Characteristics of heat insulating foam aluminum silicate under change of heat treatment parameters

I A Khristoforova1, A I Khristoforov1 and E A Timakov1

1Vladimir State University named after Alexander and Nikolay Stoletovs, Russia

E-mail: khristoforova-i@mail.ru

Abstract. In case of heat insulation, work material choice depends on the type of protected object, materials available and usage convenience. Durability match between heat insulation and basic building material lies at the core of insulation choice. Foam glass and its derivatives (e.g. foam aluminum silicate) have properties meeting the strictest requirements of today. These rigid highly porous foam insulation materials have closed cell structure, representing solidified glass foam with either polyhedral or rounded cells. For these reasons, usage of highly efficient environmentally friendly foam insulation materials is so relevant today. A process was developed for batch heat treatment using local natural raw materials found in Vladimir region. The process consists of components joint grinding, produced batch preparation, blank pressing, foaming and heat annealing, and property research for produced materials. It turns out that foaming parameters are strongly intervened with batch composition, therefore requiring constant adjustment for each batch composition change. Researches show that specific heat treatment parameters provide foam formation with high foaming ratio and closed pore structure. Furthermore, we managed to create a foam aluminum silicate having the following features: density 130-350 kg/m³, coefficient of thermal conductivity 0.07 – 0.11 W/m·K, compressive strength 0.7 – 3.0 MPa.

1. Introduction

Nomenclature extension and competition increasing are two of the highest construction priorities when it comes to domestic heat insulation materials.

Hence there is a high demand in the research field aimed at improvement of conventional heat insulation materials, as well as developing new ones that could be produced using local raw materials.

When it comes to heat insulating works and choice of material, one has to take in consideration the type of object that needs heat insulation, type of insulation itself, and the materials at hand. The basic principle for material choice is the match between durability of heat insulation and the main construction material.

Foam glass along with its modifications provides a set of properties meeting the toughest modern regulatory requirements. Foam glass is a rigid highly porous heat insulation material. It has closed cell structure and is basically a solidified glass foam that consists of polyhedral and rounded cells. Foam glass is produced using thermal heating of fine charge at temperatures around 800 – 900 °C. This charge consists of silicate and alumosilicate ingredients along with blowing agent.

Foam glass can be used in many ways due to its properties. Heat insulation of residential and industrial buildings, power plants and pipelines is one such case. This is due to the fact that material does not lose its shape and properties in temperature range between −190 and +450 °C. Also foam
glass can be used as both construction and heat insulation material thanks to the fact that foam glass is amenable to many processing methods. It is also biostable and resistant to decay. Also foam glass can be used as a floating and water resistant material.

Today Russian industry produces lightweight materials with thermal conductivity less than 0.175 W/(m·K), low average density (less than 600 kg/m³), low water absorption (less than 10%). At the same time those materials are environmentally friendly. Foam glass main advantages in comparison to other materials are water resistance, higher mechanical strength, incombustibility and biologic stability [1-20].

High cost of foam glass represents a serious problem when it comes to market expansion. High cost is cause by complexity of technology, high unit costs of both electricity and fuel, as well as extremely high capital investments. Hence the urgent need for the new material that would have all the advantages of foam glass along with a lower price

**Objective**: create a new heat insulation foam material with conductivity in range 0.07-0.11 W/(m·K), apparent density in range 180-250 kg/m³, compressive strength more than 1 MPa.

2. Methods

In this research we used the following ingredients.

1. Clay from Suvorote deposit (Russian Federation). It consists of particles less than 0.01 mm and includes several clay materials, quartz and other impurities. Career moisture is in range 19-21.5 %.

   **Table 1. Clay chemical compound.**

   | Component   | SiO₂ | Al₂O₃ | Fe₂O₃ | CaO | MgO | K₂O | Na₂O |
   |-------------|------|-------|-------|-----|-----|-----|------|
   | Mass fraction (%) | 64.3 | 8.7   | 5.32  | 0.069 | 0.74  | 1.92 | 2.2  |

   2. Soluble sodium silicate according to State Standard R 50418-92. Silicate should be produced in pieces of size in range between 10 and 150 mm. Pieces either smaller than 10 mm or more than 150 mm can be used if their total mass does not exceed 40% of the batch. This threshold decreases to 30% in case of producing liquid glass suitable for welding materials. Maximal size of each piece is 200 mm.

