ALKALINE SILICATE COMPOSITIONS OF POROUS STRUCTURE

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ABSTRACT

The article presents the results of studies of a porous heat insulating alkaline silicate compositions based on technogenic raw materials. The influence of technogenic factors on aeration of compositions made of liquid glass and technogenic filler has been detected. Aeration of the compositions has been evaluated by the bulk and stability of foam, structure and density of foamed concrete. The influence of type and concentration of foaming agent, filler content, agitation mode of raw stuff on formation of cellular structure has been studied. The structure of aerated materials has been analyzed. Reasonable ratios of solid to liquid components in molding blend have been determined. Additional aeration of cellular structure by means of addition of aerated technogenic filler and gas forming agent, hydrogen peroxide, has been demonstrated. The results of studies of a porous aggregate obtained using liquid glass and technogenic materials (glass cullet, magnetite ore dressing waste, substandard rocks) are presented. Feasibility of a multicomponent feed mixture for pyroplastic swelling of granules is substantiated. Optimization of raw mix composition provided the possibility of combining various formation mechanisms of porous granules.

Keywords: liquid glass, technogenic filler, porosity foamed concrete, pyroplastic swelling.

1. INTRODUCTION

To ensure energy-efficient construction, durable materials are required that combine high heat-shielding properties with structural stability. Numerous developments in recent years have been devoted to the development of porous concrete technology, which is represented by cellular concrete and lightweight concrete based on porous aggregates [1 - 4].

Cellular concrete is favorably distinguished by numerous existing variants, availability of raw materials, comparatively simple technology, high performances, environmental safety. Technology of cellular concretes is characterized by possibility of wide application of various raw materials, including those of technogenic origin. Portland cement and lime-silica materials (autoclave) are mainly used as binders for cellular concretes. Expensiveness of Portland cement, engineering difficulties of autoclave treatment require for alternative cement-free binders. Herewith, it is necessary to create favorable conditions for aeration, to provide increased strength of inter-pore membranes in cellular concrete.

Literature review evidences promising possibilities of alkaline silicate binders - compositions on the basis of powdered filler and solution of alkaline component (liquid glass) [5-9]. Liquid glass as the basis of such compositions used for a long time corresponds to requirements and specifications of low energy intensity technologies. Metallurgical slags, waste glass and other silicate and alumosilicate materials of various origin are used as powdered fillers in alkaline silicate binders [10-14].

Aeration is the determining stage of production technology of cellular materials. Aeration of liquid glass pastes is performed by thermal, chemical, mechanical methods [5 - 14].

Significant influence on structure of cellular materials is exerted by procedures of production of concrete mixes. Alkaline silicate foamed concretes are the most widely applied products [8-20]. Technology of foamed concrete is characterized by numerous production variants of molding blend: separate preparation of paste and foam; dry mineralization of foam; foaming of all components in high speed mixer [13, 14, 17]. Alkaline silicate foamed concretes are mainly produced by dry mineralization of foam [11, 13]. Other methods are developed mainly for cement concrete mixes and do not account for numerous components in molding blend compositions based on cement-free binders.

On the basis of liquid glass cellular concrete, porous granules for various purposes, piece products can be produced. Porization of liquid glass materials is carried out by swelling [3, 5, 9, 19]. The physicochemical features of liquid glass allow one to implement numerous expansion options. To develop effective heat-insulating materials, the choice of composition and optimization of properties of the raw mix based on liquid glass is necessary.

It can be assumed that further development of technology of alkaline silicate cellular concretes will be related with improvement of structure aeration.

This work is aimed at study of influence of technological factors on aeration of materials of liquid glass and technogenic filler.

2. MATERIALS AND METHODS OF INVESTIGATION

The object of the study was the composition of liquid glass and technogenic mineral additives.

Glass cullet is fragments of sheet glass and glass containers. The chemical composition of glass cullet is, wt. %:SiO_2 70 - 73; Al_2O_3 1- 3; Fe_2O_3 1- 3; CaO 5 - 7; MgO до 1; R_2O_3 4 - 5.

