On the results of integrated researches of the initial raw material and fenced glass structure with the application of modern physical and chemical methods

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Abstract. Studies have been conducted on the structures of the initial raw material and obtained on their basis foam glass of the “aluminosilicate rock – cullet – sodium hydroxide” systems with the use of X-ray and thermal analysis. The interrelation of the prescription-technological parameters and the physical and mechanical properties of the foam glass of the system “cullet – aluminosilicate rock – sodium hydroxide” has been established. Compositions of foam-forming mixtures have been developed, thus making it possible to obtain foam glass with the density from 412 to 1200 kg m$^{-3}$ and strength from 2.0 to 16.1 MPa.

Depending on their functional purpose, all materials used in construction have various physical and mechanical properties. Any properties of the material are influenced by their chemical and phase compositions, production methods, as well as the internal structure [1-5]. Of all the above characteristics, the structure of the thermal insulation materials is crucial. This circumstance requires a thorough study of the structure of the obtained materials and their starting components.

To study the structure, compositions and properties of the materials obtained, as well as the feedstock in a comprehensive way, the following physical and chemical methods were used: X-ray phase analysis; IR spectroscopy electron microscopy; thermal analysis. All studies were carried out on the basis of the Progress Collective Use Center of the Innovation Center of the FSBEI HE ESSUTM, as well as the Buryat Scientific Center of the RAS SB.

During the research and experimental design, clay and perlite deposits of the Buryat Republic, bar glass and sodium hydroxide were considered as raw materials.

X-ray phase analysis of cullet, as a rule, reveals their X-ray amorphous. The infrared spectra of cullet (figure 1) show the presence of a wide absorption band in the high-frequency region of the spectrum at 1050 cm$^{-1}$, which corresponds to the vibrations of relatively highly polymerized groups of [SiO$_4$] tetrahedra. At a frequency of 772 cm$^{-1}$ absorption bands indicate an increased degree of polymerization of silicon – oxygen groups upon incorporation of [AlO$_4$] groups into the silicon and oxygen network. At 600 cm$^{-1}$ the absorption band indicates the insignificant presence of various modifications of Si$_2$O– in the cullet structure, which can be attributed to Si–O–Si bonds as the second tone of stretching vibrations. The presence of effects in the range of 1510–1679 cm$^{-1}$ can be explained by the deformation
vibrations of free water molecules, adsorbed by glass. The absorption band at 2878 cm$^{-1}$ may be caused by stretching vibrations of hydroxyl groups.

According to the electron microscopic studies, the structure of cullet is homogeneous with sufficient uniformity of its chemical composition.

According to X-ray powder diffraction data (figure 2), the structure of clay rocks is represented by montmorillonite (0.457; 0.447; 0.245; 0.256; 0.223; 0.167 nm), kaolinite (0.713; 0.446; 0.357; 0.259; 0.233 nm), calcite group minerals (1.424; 0.302; 0.289; 0.279; 0.213; 0.182; 0.159 nm), low-temperature quartz (0.425; 0.334; 0.228; 0.182; 0.154 nm), hydromica (0.996; 0.446; 0.285; 0.2558; 0.197; 0.178 nm), feldspars (0.3236; 0.318; 0.259; 0.178 nm).

Figure 1. IR spectroscopy of cullet.

Figure 2. XRD clay.
IR spectroscopy of clay rocks (figure 3) shows that the presence of the absorption band at frequencies of 1000–1008 cm\(^{-1}\) in the structure of the starting materials is characterized by stretching vibrations of siloxane groups, and the presence of bands in the range 500–700 cm\(^{-1}\) is associated with the presence of silica modifications. The bands in the range of 3500-3600 relate to OH-stretching and deformation vibrations of free and bound water in the form of free hydroxyl.

![Figure 3. ICA – clay rock spectrum.](image)

In the range from 4 to 40 \(\theta\) the roentgenogram of vitreous perlite (figure 4) demonstrates the peaks of low intensity, from which reflexes with \(d/n = 4.191; 3.346; 3.223; 2.817\) Å can be distinguished. Hence it follows that glassy perlite is practically X-ray amorphous.

![Figure 4. X-ray analysis of perlite rock.](image)

Glass-perlite infrared spectroscopy (figure 5) showed the presence of a wide absorption band in the high-frequency region of the spectrum at 3500 cm\(^{-1}\). The presence of weak doubles at frequencies of 2850 and 2910 cm\(^{-1}\) characterizes hydrogen bonds formed by hydroxyls bound to silicon atoms. The absorption band with a maximum at 1650 cm\(^{-1}\) corresponds to the deformation vibrations of O–H bonds and indicates the presence of molecular water and water in the form of free hydroxyl. An intense absorption band at 1050 cm\(^{-1}\) in perlite rock indicates a strengthening of bonds in natural glass. Doubles at frequencies of 710–780 cm\(^{-1}\) are characteristic of absorption bands of crystalline quartz. The presence of various modifications of \(\text{Si}_2\text{O}^-\) in perlite is also indicated by the absorption band with a maximum at the frequency of 475 cm\(^{-1}\).
The materials studied were brought into a finely ground state by mechanical grinding in a VI 350x4 vibratory grinder. The glass charge for producing foam glass was prepared as follows: after preliminary crushing to the size of no more than 2 mm cullet pieces were dosed by weight in appropriate proportions and crushed in the vibratory grinder to the specific surface of 300–350 m² kg⁻¹.

After crushing clay rock and fine perlite were further crushed in the vibratory grinder to the specific surface of at least 350 and 450 m² kg⁻¹, respectively.

The components were closed with an aqueous solution of sodium hydroxide. The molding moisture content of the samples pressed was 16–19%. The prepared samples were subjected to heating and foaming in the EKPS-10 laboratory muffle furnace with a maximum rise temperature of up to 1100 °C in accordance with the conventional heating, foaming and cooling modes.

It has been established that the introduction of aluminosilicate rocks — clay and perlite in the presence of an alkaline component into the composition of foaming mixtures during the production of foam glass, creates conditions for the directed regulation of the phase composition and the properties of the synthesized foam glass.

Along with the previously used methods, the results of the research prove the expediency of using mechanical and alkaline activation of thermal treatment of clay rock, which contributes to the intensified process of firing foam glass due to newly formed low-melting compounds. The use of X-ray phase analysis helps to study the structures of the starting raw materials, intermediate products, and finished foam glasses of the “aluminosilicate rock - cullet - sodium hydroxide” system.

In conclusion, we have developed the compositions of foaming mixtures that make it possible to obtain foam glass with an average density of 412–542 kg m⁻³ and compressive strength of 2.0–3.6 MPa for heat-insulating products and foam glass with an average density of 588–1200 kg m⁻³ and compressive strength of 1.7–16.1 MPa for structural heat-insulating products at energetically favorable temperature conditions ranging from 850 °C to 900 °C.

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