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OPTIMIZING THE COMPOSITIONS OF REFRACTORY CEMENTS PRODUCED USING THE WASTE OF CHEMICAL INDUSTRY

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The article presents the results of optimization of the quantitative compositions of cobalt-containing calcium-aluminate and barium-aluminate special cements prepared from chemical industry wastes. Based on the obtained experimental data, the coefficients of the polynomial were computed which express the dependence of the ultimate compression strength and the melting temperature on the quantitative ratio of the $CaAl_2O_4$, $CaAl_4O_7$, and $CoAl_2O_4$ phases for calcium-aluminate cement and the quantitative ratio of the $BaAl_2O_4$, $BaAl_{12}O_{19}$, $CoAl_2O_4$ phases for barium-aluminate cement. The «composition–property» diagrams and the projections of the lines of the same level were plotted for the ultimate compressive strength and the melting temperature of the obtained cements. The following promising areas were selected for the CaO–CoO–Al_2O_3 system (wt.%): 25–55 $CaAl_2O_4$, 15–35 $CaAl_4O_7$, and 25–45 $CoAl_2O_4$; and for the BaO–CoO–Al_2O_3 system (wt.%): 60–20 $BaAl_2O_4$, 10–20 $BaAl_{12}O_{19}$, 30–60 $CoAl_2O_4$. The main physical-mechanical properties of the developed cements of optimal composition are the following: the fineness of grinding is characterized by the total passage through the sieve No 006; for calcium cements: water-cement ratio of 0.2, the setting time: the initial set of 1 h 10 min, and the final set of 5 h 40 min; the ultimate compression strength after hardening of 28 days of 63 MPa; the for barium cements: water-cement ratio of 0.16; the setting time: the initial set of 1 h 50 min, and the final set of 5 h 00 min; the ultimate compression strength after hardening of 28 days of 66 MPa; and the calculated mass absorption coefficient μ=175 cm^2/g. The refractoriness is 1630^{0}C and 1750^{0}C for calcium cement and barium cement, respectively.

Keywords: special cement, industrial wastes, refractoriness, compressive strength, optimization, regression equation, «property vs. composition» diagram.

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Introduction

The main tasks of the cement industry are to upgrade the cement quality and improve the efficiency of its production. In this context, special attention is paid to the development of the new types and compositions of refractory cements and refractory concretes based on them that possess special properties, in particular high strength, refractoriness, long time performance and a possibility to operate in high temperature ranges and at variable temperatures.

The most dynamic directions in the field of creating unshaped materials is the use of materials based on spinel compounds, the presence of which gives the final material an increased slag and metal resistance. In addition, these compounds are not hydraulically active and have high melting points. This is a prerequisite for the creation of refractory binders with reduced water demand. This technological solution is especially relevant for concretes based on alumina binders. The addition of spinel allows not only stabilizing the main characteristics of alkaline-earth cement, but also imparting to it some special properties as follows: increased strength, low porosity, reduced degree of softening at high temperatures, and resistance to aggressive media [1,2].

Over the last years, different branches of industry have been increasingly using the waste-based cements [3,4]. Today, such developments are done using either depleted raw materials or initial raw materials of a low quality. It allows reducing the consumption of fuel and energy resources, decreasing the ecological burden on the industrial regions of the country and improving simultaneously the commercial properties of the obtained materials.

Previous theoretical and experimental investigations of the three-component cobalt-bearing $CaO-CoO-Al_2O_3$ and $BaO-CoO-Al_2O_3$ systems enabled their triangulation and the detection of promising areas for the production of the cements of a new class with high operation performances based on the compositions of the above systems [5–8]. The selection of promising areas of systems was based on the assumption that the production of high-strength and refractory cements requires refractory ($BaAl_{12}O_{19}$ and $CoAl_2O_4$) and hydraulically active ($CaAl_2O_4$, $CaAl_4O_7$, and $BaAl_2O_4$) coexisting phases in their composition [9].

