## ВЛИЯНИЕ КРЕМНЕЗЕМА НА ПРОЦЕСС ПРОИЗВОДСТВА И СВОЙСТВА ПОРИСТОГО СТЕКЛОКОМПОЗИТА

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Статья посвящена разработке состава и получению пористого стеклокомпозита теплоизоляционного назначения по одностадийной технологии с пониженным содержанием NaOH при температурах не более 850 °C. Для синтеза стеклокомпозита использовано кремнеземсодержащее природное (трепел) и техногенное (микрокремнезем) сырье. Предложен состав шихты на основе трепела и микрокремнезема, обеспечивающий получение пористого стеклокомпозита по энергосберегающей технологии. Активированная гидроксидом натрия смесь трепела и микрокремнезема является альтернативой традиционному способу получения пеностекольных материалов. Высокая дисперсность сырья, аморфно-кристаллическое строение трепела и аморфное строение микрокремнезема обеспечивают высокую реакционную способность шихты. Новизна исследования заключается в снижении количества щелочи до 10,5 % и получении материала с повышенной механической прочностью (до 4 МПа). Установлено, что повышенная механическая прочность материала обусловлена растворением остаточного кварца и кристаллизацией кристобалита. а также получением однододной мелкопористой структуры пористого стеклокомпозита со средним размером пор 0,6+ 0,2 мм. Добавление в шихту SiO<sub>2</sub> в виде микрокремнезема в количестве от 10 до 50 мас.% снижает температуру вспенивания с 860 до 820 °С и повышает прочность материала с 1,5 МПа (без микрокремнезема) до 4 МПа (30 мас.% микрокремнезема). Полученный пористый стеклокомпозит отличается от традиционных теплоизоляторов типа пеностекла повышенной прочностью и рассматривается в качестве теплоизоляционно-конструкционного материала. Статья имеет большое практическое значение, так как решает две задачи. С одной стороны – расширение сырьевой базы производства пористых материалов с привлечением отходов. С другой стороны – расширение области применения за счет увеличения прочности материала.

Ключевые слова: композит на основе пористого стекла, трепел, гидроксид натрия, микрокремнезем, прочность на сжатие, кристаллизация

## INFLUENCE OF SILICA FUME ON THE PRODUCTION PROCESS AND PROPERTIES OF POROUS GLASS COMPOSITE

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The article is devoted to the development of the composition and production of a porous glass composite for thermal insulation purposes using a single-stage technology with a reduced NaOH content at temperatures not exceeding 850 °C. Silica-containing natural (tripoli) and technogenic (silica fume) raw materials were used for the synthesis of glass composite. The composition of the charge based on tripoli and silica fume is proposed, which ensures the production of a porous glass composite using energy-saving technology. A mixture of tripoli and silica fume activated with sodium hydroxide is an alternative to the traditional method of producing foam glass materials.

The high dispersion of raw materials, the amorphous-crystalline structure of tripoli and the amorphous structure of silica fume ensure a high reactivity of the charge. The novelty of the study lies in reducing the amount of alkali to 10.5% and obtaining a material with increased mechanical strength (up to 4 MPa). It is established that the increased mechanical strength of the material is due to the dissolution of residual quartz and crystallization of cristobalite, as well as the production of a homogeneous fine-porous structure of a porous glass composite with an average pore size of 0.6+0.2 mm. Addition of SiO<sub>2</sub> to the charge in the form of silica fume in an amount from 10 to 50 wt.% reduces the foaming temperature from 860 to 820 °C and increases the strength of the material from 1.5 MPa (without silica fume) to 4 MPa (30 wt.% of silica fume). The resulting porous glass composite differs from traditional foam glass type heat insulators in increased strength and is considered as a thermal insulation and structural material. The article is of great practical importance, as it solves two problems. On the one hand, the expansion of the raw material base for the production of porous materials with the involvement of waste. On the other hand, the expansion of the scope of application by increasing the strength of the material.

