One of the most effective ways to address the problem of energy saving is to reduce consumption of heat through the walls of buildings' structures, constructions, industrial equipment, heat pipelines and other facilities that exploit heat carriers. As a rule, the task of energy saving is complicated by the corresponding unresolved environmental issues. For example, efficient thermal-insulation of enclosing structures will make it possible to reduce the need for heating buildings and for fuel, accordingly. The result will be a decrease in the amount of greenhouse gas release into the atmosphere, with beneficial effects on the ecology of the environment. According to estimates from EURIMA (European Association of manufacturers of insulating materials), the amount of CO₂ emissions in Europe reaches about 3,000 million tons per year. When thermal insulation is applied, this number decreases by 10−12 %. Thus, the creation by state of favorable conditions for the development of technology of insulation processes, as well as for the production and use of these materials, will make it possible to solve a complex task on saving energy and the environment.

One of the directions to solve this problem is the development and implementation of effective thermal insulation porous materials. The relative simplicity of technology, as well as attractive thermal-physical characteristics, could enable extensive application of such materials in the industry. However, in order to produce efficient porous heat insulators, it is necessary to examine in detail the features of technologies for bloating the raw materials, which are determined by considerable energy consumption and are virtually uncontrollable. This is explained by the insufficient attention of researchers to the development of theoretical and technological basis of the given processes. In this connection, a top priority problem is to improve manufacturing technology of porous materials by selecting optimal composition from cheap raw materials.

Thus, the relevance of present work is predetermined by the need to improve the composition of raw materials, production technology, based on the devised theoretical base of bloating processes. In addition, the theoretical framework will serve as a means of control over the process of receiving materials with predictable thermal-physical characteristics.

2. Literature review and problem statement

Alkali-silicate porous materials, obtained by thermal or cold foaming of aqueous solutions of alkali metal silicates (soluble glass) or solid alkali-silicate hydrogels [1–5], refer to modern efficient inorganic isolates.

Special gas-forming agents are employed in the production of foam glass. The manufacturing process of foamed glass typically implies fabrication of a batch that consists of 95–97 % of powdered glass and 3–5 % of gas-forming agents (carbonate, such as limestone or carbon, e.g., charcoal, coke,
of sodium silicates with various additives, transformed into the gel state in advance. To transform raw materials from the fluid medium into condensed paste-like state for subsequent granulation, it is possible to add not only a hydrophobic agent, but the acidic components as well. Thus, in [12], authors propose adding boric acid. In [13], it is proposed to increase the content of acid oxides in the composition by adding not only mineral acid, but disperse acidic oxides as well, preferably SiO2 and Al2O3, and baked clay, too. In paper [14], it is proposed to add to the specified composition fly ash from thermal power plants.

Modification of additives to soluble glass is also proposed in study [15]. In the described invention, soluble glass is mixed with Portland cement and sodium hexafluorosilicate. The resulting mass is poured into molds and is exposed to heat treatment in the furnace. In the process of heating, the raw material mass is additionally bloated, thereby acquiring the necessary properties. The main tasks that the authors set implied resolving the problems of release of a gaseous pore-forming agent. They failed, however, to take into account both the energy intensity of technological process and the cost of the raw material mix.

At present, there are two technologies for manufacturing thermal insulation materials from rare-earth compositions. The main difference between them is the technique for preparing starting raw materials with liquid glass [16]. Both technologies are two-stage and include stages of granulating the gel-like raw material mixture and subsequent heating the granules in a closed mold at temperatures within 400–450 °C.

When employing technologies of granulation of raw material mixtures, the difficulties arise related to the granulation of large fillers while maintaining the necessary concentration of solutions of chlorides of Al, Ca, Mg, and compositions in the working cycle.

An interesting technical solution to this problem is the variant, proposed in paper [17], of a starting raw material mixture and a production technology of alkali-silicate insulation material. The process of obtaining granulated “aerated glass” includes homogenization by mixing the components of the above starting mixture and subsequent thermal treatment at a temperature of 110–115 °C. In the process of transformation of rheological indicators, viscosity increases significantly while the original liquid system turns into a plastic fluid mass. When cooled to room temperature, the product fully hardens and acquires fragility, required for the subsequent crushing to pieces. After crushing, it is fractionated with the formation of microspheres. Heat treatment is performed in a boiling layer or in a drum furnace at 350–600 °C. The application of such a procedure results in a number of technological problems related to the rheological and environmental difficulties during introduction of hydrophobic agents into composed system.