Calculations

In order to create high-strength refractory cobalt-containing calcium aluminate and barium aluminate cements, we selected the $CaAl_2O_4-CaAl_4O_7-CoAl_2O_4$ section of the $CaO-CoO-Al_2O_3$ system and the $BaAl_2O_4-BaAl_{12}O_{19}-CoAl_2O_4$ section of the $BaO-CoO-Al_2O_3$ system.

The raw mixtures contain barium-bearing waste of the production of aminocaproic acid by the State Company «Chemical Reagent Plant», Science and Engineering Company «Institute for Single Crystals» (Kharkiv), and calcium-bearing water purification waste and spent corundum cobalt-bearing catalysts of the PJSC «Severodonetsk Association «Azot» (Severodonetsk, Luhansk region, Ukraine). To adjust the compositions, the following chemicals were used: barium carbon dioxide (the state standard GOST 2149-75), metallurgical alumina (the state standard GOST 30558-98), cobalt(II) hydroxide carbonate aqueous (the technical regulations TU 6-09-5352-89), and technical calcium dioxide (the state standard GOST 4530-76). The raw mixtures were fired at the temperatures of 1300°C to 1650°C, depending on the phase compositions, and then kept at a maximum temperature for 3 hours.

The physical and mechanical properties of the specimens were studied using the Strelkov method of small samples [10].

To obtain the cementing material of a high strength and refractoriness, the optimal phase ratio was selected for the end product. The quantitative phase ratio was optimized using the simplex-lattice method of the experimental design [11]. To describe the dependence of the developed material properties on the quantitative phase ratio, we used the following polynomial of incomplete third order:

$$Y=\beta_1x_1+\beta_2x_2+\beta_3x_3+\beta_{12}x_1x_2+ \\ +\beta_{13}x_1x_3+\beta_{23}x_2x_3+\beta_{123}x_1x_2x_3. \quad (1)$$

The matrices of experimental design are given in Tables 1 and 2.

Table 1

Experimental design matrix for calcium cements

Polynomial coefficients	Designation and physical content of the fact				
	phases			experimental dat	
	$CaAl_2O_4$	$CaAl_4O_7$	$CoAl_2O_4$	$Y_{\sigma\ str}$	Y_{Tm}
η_1	1	0	0	60.0	187
η_2	0	1	0	32.0	202
η_3	0	0	1	0.0	223
η_{12}	0.5	0.5	0	48.0	198
η_{13}	0.5	0	0.5	26.0	207
η_{23}	0	0.5	0.5	18.0	217
η_{123}	0.33	0.33	0.33	46.0	184
Test point	0.3	0.4	0.3	45.0	185

Note: * – $Y_{\sigma\ str}$ is the ultimate compression strength at the age of 28 days (MPa); and Y_{Tmel} is the melting temperature (K).

Table 2

Experimental design matrix for barium cements

Polynomial coefficients	Designation and physical content of the factors				
	phases			experimental data *	
	$BaAl_2O_4$	$BaAl_{12}O_{19}$	$CoAl_2O_4$	$Y_{\sigma\ str}$	Y_{Tmel}
η_1	1	0	0	56.3	2105
η_2	0	1	0	0.0	2170
η_3	0	0	1	0.0	2230
η_{12}	0.5	0.5	0	48.0	2150
η_{13}	0.5	0	0.5	55.0	2200
η_{23}	0	0.5	0.5	0.0	2365
η_{123}	0.33	0.33	0.33	45.0	2070
Test point	0.3	0.4	0.3	41.0	2080

Note: * – $Y_{\sigma\ str}$ is the ultimate compression strength at the age of 28 days (MPa); and $Y_{Tmel.}$ is the melting temperature (K).

Using the obtained experimental data, we computed the following polynomial coefficients, which express the dependence of the ultimate compression strength and the melting temperature on the quantitative phase ratio:

– for calcium cements:

$$Y_{\sigma\ str}=60x_1+32x_2+8x_1x_2-16x_1x_3+8x_2x_3+414x_1x_2x_3; \quad (2)$$

$$Y_{Tmelt}=1870x_1+2020x_2+2230x_3+ 140x_1x_2+100x_1x_3+200x_2x_3-6720x_1x_2x_3, \quad (3)$$

where x_1, x_2, and x_3 are the relative phase ratio of $CaAl_2O_4$, $CaAl_4O_7$, and $CoAl_2O_4$, respectively.