   3. Foaming agents:
   - Ground dolomite according to state standard 23672-79.
   - Carbon black according to state standard 7885-86 type P-803.

   Preparation and production process for foam material consists of the following stages:

   1. Joint grinding of components.
   2. Preparation of the charge (moisturizing).
   3. Pressing the workpiece before foaming.
   4. Foaming and annealing in muffle furnace.
   5. Mechanical processing of foamed workpiece.
   6. Trial check of operational properties.

   During joint grinding particles of glass forming components are activated mechanically. This happens along with dispersion and stirring. All this leads to mutual diffusion as the powder softens and melts. Moisturizing of the charge is necessary for achieving specific properties, such as compressibility and lack of dusting. Besides that water is a necessary reagent for pore formation.

   During the experiment we researched impact of molded briquette density on thermal processing mode and final product properties. In order to achieve this samples pressed using various specific pressures were foamed on a layer of quartz sand. Foaming was carried out in laboratory muffle furnace managed programmatically by MIMP-P during various temperature conditions. Upon reaching foaming temperature samples were annealed in the same furnace for 12-14 hours. This was done in
In order to prevent residual stress in foamed unit after pore structure fixation. Once the sample is cooled down it is removed from oven and processed with abrasive tool. Finally the sample goes through a series of physical and chemical tests.

According to state standard 4.201-79 every heat insulation material has to meet several quality requirements: maximal usage temperature, moisture, thermal conductivity, flammability, nominal sizes and variations, density (bulk mass). Foam glass blocks have to meet the following requirements — density, total porosity, water absorption, compressive strength, appearance defects and structure uniformity.

Due to the fact that technology process of foam materials (thermal processing at 900°C) rule out water and combustible elements in the structure, there is no need to define moisture and flammability.

Produced samples were tested according to the following parameters: coefficient of thermal conductivity, compressive strength, total porosity, water absorption, average density. Compressive strength (σ, MPa), average density (ρ, kg/m³) and water absorption (B, %) were measured using methods of state standard 17177–94. Coefficient of thermal conductivity (λ, W/m·K) was measured by additive contribution of ingredients thermal conductivity (charge components and air inside pores).

3. Results and Discussion

We managed to define a more narrow temperature range suitable for foaming. This happened thanks to a research where prepared samples were placed in the furnace with temperature range 10°C and starting temperature 800°C.

Briquette warming speed was defined empirically and is equal to 5°C per minute. Recommended speed for warming foam glass (from sintering start to foaming temperature) depends on compound, glass dispersion, blowing agent type, agent activity, furnace gas medium. This temperature is in range 2.5 – 12 °C per minute in case of carbonaceous blowing agents. As for the neutral blowing agents, recommended speed range is 8 – 20 °C per minute.

Figure 1 shows structure of the samples produced for various temperature peaks.

![Figure 1](image)

Figure 1. Porous structure of the samples produced at various foaming temperature peaks, °C: a) 850; b) 860; c) 870; d) 880; e) 890; f) 900 (charge moisture 10%, specific pressure 0.4 MPa)

Increase in speed, as well as warmup time above the specified threshold causes increase in foaming agent consumption, specifically in the surface layer before the charge starts sintering. Figure 2 shows the structure of material that was produced from a charge with grounded dolomite and carbon black serving as the foaming agent, at the warmup speed 20 °C per minute.
As one can see in figure 2, there forms indeed a significant layer of glazed shell due to high warmup speed with exposure time being stable.

Table 2 shows material properties at various temperature peaks.

| Foaming temperature (°C) | Material properties | Compressive strength (MPa) |
|--------------------------|---------------------|---------------------------|
| Foaming temperature (°C) | Average density (kg/m³) | Water absorption (%) | Compressive strength (MPa) |
|--------------------------|---------------------|---------------------|---------------------------|
| 850                      | 340                 | 2.1                 | 3.2                       |
| 860                      | 292                 | 2.6                 | 2.5                       |
| 870                      | 246                 | 3.2                 | 1.8                       |
| 880                      | 198                 | 4.1                 | 1.5                       |
| 890                      | 156                 | 4.3                 | 1.3                       |
| 900                      | 130                 | 5.2                 | 1.1                       |

* Measurement error ±5%

In present technology of foam glass production has not undergone significant changes [21]. Conventional powdery technology implies usage of metallic refractory foaming forms. According to this method there are several stages before sending form to the furnace. First, internal surface of the form is sprayed by pulverizer in order to make a protecting cover. Subsequently, the form is filled with foaming mixture. The furnace temperature at start varies in range 500 – 750°C. Once the foaming process is over, a ready block of foaming glass is removed from the form and the whole process repeats.