Key words: porous glass composite, tripoli, sodium hydroxide, silica fume, compressive strength, crystallization

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#### INTRODUCTION

Currently, the construction and engineering industries have growing demand for eco-friendly, nonflammable and durable thermal insulation materials. These requirements are met by silicate materials with developed pore macrostructure, such as foam glass, foam concrete, aerated concrete, foamed silicate and porous glass-ceramics [1-5]. Main fields of application for the materials are: construction of residential, public and industrial buildings, roads, and, in the granular form, as filler in a variety of composites and concrete. Porous composites with glassy and glass-ceramic matrix are high-performance, chemically inert and resistant to water and steam. These materials are used for sound and heat insulation, as catalysts carriers and absorbers of electromagnetic radiation [6-10].

The traditional way of foam glass manufacturing is the powder method based on foaming of the mixture prepared from a glass powder with a blowing agent [11]. Using cullet allows us to recycle non-conforming glass products, part of the municipal waste, and also reduces energy costs for production of the material [12-14].

Using different types of recycled cullet has difficulties associated with the stability of the chemical composition of the glass, deviation from which leads to variations in the foam glass properties [15]. In addition, the glass can be contaminated by waste paper, iron, ceramics, polymers, and other organic substances. The costs associated with the removal of impurities and colour classifications of the glass are relatively high. In some countries, such as in Russia, there is a resource limitation of the cullet, so the issues of expanding the raw material base for the production of foam glass remain relevant. For this reason, recycled cullet is replaced with synthesized quenched cullet [16].

Relatively cheap and easily available raw materials, including partial replacement of the treated cullet by the industrial waste, in particular by ash, can be used as the feedstock [17-18]. Production of foam glass from the granulate is a two-step technology. In the first phase, the granulate is synthesized, followed by preparation of the foaming mixture and the foaming process. The basis of foam glass is the glass phase, its composition and quantity are controlled by the process parameters and by using various raw materials. Selection of raw materials is determined by foam glass production technology, as well as its availability and cost.

Currently, the development of low-temperature methods for obtaining porous glass composites based on industrial waste, including silica fume [19-21], is topical. This will solve environmental problems associated with the disposal of industrial waste and reduce economic costs. Moreover, it allows reducing consumption energy resources and lessens harmful industrial emissions. A single-stage process for the synthesis of a porous glass composite, for example, using sodium hydroxide, is more economically feasible. Natural materials [22, 23] and cullet [24, 25] are used to synthesize foam glass with sodium hydroxide.

The sodium hydroxide not only reduces the temperature of the glass phase synthesis, but also acts as a blowing agent. However, there are disadvantages of the alkali-based technology. First, it requires strict adherence to the safety rules, since sodium hydroxide is a very aggressive agent. Second, high sodium hydroxide content (above 20%) leads to reduction of water resistance of the finished material. This limits the possibility of using the material in building composites, for example, based on Portland cement. Therefore, important are the studies aimed at reducing the sodium hydroxide content in the mixture to obtain a porous glass composite from natural or man-made silica raw materials.

For production of foamed glass with high mechanical strength, special additives are inserted into the feed mixture; this either leads to the crystallization of the new phase, or acts as a reinforcing agent of the glass matrix. Paper [26] confirms high mechanical strength of the foam glass produced from a glass of cathode ray tubes with the addition of silicon carbide in the amount up to 7 wt. %. The mechanical strength of foam glass is increased due to the crystallization of wollastonite and diopside in the vitreous matrix.

Potential for foam glass hardening by crystallization is shown in [27, 28]. Using as Na-Ca silicate glass with the addition of other glass compositions as the matrix and the blowing agent in the form of SiC with the addition of  $MnO_2$  allows us to increase the strength of the foamed glass by partial crystallization of wollastonite. The resulting material has closed pores, an apparent density of 0.5 g/cm<sup>3</sup>, an overall porosity of 78-79%, and it is recommended for industrial use for thermal insulation [27].