In paper [18], authors report research findings related to the creation of microporous ceramics from silicon carbide and to the study into properties of the obtained materials. The technology is based on the thermal bloating of the raw material mass, but the study did not take into account the possibility of a temperature decrease of bloating due to the optimization of composition of a raw material mixture, as was the case, for example, in [19]. In the given studies, the authors examined the mechanisms of dehydration and recrystallization of minerals of hydrated calcium silicate using a TGA method. The main stages of recrystallization underlie
the present work. However, the process of formation of a porous structure was not considered by authors of [19].

The authors of article [20] explored the possibility of using solid residue of wastewater and coal ash as a mineral filler. The processes of physical-chemical transformations in the raw mixture, occurring when heated, have not been studied. Similar studies are presented in [20] in which light aggregates were obtained based on the sandy sediment and zeolite rocks. The modes of heat treatment, materials properties were not investigated, as was the case, for example, in paper [21]. However, the specified work, similar to all reviewed above, was not intended to carry out a comprehensive study into:

- selection of a raw mix composition, based on the low-cost raw materials, in particular, utilizing a technogenic waste – ash from thermal power plants;
- optimization of the mixture to reduce temperatures of plasticization and bloating;
- examination of the kinetics of physical-chemical transformations at heating the mixture, as well as the properties of the obtained material.

3. The aim and objectives of the study

The aim of present study was to create highly efficient porous thermal insulation materials based on cheap raw materials.

To accomplish the set aim, the following tasks have been formulated:

- to choose and explore the composition of a raw mixture, which is easily plasticized and bloated at relatively low temperatures;
- to examine a possibility of replacing silicate components of the mixture with ash from thermal power plants;
- to determine optimal temperature regimes of bloating.

4. Special features of thermal bloating of the raw mixture

An analysis of the compositions of starting mixtures and techniques, proposed by various authors for producing thermal insulation materials, proves that the introduction of a significant number of gel-forming agents has a serious shortcoming. A gel-forming agent destroys the structure of soluble glass with the formation of gel of hydrosilicic acid that can retain less water. This adversely affects porosity of the derived compositions. It is therefore necessary to introduce such substances, which are inert relative to the soluble glass at a normal temperature.

At relatively low temperatures, the process of pore formation is complicated due to the low speed of heating the internal volumes of a raw mass. This leads to an increase in the duration of the process of formation of porous structure. Slow heating of mixtures also leads to significant losses of chemically bound water. High speed and uneven heating affect the size, pore regularity, and the strength of porous structure. Therefore, an important prerequisite for obtaining a porous material with a set of required properties is compliance with the principle of matching the rate of crystallization and the removal of chemically bound water.

In all the techniques described above, the first stage of technology implies obtaining a hard or plastic composition from soluble glass, which can further be exposed to heat treatment. That is why there is no need to use different additives that cause coagulation of the mixture. It is possible to get a plastic composition using soluble glass, simply by adding an inert particulate component.

The studies conducted are aimed at searching for and developing of the optimal variant of a mixture of starting materials of silicon oxide that contains a technogenic component – ash from thermal power plants. A new method for obtaining porous alkaline silicate composite thermal insulation materials differs from analogues by the composition of the mixture, the content of starting bulk, consistency and the modes of formation of the target product, as well as by the applied technological equipment.

The DTA curves of hydrated mixture based on ash, Portland cement, silicate registered four endothermic effects, accompanied by a decrease in mass (Fig. 1).

Significant endoeffect was observed at temperatures of 50–250 °C with an extremum from 115 °C to 123 °C and is associated with the removal of adsorbed water from gel-like hydration products, such as calcium hydrosilicates of the type CSH (I), as well as crystallization water from calcium hydrosulfoaluminate of the AFT-phase.

The next endothermic effect that was registered in the temperature range of 370–420 °C characterizes the process of dehydration of calcium hydroxide by the scheme: \( \text{Ca(OH)}_2 \rightarrow \text{CaO} \cdot \text{H}_2\text{O} \). Minor endoeffects at 652 and 780 °C are associated with the processes of decomposition of calcium hydrosilicate (CSH (II)), as well as decarbonization of calcite. In the region of temperatures close to 930 °C – also an endoeffect caused by the crystallization of tobermore-like gel (C–S–H) into wollastonite (\( \text{Ca}_3\text{Si}_2\text{O}_6\)). Curve 2 in Fig. 1 corresponds to the results of DTA of the mixture with lower ash content.

Based on an analysis of endothermic processes occurring during bloating, we expected certain differences from the diagram (Curve 1). Indeed, when substituting chemical compounds in the mixture with ash, the bloating processes do not change qualitatively, but energy characteristics become significantly different.

