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Porous composite material based on liquid glass

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ABSTRACT

The article presents the results of experimental studies of a composite material obtained on the basis of liquid glass and mineral fillers of technogenic origin. The structure of the composite material is formed by porous granules bonded with a liquid-glass matrix. The porous filler is synthesized from a mixture of liquid glass and combined filler (cullet, flake overburden, coal mining waste, and ash microsphere). Regulation of composition and content of the filler in the raw mixture ensures porous granules production with a bulk density of 270 – 330 kg/m³. Analysis of mathematical models reflecting the dependence of the density and strength of the composite material on the composition of the moulding mixture allowed us to establish a reasonable ratio between the liquid glass and the filler, the matrix, and the porous filler. Optimal proportions of the composite material are characterized by a density of 450 – 600 kg/m³ and compressive strength of at least 5.5 MPa. Strong adhesion of the liquid-glass matrix to the surface of the porous filler ensures the resistance of the composite material to diverse effects. The structure of the materials was studied by electron microscopy. The development of composite material is aimed at improving the energy efficiency of construction.

Keywords: composite material, liquid glass, technogenic filler, porous granules, lightweight concrete.

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Introduction

The energy efficiency of construction is largely ensured by the use of composite materials with a porous structure. The advantages of cellular concretes are extensive raw material base, a variety of products and their cost-effectiveness. However, the porous structure of cellular concrete is unstable, which often leads to a decrease in the strength and heat-shielding performance of products. This limits cellular concretes' application [[1], [2], [3], [4]].

Lightweight concretes based on porous fillers are promising composite materials for energy-efficient construction [[5], [6], [7], [8], [9]]. Porous filler is a dominant component, which makes up to 80% of concrete's mass. Structural characteristics of porous filler affect the structure of composite materials, and determine thermal and physicomaterial properties of lightweight concretes. The development of effective porous concrete technology is associated with the

modification of raw materials, and improvement of moulding sand composition.

The structure of composite materials consists of a matrix and a discrete component distributed in the matrix. The block of hardened binder serves as a matrix in light concretes, the discrete component is represented by grains of porous filler. Thermal and physicomaterial properties of lightweight concrete depend on the characteristics of porous filler and a matrix. The matrix connects the filler particles into a monolith and provides uniform distribution of stresses throughout the volume of the material. Types of cement are mainly used as a matrix of lightweight concretes.

Liquid glass is an aqueous solution of alkaline (often sodium) silicates. Liquid glass is characterized by chemical activity and has a high adhesive ability. The rheological properties of liquid glass are regulated by additives and thermal effects. The formation of a highly porous structure of liquid glass

is carried out by swelling, which is accompanied by the formation of cells of various sizes. The introduction of powdered filler into liquid glass can change the nature of swelling. Filled liquid glass compositions serve as a binder for moulding sand to produce porous filler.

Analysis of the last year's achievements indicates the creation of effective porous products based on liquid glass. The technology of granular foam-glass-ceramic materials from industrial waste is being actively developed. Thermal power engineering waste, waste from ores mining and processing, and metallurgical slags are used to obtain porous fillers [[10], [11], [12], [13], [14], [15], [16], [17], [18], [19], [20]]. A number of developments are devoted to highly porous granular materials based on the thermal swelling of liquid glass [[14], [15], [18], [19]]. Polarization of liquid glass composites is carried out mainly by swelling. The choice of fillers, aimed at optimizing compositions' technological state is of importance for the development of effective liquid glass materials. Information about lightweight concretes obtained on the basis of liquid glass is scarce.

The aim of the research is to study composite materials with porous filler; their main component is sodium liquid glass.

Experimental part

The object of the study is composite materials based on liquid glass, of which both components of lightweight concrete were synthesized.

Sodium liquid glass ($\text{Na}_2\text{O} \cdot m\text{SiO}_2 + n\text{H}_2\text{O}$) – an aqueous solution of sodium silicate with a density of 1430 kg/m^3 was used in the experiments. Liquid glass provides binding of components powdered and promotes the thermal expansion of the raw mass.

Materials of various origins were introduced into the liquid glass as filler.

A finely ground cullet of quartz glass (cullet) is fragments of sheet and container glass. The basis of technogenic glass is amorphous silica. Chemical composition of cullet, wt. % as follows: SiO_2 –74; Al_2O_3 – 4; Fe_2O_3 – 2; CaO – 6; MgO – 1; R_2O –13.

