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Biostable silicic rock-based glass ceramic foams

Биостойкие пеноситаллы
на основе кремнеземсодержащих пород

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Abstract. The search for the possibility of expanding the resource base through the use of local rocks, as well as reducing the cost of final product, is one of the scientific research areas in the field of obtaining foam glass-based building materials. The aim of the research is the development of compositions and recommendations for the production of silicic rock-based glass ceramic foams. These studies will allow to create strong and durable building materials with low density and thermal conductivity, as well as increased biological stability. The results of studying the phase transformations occurring in the charge (tripoli : soda ash) during heating, obtained by thermal analysis methods are presented as well as the production technology, physico-mechanical and thermophysical properties of the developed glass ceramic foams. As a result, construction materials resistant to aggressive media with a density of 200 to 600 kg/m³, thermal conductivity from 0.053 to 0.115 W/m·°C, compressive strength from 1.2 to 9.8 MPa, have been developed. Due to its properties, developed glass ceramic foams will be used primarily as insulants for the construction of nuclear power plants, in the gas and oil industries, industrial and civil engineering.

Аннотация. Расширение сырьевой базы за счёт применения местных горных пород, а также снижение стоимости готовой продукции, является одним из научных направлений исследования в области получения строительных материалов из пеностекла. Целью исследований является разработка составов и рекомендаций по получению пеноситаллов на основе кремнеземсодержащих пород. Данные исследования позволят создать прочные и долговечные строительные материалы с низкой плотностью и теплопроводностью, повышенной биологической стойкостью. Представлены результаты исследований фазовых превращений, происходящих в шихте (трепел : кальцинированная сода) при нагревании, полученные методами термического анализа; а также технология получения, физико-механические и теплофизические свойства разработанных пеноситаллов. В результате разработаны строительные материалы плотностью от 200 до 600 кг/м³, теплопроводностью от 0,053 до 0,115 Вт/м·°С, прочностью при сжатии от 1,2 до 9,8 МПа, стойкие в условиях агрессивного воздействия биологических сред. Разработанные пеноситаллы благодаря своим свойствам найдут достойное применение в первую очередь в качестве утеплителя при строительстве АЭС, в газо- и нефтепромышленности, промышленном и гражданском строительстве.

1. Introduction

Foam glass is a unique building material, which consists of glass cells for almost 100 %. Foam glass-based materials are light, have very low thermal conductivity, sufficient operational durability, do not shrink and do not change the geometric dimensions over time under the influence of operational loads, withstand high temperatures during operation, corrosion-resistant. All this ensures the reliability and quality of the final

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product, which allows us to recommend this material primarily as an insulant for the construction of nuclear power plants, in the gas and oil industries, industrial and civil construction [1]. The tightening of thermotechnical requirements for enclosing structures became an additional reason for the massive use of this material in the reconstruction of existing building projects and the construction of new ones [2–4].

Modern world scientific research in the field of producing the foam glass-based building materials has the following directions: search for not material-intensive production methods of foam glass [5–7]; low-temperature synthesis of glass mass without the use of glass-melting furnaces [7–9]; foaming of the charge mixture while bypassing the process of high-temperature melting glass [7–11]; expansion of the resource base through the use of various types of glass, cullet and local rocks, which allows to significantly increase the availability of raw materials and, at the same time, reduce its cost [5, 7, 9–19]; use of various gasifiers [6, 20–25]; optimization of foaming and annealing thermal modes [26].

The production technology of foam glass is quite complex. The first step is melting the glass. Then, the cooled glass is grounded with gas-forming additives and reheated, followed by annealing of the material obtained. The line of scientific research we offer implies the abandonment of the first stage (glass melting), the founding and foaming of the charge mixture should be carried out for one heating, which will allow to significantly reduce production costs. In addition, this technology allows the use of cheap components in the production, which are available in Russia in large quantities, (diatomite, tripoli, flask, etc.).

Physical-chemical bloating processes of zeolites, clay and perlitic rocks, slag and glass are well studied. These mechanisms cannot be correlated with the processes occurring during the bloating of silica-containing rocks, such as diatomite, tripoli and flask. However, science knows cases of foaming of tripoli with the addition of alumina to its composition, as well as foaming of diatomites with the addition of NaOH or KOH aqueous solutions [7, 10, 11].