– for barium cements:

$$Y_{\sigma\ str}=56.3x_1+79.4x_2+107.4x_1x_2+147x_1x_2x_3; \quad (4)$$

$$Y_{Tmelt}=2105x_1+2170x_2+22303x_3+ 50x_1x_2+120x_1x_3+650x_2x_3-5160x_1x_2x_3, \quad (5)$$

where x_1, x_2, x_3 are the relative phase ratio of $BaAl_2O_4$, $BaAl_{12}O_{19}$, and $CoAl_2O_4$, respectively.

Results and discussion

The adequacy of equations was verified using the Student's test and carrying out additional test experiments. Regression equations were calculated by means of the specially developed software based on Microsoft Excel, the variation step being 10 wt.%. Based on the computational data and mathematical treatment of the experiment data by using the TRIANGLE V.1.0 software, the «property vs. composition» diagram and the projection of the lines of the same level were plotted for the compression strength and melting temperature of the prepared cobalt-containing calcium aluminate and barium aluminate special cements (Fig. 1–4).

Using the obtained calculated data, we selected the optimal compositions for the cobalt-bearing calcium aluminate and barium aluminate cements with the following phase composition (wt.%): $CaAl_2O_4$ 25–55, $CaAl_4O_7$ 15–35, and $CoAl_2O_4$ 25–

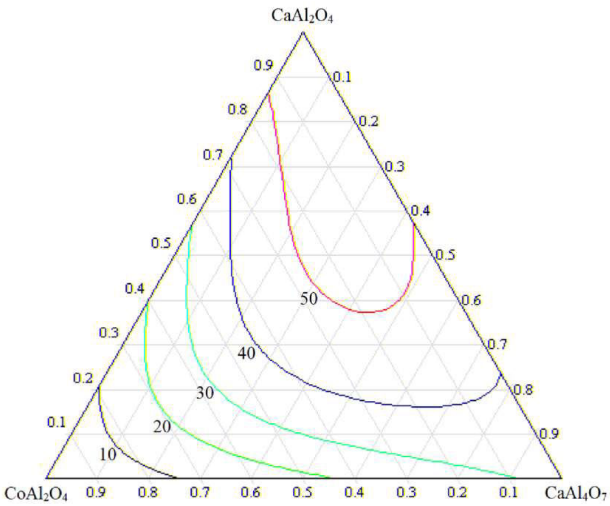

Fig. 1. Diagram «compression strength (MPa) vs. composition» of the cobalt-containing calcium aluminate cement

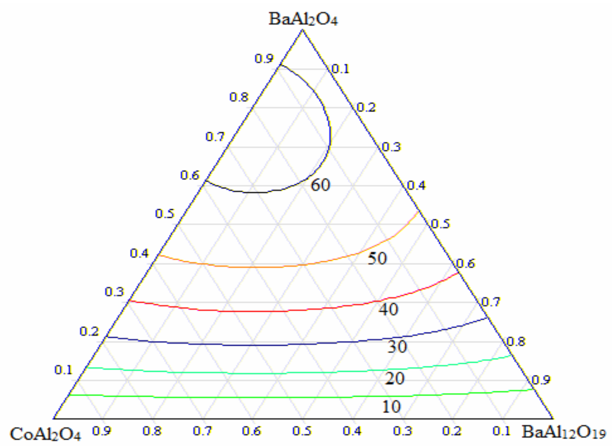

Fig. 3. Diagram «compression strength (MPa) vs. composition» of the cobalt-containing barium aluminate cement