Flask sedimentary rock is the overburden during mining. The flask rock is predominantly composed of

amorphous opal and includes cristobalite, tridymite, and admixtures of clay minerals. Chemical composition, wt. % as follows: SiO_2 – 81; Al_2O_3 – 5; Fe_2O_3 – 2; CaO – 2; MgO – 1; R_2O –1. oth. – 7.

Overburdened rocks of the coal basin are coal mining waste. The mineral composition is represented by quartz, kaolinite, pyrite, and siderite; there are feldspars, montmorillonite, anhydrite, and calcite. Chemical composition of overburden rocks, wt %: SiO_2 – 65; Al_2O_3 – 10; Fe_2O_3 –7; CaO – 3; MgO –1; S –3; C – 6; oth. - 5.

Ash microsphere is a loose material consisting of hollow spherical particles with a diameter of 100 – 350 microns. It is formed as a light fraction of ash from the combustion of solid fuels. The bulk density is 400 kg/m^3 . Chemical composition, %: SiO_2 – 83; Al_2O_3 – 5; Fe_2O_3 – 2; CaO – 10.

Moulding compounds were prepared from liquid glass binder and porous filler was developed. The porous filler was synthesized by thermal treatment of the granular material. Granules were moulded on a laboratory drum pelletizer from a mixture of liquid glass and mineral filler.



Discussion of results

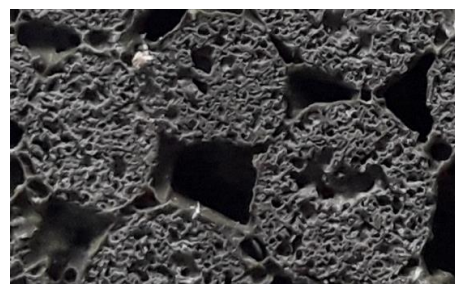
The combination of fillers of different proportions makes it possible to control the viscous and plastic properties of liquid glass and determines the nature of the thermal swelling of the granules. Regulation of the content and composition of the mineral filler affects the temperature of swelling and properties of granular material (Table 1).

Porous granules with a diameter of 6 – 10 mm are characterized by a porosity of 75 – 80%. The porosity of granules fired at 750°C is represented by swelling cells. In granules fired at 350°C , the porous structure is formed by microsphere cavities and swelling cells in the liquid glass component. The efficiency of liquid glass granules is determined by improved thermal performance.

Calculations show that in order to provide thermal resistance of $3.3 \text{ (m}^2 \cdot ^\circ\text{C)/W}$, the heat-insulating layer of liquid glass granules (thermal conductivity coefficient is $0.06 \text{ W/(m} \cdot ^\circ\text{C)}$) should be 20 cm; and the expanded clay layer (thermal conductivity coefficient is $0.09 \text{ W/(m} \cdot ^\circ\text{C)}$) is 36 cm.

Table 1 – Characteristics of the raw mixture and porous granules based on it

Composition of filler in the raw mix, %				Swelling temperature, °C	Bulk density, kg/m ³	Structure of the granules
cullet	flask rock	coal mining waste	ash microsphere			
50	20	30	–	750	270	
–	–	60	40	350	330	

**Picture 1** – Granules grouting by the matrix and porous matrix

Porous liquid glass filler forms the basis of lightweight concretes' structure. A liquid glass binder was used to connect porous filler grains. Preference of liquid glass binder as a matrix for a composite material is due to the high binding ability of liquid glass; ability to control rheological properties of moulding sand; and reliability of adhesion with filler particles having a related origin with the matrix.

A liquid glass binder was obtained by mixing liquid glass with filler, which was used as a substance of various origins. The materials investigated are conditionally divided into the following groups. The first group is formed by materials, the firing of which is accompanied by dehydration and the formation of water vapour (clay, flask, bauxite, expanded clay dust, volcanic glass). Fillers of the second group contain minerals (pyrite, calcite, dolomite), which emit a gas phase during thermal transformations. Fillers of the third group (oil shale, wood flour, coal-

bearing rocks, ash from coal combustion, expanded clay dust) contained a burnable component.