The porous glass composite with good thermal insulation properties is obtained from waste borosilicate glasses [28]. This material is different from the traditional foamed glass by high mechanical strength. The authors explain this by the crystallization of wollastonite and cristobalite in the interpore wall of the material.

A high-strength porous glass composite was also obtained by crystallization of anorthite and diopside in a glass matrix [29]. Foaming of the composition is conducted at 900 °C for 30 min, with the blowing agent in the form of silicon carbide. The authors note that even a very small addition of silicon carbide has a significant impact on the foaming process.

Thus, there are various approaches to the preparation of porous glass composite with improved characteristics, in particular, with high mechanical strength. Process optimization of porous glass composite technology provides for: determination of the optimal foaming mode (low-temperature method); choice of feedstock (use of available and common raw materials, including waste); the introduction of various additives that affect the process of foaming and crystallization of the glass phase.

The goal of the work is to establish the effect of silica fume on the properties of a porous glass composite obtained from tripoli with a low content of sodium hydroxide (no more than 11%) at foaming temperatures not exceeding 800-850 °C, and the strength of the finished material up to  $4 \pm 0.5$  MPa.

#### MATERIALS AND EXPERIMENTAL METHOD

#### Materials

Samples of porous glass composite were obtained by one-stage technology. A one-step technique includes a targeted synthesis of hydrated polysilicates from a mixture of silica raw materials with sodium hydroxide. In order to evaluate the potential of this technology, six compositions of the foaming mixture have been tested. Selection of the siliceous raw material was based on the following requirements: the materials should have high dispersion (average particle size of less than 100 µm) and sufficient amount of oxide SiO<sub>2</sub> for the vitrification (at least 70 wt.%). These requirements are met by natural tripoli and man-made metallurgical wastes such as silica fume. According to the data presented in the Table 1, tripoli differs from that of graded quartz sands, normally used for glass melting, as it contains less glass-forming oxide SiO<sub>2</sub> and more Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>. The composition of silica fume is similar to the sand grade, which is used for the production of foam glass, glass containers, insulators, pipes. The chemical composition of the silica raw material indicates its potential suitability for the production of a porous glass composite.

The results of dispersion analysis revealed that natural tripoli contains coarse particles in the amount of 3-7 wt %. Therefore, tripoli has been preliminarily dried and ground in a ball mill. Silica fume is presented mainly as particles of less than 0.5  $\mu$ m and requires no additional preparation. Evaluation of siliceous materials size distribution confirmed that the raw material is finely dispersed and can be considered as a basis for producing a porous glass composite using the one-step process.

Tripoli is a kind of amorphous-crystalline natural siliceous materials. Slight amorphous halo and reflection peaks are found on the XRD, it correspond to quartz and small reflection peaks which are typical for albite (Fig. 1). Phase analysis revealed that the silica fume is amorphous material. Crystal phase is almost missing and presented as silicon carbide. It should be borne in mind that the presence of even small amounts of SiC affects the foaming process and the mechanical properties of the porous material [29]. In this study, crystalline powder of sodium hydroxide is used as the alkaline component, it has the following composition, wt. %: NaOH – 99.5; Na<sub>2</sub>CO<sub>3</sub> – 0.5; NaCl, Na<sub>2</sub>SO<sub>4</sub>, Fe<sub>2</sub>O<sub>3</sub> – trace.

Table 1 The chemical composition of siliceous components Таблица 1. Химический состав кремнистых компо-

нентов							
Description of the	Content of oxides by weight %						
material	SiO <sub>2</sub>	$Al_2O_3$	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO		
tripoli	85.0	8.4	4.6	1.2	0.8		
silica fume	97.8	0.5	0.4	1.3	-		



Fig. 1. The XRD pattern of tripoli:  $Q - \beta$  quartz (0.426; 0.334; 0.182 nm); A – albite (0.318; 0.375; 0.321 nm)

Рис. 1. Рентгенограмма трепела: Q – β кварц (0,426; 0,334; 0,182 нм); А – альбит (0,318; 0,375; 0,321 нм)

## Experimental method

The following characterization methods were used. The study of the material's phase composition as well as finished materials was conducted using XRD on the diffractometer DRON-3M in copper radiation, whilst the quantitative XRD analysis was performed using «Match» software.