Slag of ferrous metallurgy - blast furnace granulated slag with the following chemical composition, wt %: SiO_2 38-45; $A1_2O_3$ 8-12; FeO 0.5-0.7; CaO 23-29; MgO 7-12.

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Gaize consists mainly of silica gel; SiO_2 content is 80 - 85%. Gaize is a part of overburden formed during the development of magnetite ore deposits.

Magnetite ore dressing waste is fine sand containing calcium silicates and aluminosilicates (pyroxenes, garnets, epidote, scapolite, chlorites, feldspars). The waste contains pyrite, calcium carbonate. The chemical composition of the waste is, wt. %: SiO_2 39 - 41; Al_2O_3 11 - 13; Fe_2O_3 14 - 16; CaO 10 - 12; MgO 5 - 6; SO_3 5 - 7; R_2O_3 2 - 3; others 2 - 4.

Oil shale is a metamorphic rock comprising from 10 to 30% of bituminous substances. The chemical composition of oil shale is, wt. %: Si 17 - 19; Al 7 - 10; Fe 10 - 12; Ca 17 - 20; S 4 - 6; Mg 1 - 3; C 29 - 32; H 2 - 4.

Sodium liquid glass with a density of 1400 kg/m^3 was used as a blowing agent and connection of components of the molding mass.

The pastes were aerated using various surfactants: Unipor protein foamed concentrate, foaming agents on synthetic base, Fairy and Zelle-1.

Foamed paste was produced by one-stage method: slurry obtained by mixing of all components was foamed in mixer in 2 min. Rotation frequency: 600 - 1000 rpm. Expansion ratio and density of foamed paste were measured. Expansion ratio of foamed paste was determined as the ratio of blend volume before and after foaming. Foamed concrete samples with the sizes of 40×40×40 mm were solidified under normal conditions.

Pore sizes, homogeneity and stability in time of the obtained foams were compared visually. Foam with pore sizes of 0.5 mm were considered as fine; those with pore sizes more than 1 mm were considered as coarse. Homogeneous porous structure was characterized by uniform pore distribution and no large air cavities. Stability of foamed paste was determined by the time of retention of initial volume: high stability - at least 30 min; low stability - breakage after extraction from mixer.

Thermal transformations in the raw material mass were evaluated by the nature of porous structure and granules' density. The granules were dried and fired at a temperature of 700 - 850 ^{0}C. When firing, the granules swell. The coefficient of expansion was determined as the ratio of granules' size before and after firing.

Structure of the materials was studied using an electron microscope.

3. RESULTS AND DISCUSSIONS

3.1 Aerated Concrete Based on Compositions of Water Glass

Peculiar of the considered compositions: mixing by means liquid with adjustable density. Liquid glass has two functions: in combination with foaming agent it is a component of technogenic foam and, at the same time, a component of alkaline silicate binder. This stipulated necessity to study the influence of density of liquid glass on the properties of foam (Table-1). The properties of foamed pastes obtained on the basis of various raw materials were studied (Tables 2, 5; Figures 1, 2). Possibility to increase porosity of alkaline silicate compositions by combinations of various methods of formation of cellular structure was demonstrated (Tables 6, 8; Figures 3, 4).

Comparison of properties of foams obtained under equal conditions on the basis of various liquids revealed decreased expansion ratio (water - 12, liquid glass - 5) and increased average density (water - 80 kg/m^3, liquid glass - 200 kg/m^3) of foam based on liquid glass. Effluent of liquid from foam as a consequence of syneresis in foamed pastes differed insignificantly.

Study of foam formed of liquid glass of various state evidenced higher preference of $Na_2O(SiO_2)_n$ solution with the density of 1200-1300 kg/m^3, when foam of required quality was formed and substantiated rate of material solidification was achieved (Table-1). Increased densities of liquid glass reduced the yield of foamed paste, at low densities the concrete structure hardened slowly.

Table-1. Influence of liquid glass density on properties of foam.