Liquid glass binders containing 40 – 45% additives of the first and third groups are evenly distributed between the filler grains and provide the necessary viscosity of moulding mass. Binding compositions with first group fillers form thin shells around porous filler grains. This ensures the minimization of the matrix in the lightweight concrete structure (Figure 1). Binding compositions with 30 – 35% fillers from all groups exhibit the ability to swell at 200 – 300°C.

Moulding sands were prepared in the following order: liquid glass was mixed with filler (overburdened rocks of the coal basin); granular filler was introduced into the resulting liquid glass suspension. Moulding sand of moderate mobility was placed in cube moulds with a 70 mm edge. To accelerate hardening and ensure water resistance, the samples were subjected to heat at 250°C for 40 min. The strength of samples of composite materials was determined by testing on a hydraulic press. Electron microscopy was used to study the composition of composite materials structure.

As a result of preliminary experiments, approximate ratios between the porous filler and the liquid glass binder were determined, which ensures low-density composite materials production. The mathematical planning method was used to optimize the composition of moulding mass. The conditions of the experiment are shown in Table 2. The moulding sands consisted of pellets fired at 350°C; liquid glass and filler were from coal mining waste. The following factors were taken as the factors studied: the proportion of grains of coarse filler fraction; the ratio between the liquid glass and the filler. Coarse filler is represented by grains 9 – 10 mm in size, fine filler consisted of grains 6 – 7 mm in size.

The planning matrix and the results of the experiment are shown in Table 3.

After statistical processing of experimental data, mathematical models were obtained.

Mathematical model reflecting dependence of composite materials’ density, ρ (kg/m³), on the studied factors as follows:

$$\rho = 635.857 - 0.377 \cdot x_1 + 0.107 \cdot x_1^2 - 475.13 \cdot x_2 + 2.708 \cdot x_2^2 - 1.920 \cdot x_1 \cdot x_2 \quad (1)$$

A geometric illustration of the function is shown in Figure 2.

The dependence of composite materials’ strength (R, MPa) on the composition of the moulding material is obtained in the form of a regression equation of the full quadratic model:

$$R = 5.857 + 0.145 \cdot x_1 - 0.002 \cdot x_1^2 - 3.68 \cdot x_2 + 22.370 \cdot x_2^2 - 0.140 \cdot x_1 \cdot x_2 \quad (2)$$

A geometric illustration of the function is shown in Figure 3.

Table 2 – Conditions of the experiment

Factors		Levels of variation				
Natural	Encoded	- 1.41	- 1	0	+ 1	+ 1.41
Share of coarse filler fraction, %	X ₁	0.0	14.5	50.0	85.5	100.0
Liquid glass / Filler	X ₂	0.55	0.59	0.70	0.81	0.85

Table 3 – Mathematical planning of the experiment

	Factors researched		Share of coarse filler fraction, %	Liquid glass Filler	Density, kg/m ³	Compressive strength, MPa
	X ₁	X ₂	X ₁	X ₂	ρ	R
1	-1	-1	14.5	0.59	650	6.42
2	+1	-1	85.5	0.59	615	5.84
3	-1	+1	14.5	0.81	485	4.96
4	+1	+1	85.5	0.81	420	4.16
5	-1.41	0	0.0	0.70	570	5.17
6	+1.41	0	100.0	0.70	510	4.05
7	0	-1.41	50.0	0.55	680	6.20
8	0	+1.41	50.0	0.85	480	4.10
9	0	0	50.0	0.70	505	4.87
10	0	0	50.0	0.70	500	4.93

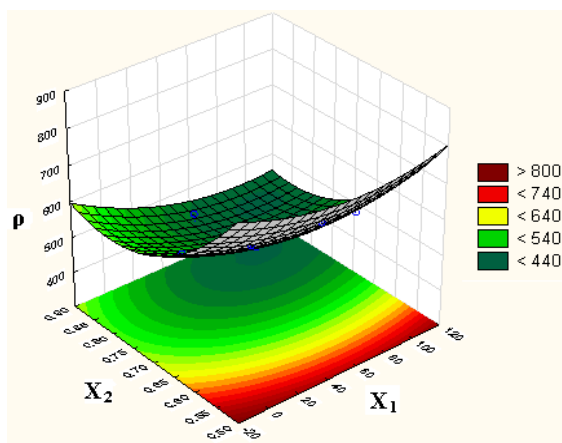


Figure 2 – Response surface of the dependence of composite materials’ density on moulding mass’ composition

