperature of 1500°C for 6 hours

a)





For BACr-concrete (Fig. 10), the peaks $BaAl_2O_4$ (d = 0.315; 0.261; 0.226; 0.201; 0.179 nm) and $Ba_3Cr_2O_6$ (d = 0.476; 0.358; 0.320; 0.303; 0.213; 0.195; 0.173 nm) are characteristic, which are the basis of cement and MgO (d =0.243; 0.210; 0.149; 0.127; 0.121 nm) aggregate. Slight peaks are also observed on the X-ray diagram chromium-magnesia spinel MgCr₂O₄ (d = 0.481; 0.251; 0.208; 0.160; 0.147 nm), which is formed by the interaction of periclase with chromium hydroxide of hydrated cement stone.

The heat resistance of unshaped materials is of paramount importance, since failure at high temperatures determines their operational application. In this work, the heat resistance of concrete based on aluminochromite cements with periclase was determined (heating – up to 1300°C, cooling of the samples was carried out in air). As a result of the tests, it was found that all concrete structures withstood more than 2-5 cycles, while retaining more than 80% of the original strength.



One of the important performance characteristics of unshaped materials is the degree of its softening when heated. To study the mechanical strength of concrete under the influence of elevated temperatures, samples of $5 \times 5 \times 5$ cm in size were made, which were tested after 28 days of hardening. Isothermal exposure – 4 hours at a given temperature. As a filler, used periclase. The results of tests of concrete under the influence of elevated temperatures are presented in Fig. 11.



---- CACr-concrete ---- SrACr-concrete ---- BACr-concrete

Fig. 11. Dependence of the degree of softening of aluminochromite concretes from temperature

As can be seen from the presented results, concrete with periclase on aluminochromite cements behaves approximately the same. The greatest increase in the degree of softening of concretes is observed in the temperature range from 500 to 700°C, which is associated with the removal of bound water from cement

stone. Above 700°C, the increase in the degree of softening slows down and stabilizes by 800°C in the region of 11-17%. Over 1000°C begins the sintering of the material with the production of a strong ceramic structure and an increase in strength. The increased degree of softening for concrete on calcium aluminochromite cement is explained by the significant removal of bound water from cement hydrate neoplasms in a limited temperature range compared to strontium and barium analogues. The loss of strength of concrete based on alumina and high-alumina cement with traditional aggregates in the temperature range up to 500°C is about 30-40 % of the original, which is 4 times higher than that of the developed unshaped materials¹⁰.

An important characteristic of a material operating at high temperatures is the thermal coefficient of linear expansion (TCLE), since it must be close to the TCLE of the material during repairs and suture work. In this regard, the TCLE of concretes based on aluminochromite cements with periclase was determined. Samples of size d = 8 mm, h = 50 mm were made to determine the TCLE. The cement: aggregate ratio in the test samples was 1:4. Studies of the thermal coefficient of linear expansion of concrete based on aluminochromite cements with periclase show that in the temperature range from 20 to 1000°C, the TCLE is equal to $(8.4 \div 11.6) \cdot 10^{-6} \text{ K}^{-1}$, which satisfies the requirements of the pits for suture materials. The size of the sample volume is 1.0 - 1.1%.

To determine the corrosion resistance of the obtained concretes, the following were used as corrosive agents: the alkali blast furnace granulated slag of JSC «MP Zaporizhstal» ($M_o = 1.15$), iron ore pellets of JSC «Poltava Mining and Processing Plant», precision heat-resistant alloy with high electrical resistance (kanthal) X23Yu5T. To determine the depth of impregnation with a corrosive agent from a concrete mixture, samples were pressed in the form of cubes with a rib size of 5 cm with a cylindrical recess 25 cm in diameter and 30 cm deep. The obtained crucibles hardened for 3 days in air conditions. The resulting crucibles hardened for 3 days in air conditions. After that, the following was melted: slag – at a temperature of 1450°C for 2 hours, iron ore pellets – at a temperature of 1550°C for 3 hours, kanthal – at a temperature of 1600°C for 3 hours. After cooling, the crucibles were sawn through the center of the recess and the depth of impregnation was assessed. Microfotographs of concrete samples after melting of corrosive agents are shown in Fig. 12.

It is established that for the alkali slag the depth of corrosion for all types of concrete is 2 mm. During the melting of iron ore pellets and kanthal, no interaction between the material and the metal was observed.

 $^{^{10}}$ Composite Materials: Design and Applications (3rd Edition) / Ed. by D. Gay. Boca Raton: CRC Press, 2014. 635 p.

Thus, the developed concretes based on aluminochromite cements are corrosion-resistant to the action of metal melts and slags and can be used to create linings for the metallurgical industry.



a) the boundary of CACr-concrete - slag



c) the boundary between BACr-concrete and slag



b) SrACr-concrete - slag boundary



d) the boundary of CACr-concrete – metal

Fig. 12. Microfotographs of concrete samples after melting corrosive agents (magnification ×22)

The main physical, mechanical and technical properties of the developed unshaped materials based on aluminochromite cements are presented in Table 4.