Suggested foaming method of silica-containing rock is based on the uniqueness of its natural composition, or on rationally selected one during the production. The chemical composition of diatomite, tripoli and flask does not contain a sufficient amount of elements included in the composition of fluxes flows (Na, K, etc.), which are required in order to obtain low-temperature eutectics, to reduce the viscosity of the glass phase, etc. The main rock component is cristobalite (SiO_2), which, in the presence of calcite (CaCO_3) microcrystalline structure (regulates the melting temperature, viscosity, improves the mechanical and chemical properties of the future material), as well as of soda ash (Na_2CO_3) (reduces the melting temperature) begins to react when heated to a temperature of about 400 °C with the formation of silicates. The silication rate is the higher, the higher is the activity of the charge mixture components, and it also depends on the amount of alkaline and alkaline-earth components in the composition. Silication also accelerates in the presence of moisture in the composition, especially hydrate one, as well as it depends on the fineness degree of the charge mixture. It is known that the formation of devitrite-type ternary compounds is already finished at a temperature of 600–650 °C in mixtures consisting of silica, soda ash and a sufficient amount of alumina. The eutectics formed by these compounds and sodium silicates melt already at a temperature of 710–760 °C [27]. There are no bloating components in many silica-containing rocks. The rock presented in this work has at least two components that can be classified as bloating ones: muscovite, which releases structural water at a temperature of about 700 °C (Figure 1, Differential thermogravimetric analysis (DTG) curve 2) and heylandite, which is characterized by stepwise dehydration up to 700 °C temperature [28]. Therefore, it can be assumed that the charge mixture consisting of silica-containing rock and soda ash, which is presented in this paper, will foam when heated to a 750–800 °C temperature.

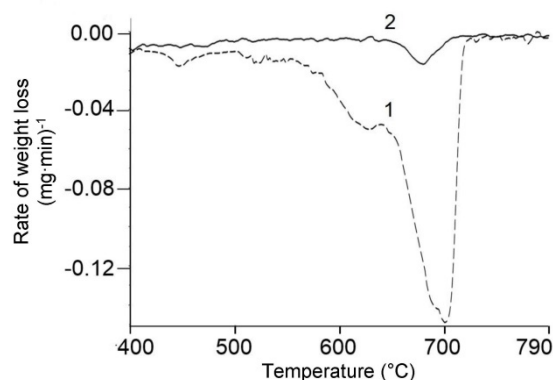


Figure 1. DTG curves of the rock (1) and muscovite (2).

The conducted researches are aimed to the development of the compositions and recommendations for the production of silicic rock-based glass ceramic foams. The following problems were solved:

– the phase transformations occurring in the charge mixture during heating, as well as the phase composition of glass ceramic foams were studied;

– the physical, mechanical and thermophysical properties of the glass ceramic foams, as well as their biological resistance were studied.

2. Methods

The following materials were used as raw materials for the production of highly biostable glass ceramic foams:

– silica-bearing rock (tripoli), which deposit is near the Engalychevo village, Dubensky District, Republic of Mordovia, chemical composition: SiO_2 – 71.00 %, CaO – 9.01 %, Al_2O_3 – 8.90 %, Fe_2O_3 – 2.86 %, K_2O – 2.06 %, MgO – 1.61 %, TiO_2 – 0.444 %, Na_2O – 0.252 %, P_2O_5 – 0.171 %, SrO – 0.064 %, BaO – 0.029 %, SO_3 – 0.027 %, ZrO_2 – 0.017 %, V_2O_5 – 0.012 %, MnO – 0.012 %, Cr_2O_3 – 0.009 %, Rb_2O – 0.010 %, CuO – 0.008 %, ZnO – 0.005 %, other impurities – 3.50 %, mineralogical composition: cristobalite (SiO_2) – 42.1 %, heylandite ($(\text{Ca}, \text{Sr}, \text{K}_2, \text{Na}_2)[\text{Al}_2\text{Si}_6\text{O}_{16}] \cdot 5\text{H}_2\text{O}$) – 17.7 %, muscovite ($\text{KAl}_2[\text{AlSi}_3\text{O}_{10}](\text{OH})_2$) – 14.4 %, calcite (CaCO_3) – 13.9 %, quartz (SiO_2) – 11.2 %, tridymite (SiO_2) – 0.7 %. DTG of the rock is presented in Figure 1, curve 1.

– first grade industrial soda ash, which meets the requirements of all-Union State Standard 5100-85. Its chemical formula is Na_2CO_3 .