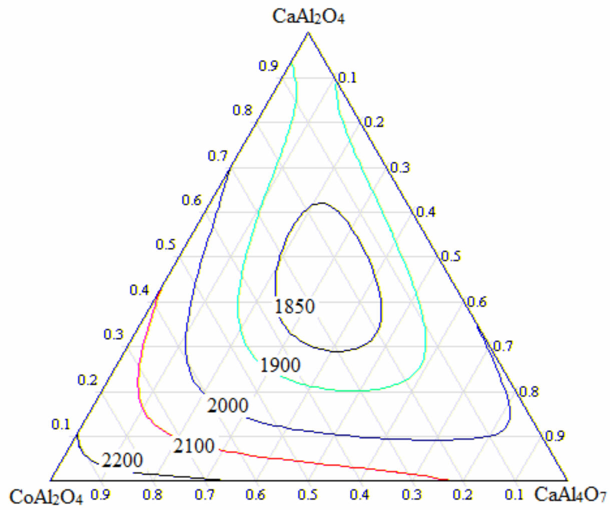

Fig. 2. Diagram «melting temperature (K) vs. composition» of the cobalt-containing calcium aluminate cement

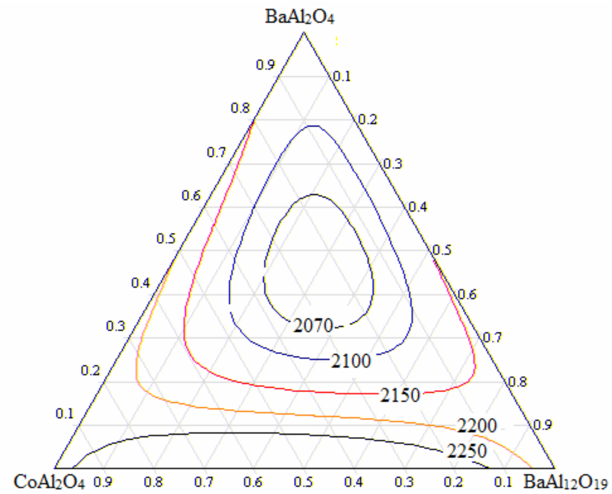

Fig. 4. Diagram «melting temperature (K) vs. composition» of the cobalt-containing barium aluminate cement

45 for calcium cement; and $BaAl_2O_4$ 60–20, $BaAl_{12}O_{19}$ 10–20, and $CoAl_2O_4$ 30–60 for barium cement, accordingly. When developing cobalt-containing barium aluminate cements, it is planned to introduce barium hexaaluminate as a filler.

In order to confirm the results obtained, the synthesis of refractory cobalt-containing calcium-aluminate and barium-aluminate cements was carried out. Raw mixtures were prepared, chemical and phase compositions of which are given in Table 3.

The results of studies of the main physical-mechanical properties of the synthesized cements are given in Table 4. The coefficients of mass absorption were calculated for barium compositions.

The physical-mechanical properties of the cements of developed compositions were studied according to EN 196-6:2019, EN 196-1:2019, EN 196-3:2017; the refractoriness was defined by using the method of pyrometric cone failure [12]; and the mass absorption coefficient was calculated by the method described elsewhere [13]. Analysis of the results obtained (Table 4) shows that an increase in

Table 3

Chemical and phase compositions of cobalt-containing calcium-aluminate and barium-aluminate cements

Chemical compositions, wt.%				Phase compositions, wt.%			
BaO	CaO	Al_2O_3	CoO	$CaAl_2O_4$	$CaAl_4O_7$	$BaAl_2O_4$	$CoAl_2O_4$
–	24.83	62.46	12.71	70	–	–	30
–	17.73	61.09	21.18	50	–	–	50
–	10.64	59.71	29.65	30	–	–	70
–	11.41	63.18	25.41	20	20	–	60
–	14.19	60.40	25.41	40	–	–	60
–	14.96	63.86	21.18	30	20	–	50
–	7.10	59.02	33.88	20	–	–	80
–	21.29	61.77	16.94	60	–	–	40
–	28.38	63.15	8.47	80	–	–	20
12.0	–	54.1	33.9	–	–	20	80
18.0	–	52.4	29.6	–	–	30	70
24.0	–	50.6	25.4	–	–	40	60
30.0	–	48.8	21.2	–	–	50	50
36.0	–	47.1	16.9	–	–	60	40
42.0	–	45.3	12.7	–	–	70	30
48.0	–	43.5	8.5	–	–	80	20