With less ash content, all endothermic effects increase and slightly shift towards the region of higher temperatures. This is explained by the activation in the processes of hydration of clinker minerals of cement with the accumulation of a large number of new formations under conditions of a growing degree of crystallization. Total differential loss of mass of the sample in the temperature range of 25...800 °C amounted to 23 %.

Further experiments were performed using the DTA method.
5. Discussion of results of research into the processes of bloating

The method of thermal bloating of a bulk of raw materials includes four basic stages:
1) preparation and homogenization of the starting components of the mixture;
2) mixing of the composite system with soluble glass and formation of stable gel, fragmentation of the bulk of raw materials and pouring of the granulate into molds;
3) heating and transformation of the mixture into the pyroplastic state (110–115 °C);
4) further foaming and formation of porous macro structure of composite systems (110–160 °C), formation of properties of the target treated product (200–500 °C).

In this case, the foaming agent is water (mostly silanol or molecular, bound with hydrogen bonds to the unmodified oxygen atoms), which is released during heat treatment of composite systems.

The raw mixture may contain industrially soluble glass, power stations ash of mixed chemical composition (Table 1), Portland cement and, in addition, a thickener (partially dehydrated tempered “dry glass” prepared in advance).

| Chemical composition of ash from thermal power plants, % by weight |
|----------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|
| SiO₂                 | 51.68            |
| Al₂O₃                | 16.75            |
| Fe₂O₃                | 4.47             |
| MgO                  | 0.88             |
| CaO                  | 4.38             |
| Na₂O                 | 0.35             |
| K₂O                  | 2.58             |
| MnO₂                 | 0.04             |
| TiO₂                 | 0.86             |
| SO₃                  | 4.24             |
| P₂O₅                 | 0.49             |

In the prepared samples, fly ash shows good reinforcement properties, high thermal stability, sufficient resistance to aggressive media, has low bulk density.

Results of the studies by authors of [22, 23] (on the ability of alkali-silicate systems with Al₂O₃ to form insoluble products of Na₂O-Al₂O₃·2SiO₂·nH₂O in alkaline media) specify aluminum oxide of ash as a modifying component. It provides raw mixture with qualities required for the target product creation.

When forming a raw mixture, we considered results of improving the water resistance of an alkali-silicate composition by replacing hydrophobic components of dicalcium silicates (belite) with Portland cement; results of these studies are reported in [24, 25].

Controlling the rate of formation of xerogel of hydrosilic acid was performed using various properties of the applied thickener. The proposed starting mixture makes it also possible to overcome the difficulties related to drying a viscous mixture with liquid glass when removing large quantities of water (56–62 %). The resulting water content is within 33–38 %, which ensures certain properties of the hydrogel, required for thermal bloating. The designed composition of a raw mixture makes it possible to form thermal insulation materials with different thermal-physical characteristics. An important prerequisite for reproducibility is strict compliance with technological parameters, established as a result of experimental research.

In parallel with the development of formulation, we conducted study into technology of manufacturing the samples. In this case, in contrast to technology in [17], we exclude the stage of granulation of a raw mixture after heat treatment at a temperature of 110–115 °C and the usage of closed molds during thermal annealing of the bloated material.

The determining factor in the process of thermal treatment of the mixture was the speed of its heating. A choice of optimal thermal regime was motivated by empirical data obtained during thermal bloating and by the resulting properties of material. We have employed a method of differential thermal analysis (DTA) for experimental studies. Research results are shown in Fig. 2.

The technological process includes three main stages whose duration and character depend on the type and amount of water contained in the starting mixture:
– in the temperature range of 100–110 °C, a composite system is partially transformed into a pseudo-plastic state and starts to bloat with an increase in volume;
– at temperatures of 116–147 °C, an intensive release of free and adsorbed water occurs;
– at temperatures above 147 °C, a removal of the bound moisture is observed, the restructuring is completed, as well as the physical and chemical transformations in the material.

Based on an analysis of thermographic data and macrostructure of the obtained samples, it can be concluded that the bound water exerts the greatest influence on the formation of a homogeneous porous structure of the product. Removal of excessive adsorption moisture at the initial stages leads to the formation of large pores and capillary channels in the bulk of raw materials. That is why the starting composition from liquid glass and ash must contain a minimum quantity of free and adsorbed water.

It is possible to recommend the following effective techniques to reduce the effects of free water:
– direct thermal dehydration and transformation of soluble glass into a xerogel;
– liquid granulating of composite systems (for example, in the solutions of chlorides Al, Ca, Zn, Mg, or mixtures);
– introduction of mineral fillers or chemical additives to the composite system from liquid glass.