The charge mixture for the manufacture of glass ceramic foam was obtained by mixed grinding of the above-mentioned rock dried to constant weight at $t = 105$ °C, and soda ash until the specific surface was equal to 1000–1100 m^2/kg . The obtained mixture was then poured into a metal mold, pre-treated with kaolin coating and compacted. The form with the mixture was set in a muffle furnace and heated at a speed from 1.5 to 4.5 °C/min to a temperature from 750 to 950 °C with soaking for 30 minutes at the maximum temperature. After cooling the mold with the obtained material and the furnace to 40 °C, it was disassembled, and the material was removed for further testing.

Phase transformations occurring in the charge mixture during heating were studied using the TGA/DSC1 device. 0.15–0.16 g of the mixture was weighed to the accuracy of 0.0001 g and poured into an alundum thimble with a volume of 150 mcl. The sample was compacted by tapping the thimble on the table. Next, the thimble was placed on the holder and then in an oven. The sample was heated from 30 to 900 °C at a rate of 10 °C/min.

The phase composition of glass ceramic foams was determined by X-ray phase analysis (XRD) using an ARL X'tra diffractometer (Switzerland). Diffractograms were recorded on $\text{CuK}\alpha_{1+2}$ radiation in the angle range $2\theta = 4$ –70° at a speed of 2 °C/min. During shooting, the sample was rotating at a speed of 60 revolutions/min. Qualitative phase analysis was performed according to the Hanavalt method using the ICDD PDF-2 database. Quantitative X-ray phase analysis was performed according to the Rietveld method using the software Siroquant 3 Sietronics Pty Ltd.

The physical and mechanical properties of the developed material were determined in accordance with all-Union State Standard 33949-2016.

The thermal conductivity index was determined by the probe method in accordance with all-Union State Standard 30256-94.

The mold fungi treeing of glass ceramic foams obtained from charge mixtures with different compositions was determined using beam samples of 10×10×30 mm in size according to all-Union State Standard 9.049-91. Methods 1 (without additional sources of carbon and mineral nutrition) and 3 (using solid Czapek's nutrient medium) were used, the funginertness and fungicity were determined.

3. Results and Discussion

In order to approve the above-mentioned statement, thermal analysis methods (differential thermal analysis (DTA) and differential thermogravimetric analysis (DTG)) were used to study phase transformations of the charge mixture that was ground to a specific surface area of 1000–1100 m^2/kg (a mixture of tripoli and soda ash in the ratio from 85:15 to 76:24). The methodology of the experiment is described above. The compositions and research results are presented in Figure 2.

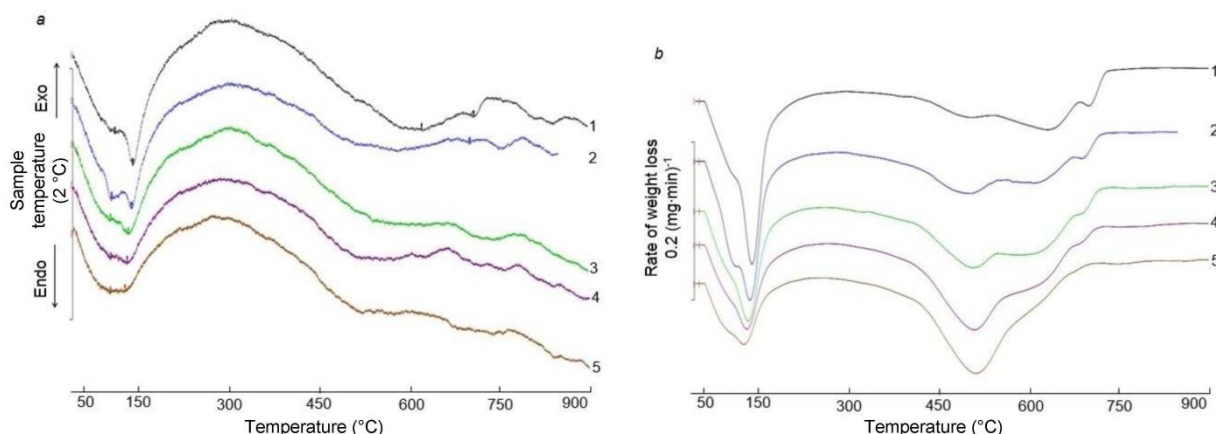


Figure 2. DTA (a) and DTG (b) curves of the mixture of tripoli and Na_2CO_3 with ratios: 1, 2, 3, 4, 5 – 85:15; 82.5:17.5; 80:20; 78:22; 76:24 respectively.

