POROUS GRAIN MATERIAL BASED ON ALKALINE SILICATE COMPOSITION

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ABSTRACT

The porous granular material synthesized by firing silicate compositions based on cullet, water glass and mineral additives was studied. Introduction of additives into the glass mixture provides an additional source of gas formation in a pyroplastic material. Effectiveness of technological solutions is shown: mechanical activation of the raw mixture, increasing proportion of the blowing agent in the molding material, preliminary heat treatment of raw granules.

Keywords: cullet, alkali-silicate granules, lightweight concrete, firing, porosity.

1. INTRODUCTION

Prospects for energy-efficient construction are associated with the use of lightweight concrete [1 - 4]. The structural and geometric characteristics of the porous aggregate make it possible to regulate the combined cellular-granular structure of composite materials, and determine thermo technical and physical and mechanical properties of lightweight concrete.

Numerous developments of recent years are devoted to the problems of porous concrete fillings. Technologies of granular foam glass [5 - 10], highly porous granular materials based on thermal expansion of liquid glass [1, 5, 7, 9, 11, 12] are actively developing. Expansion of raw material base and improvement of technology allowed creating of new generation aggregates: highly porous silicate material [12]; foamed glass ceramic multifunctional materials [8, 13]. Porization is a defining stage of lightweight concrete aggregate technology. To realize the thermal expansion of granular material, gasforming processes are used in main raw materials or specially introduced additives; compositions porosity of which is achieved by the use of liquid glass are effective [1, 5, 7, 9, 12, 14 - 16]. For porous granular materials of pyroplastic synthesis, resource-saving technologies using technogenic raw materials are preferred: cullet, ash of thermal power plant, metallurgical slag, and others [2, 4, 7, 10, 15, 17]. The widespread distribution of porous granular aggregates in construction is hindered by the following problems: limited list of raw materials used; need for high-temperature processes; low efficiency of porosity techniques.

The purpose of the work is to study the effect of material composition of alkaline silicate raw material mixtures on the porosity of granules.

2. MATERIAL AND METHOD

The object of the study is a porous granular material synthesized during the firing of silicate compositions based on cullet, liquid glass and mineral additives.

The basis of investigated raw material mixtures is cullet - fragments of sheet and container glass, crushed into powder (Table-1). Liquid glass is an aqueous solution of sodium silicate with a density of 1410 kg/m³, introduced to thermally expand the granules and ensure molding properties of the powdered raw material. As additives there were used materials containing burnable or gas-forming minerals (Table-1).

	Containing of the oxide, %								
Material	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	R ₂ O	SO ₃	Other	Loss during sintering
Cullet	71.8	2.5	0.2	6.8	3.9	14.5	0.2	0.1	-
Silica clay	77.2	5.7	2.5	0.7	1.3	1.2	1.8	0.2	9.4
Lignite-bauxite	13.4	50.2	3.2	0.9	0.4	0.2	5.1	0.1	26.5
Magnetite skarn ores tails	37.5	9.1	16.8	10.4	5.1	1.2	14.3	1.5	4.1

Table-1. Chemical composition of raw materials.

Silica clay is a micro porous gaize, consisting mainly of opal (amorphous silica); clay minerals are present (hydro mica, feldspars). Endothermic effect at a temperature of 510 0 C corresponds to dehydration of opal and clay minerals. The choice of silica clay for research is

justified by information on the effectiveness of using siliceous rocks in the production of foam glass [11, 18].

Lignite-bauxite is a substandard rock for the production of aluminum, enriched with finely dispersed carbonized wood residues with preserved fiber. Organic



carbon content reaches 10 %. The mineral base of the rock is formed by aluminum hydroxides: hydrargillite and boehmite. The mass loss noted on the thermogram at temperatures of 300 and 520 ^oC is associated with the burnout of the organic component and dehydration of hydrargillite, respectively.

Skarn-magnetite ores tails are fine particles with an average size of 0.55 mm, which are formed during magnetic separation of rock mass. Mineral composition of the waste is represented mainly by calcium silicates and aluminosilicates (diopside, grossular, epidote, scapolite, chlorites, feldspars). The waste contains 8 - 10 % pyrite, up to 7 % of calcite and up to 4 % of magnetite. Exothermic effect on the thermogram at 500 ^oC characterizes the oxidation of pyrite. In the temperature range of 600 - 800 ^oC, endothermic reactions of dehydration of aluminosilicates and decomposition of anhydrite, formed during interaction of decomposition products of pyrite and calcite occur [19].

Raw mixtures were prepared by thoroughly mixing pre-ground components. Raw grains were obtained in the form of pills or granules with a diameter of 10 mm, taking into account the state of the molding material. The dried samples were fired in a laboratory furnace at a given temperature with a holding time of 15 minutes. Cooling of samples was done in an oven up to 600 0 C. To determine the composition of materials, differential thermal and diffractometric methods of analysis were used. Microstructure of calcined granules was investigated by electron microscopy. Structural transformations of materials were evaluated by the coefficient of expansion (the ratio of the diameters of the samples before and after firing), density. The average pore size is the result of nine measurements in a photograph of chip cleavage.