According to [22,23] thermal heterogeneity of foaming mixture causes a large number of deformed cells. This heterogeneity is caused by both insufficient averaging of mixture and high warmup speed in time range from sintering start to foaming temperature peak. Increase in mixture thermal conductivity gives a possibility to briquette warmup without damaging structure of resulting material [21].

We managed to solve problems with form usage by shaping blocks in required way using pressing.

In order to make block capable of retaining its form at minimal pressure we moisturized the charge at the level found empirically. Our research data indicates that initial charge moisture at 20% causes significant deterioration in structure of produced samples. With such a moisture level, once the
foaming process is over, there forms a large cavity under the top of the surface. Figure 3 shows such samples.

![Image](image_url)

**Figure 3.** Sample produced using charge with 20% moisture.

The research clearly indicates a significant impact of water vapor partial pressure on viscosity, surface tension and gas formation temperature. Increase in partial pressure causes foaming process start to shift at a lower temperature. As a result, under the same foaming temperature peak, viscosity of silicate melt will have a lower value.

We studied the impact of initial moisture and pressure on resulting material properties. Table 3 shows the numbers.

| Briquette moisture (%) | Water absorption at specified pressure (%) | Density at specified pressure (kg/m³) |
|------------------------|------------------------------------------|-------------------------------------|
|                        | 0.2 (MPa) | 0.4 (MPa) | 0.6 (MPa) | 0.2 (MPa) | 0.4 (MPa) | 0.6 (MPa) |
| 5                      | 5.2       | 4.4       | 3.5       | 208       | 204       | 194       |
| 7.5                    | 5.8       | 5.6       | 3.8       | 201       | 194       | 184       |
| 10                     | 6.4       | 6.2       | 4.3       | 198       | 186       | 156       |
| 12.5                   | 7.6       | 6.8       | 4.6       | 190       | 182       | 142       |
| 15                     | 8.2       | 7.2       | 5.2       | 182       | 174       | 130       |

*Measurement error ±5%*

As table 3 clearly shows, increase in moisture from 5% to 15% along with 0.2 MPa pressure causes an increase in water absorption due to forming of open pores at initial pressure stage. This is reaffirmed by a higher average density of material produced from briquettes having higher moisture.

Due to internal friction (caused by low mobility of charge particles) pressure of 0.2 MPa is not enough for dense packing. As the moisture rises, the density decreases, but due to open pores water absorption also rises. The same thing is true for pressure levels of 0.4 MPa and 0.6 MPa. But once the pressure rises, water absorption decrease is observed in the entire temperature range, as well as density decrease.

Mechanical strength of foam material depends on a lot of factors. The cooling type serves as the main cause in case of the strength. Since the material has extremely low coefficient of thermal conductivity and is formed at high temperatures, the cooling process should be performed in a way that allows to avoid significant speed of temperature change.

Therefore a uniform highly porous cellular structure of the material just is not enough to obtain required mechanical strength. One also needs to maintain the resulting block in such a temperature
range that ensures the silicate melt will undergo a structural transition from a plastic-viscous to a glassy, solid, elastic-brittle state, avoiding formation of residual stress in material.

Since the resulting foam material is similar (in sense of thermoplastic properties) to industrial foam glass for the formation of a solid foam with a strong structure, we should expect that its cooling parameters correspond to the already known parameters of foam glass cooling [23, 24].

Material exposure at the foaming temperature helps to keep the foaming. The foaming agent reacts with the clay ingredients releasing gas. Also there is an assimilation of separate cells with each other, leading to formation of pores with enlarged size.

It is likely that the cell connection prevails over the release of gas and cell expansion as foaming starts. However, as foaming goes on, cell expansion starts to prevail causing in a larger sample volume. As the foaming gets close to an end, the release of gas from the foaming agent is suspended under the influence of its concentration decrease. Therefore the process of mutual cell assimilation starts prevailing again. At this point the foaming process should be stopped.