According to the data presented in Figure 2, the following main phase transformations were identified as a result of heating the mixture. The first peak in the 25 to 100 °C temperature range (endoeffect) corresponds to the dissociation of crystalline unbound water. The second peak at a temperature near 150 °C corresponds to the dissociation of the NaHCO_3 formed due to the presence of an insignificant moisture amount in the charge mixture. The third and fourth endoeffects at temperatures of 240 and 350 °C correspond to the dehydration of heylandite in the rock. The following endothermic effect and a significant mass loss in the 400 to 550 °C temperature range corresponds to the formation of sodium silicates, the intensity of which increases with an increase of Na_2CO_3 content in the mixture. The endothermic effect in the 550 to 700 °C temperature range corresponds to the decarbonization of unreacted calcite, as well as to the dehydration of structurally bound water in the rock. The intensity of this peak decreases with an increase of soda ash content in the mixture. The last peak (endoeffect) and mass loss in the 700 to 720 °C temperature range corresponds to the release of structural water from muscovite. Melting of the charge mixture, according to the data in Figure 2, a, begins at a temperature of about 650 °C. All the foregoing confirms the assumption that obtaining foam material from silica-containing rock during one mixture heating is possible.

After heating the charge mixture (consisting of tripoli and soda ash at a 80:20 ratio, respectively) to a maximum temperature of 750 °C, the composition of the calcine consisted of 60 % amorphous phase and 40 % quartz according to the result of the XRD (Figure 3). With an increase of the maximum temperature by 100 °C, the calcine consisted of 55 % of amorphous phase, 26 % of wollastonite, albite and devitrite of 6.5 % each, and 6 % of quartz. When the maximum temperature was increased by 100 °C more, the composition of the material was represented by the 60 % of amorphous phase and 40 % of wollastonite. According to the data obtained, the developing material was named as glass ceramic foam. The photo of the experimental sample of glass ceramic foam is exposed in Figure 4.

Studies of the physical, mechanical and thermophysical properties of the developed materials were carried out in order to confirm the foregoing. The compositions and research results are presented in Table 1.

The research was conducted in order to determine the correlations between the changes in average density as well as compressive strength of the obtained material and the quantitative content of soda ash in the composition (Table 1, C1-C3). The mixture was heated to 850 °C at a rate of 4.5 °C/min. According to the data obtained, the average density of glass ceramic foam reduced slightly from 600 to 570 kg/m^3 with an increase in the content of soda ash in the mixture from 15 to 17.5 %. A further increase of the soda content up to 20 % leads to a directly proportional material density decrease to 220 kg/m^3 .

The compressive strength of the obtained material has a similar correlation regarding the above-mentioned factors. With an increase of the Na_2CO_3 content in the mixture from 15 to 17.5 %, the compressive strength decreases slightly from 9.8 to 8.2 MPa. With an increase of the quantitative content of soda up to 20 %, the compressive strength decreases to 1.2 MPa.

According to the conducted research, the rational content of the soda ash should be in the range of 15 to 20 % during the production of glass ceramic foam based on a mixture of silica-containing rock (tripoli) and soda ash. A further increase of Na_2CO_3 will lead to a significant increase in the liquid phase, as well as in the cost of the final product.