Density of liquid glass $Na_2O(SiO_2)_n$, kg/m^3	Foam expansion ratio	Density of foam, kg/m^3
1100	8.5	115
1150	8.1	170
1200	7.7	195
1250	6.4	230
1300	5.3	250
1350	4.2	320
1400	3.8	470

The structure of cellular materials depends significantly on the essence of pore generating component. The foams were analyzed obtained with various foaming agents added to liquid glass in amount of 3% (Table-2).

Application of Unipor protein foaming agent is accompanied by coagulation and formation of clusters in liquid glass. Foam obtained with Unipor is heterogeneous and highly unstable. Protein cationic or amphoteric surfactants are usually efficient only ion weak acidic medium.

Foam on the basis of Fairy foaming agent is favorably distinguished by fine porous structure, low porosity and good stability. Synthetic foaming agents for liquid glass are more preferable due to their anionic or non-ionic type. Such foaming agents contain sodium salts of alkyl sulfonates and alkyl benzene sulphonic acids, they are efficient in the range of pH = 7.0 - 10.5.

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Table-2. Influence of foaming agent composition on properties of liquid glass foam.

Type of foaming agent	Foam expansion ratio	Density of foam, kg/m^3	Foam properties		
			size	homogeneity of porosity	stability
Fairy	7.0	180	very fine	homogeneous	high
Zelle – 1	6.5	190	fine	homogeneous	high
Unipor	4.0	210	average	heterogeneous	low

Alkaline silicate compositions are comprised of liquid glass and powdered filler (metallurgical slag or waste glass) which influences on rheological properties and foamability. Alkaline silicate composition was aerated by Fairy foaming agent. Increased portion of filler increased density of the paste due to decrease in the material aeration (Table-3 and Table-4). In order to obtain sediment resistant foamed paste with low density the liquid glass to filler ratio should be reasonably set to 1:1.85 -1:2.00. Compositions on the basis of waste glass are less sensitive to variation of filler portion and retain the same expansion ratio of foamed paste.

The structure of foamed concrete is sensitive to variation of molding blend composition. Comparison of properties of alkaline slag compositions with various synthetic foaming agents demonstrated that the use of Zelle -1, other conditions of foamed paste production being equal, provided formation of coarser cells with average size of 0.8 - 1.0 mm (Figure-1) and decrease in density of aerated concrete (Table-5). Foamed concrete on the basis of metallurgical slag was characterized by finer cells in comparison with the composite material on the basis of waste glass (Figure-2).

Table-3. Influence of slag portion on properties of aerated material.

Liquid glass : slag	Foam expansion ratio	Density of foam, kg/m^3	Density of foamed concrete, kg/m^3	Compression strength of foamed concrete, MPa
1: 1.45	6.4	430	260	0.3
1: 1.65	6.3	470	320	0.4
1: 1.85	6.1	490	350	0.5
1: 2.00	5.8	550	460	0.7

Table-4. Influence of waste glass portion on properties of aerated material.

Liquid glass : slag	Foam expansion ratio	Density of foam, kg/m^3	Density of foamed concrete, kg/m^3	Compression strength of foamed concrete, MPa
1: 1.45	5.1	570	400	0.8
1: 1.65	5.2	620	420	1.0
1: 1.85	5.1	640	430	1.1
1: 2.00	5.0	670	480	1.2

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Table-5. Influence of blend composition on properties of slag alkaline foamed concrete.

Foaming agent		**Liquid glass : slag**					
		1 : 1.85			**1 : 2.00**		
type	**content in the blend,%**	**foam expansion ratio**	**density of foam, kg/m^3**	**compression strength of foamed concrete, MPa**	**foam expansion ratio**	**density of foam, kg/m^3**	**compression strength of foamed concrete, MPa**
Fairy	3	5.4	393	0.68	5.2	402	0.73
	4	5.8	296	0.63	5.8	369	0.72
	5	6.2	255	0.62	6.2	322	0.61
Zelle-1	3	5.6	309	0.59	5.8	369	0.68
	4	5.9	237	0.53	6.2	336	0.63
	5	6.3	205	0.50	6.7	276	0.60

In order to improve heat insulating properties of the compositions the possibility of additional aeration was investigated.