Timing is the key factor in termination of the foaming process in order to preserve the shape of the finished block without visible and, more important, non-uniform deformations. Perfect timing is one of the most difficult phases in the entire technology for producing foam glass. The key to correct and complete stabilization is in stopping the foaming process precisely when the expansion of the cells of the outer layer of the block has already stopped. But if we take the block as a whole it should still be under the influence of a significant pressure from its inner part.

If you can stop the foaming process at this point quickly enough and cool the block is cooled to a sufficiently low temperature, then the block surface crust will harden and like a shell will enclose the internal parts of the block. Those parts still have a significant excess pressure at the given moment. After that, you can adjust the temperature of the entire block to such a value that prevents any noticeable temperature deformations. The block still retains its original shape.

The gas release in the internal parts of the block after temperature decrease suspends smoothly. The block is removed from the foaming furnace having a state close to equalized pressure. To carry out this foaming process, you can use a technological solution, which implies that a special cooling zone between the foaming and annealing ones is included in the process. This zone is the one where the stabilizing process is carried out. Foaming process is stopped and the outer layer is cooled down.

Physical and chemical transformations that occur in the charge during the entire heat treatment cycle are the key feature. Because of this, the temperature field in the charge layer is affected by exo- and endo-thermic effects occurred during heating, making its modeling becomes extremely complex. Figure 4 shows the research results.

Figure 4 shows temperature distribution both inside furnace and the surface of the sample being heated.

The temperature drop in both the furnace and sample surface at the initial stage of heating is associated with sample loading. The temperature inside compressed briquette at this stage of heating is lower than the surface temperature. As the heating goes on there is an observable fairly rapid temperature increase in sample inner layers. This is caused by an increase in the thermal conductivity of the charge due to both pressing and sintering of the material.

The next section is defined by drastic drop in furnace temperature to 500°C. This is necessary for fixing the formed porous structure. The temperature of briquette surface also starts to drop, however, due to the ash accumulated by the sample during heating phase, it reaches slightly lower values. The temperature in the briquette inner layers also drops, but this temperature curve is smooth and does not have drastic changes. Figure 5 shows the materials obtained at different values of the exposure time at the maximum foaming temperature.
Figure 5 shows that an increase in the exposure time at the maximum foaming temperature up to 10 min followed by rapid cooling (down to 510 °C, figure 4) allows to stabilize the porous structure. This is much more stable in comparison with the structure of the foam material extracted from furnace immediately after foaming. This is due to the temperature of foam glass inner layers (figure 4)

Figure 4. Temperature distribution during heating: 1 – surface internal; 2 – sample surface; 3 – furnace itself

Figure 5. Exposure time impact on the porous structure: a) 0; b) 2; c) 4; d) 6; e) 8; f) 10 min.
reaching level sufficient for increasing the strength of cell barriers. This eliminates the residual shrinkage stresses are leveled. The temperature lag of sample internals is caused by the introduction of the liquid phase and endothermic dissolution of the clay components in the silicate melt. Part of the absorbed heat is spent in gas formation. The eventual pattern of changes in sample temperatures matches the change in the furnace temperature. It is especially worth noting that furnace temperature at the end of exposure time at the temperature of foaming is accompanied by a temperature drop both inside sample and on its surface. This is a clear indication of all chemical reactions completed in the mixture, since those reactions are accompanied by thermal effects.

4. Conclusions

Our research shows moisture increase (the lubricant between charge particles) and specific pressing lead to the production of foam having density of 130 kg/m³ and a compressive strength of 1.1 MPa. On the other hand, an increase in water concentration to 20% leads to block ruptures.

Additive calculation of resulting material thermal conductivity allowed us to determine the following values. A material with density 130 kg/m³ and \( \lambda = 0.07 \text{ W/m·K} \). As for the material with density of 340 kg/m³, it has \( \lambda = 0.11 \text{ W/m·K} \).

This heat insulation material can be used in the construction of buildings and structures for both civil and administrative purposes, low-rise construction, and various types of pipelines. Material can be produced both in the form of blocks and in the form of pellets that can be used for heat insulation filling.

Further research should be aimed at density increase for particle packaging in the briquette. This will allow to reduce the number of open pores and average density of foam glass causing reduce in thermal conductivity. In order to use this in industrial furnaces further researches should focus on foaming modes, drastic furnace temperature drops (below the glass transition temperature).

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