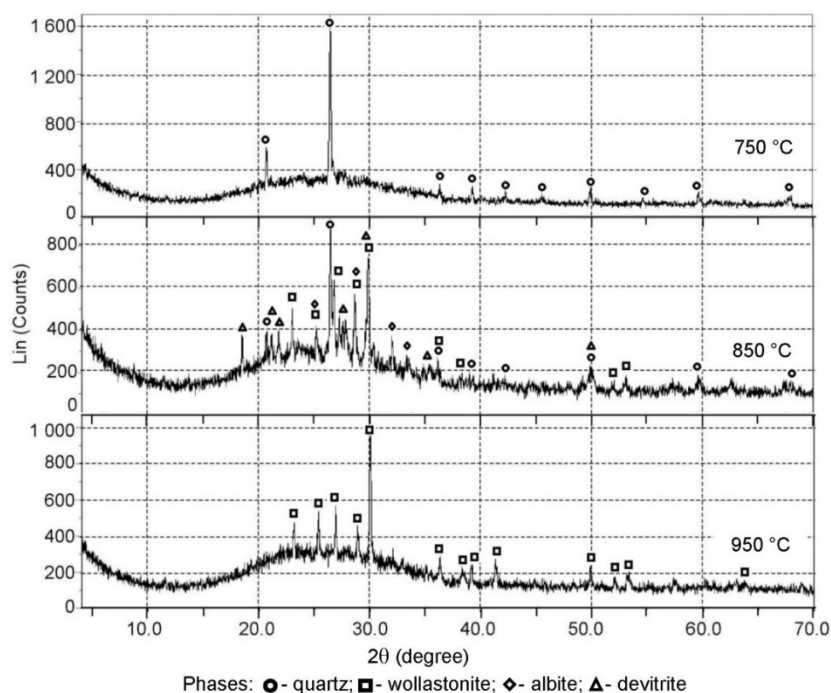


Figure 3. XRD of glass ceramic foams obtained with various maximum heating temperatures of the mixture.



Figure 4. Experimental glass ceramic foam sample.

Table 1. The compositions and properties of the developed materials.

The compositions and properties		Indicators for compositions										
		C1	C2	C3	C4	C5	C6	C7	C8	C9	C10	
Composition (%)	tripoli	85	82.5	80								
	Na ₂ CO ₃	15	17.5	20								
Maximum mixture heating temperature (°C)		850				750	800	820	850	900		
Heating rate (°C/min)		4.5		3	1.5	4						
Properties												
Average density (kg/m ³)		600	570	220	240	285	305	250	200	230	260	
Compression strength (MPa)		9.8	8.2	1.2	1.5	1.9	1.8	1.42	1.28	1.25	1.2	
Water absorption (%)		–	–	20	22.5	34	5.1	10.3	15.9	20	7	
Thermal conductivity (W/m·°C)		0.115	0.104	0.053	0.054	0.068	0.063	0.055	0.053	0.054	0.061	

Further studies were devoted to determining the correlations between the change in the average density, as well as water absorption and compressive strength of the obtained material, and the heating rate of the charge mixture (Table 1, C3-C5). In this regard, the mixture containing 20 % soda ash was heated in a muffle furnace to a temperature of 850 °C at a 1.5 to 4.5 °C/min rate with a soaking at the maximum temperature for 30 minutes. According to the data obtained, the average density of glass ceramic

foam increases almost positively associated from 220 to 285 kg/m³ while the heating rate decreases from 4.5 to 1.5 °C/min.

The water absorption of the obtained material after 1 day of soaking in water was slightly increased from 20 to 22.5 % by volume while reducing the heating rate from 4.5 to 3 °C/min. A further decreasing the heating rate to 1.5 °C/min leads to a water absorption increase up to 34 %.

According to the obtained data, the compressive strength of the developed material in the dry state increases from 1.2 to 1.5 MPa with a decrease in the heating rate from 4.5 to 3 °C/min. A further decreasing the heating rate to 1.5 °C/min leads to an increase in compressive strength of glass ceramic foam to 1.9 MPa. The compressive strength of a material in a water-saturated state slightly decreases regardless of the heating rate of the charge mixture.

According to the conducted research, the heating rate should vary from 3 to 4.5 °C/min during the production of glass ceramic foams based on a mixture of silica-containing rock (tripoli) and soda ash.

Studies aimed at determining the correlations between changes in the average density, water absorption, compressive strength of the developed materials, and the maximum heating temperature of the mixture are important (Table 1, C6-C10). For this purpose, the mixture containing 20 % of soda ash was heated in a muffle furnace at a rate of 4 °C/min to a 750 to 900 °C temperature with 30 minutes soaking time. According to the obtained data, the average density of the developed material decreases from 305 to 200 kg/m³ with an increase in the maximum mixture heating temperature from 750 to 820 °C. A further increasing the heating temperature up to 900 °C leads to the average density increase up to 260 kg/m³.

The water absorption of the material by volume after 1 day of soaking in water increases from 5.1 to 20 % with an increase in the heating temperature from 750 to 850 °C, and its increase from 850 to 900 °C leads to a decrease in water absorption to 7 %.