Based on the study results [26], alkali-silicate compositions in solutions form at heating a series of hydrated
associates with various properties. This makes it possible (Fig. 3) to modify properties of the “dry glass” by partially removing water in the liquid state at different temperatures.

Excessive amount of liquid glass in the mixture, on the one hand, improves rheological properties, plasticity of the treated raw mixture, while, on the other hand, increases additional viscosity of the system. This worsens heat transfer conditions, requires maintaining the temperature longer at its higher values, and leads to an increase in energy consumption. That is why there is a need to find an effective technique to control the rate of gel formation. To do this, we shall use the method of “shift” in the balance of physical and chemical processes of dehydration of disperse systems by adding less hydrated forms of dried soluble glass.

This method for preparing a starting mixture enables obtaining thermal insulation materials for different purposes: granular insulating filler (Fig. 4), materials for thermal insulation of complex configuration (Fig. 5), lamellar and film types of insulation materials (Fig. 6). A choice of the type and shape of thermal insulation design can be made at the final stages of technological process by employing several techniques for obtaining the products.

Advantages of the proposed method are:
– the availability of components, the ease of receiving them and preparing a starting mixture;
– fast obtaining of thermal insulation;
– easy formation and fragmentation of billets (possibly, at a construction site);
– low shrinkage of starting mixture;
– stability of properties of thermal insulation material, high thermal and chemical resistance, incombustibility, ability to withstand considerable temperatures;
– a winning combination of consumer characteristics: low coefficient of thermal conductivity, durability, low cost, low energy consumption in the technology of bloating.

Fig. 7 shows a flow chart of the technological process of producing porous thermal insulation.

Thermophysical properties of materials are given in Table 2.

![Fig. 3. Solubility in the system of Na$_2$O–SiO$_2$–H$_2$O](image)

![Fig. 4. Samples of granular thermal insulation fillers received in the lined molds without volumetric limitation: a, b – isometric elements; c – molded materials of complex configuration; d – thermally treated materials in open molds](image)

![Fig. 5. Fragments of sections in the thermal insulation zones of structures with complex shape, produced by filling a working zone with fragmented elements and subsequent thermal treatment in demountable molds of varying complexity: a – without limiting the free volume; b – with constrained space of formation](image)

![Fig. 6. Fragments of materials: a – in the form of films; b – in the form of plates](image)

Table 2

| Indicator                                  | Bloated material | Porous concrete |
|--------------------------------------------|------------------|-----------------|
| Heat conductivity, W/(m·K)                 | 0.045–1.3        | 0.09–1.7        |
| Application temperature, °C               | to 1200          | to 1000         |
| Resistance to heat transfer, m$^2$/S/W     | 3.8–4.7          | 3–4             |
| Compression strength, MPa                | 0.6–3            | 3–8             |
| Water absorption, %                       | 3                | 6               |
| Bulk density, kg/m$^3$                     | 160–180          | –               |
| Density, kg/m$^3$                         | 700–800          | 860–920         |
| Coefficient of linear temperature expansion, µm | 0.4             | 0.2             |
| Porosity, %                               | 75–80            | 65–75           |
| Coefficient of bloating                   | 7–8              | 3.5             |
The materials proposed could be applied in various technological processes for thermal protection of the surfaces.

6. Conclusions

1. We proposed a composition of the raw mixture that contains soluble glass, power plants ash, Portland cement, and a thickener. The optimum ratio of components was experimentally determined, at which the mixture is easily transformed into a plastic gel-like state and bloats at relatively low temperatures (110–150 °C).

2. Using a differential thermal analysis, we determined temperatures of the basic stages of heat treatment and studied dynamics of physical-chemical transformations occurring in the process of heating and bloating; the possibility is shown of substituting silicate components of the mixture with ash from thermal power plants up to 70 %. At such amount of ash in the raw mixture, plasticization occurs in the temperature range of 100–110 °C; bloating – at 110–147 °C.

3. We determined basic technological parameters of the process of bloating for obtaining new alkali-silicate compositions. The greatest intensity is achieved in the temperature range of 115–123 °C. This endothermic effect is associated with the removal of adsorbed water from the gel-like products of hydration. Heat treatment at temperatures in the range of 140–150 °C makes it possible to form strength characteristics in a range of values of compressive strength of 3–8 MPa. Heating intensity determines the form of porosity: when heated to 5°C, we obtained a material with closed cellular porosity.

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