Table 4

Physical-mechanical properties of cobalt-containing calcium-aluminate and barium-aluminate cements

Water/cement ratio	Setting time, hours–minutes		Compressive strength, MPa, after hardening, days			Refractoriness, ^0C	μ, cm^2/g
	initial set	final set	3	7	28		
0.23	1–20	6–00	18	22	29	1580	–
0.23	0–55	5–50	21	30	36	1670	–
0.23	0–45	5–40	26	31	33	1760	–
0.22	0–37	5–45	46	51	62	1620	–
0.24	1–05	5–30	20	41	59	1680	–
0.20	1–10	5–40	31	53	63	1630	–
0.21	1–10	5–20	25	50	56	1670	–
0.20	1–10	5–00	32	48	55	1620	–
0.22	1–20	5–40	30	40	51	1580	–
0.15	3–50	7–45	22	36	40	1770	150
0.15	2–45	6–55	33	37	44	1760	163
0.16	1–50	5–00	48	55	66	1750	175
0.16	1–00	3–55	40	48	56	1720	187
0.18	1–05	2–40	41	46	50	1690	198
0.2	0–45	1–30	39	44	46	1580	207
0.28	0–28	0–55	36	40	43	1600	217

the cobalt spinel content of the clinker compositions contributes to an increase in the refractoriness of the material being synthesized. However, due to the fact that $CoAl_2O_4$ is a hydraulically inert compound, an increase in its content above 60 wt.% leads to a decrease in the compression strength of samples of both types of cements.

This, in our opinion, the most promising compositions are the following: composition No. 6 containing (wt.%) 30 $CaAl_2O_4$, 20 $CaAl_4O_7$ and 50 $CoAl_2O_4$; and composition No. 12 containing (wt.%) 40 $BaAl_2O_4$, and 60 $CoAl_2O_4$. They are characterized by a reduced water demand of the cement paste, increased values of compressive strength and refractoriness.

The main physical-mechanical properties of the developed cements of optimal composition are the following: the fineness of grinding is characterized by the total passage through the sieve No 006; for calcium cements: water-cement ratio of 0.2; the setting time: the initial set of 1 h 10 min, and the final set of 5 h 40 min; the ultimate compression strength after hardening of 28 days of 63 MPa; for barium cements: water-cement ratio of 0.16; the setting time: the initial set of 1 h 50 min, and the final set of– 5 h 00 min; the ultimate compression strength after hardening of 28 days of 66 MPa; the calculated mass absorption coefficient μ=175 cm^2/g. Refractoriness is 1630^{0}C and 1750^{0}C for calcium cement and barium cement, respectively.

Conclusions

Comparative analysis of the data obtained indicates that the physical-mechanical characteristics of the developed binders meet the requirements for alumina and high-alumina cements (the state standard DSTU EN 196:2019). It was established that the developed cements are attributed to fast setting, fast hardening, high strength materials with high refractoriness and can be recommended for the production of refractory concretes, gunning mixtures and also refractory mortars that can be used in high-temperature aggregates for different branches of industry. The developed barium aluminate binders are characterized by protective properties against radiation (μ=150–217 cm^2/g), which also makes it possible to recommend these materials for the manufacture of protective screens, structural elements of biological protection of nuclear energy systems, and containers for the disposal of radioactive waste.

The developed procedures for the production of high-strength refractory materials of a special purpose that are based on the chemical industry waste are considered to be resource-saving. The adoption of this technology will enable a considerable improvement of the ecological situation in the industrial regions of Ukraine, as well as an opportunity for a considerable decrease in the cost and the synthesis of products requiring no hefty capital investments.