The compressive strength of the obtained material in the dry state decreases from 1.8 to 1.2 MPa with an increase in the maximum mixture heating temperature from 750 to 900 °C. The compressive strength of glass ceramic foam in a water-saturated state, reached at the mixture maximum heating temperature of 750 °C, is almost 20 % more than the strength of the material in the dry state. This can be explained by the ability of this material to interact with water as a result of its insufficient heat treatment. At the maximum heating temperature of the charge mixture from 800 to 900 °C, the compressive strength of a water-saturated glass ceramic foam is almost equal to the compressive strength in a dry state.

According to the conducted research, the production of glass ceramic foam based on a mixture of silica-containing rock (tripoli) and soda ash requires the maximum heating temperature from 800 to 850 °C.

The studies of the correlation between thermal conductivity change of the obtained material and the temperature as well as the heating rate of the mixture are set out below (Table 1).

According to the obtained data, the thermal conductivity of the developed material decreases from 0.063 to 0.053 W/m·°C with an increase of the maximum mixture heating temperature from 750 to 820 °C. With a further increasing the maximum temperature up to 900 °C, the thermal conductivity increases to 0.061 W/m·°C.

The thermal conductivity of glass ceramic foam is decreasing from 0.068 to 0.054 W/m·°C with an increase in the heating rate of the mixture (maximum heating temperature equals 850 °C) from 1.5 to 3 °C/min. A further increase of the heating rate to 4.5 °C/min does not have a significant effect on the thermal conductivity of the obtained material.

It was determined that in order to obtain the lowest thermal conductivity of the developed material, the heating rate of the charge mixture should vary from 3 to 4.5 °C/min, and the maximum heating temperature from 800 to 850 °C.

Recently, the problem of biological corrosion of materials has become especially urgent. The corrosion increases with high humidity, cyclically operating temperatures and other environmental factors. The world annual economic damage from biodeterioration reaches tens of billions of dollars. The appearance of buildings and the indoor ecological situation are getting worse, and the list of human diseases caused by microscopic organisms is expanding [29–31]. Further research results (Table 2) are devoted to studying the impact of the mixture composition on treeing and the dominant microorganism species on the glass ceramic foam sample.

According to the research results, the developed glass ceramic foams with a density of 200 to 600 kg/m³ are funginert, i.e. they are not a nutrient source for mold fungi, and after a month of testing in a standard filamentous fungi environment, 2 types of micromycetes of the genus *Penicillium* (*Penicillium cyclopium* and *Penicillium chrysogenum*) were identified on their surface as well as 1 species of the genus *Trichoderma* (*Trichoderma viride*). It was determined that the fungi on the samples develop very slowly, which indicates the antifungal properties of the obtained material.

Table 2. The impact of the mixture composition on treeing and dominant microbial species on glass ceramic foam sample.

No.	Mixture composition, %		Characteristic according to all-Union State Standard 9.049-91		Dominant microorganism species on the sample
	Tripoli	NaCO ₃	Method 1	Method 3	
1	85	15	0	5	Penicillium cyclopium, Penicillium chrysogenum, Trichoderma viride
2	82.5	17.5	0	4	Penicillium cyclopium, Penicillium chrysogenum, Trichoderma viride
3	80	20	0	4	Penicillium cyclopium, Penicillium chrysogenum, Trichoderma viride

4. Conclusions

1. Thermal insulating materials based on tripoli and soda ash were developed using only one charge mixture heating with a glass-ceramic structure and 200 to 600 kg/m³ density, 0.053 to 0.115 W/m·°C thermal conductivity, compressive strength from 1.2 to 9.8 MPa, as well as increased biological resistance.

2. The phase transformations occurring in the mixture (tripoli : soda ash) during heating were determined using the thermal analysis. The silicate formation in the mixture begins at a temperature of about 400 °C, and melting mixture is at about 650 °C. The foaming process is carried out due to separating the structural water from muscovite, which is a part of the tripoli, at a temperature of about 700 °C.

3. The phase composition of the crushed baked material was determined using the XRD method, which consists of crystalline phases by 40–45 % and of amorphous phases by 55–60 %. The obtained material was named as glass ceramic foam.

4. It was determined that the production of glass ceramic foam based on a mixture of silica-containing rock (tripoli) and soda ash requires the rational content of the soda ash in ranges from 15 to 20 %, the heating rate of the mixture is about 4 °C/min, and the maximum heating temperature is about 820 °C.

5. The developed glass ceramic foams are funginert, which makes it possible to recommend their use in buildings and structures with aggressive biological media.

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