Table 4

Indicators	Concrete based on periclase aggregate and aluminochromite cement				
	calcium	strontium	barium		
Compressive strength at the age of 28 days of hardening, MPa	52	49	56		
Open porosity, %	11,6	11,2	10,9		
Refractoriness, °C	2000	1900	1800		
Thermostability 1300°C – water, flat		> 25			
Temperature of the beginning of deformation under load 0.2 MPa, °C	1560	1540	1510		
Thermal coefficient of linear expansion, K^{-1}	$(8,4 \div 11,6) \cdot 10^{-6}$				
The degree of softening in the temperature range 20–1000°C,%	17,6	14,3	11,4		
Corrosion resistance (depth of					
penetration), mm					
 alkali blast furnace slag 	2				
– metal melt	_				

Basic physical, mechanical and technical properties of the developed refractory unshaped materials on the basis of aluminochromite cements

Thus, developed refractory concretes based on aluminochromite cements and electrofused periclase can be recommended for further use in thermal units and installations of refractory and metallurgical enterprises.

CONCLUSIONS

1. Compositions of high-strength refractory concretes based on aluminum chromite cements with various aggregates have been developed, which are characterized by the following indicators: compressive strength at the age of 28 days of hardening -49 - 56 MPa; open porosity -10.9 - 11.6%; refractoriness $-1800 - 2000^{\circ}$ C; heat resistance 1300° C - water - more than 25 heat shifts; the temperature of the beginning of deformation under load 0.2 MPa $-1510 - 1560^{\circ}$ C; thermal coefficient of linear expansion $-(8.4 \div 11.6) \cdot 10^{-6}$ K $^{-1}$; degree of softening in the temperature range 20 -1000° C -11.4 - 17.6%; slag and metal resistance (by penetration depth): for the alkali blast furnace slag -1.8 - 2 mm, for molten metal - none.

2. Physical and chemical studies of concretes based on aluminochromite cements have been carried out and it has been established that before firing, all concrete samples have a dense, almost pore-free structure. In the total mass, colorless grains of the correct form are distinguished, related to the large fraction of the aggregate. The bulk is represented by small aggregate grains and hydration products of aluminochromtin cement. After firing, it can be seen that only the binder part, represented by hydrated cement and fine aggregate, undergoes a change. There is a residual interaction, leading, especially in barium concrete, to the formation of a melt. Results of the X-ray analyses of concrete after firing identify peaks related to the main phases of both the aggregate and the hydraulic component. For CACr-concrete, the presence of aluminum-magnesia spinel is observed, which is formed by the interaction of periclase with aluminum hydroxide of hydrated cement stone. For SrACr-concrete, no traces of interaction between cement and aggregate are observed. For BACr-concrete, the presence of chromium-magnesia spinel is observed, which is formed by the interaction of periclase with chromium hydroxide of hydrated cement stone.

SUMMARY

Based on the regression analysis data and the results of physical and mechanical tests, the compositions of aluminochromite cements were optimized and the compliance of their characteristics with the requirements of regulatory documentation was established: the normal density of the cement paste is 23-29%; setting time: start – from 40 minutes to 2 hours, end – from 55 minutes to 4 hours 15 minutes; compressive strength at the age of 28 days of hardening 57 – 75 MPa; ultimate strength in bending at the age of 28 days of hardening 6.2 - 6.8 MPa, fire resistance – $1700 - 1900^{\circ}$ C.

Compositions of high-strength refractory concretes based on aluminochromite cements with various aggregates have been developed, which are characterized by high performance: compressive strength at the age of 28 days of hardening -49 - 56 MPa; open porosity -10.9 - 11.6%; fire resistance -1800 - 2000 ° C; heat resistance 1300 °C - water - more than 25 thermal cycles; the temperature of the beginning of deformation under a load of 0.2 MPa -1510 - 1560 ° C; thermal coefficient of linear expansion $-(8.4 \div 11.6) \cdot 10^{-6}$ K⁻¹; the degree of softening in the temperature range 20 - 1000 °C - 11.4 - 17.6%; slag and metal resistance (according to the penetration depth) for the alkali blast-furnace slag - 2 mm, for the metal melt - none.

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Information about the authors: Korohodska Alla Mykolaivna,

Doctor of Technical Sciences,

Head of the Department of General and Inorganic Chemistry National Technical University «Kharkiv Polytechnic Institute» 2, Kyrpychova str., Kharkiv, 61002, Ukraine

Shabanova Halyna Mykolaivna,

Doctor of Technical Sciences, Professor, Professor at the Department of Ceramics, Refractory Materials, Glass and Enamels Technology National Technical University «Kharkiv Polytechnic Institute» 2, Kyrpychova str., Kharkiv, 61002, Ukraine

Logvinkov Sergii Mykhaylovych,

Doctor of Technical Sciences, Senior Researcher, Professor at the Department of Hotel and Restaurant Business Simon Kuznets Kharkiv National University of Economics, 9-A, Nauky ave., Kharkiv, 61002, Ukraine