The study of the macro- and micro structures of the porous samples was performed using scanning electron microscope (JCM-6000) with an attachment for energy dispersive analysis.

Density and water absorption have been determined in accordance with DIN V 18004. This standard determines that the water absorption is calculated as the difference in the mass of material in the dry state and after 1-hour immersion in water. The compressive strength of the pellets was determined by the following formula [30]:

$$\mathbf{S} = \frac{2.8 \cdot \mathbf{P}_c}{\pi \cdot d^2} \tag{1}$$

where, S is the compressive strength,  $N/mm^2$ ; d is the diameter of the pellets, mm;  $P_c$  is the failure load, N. The tests were carried out on five different samples of each composition.

The value of the porosity of the glass composite was calculated by the formula (2).

$$\mathbf{P} = \left(1 - \frac{d_a}{d_t}\right) \cdot 100\% \tag{2}$$

where, P is the material porosity, %;  $d_a$ ,  $d_t$  is the average and true material density, kg/m<sup>3</sup>.

A comparative analysis of the properties of the obtained samples was carried out by the value of the strength factor, which is calculated as the ratio of the compressive strength to the apparent density of the material (3).

$$\mathbf{K}_f = \frac{\sigma}{d} \cdot 100\% \tag{3}$$

where,  $K_f$  is the strength factor, %;  $\sigma$  is the compressive strength, MPa; d is the apparent density of material, kg/m<sup>3</sup>.

### RESULTS AND DISCUSSION

Optimization of the composition of the charge for the porous glass composite was carried out under the following conditions [16]: 1) the formation temperature of the liquid phase (melt) should not exceed 900 °C; 2) the amount of the melt should be not less than 70%, it is necessary to provide conditions for pyroplastic state at the foaming stage; 3) the liquid phase should have optimum viscosity ( $10^3$ - $10^6$  Pa s) at the temperature range of foaming.

For the pre-selection of the charge composition, the diagram of the ternary system was analyzed Na<sub>2</sub>O–Al<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub>. The choice of the diagram is due to the oxide composition of the tripoli. Concentration range of compositions that have at least 70% of the melt at temperatures below 900 °C contains: SiO<sub>2</sub> (65-80%), Al<sub>2</sub>O<sub>3</sub> (5-15%) and Na<sub>2</sub>O (13-20%). All compositions fall into the same element phase triangle Na<sub>2</sub>O  $2SiO_2$ –Na<sub>2</sub>O Al<sub>2</sub>O<sub>3</sub> 6SiO<sub>2</sub>-SiO<sub>2</sub>. Most fusible eutectic system is in this area, between disilicate, albite and silica with a melting temperature of 740 °C. For comparison, selected are three compositions of tripoli with so-dium hydroxide, which fall into this area (Table 2). NaOH content varied from 10.5 to 14.3 wt. %.

Melting curves of mixtures, calculated from the system Na<sub>2</sub>O-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>, show that 70% of the content of the melt is achieved for the first composition at a temperature of 830 °C, for the second one at 885 °C, and for the third one at 920 °C (Fig. 2). With a decrease in the content of caustic soda in the mixture by 3%, the melting point rises by 90 °C. Temperature increase the melting of the mixture with the decrease in the amount of sodium hydroxide is compensated by addition of the silica fume into the mix.

 Table 2

 Chemical and component composition of the batch

 Таблица 2. Химический и компонентный состав

 шихты

	Componen	Oxide composition of the					
N⁰	of the charge, wt %		glass phase, wt. %				
	tripoli	NaOH	SiO <sub>2</sub>	Na <sub>2</sub> O	$Al_2O_3$		
1	85,7	14,3	76	17	7		
2	87,8	12,2	78	14	8		
3	89,5	10,5	80	8	12		





It is known that the silica fume has a high reactivity and improves the properties of various materials [30]. The porous material, obtained from mixing of silica fume and sodium hydroxide, foams at a temperature as low as 200 °C, but has no water resistance. Therefore, silica fume can be used only as an additional component. Its amount in the mixture was varied from 10 to 50% by weight in 10% increments.