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ОПТИМІЗАЦІЯ СКЛАДУ ВОГНЕТРИВКИХ ЦЕМЕНТІВ, ОДЕРЖАНИХ ІЗ ВИКОРИСТАННЯМ ВІДХОДІВ ХІМІЧНОЇ ПРОМИСЛОВОСТІ

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Наведено результати оптимізації складів кобальтвмісних кальцій-алюмінатних і барій-алюмінатних спеціальних цементів, одержаних на основі відходів хімічної промисловості. За результатами експериментальних даних розраховані коефіцієнти поліному, що виражають залежність межі міцності при стисканні і температури плавлення від кількісного співвідношення фаз $CaAl_2O_4$, $CaAl_4O_7$, $CoAl_2O_4$ для кальцій-алюмінатного і $BaAl_2O_4$, $BaAl_{12}O_{19}$, $CoAl_2O_4$ для барій-алюмінатного цементу; побудовані діаграми «склад–властивість» та проекції ліній однакового рівня для одержаних цементів. Обрано перспективні ділянки для системи CaO–CoO–Al_2O_3 (мас.%): $CaAl_2O_4$ 25–55, $CaAl_4O_7$ 15–35; $CoAl_2O_4$ 25–45 та для системи BaO–CoO–Al_2O_3 (мас.%): $BaAl_2O_4$ 60–20; $BaAl_{12}O_{19}$ 10–20; $CoAl_2O_4$ 30–60. Основні фізико-механічні властивості розроблених цементів оптимальних складів: тонкість помелу – повний прохід через сито № 006; для кальцій-алюмінатного цементу: водоцементне відношення – 0,2; терміни тужавіння: початок – від 1 год 10 хв, кінець – від 5 год 40 хв; межа міцності при стисканні протягом 28 діб тверднення – 63 МПа; для барій-алюмінатного цементу: водоцементне відношення – 0,16; терміни тужавіння: початок – 1 год 50 хв, кінець – 5 год 00 хв; межа міцності при стисканні протягом 28 діб тверднення – 66 МПа, розрахунковий коефіцієнт масового поглинання μ=175 см^2/г. Вогнетривкість становить 1630^{0}C для кальцієвого та 1750^{0}C для барієвого цементу, відповідно.

Ключові слова: спеціальний цемент, промислові відходи, вогнетривкість, міцність при стисканні, оптимізація, рівняння регресії, діаграма «властивість–склад».

OPTIMIZING THE COMPOSITIONS OF REFRACTORY CEMENTS PRODUCED USING THE WASTE OF CHEMICAL INDUSTRY

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The article presents the results of optimization of the quantitative compositions of cobalt-containing calcium-aluminate and barium-aluminate special cements prepared from chemical industry wastes. Based on the obtained experimental data, the coefficients of the polynomial were computed which express the dependence of the ultimate compression strength and the melting temperature on the quantitative ratio of the $CaAl_2O_4$, $CaAl_4O_7$, and $CoAl_2O_4$ phases for calcium-aluminate cement and the quantitative ratio of the $BaAl_2O_4$, $BaAl_{12}O_{19}$, $CoAl_2O_4$ phases for barium-aluminate cement. The «composition–property» diagrams and the projections of the lines of the same level were plotted for the ultimate compressive strength and the melting temperature of the obtained cements. The following promising areas were selected for the CaO–CoO–Al_2O_3 system (wt.%): 25–55 $CaAl_2O_4$, 15–35 $CaAl_4O_7$, and 25–45 $CoAl_2O_4$; and for the BaO–CoO–Al_2O_3 system (wt.%): 60–20 $BaAl_2O_4$, 10–20 $BaAl_{12}O_{19}$, 30–60 $CoAl_2O_4$. The main physical-mechanical properties of the developed cements of optimal composition are the following: the fineness of grinding is characterized by the total passage through the sieve No 006; for calcium cements: water-cement ratio of 0.2, the setting time: the initial set of 1 h 10 min, and the final set of 5 h 40 min; the ultimate compression strength after hardening of 28 days of 63 MPa; the for barium cements: water-cement ratio of 0.16; the setting time: the initial set of 1 h 50 min, and the final set of 5 h 00 min; the ultimate compression strength after hardening of 28 days of 66 MPa; and the calculated mass absorption coefficient μ=175 cm^2/g. The refractoriness is 1630^{0}C and 1750^{0}C for calcium cement and barium cement, respectively.

Keywords: special cement; industrial wastes; refractoriness; compressive strength; optimization; regression equation; «property vs. composition» diagram.

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