Porosity in molding blend was increased by introduction of aerated filler: alumosilicate ash microsphere. Variation of microsphere content makes it possible to adjust the type of aerated structure of the compositions. The influence of alumosilicate microsphere on state of liquid glass composition is summarized in Table-6. It had been determined that the content of alumosilicate microsphere should be restricted by 10%.

Increased concentration of ash particles influences variously on properties of the composition which is stipulated by increase in viscosity of blend with the same liquid to solid ratio. This explains certain increase in average density of aerated composition upon increase in the portion of fine hollow granules with low bulk density.

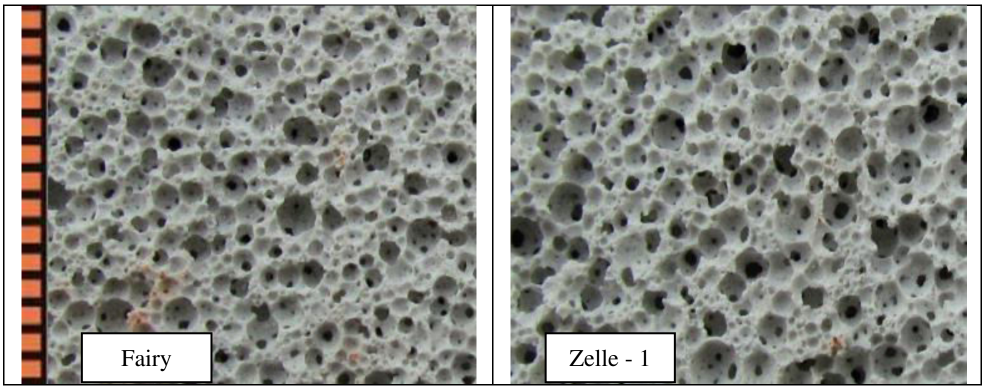

Figure-1. Structure of foamed concrete with various foaming agents.

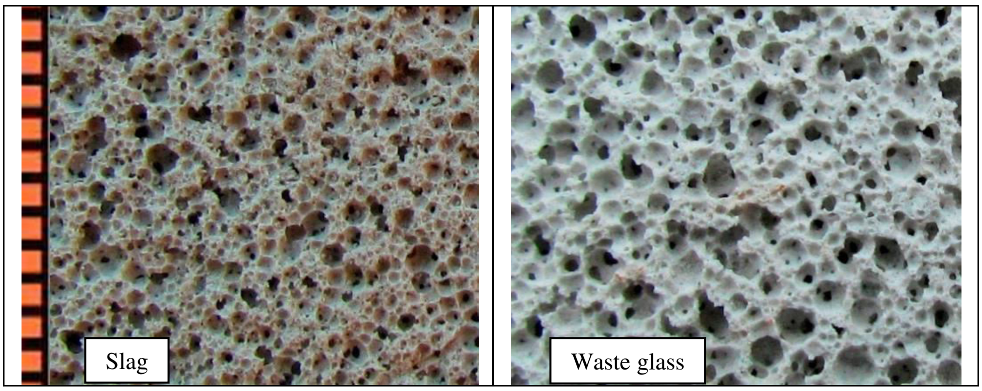

Figure-2. Structure of foamed concrete with various fillers.

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Table-6. Influence of microsphere on aerated compositions' properties.

Content of microsphere, %	Foam expansion ratio	Average density, kg/m^3	Compression strength, MPa	Efficiency coefficient
0	2.5	370	0.7	0.18
2.5	2.8	325	0.7	0.18
5.0	2.1	330	0.6	0.14
10.0	2.4	345	0.6	0.17

Another approach to decrease density of the composition is additional aeration using gas forming agent. Powdered aluminum is a conventional gas forming agent of cellular concretes, it also can act as aqueous curing agent, since the products of interaction between aluminum and liquid glass additionally modify alkaline silicate matrix increasing its water resistance.