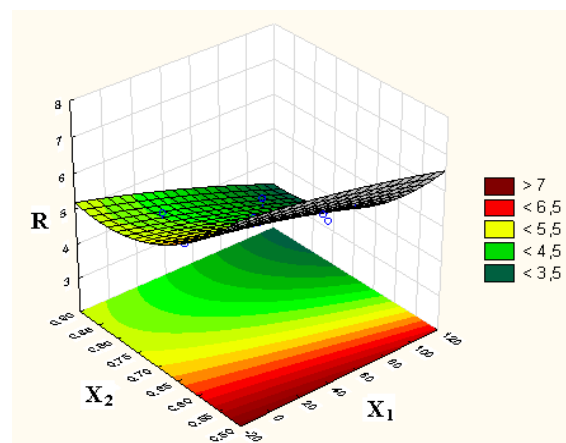


Figure 3 – Response surface of the dependence of composite materials’ compressive strength on moulding mass’ composition

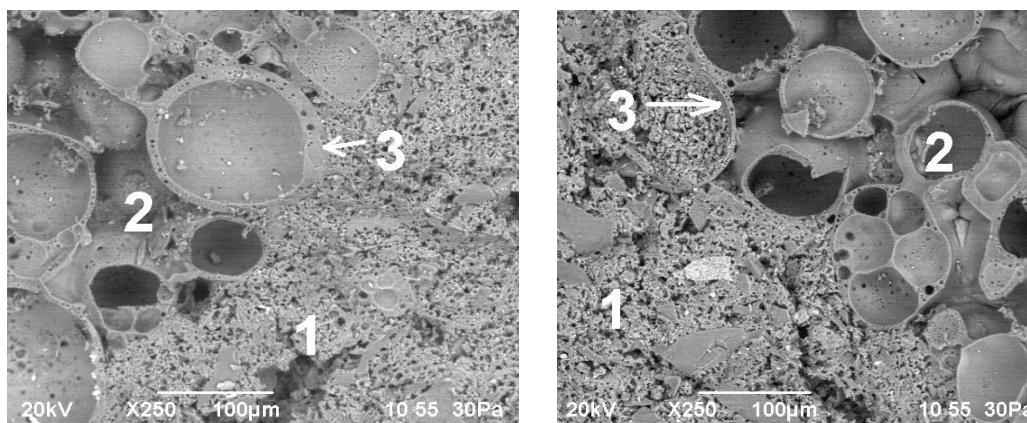


Figure 4 – Microstructure of the composite material: 1 - binder block; 2 - filler; 3 - contact zone



Figure 5 – Composite material sample before testing



Figure 6 – The nature of destruction of the composite material during the strength test



Figure 7 – Fragment of a chip of composite material's sample

The obtained mathematical models indicate that the ratio between liquid glass and filler has a decisive influence on the density of composite materials. An increase in the proportion of filler increases the density of composite materials. The influence of the fractional composition of the filler is less significant and is expressed in a decrease in density of the composite as coarse grains of the filler increase. An increase in the strength of composite materials is facilitated by an increase in the proportion of coarse filler, limiting the content of the binder in the moulding sand.

The analysis of mathematical dependences makes it possible to single out the range of compositions of composite materials with density values of not more than 600 kg/m^3 . This is ensured when the content of the coarse filler is not less than 20%, and the ratio of liquid glass to filler is not less than 0.65. Requirements for strength indicators of composite materials (not less than 5.5 MPa) thin the area of preferred mixes of composite materials: the proportion of coarse filler is 50 – 80%, the ratio between the binder and the filler is 0.65 – 0.75.

The results of the mathematical model analysis

are consistent with the conclusions of numerical studies of the packing of filler in a composite material. A comparison of various porous filler packing schemes showed that the minimum stress and deformation in a composite material are typical for a two-component model that contains 60 – 70% porous filler granules. At the same time, the proportion of large grains in the filler composition is 50 – 60%

The study of the contact zone between the matrix and the surface of the porous filler indicates a close contact of the components (Figure 4). The nature of the destruction of the samples during the strength test confirms the high adhesion strength of the filler and the block of the liquid glass binder (Figures 5 and 6). The fracture of the samples occurs along the binder block and along the filler grain (Figure 7).