3. RESULTS AND DISCUSSIONS

3.1 The effect of mineral additives on the expansion of the glass mass

Glass blends with different flask contents and 35 % of liquid glass was investigated. Addition of siliceous microporous rock to the glass mass is accompanied by a decrease in plasticity of the moldable mixture. As the flask content in the mixture increases, the ability of samples to swell decreases (Table-2). To ensure a highly porous structure and a reduced density of granules, it is advisable to add 20 % of the flask (Figure-1).

Flask content in mixture,%	Expansion coefficient	Granule's density, kg/m ³	Average pore size, mm
0	1.2	550	0.1
20	1.5	480	0.7
40	1.1	570	0.5
60	1.0	630	0.3
80	0.9	880	0.2
100	0.8	940	0.1

Table-2. The influence of flask on structural properties of the calcined granules.



Figure-1. The structure of samples from glass mixtures with different flask contents.

Lignite-bauxite was added to the glass powder in an amount of 20 - 40 %. A glass mixture with the addition of 20 % lignite-bauxite differs from the unloading mass by a lower coefficient of expansion, equal to 1.1. The introduction of 20 % carbonated rock provides an increase in porosity and a decrease in the density of the granule by 40 kg/m^3 in comparison with the material without additives, mainly due to burning of wood residues. A further increase in proportion of lignite-bauxite in the raw



material mass is accompanied by compaction of the samples.

The effect of skarn-magnetite ore dressing wastes is manifested in decrease in softening temperature of the mass by 25 - 30 0 C, pore enlargement. The coefficient of expansion of samples from 20, 30 and 40 % of ore dressing waste is 1.3; 1.8 and 2.1, respectively. The optimum content of ore dressing wastes in the mixture is 20 % provides the lowest density of the material (420 kg/m³) with closed pores and a uniform cellular structure. With an increase in the content of ore dressing wastes, large cavities form in the structure (2.5 - 3.0 mm); burnt samples are easily destroyed.

The study of microstructure of materials showed that introduction of lignite-bauxite and ore dressing waste contributes to the formation of additional pores in the glass mass. Along with the main cells, structure of the calcined materials is saturated with small pores in the partitions between the cells (Figure-2).



Figure-2. Microstructure of porous granules of various compositions.

3.2 The effect of combined additives on the expansion of the glass mass

At the next stage, the joint effect of mineral additives was investigated. The raw mixes contained 56 % of cullet, 14 % of flask and 30 % of a combined additive from lignite-bauxite and ore dressing wastes (Table-3). Molding properties of raw materials are ensured by introduction of 35 % of liquid glass. Calcination of samples at various temperatures contributed to

optimization of the composition of a raw mix. Preferred are raw mixes with a combined additive of lignite-bauxite and ore dressing with a ratio of 1: 1. Mixtures of optimal composition are characterized by a low temperature of intense expansion, uniform porosity with a mesh size of 0.3 - 0.5 mm. At a firing temperature above $850 \, {}^{0}$ C, voids are formed in the structure due to ruptures of inter-pore septa (Figure-3).

Composition of the combined additives,%		The coefficient of expansion at a firing temperature, ⁰ C				
lignite-bauxite	skarn - magnetite ores waste	800	850	900		
0	0	1.05	1.20	1.40		
10	20	1.30	1.45	1.70		
12	18	1.35	1.45	1.80		
14	16	1.40	1.55	1.90		
16	14	1.35	1.55	1.80		
18	12	1.20	1.50	1.70		
20	10	1.15	1.40	1.60		

Table-3. Effect of combined additives on expansion of glass mass.





Figure-3. Structure of samples of optimal composition at different firing temperatures.

3.3 The influence of the method of preparation of glass mass on the formation of the porous structure

Porization of a glass mixture depends on reactivity of raw materials and uniformity of their distribution in the molding material. Multicomponent composition of the studied raw material composition requires careful preparation, which can be achieved with mechanical activation [20]. Raw materials and their mixture were processed in an «Emax» laboratory a high speed mill. Duration of mechanical activation of the components is individual, taking into account a particle size of the starting particles and hardness of a material. The dispersion of powders was determined on a photosedimentometer. The effect of high-speed grinding on reactivity of the test mixture was evaluated by reflection intensity d = 3.36 nm in the diffraction patterns of the calcined materials and coefficient of expansion of the granules (Table-4). A comparative analysis of the obtained results testifies to the advantages of joint mechanical activation of raw materials, which provides the greatest amorphization of the siliceous component, lowering temperature, and increasing intensity of expansion of granules.