The method of producing the material comprises mixing tripoli and silica fume in a bowl of the mixer-granulator, followed by addition of the alkali component. Two variants were tested using alkali in solid form and in the solution. According to the first option, the batch preparation sequence is as follows: the prepared mixture of solid components was wetted with water, mixed and then dry sodium hydroxide was injected into the mixture.

According to the second option, the prepared mixture of components was wetted with 45% solution of NaOH in the amount of 23 wt. %, stirred for two minutes, after this water was added to the granulator in the amount of 7 wt. %, the rotation speed was reduced,

and granulation continued for two minutes more. It was experimentally established that, when using a solid alkali, its dissolution is accompanied by considerable release of heat and local heating of the charge. This disrupts the uniformity of charge and formation of the pellets that have a wide variety of sizes, from 0.3 to 10 mm.

Introduction of the alkaline component in the form of a solution does not disturb the uniformity of the charge reached at the component mixing step, providing intimate contact of the mixture components through the interlayer of alkali solution. Using 45% alkali solution in an amount of 23 wt. % corresponds to the lowest possible content of sodium hydroxide in a mixture, 10.5 wt. %. Experiments were conducted on compositions with a minimal amount of alkali, which was introduced into the mixture in the solution form, this ensured better condition for silicate formation (Table 3).

Table 3

Component composition of the raw mixture *Таблица 3*. Компонентный состав сырьевой смеси

Mixture compo-	Components content in the mixture, wt. %					
nents						
tripoli	89,5	79,5	69,5	59,5	49,5	39,5
silica fume	0	10	20	30	40	50
NaOH	10,5	10,5	10,5	10,5	10,5	10,5

When adding an alkali solution at the stage of preparation and the batch granulation, it is subject to self-heating to 80 °C. This allows to form of sodium silicate and silica gel in the colloidal state.

Interaction of alkali solution with siliceous component of the charge follows reaction 1.

$$3SiO_{2} + 2NaOH + 3H_{2}O \xrightarrow{80 \ C} \\ \xrightarrow{80^{\circ}C} Na_{2}O \cdot 2SiO_{2} \cdot 3H_{2}O + SiO_{2} \cdot H_{2}O$$
(1)

The resulting raw granules are dried at the temperature of 200 °C to a final moisture content of than 1.5%. The erestrictions of pellets humidity is necessary for the subsequent foaming process. Increased water content leads to inhomogeneous structure of the foamed materials, presence of large cavities, which affect the strength of the finished porous material. In the drying step, a dehydration reaction of sodium silicate hydrates (reaction 2).

$$Na_{2}O \cdot 2SiO_{2} \cdot 3H_{2}O \xrightarrow{200\ C}$$

$$\xrightarrow{200\ C} Na_{2}O \cdot 2SiO_{2} + 3H_{2}O \qquad (2)$$

When heating the granulated material to 750 °C and above, vitrification process is observed at the foaming stage, after which the finished porous material has vitrified surface. The process has the stages nature. First, there is formation of the eutectic melt by

melting the ternary eutectic formed between  $Na_2O$   $2SiO_2$  and  $SiO_2$  and albite at temperature 740 °C and the binary eutectic between  $Na_2O$   $2SiO_2$  and  $SiO_2$  at the temperature 793 °C (reaction 3).

$$Na_2O \cdot 2SiO_2 + SiO_2 \xrightarrow{793 C} Eutectic melt$$
 (3)

Then, at the foaming temperatures (800-900 °C), the residual silica is dissolved in the primary melt, and the formation of sodium alumino-silicate glass occurs during the subsequent cooling (reaction 4).