However, increased dependence of gas evolution on the state of raw mix and surface of pore forming agent as well as the powder price required another gas forming component.

Concentrated aqueous solution of hydrogen peroxide H_2O_2 (commercial perhydrol, weight concentration 37%) was used as pore forming agent; this substance was efficient and available. Gas formation with hydrogen peroxide is independent on the medium pH; oxygen is evolved upon H_2O_2 decomposition.

The agent was added to raw blend before foaming. The composition was characterized by the liquid glass to waste glass ratio of 1:2. Gas evolution took place in 30-40 min after mold filling with foamed paste; it was accelerated upon heating (30^{0}C). The ratio of weights before and after gas saturation was characterized by swelling index which was calculated after completion of aeration. The experimental results evidenced that the bulk swelling which increased with the increase in the portion of hydrogen peroxide depended on the type of foaming agent (Table-7). Decreased swelling index of the mix based on Zelle - 1 was stipulated probably by lower gas retaining capacity of foamed paste. This was confirmed by perforation of interpore membranes in foamed concrete (Figure-3) which increased with the increase in content of gas forming agent. Hence, the content of hydrogen peroxide in the mix should be restricted to 1.75-2.25%.

Hydrogen peroxide, providing formation of additional pores, reduced density of the composition. However, the structure formed with hydrogen peroxide was characterized by discontinuities in cells, heterogeneous porosity. The structural defects were attributed to mismatching of binder structure formation and gas filling of the bulk, which was hindered in cured material. Necessity to heat liquid glass in order to obtain fault-less fine porous structure of liquid glass compositions is demonstrated (Table-8, Figure-4).

Table-7. Influence of hydrogen peroxide on properties of alkaline compositions on the basis of waste glass

Foaming agent	Content of hydrogen peroxide in the blend, %	Foam expansion ratio	Swelling number, %	Average density of foamed aerated concrete, kg/m^3
Fairy	0	5.4	-	500
	0.75		14	440
	1.25		20	400
	1.75		28	340
	2.25		42	280
	2.75		57	230
Zelle – 1	0	5.6	–	480
	0.75		10	420
	1.25		18	380
	1.75		22	340
	2.25		31	270
	2.75		37	210

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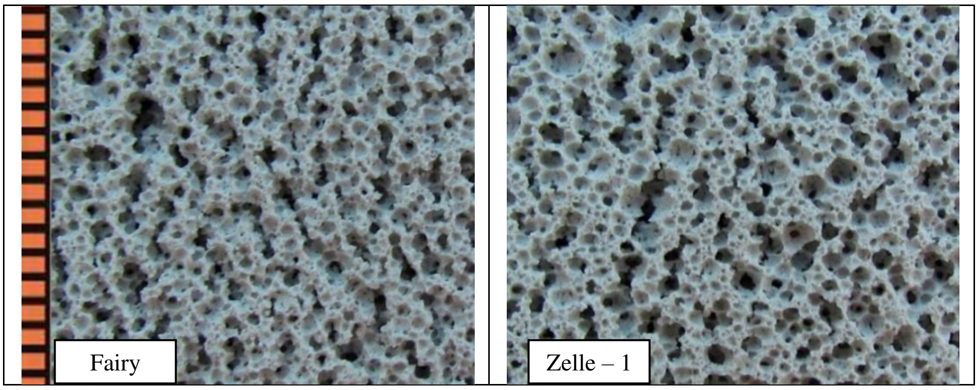

Figure-3. Structure of foamed concrete on the basis of waste glass with various foaming agents and hydrogen peroxide (1.75%).

Aeration depends on paste foaming mode. Investigation into the influence of mixer rotation speed on properties of foamed paste provided the following results.

Table-8. Influence of molding blend production on properties of foamed aerated concrete based on liquid glass.