Prenaration method of the	Average dia particles, n	meter of nicrons	Height	The coefficient of expansion at a firing temperature, ⁰ C	
mixture	before	after	d = 3.36 nm,		
	grinding	grinding	70	825	850
Without mechanical activation	65	-	100	1.35	1.55
Cullet mechanical activation	72	15	67	1.48	1.58
Mechanical activation of the flask	63	13	69	1.57	1.65
Mechanoactivation of lignite bauxite	64	18	75	1.38	1.47
Mechanical activation of ore dressing waste	73	15	73	1.52	1.57
Mechanoactivation of the raw mix	65	17	64	1.65	1.78

Table-4. The effect of mechanical activation on reactivity of a glass mixture.

Liquid glass is a multifunctional component of molding materials. Increase in the proportion of alkaline silicate ensures decrease in the temperature of pyroplastic state and increases porosity. With the introduction of liquid glass in excess of 40%, the molding mixture is characterized by increased fluidity, making it difficult to obtain stable raw samples. Modification of liquid glass with sodium chloride makes it possible to effectively control viscosity of the molding material [5, 12]. However, the introduction of salt into the studied multicomponent mixture can affect the nature of thermal transformations of the constituents and change conditions of porosity.

Influence of the temperature of liquid glass on the molding properties of a glass mixture was investigated. It was revealed that heating an aqueous solution of sodium silicate to 60 - 70 ⁰C increases viscosity and favours pelletization of a raw material mass, accelerates hardening of granules. The use of heated liquid glass made it possible to increase the proportion of the main gas-forming component to 55 % of the dry mix weight.

To optimize the expansion temperature of the developed raw material mixture, transformations during the firing of granules in the range of 200 - 950 ⁰C were studied (Figure-4).



Figure-4. The effect of firing temperature on the properties of the granules.

Samples were heated to a predetermined temperature; after 5 minutes of isothermal heating, they were removed from the furnace. The stepwise nature of the swelling of granules is due to the phased participation of the components of a raw material mixture in the formation: foaming of liquid glass, fading of organic substances, saturation of a softened mass with gaseous decomposition products of mineral additives and intermediate formations. Swelling of granules is intensified in the pyroplastic mass formed at a temperature of 700 °C and reaches a maximum during firing in the temperature range of 800 - 900 °C. Decrease in the coefficient of swelling of granules calcined at a temperature of 950°C is due to the low gas-holding ability of samples and due to decrease in viscosity of the mass. This corresponds to the nature of change in pore size in the

structure of granules: 0.01 - 0.02 mm at 200 - 300 $^{\circ}\text{C}$; 0.1 - 0.2 mm at 400 - 600 $^{\circ}\text{C}$; 0.3 - 0.4 mm at 550 - 850 $^{\circ}\text{C}$; 0.5 mm at 900 $^{\circ}\text{C}$; 0.9 mm at 950 $^{\circ}\text{C}$.

The increased sensitivity of the alkali silicate composition to low-temperature exposure (Figure-4) determined feasibility of two-stage firing. The low-temperature stage of heat treatment at 250 0 C creates conditions for preliminary swelling and hardens granules, increasing their technological properties. The second stage of firing is characterized by active swelling of granules, the maximum temperature of which can be reduced to 775 - 800 0 C.

The structure of the granules is characterized by uniform distribution of cells with an average size of 0.2 - 0.3 mm; the pore polymodality is a characteristic of the microstructure of a material (Figure-5).



Figure-5. The structure of alkaline silicate granules.

The basic properties of the developed granular glassy crystalline alkali silicate material are determined: a granule density is $320 - 370 \text{ kg/m}^3$; bulk density is $230 - 280 \text{ kg/m}^3$; compressive strength of granules is 0.4 - 0.7 MPa; coefficient of thermal conductivity is $0.05 - 0.06 \text{ W} / (\text{m}^{-0}\text{C})$; water absorption of 17 - 20%; water resistant.

Preliminary technical and economic calculations indicate that the use of a developed raw material mass will reduce material costs by 24 % in production of a granular material compared to the foam-glass analog obtained from cullet and liquid glass. The increased heat engineering characteristics in comparison with the expanded clay provide a porous granular material from an alkaline

silicate composition with economic advantages when used in construction.

4. CONCLUSIONS

Composition of an alkaline silicate mixture has been developed to obtain a highly porous granular material by expansion at a temperature of 775 - 800 ⁰C.

Pyrogenic transformations of polymineral additives are accompanied by gas formation in a wide temperature range, thereby enhancing the porosity of liquid glass.

Mechanical activation of a raw material mixture, increasing the proportion of blowing agent in the molding material to 55 %, pre-treatment of raw granules at 250 0 C stimulate formation of a porous structure and completion of expansion at a low temperature.

Heated liquid glass provides the formation of stable granules from a mixture with a high content of an aqueous solution of sodium silicate.

The developed granular material can be used as an aggregate of lightweight concrete and insulation.

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