In terms of physical and mechanical parameters, liquid glass composite materials are not inferior to cement lightweight concretes of comparable density. Test results testify to the resistance of composite materials under conditions of variable values of humidity and temperature.

Conclusions

Proportions of the composite material based on liquid glass are proposed. The matrix and porous granules of composite materials are synthesized from mixtures of liquid glass and mineral fillers of technogenic origin.

Polarization of liquid glass granules is provided by thermal expansion and the presence of a hollow microsphere.

The range of proportions of the composite material, characterized by a density of 450 – 600 kg/m³ and compressive strength of at least 5.5 MPa, has been determined. Composite material's proportions with a binder-filler ratio of 0.25 – 0.40

are preferable when large particles of the filler dominate.

Genetic similarity of the raw basis of the matrix and porous granules ensures the reliability of the adhesion of the components and the high technical properties of the composite material.

Conflict of interest. The author declares that there is no conflict of interest.

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Сұйық шыны негізіндегі кеуекті композициялық материал

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ТҮЙІНДЕМЕ

Мақалада сұйық шыны және техногендік текті минералды толтырғыштар негізінде алынған композициялық материалдың эксперименталды зерттеулерінің нәтижелері берілген. Композиттік материалдың құрылымы сұйық шыны матрицамен бірге ұсталған кеуекті түйіршіктерден тұрады. Кеуекті толтырғыш су шынысының және аралас толтырғыштың (куллет шыны, колбаның үстіңгі қабаты, көмір өндіру қалдықтары, күл микросферасы) қоспасынан синтезделді. Шикізат қоспасындағы толтырғыштың құрамы мен мазмұнын реттеу 270 – 330 кг/м³ көлемді тығыздығы бар кеуекті түйіршіктердің алынуын қамтамасыз етеді. Композиттік материалдың тығыздығы мен беріктігінің қалыптау құмының құрамына тәуелділігін көрсететін математикалық модельдерді талдау сұйық шыны мен толтырғыш, матрица және кеуекті толтырғыш арасындағы ұтымды байланыстарды орнатуға мүмкіндік берді. Композиттік материалдың оңтайлы композициялары 450 – 600 кг / м³ тығыздықпен және кем дегенде 5,5 МПа қысу күшімен сипатталады. Кеуекті толтырғыштың бетіне сұйық шыны матрицасының күшті жабысуы композиттік материалдың әртүрлі әсерлерге төзімділігін қамтамасыз етеді. Материалдардың құрылымы электронды микроскоп арқылы зерттелді. Композиттік материалды әзірлеу құрылыстың энергия тиімділігін арттыруға бағытталған.

Түйін сөздер: композициялық материал, сұйық шыны, техногендік толтырғыш, кеуекті түйіршіктер, жеңіл бетон

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Мирюк Ольга Александровна

Поризованный композиционный материал на основе жидкого стекла

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АННОТАЦИЯ

В статье приведены результаты экспериментальных исследований композиционного материала, полученного на основе жидкого стекла и минеральных наполнителей техногенного происхождения. Структура композиционного материала сформирована пористыми гранулами, скрепленными жидкостекольной матрицей. Пористый наполнитель синтезирован из смеси жидкого стекла и комбинированного наполнителя (стекольный бой, опоковая вскрышная порода, отходы добычи угля, зольная микросфера). Регулирование состава и содержания наполнителя в сырьевой смеси обеспечивает получение пористых гранул с насыпной плотностью 270 – 330 кг/м³. Анализ математических моделей, отражающих зависимость плотности и прочности композиционного материала от состава формовочной смеси, позволил установить рациональные соотношения между жидким стеклом и наполнителем, матрицей и пористым наполнителем. Оптимальные составы композиционного материала характеризуются плотностью 450 – 600 кг/м³ и прочностью при сжатии не менее 5,5 МПа. Прочное сцепление жидкостекольной матрицы с поверхностью пористого наполнителя обеспечивает стойкость композиционного материала к различным воздействиям. Структуру материалов исследовали методом электронной микроскопии. Разработка композиционного материала направлена на повышение энергетической эффективности строительства.

Ключевые слова: композиционный материал, жидкое стекло, техногенный наполнитель, пористые гранулы, легкий бетон.

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