 $melt + SiO_2 \xrightarrow{850^{\circ}C} melt \xrightarrow{coo \ ling} glassphase \quad (4)$ 

Foaming of the silicate mass is from the gases formed by chemical interaction of the various impurities contained in the silicate mass and by steam, obtained by partial dehydration of hydroxide in the indicated temperature range. It was established experimentally that the optimum heating rate of 15 °C/min, holding time of 10 min. Cooling the foamed material was carried out by gradually reducing the temperature the rate of 5 °C/min.

According to the XRD phase analysis, the dried (up to 200 °C) pellets of all compositions have reflection peaks corresponding to sodium disilicate. With an increase in silica fume content in the charge, disilicate diffraction peak intensity increases, this indicates more intensive silicate formation. The XRD pattern of the dried granules obtained from the batch without silica fume, exhibits diffraction peaks corresponding to the quartz and two diffraction peaks characteristic for sodium disilicate (Fig. 3). When silica fume is added to the initial charge, maximum intensity of reflex, corresponding to quartz, decreases.While new reflexes corresponding to sodium disilicate are profound, as well as the amorphous halo of larger area (Fig. 3).



Fig. 3. The XRD pattern of the dried granules obtained from the charge without (1) and with 50% of silica fume (2): Q – quartz (0.426; 0.334; 0.182 nm); D – sodium disilicate (0.478; 0.315 nm) Рис 3. Рентгенограммы гранул, полученных из шихты без (1) и с 50% микрокремнезема (2): Q – кварц (0,426; 0,334; 0,182 нм); D – дисиликат натрия (0,478; 0,315 нм)

Quantitative XRD analysis of the synthesized porous glass composite showed that the amount of the

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glass phase in the finished product depends on the silica fume content in the raw charge. With increasing silica fume content from 10 to 20%, the glass phase content increases from 74% to 78%, respectively (Table 4). The amount of silica fume in the starting material of 30% results in a decrease of glass phase content to 64%, and when the content of silica fume is 40 and 50%, the content of the glass phase is increased to 69%.

Table 4

Characteristics of porous glass composite Таблица 4. Характеристики пористого стеклокомпо-

Shia							
The sil- ica fume content, wt.%	The content of the phases in the porou glass composite, % glass crystallir phase phase		The po- rosity, %	The av- erage pore size, mm	The strength factor		
0	71	29	74	0.1	0.4		
10	74	26	75	0.3	0.7		
20	78	22	77	0.5	0.8		
30	64	36	78	0.7	1.0		
40	69	31	79	0.6	0.9		
50	70	30	80	0.8	0.9		

Different content of the glass phase in the final product is due to the glass crystallization processes occurring at the stage of pellet foaming. The XRD patterns of all samples contain diffraction peaks corresponding to quartz and new diffraction peaks corresponding to cristobalite (Fig. 4). With an increased content of silica fume in the charge, intensity of cristobalite diffraction peaks increases. If the new formations are not centers of stresses, then crystallization processes should increase the strength of the finished material.



Fig. 4. The XRD pattern of the foam samples as the function of silica fume content in the mixture: Q – quartz (0.426; 0.334; 0.182 nm); C – cristobalite (0.4083; 0.251; 0.2119 nm); A – albite (0.3188 nm) Рис. 4. Рентгенограммы образцов в зависимости от содержания микрокремнезема: Q – кварц (0,426; 0,334; 0,182 нм); C – кристобалит (0,4083; 0,251; 0,2119 нм); A – альбит (0,3188 нм)

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The material strength values indicate the following. With increasing content of silica fume in the charge of up to 30%, the mechanical strength is increased to 3.9 MPa, above this value, the strength is reduced to 3.3 MPa, but still higher than the porous glass composite strength without silica fume addition which is 1.5 MPa (Fig. 5). The strength of the material depends not only on the amount of the crystalline phase therein, but also on the ratio of residual guartz and cristobalite formed. With increasing content of silica fume in the charge, intensity of maximum cristobalite reflection increases, and the intensity of quartz reduces. For the sample with silica fume content of 30%, there are the same intensity maximums of quartz and cristobalite reflection. These samples are characterized by high mechanical strength, which is caused both by dissolving of residual quartz in the melt and crystallization of cristobalite.