Production method	Spread diameter, mm		Average density, kg/m^3	Ultimate compression strength, MPa	Efficiency coefficient
	before foaming	after foaming			
W/o heating	85	55	515	1.1	0.21
With heating	85	60	417	1.2	0.28

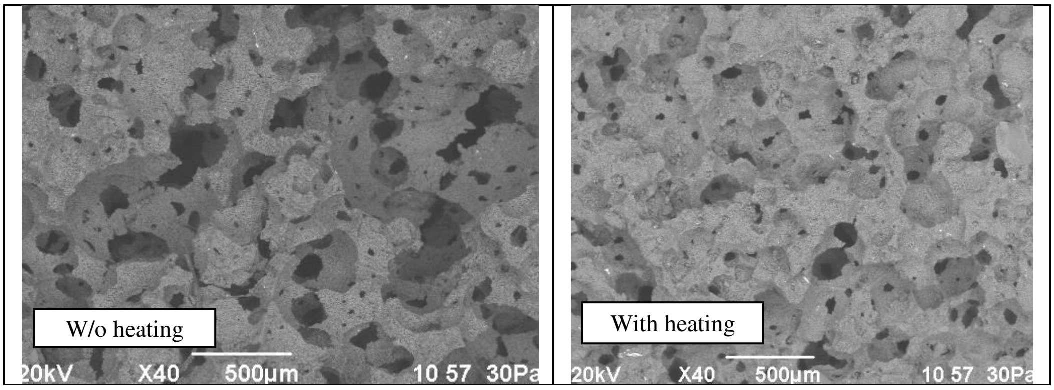

Figure-4. Microstructure of liquid glass compositions with hydrogen peroxide.

In order to obtain steady fine porous foamed paste with high expansion rate the reasonable speed is 600-1000 rpm. Actual speed depends on viscosity of initial paste and increases with its increase.

The quality of foamed paste significantly depends on design of mixer moving part. The influence of contact surface of mixer working unit on the state of foamed paste has been studied. The experiment has been carried out with different length of wire on the working units used for increase in contact surface area. It has been determined that with the increase in the wire length the quality of foamed paste improves. Expansion ratio of foamed paste: without wire - 5.0; with wire, 100 mm - 6.0; with wire, 200 mm - 7.5; with wire, 300 mm - 8.5. However, further increase in the wire length leads to decrease in expansion ratio and quality of foamed paste.

3.2 Porous granules based on liquid glass compositions

Swelling of materials based on liquid glass is due to removal of water, which is a part of sodium hydrosilicates. The amount of water in sodium hydrosilicates depends on the temperature of formation of a crystalline phase, silicate module, and alkali concentration. The content of liquid glass significantly affects porous aggregate's structure.

Liquid glass is a multifunctional component of the composition studied. At the stage of molding it provides powder mass pinning and formation of granules. During heat treatment it reduces the sintering temperature, provides porosity formation. Compositions with different liquid glass content were studied. Firing was carried out at a temperature of 800^{0}C (Table-9). An increase of liquid glass from 40 to 45% in the composition leads to a decrease in the density of granules by 210 kg/m^3.

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Table-9. Liquid glass effect on granules' structure and properties.

Content of liquid glass, %		
40	**45**	**50**
Granules density, kg/m^3		
820	**610**	**590**
		

Granules from a composition containing 45 % of liquid glass are characterized by intense expansion and high porosity. The average pore size is from 0.5 to 1.5 mm. Increase in the content of liquid glass is accompanied by a decrease in granules' strength and the formation of large pores. Therefore, the content of liquid glass in the composition should not exceed 45 %.

The basic raw material composition contained liquid glass and ground glass break. This composition of the raw mix made it difficult to form granules. Fired material was characterized by uneven porosity, brittleness. To improve properties of a composition and granules, various mineral additives effect was investigated. Introduction of mineral additives has improved molding properties of the composition. The flocking rock provides high plasticity of the composition and strength of granules before firing.

To clarify the content of mineral additives, the structure of fired granules was investigated (Figure-5).

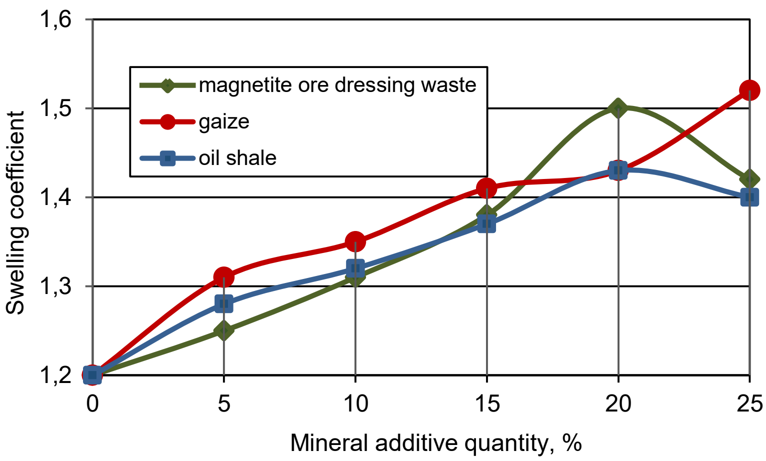

Figure-5. Mineral additives effect on granules swelling.

The pore-forming ability of magnetite ore dressing wastes is due to dehydration of aluminosilicates, decomposition of pyrite, calcite. Oil shale contains organic matter; during its combustion a gas phase is formed. Flask rock provides formation of pores in the granules due to evaporation of water, released during dehydration of minerals.

Influence of mineral additive type is observed at a concentration of 20 %, along with that the largest swelling of granules is provided by magnetite ore dressing waste. For compositions containing 25 % of mineral additives, a high coefficient of expansion is achieved when using gaize. At high concentrations of magnetite ore dressing waste the melting point decreases and the gas-holding ability of the composition decreases. As a result, the number of pores in the granules decreases, while the size of the cells increases. When firing a composition containing oil shale, part of the organic matter burns out until the mass softens, therefore, the coefficient of expansion of granules is small.

The type of mineral additives affects the nature of porosity (Figure-6).

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Figure-6. Effect of mineral additives on granules' structure.

Comparison of granules structure obtained from mixtures with the optimal content of mineral additives showed that the most uniform distribution of pores is achieved when using gaize. In this case, the highest porosity is formed using wastes from enrichment of magnetite ores.

This served as a prerequisite for combination of various mineral additives in the composition. It is established that combining gaize and magnetite ores enrichment waste, the best conditions for granules' formation are provided. Expanded granules have a high and uniform porosity. When combining gaize and oil shale, the coefficient of expansion increases with a lower content of mineral additives.

Therefore, to improve technological properties of the composition and increase the porosity of granules, a multicomponent feed mixture is preferred. The properties of granules synthesized by the developed technology are investigated (Table-10).

Table-10. Comparative characteristics of porous granules.

Characteristics	Types of filling aggregates	
	lightweight aggregate concrete	synthesized granules
Firing temperature, °C	900-1200	725
Density of a granule, kg/m^3	790	490
Bulk density, kg/m^3	450-500	250-300
Heat conductivity coefficient, W /(m·°C)	0.09	0.06

Compared with expanded clay gravel, a new porous aggregate obtained from industrial raw materials at a low firing temperature, has improved thermal characteristics.

4. CONCLUSIONS

Aeration of alkaline silicate compositions is sensitive to variation of state of raw components, recipe and foaming modes of molding blend.

Steady fine porous liquid glass foamed paste is obtained using synthetic foaming agents of anionic or non-ion types characterized by high foaming ability and stability in liquid with expressed chemical activity and adjustable density.

Complex aeration of liquid glass compositions by foaming in high-speed mixer and subsequent aeration using hydrogen peroxide promotes formation of polydisperse structure of foamed aerated concrete with average density of 300-400 kg/m^3.

It has been established that the use of a multicomponent composition based on liquid glass provides a highly porous granule structure due to a combination of various pore formation mechanisms. Possibility of regulating the porosity of granules through a combination of various mineral additives of technogenic origin was revealed.

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