It has been established that with an increase in silica fume in the charge, an increase in the average pore size from 0.1 mm (composition without silica fume) to 0.8 mm (composition with 50% silica fume) is observed (Table 4). The value of the porosity of the glass composite, calculated by formula (2), also shows a slight increase with an increase in the amount of silica fume in the charge. The porosity of the glass composite obtained from a mixture without silica fume and a mixture with 50% silica fume is 74% and 80%, respectively. At the same time, the charge foaming temperature decreases from 860 °C (composition without silica fume) to 820 °C (composition with 40 and 50% silica fume) (Fig. 5).



Fig. 5. The compression strength of the porous glass composite and foaming temperature as function of the silica fume content in the charge: 1 - compressive strength, 2 - foaming temperature Рис. 5. Прочность пористого стеклокомпозита и температура вспенивания в зависимости от содержания микрокремнезема в шихте: 1 - прочность на сжатие, 2 - температура вспенивания

The value of water absorption of the obtained samples is  $8.3 \pm 0.2\%$  maximum value is for the composition with 50 wt. % addition of silica fume (Fig. 6). Higher water absorption may be explained by the high

microporosity of the material and the formation of interconnected pores.

Thus, samples of porous glass composite were produced from natural and man-made silica raw materials using one-step alkaline technology. These studies have shown the possibility of reducing the amount of NaOH and use of the waste material as one of the components. Foaming of the mixture of tripoli and silica fume, activated by NaOH, is conducted at relatively low temperatures (at temperature of 830-850 °C). All of this combined allows us to reduce the cost of the process. In addition, the final product of porous glass ceramic is non-combustible and fireproof, unlike commonly used at present organic thermal insulation materials, and it is recommended as an eco-friendly material for thermal insulation purposes. Further adjustment of the material properties can be achieved by using a variety of blowing agents (carbon black, aluminium powder, glycerol), and their combinations.



Fig. 6. The density of porous glass composite and water absorption upon the silica fume content in the mixture: 1 - average pellet density; 2 - water absorption

Рис. 6. Плотность пористого стеклокомпозита и водопоглощение в зависимости от содержания микрокремнезема в смеси: 1 - средняя плотность гранул; 2 – водопоглощение

#### CONCLUSIONS

Analysis of the results allows to draw the following conclusions:

To obtain a porous glass-crystalline material based on natural and man-made silica raw materials using a single-stage technology with a reduced content of sodium hydroxide, the following composition of the charge was developed: 59.5 wt. % of tripoli, 30 wt. % silica fume and 10.5% NaOH. When foaming a charge of this composition (at 830 °C), a porous material (porosity 78%) with an average density of 380 kg/m<sup>3</sup>, water absorption of 8.3% and an average pore size of 0.6 + 0.2 mm is obtained.

Increasing the amount of silica in the charge reduces the foaming temperature from 860  $^{\circ}$ C (charge

without silica) to 820 °C (charge with 40 and 50% silica). With the addition of highly dispersed amorphous silica, the processes of silicate formation are more active, which is confirmed by an increase in the intensity of the diffraction peaks of sodium disilicate. Sodium disilicate has a relatively low melting point (790 °C) and contributes to the formation of the amount of melt necessary for foaming.

When foaming the charge with 30% silica (foaming temperature 830 °C), a porous material with an increased strength of 3.9 MPa is obtained, compared with the strength of samples obtained from other studied compositions. This is due on the one hand to obtaining a uniform fine-pored structure (pore size 0.6 + 0.2 mm). On the other hand, the increased strength of the glass composite is due to the high content of the crystalline phase of 36% due to the crystallization of cristobalite and the dissolution of residual quartz.

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The authors declare the absence a conflict of interest warranting disclosure in this article.

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