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The choice of the grouting method for liquid glass granulate while obtaining composite thermal insulation materials

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The paper presents the research on the choice of the most effective grouting method for the liquid glass granulate by the binder based on liquid glass during production of composite insulation materials by their bloating under the microwave irradiation. The choice of the grouting method is carried out by determining the basic physical and mechanical properties of thermal insulation materials depending on the ratio of the granular filler to the binder. The research has shown the feasibility of obtaining composite insulation materials by volume grouting, i.e. simultaneous bloating of the granular filler and the liquid glass binder, which prevents shrinkage, cracks, foam collapse and formation of large voids due to close packing of contacting granules and uniform distribution of the bloated binder that fills the intergranular space. Therefore, such materials are characterized by better physical and mechanical properties.

Keywords: composite insulation material, liquid glass, bloating, microwave irradiation, physical and mechanical properties, volume grouting, contact grouting.

Вибір способу омонолічування рідкоскляного грануляту при одержанні композиційних теплоізоляційних матеріалів. *Т.Е.Римар, О.В.Суворін*.

Проведено дослідження з вибору найбільш ефективного способу омонолічування рідкоскляного грануляту зв‘язуючим на основі рідкого скла при виробництві композиційних теплоізоляційних матеріалів шляхом їх спучування під дією НВЧ-випромінювання. Вибір способу омонолічування проводився шляхом визначення основних фізико-механічних характеристик теплоізоляційних матеріалів в залежності від співвідношення гранульованого наповнювача до зв‘язуючого. Проведені дослідження показали доцільність одержання композиційних теплоізоляційних матеріалів шляхом об’ємного омонолічування, тобто одночасним спученням гранульованого наповнювача і рідкоскляного зв’язуючого, що дозволяє запобігти усадкових явищ, утворення тріщин, осідання піни і утворення великих пір завдяки щільній упаковці гранул, які спікаються між собою, і рівномірному розподілу спученого зв’язуючого, що заповнює міжгранульний простір. Як наслідок, такі матеріали характеризуються більш високими фізико-механічними показниками.

Проведено исследование по выбору наиболее эффективного способа омоноличивания жидкостекольного гранулята связующим на основе жидкого стекла при производстве композиционных теплоизоляционных материалов путем их вспучивания под действием СВЧ-излучения. Выбор способа омоноличивания проводился путем определения основных физико-механических характеристик теплоизоляционных материалов в зависимости от соотношения гранулированного наполнителя к связующему. Проведенные исследования показали целесообразность получения композиционных теплоизоляционных материалов путем объемного омоноличивания, то есть одновременным вспучиванием гранулированного наполнителя и жидкостекольного связующего, что позволяет предотвратить усадочные явления, образование трещин, оседание пены и образование больших пор благодаря плотной упаковке гранул, которые спекаются между собой, и равномерному распределению вспученного связующего, которое заполняет межгранульное пространство. Как следствие, такие материалы характеризуются наиболее высокими физико-механическими показателями.

1. Introduction

All thermal insulation materials (TIMs) can be divided into three groups by their structure: materials with a rigid, cellular structure, materials with a granular, unbound structure, and materials with a fibrous structure. The choice of materials for thermal insulation is primarily determined by the nature of the thermal protection object, the appropriacy of the protection method, the availability of materials, and the ease of their use during the work [1].

To create materials with the given highly porous structure, several dozen methods are used depending on the type of raw materials and the specified properties of products. However, there are six main porization methods: removal of the pore-forming agent, loose packing, contact grouting, volume grouting, creation of combined structures, and bloating [2].

Contact grouting is based on the grouting of granular and fibrous elements of the structure in places of their mutual contact with thin adhesive layers. These layers are created by adding low-viscous binder compositions into the frame-forming material. They are distributed over the surface of grains or fibres evenly and thinly. Thus, it leads to adhesion by applying small pressing forces to them. Liquid compositions (preferably aqueous solutions) of polymers, cement, clay, and soluble glass are used as binders.

Volume grouting differs from the previous method since the binder fills all the voids in the frame-forming material. To increase the overall porosity of the material, a porous binder in the form of foam or polyfractional highly porous grains is mostly used to achieve their largest amount in the material volume. In this case, the obtained material has cellular porosity consisting of the grain porosity and the binder porosity.

Creation of combined structures results in obtaining porous products with more than two types of porosity: fibrous-cellular, granular-cellular, fibrous-cellular-capillary, etc. The purpose of creating the combined structures is to increase the overall porosity of products as well as their flexural strength [2].

The vast majority of composite materials based on liquid glass granulate are obtained by contact grouting of granular fillers with a binder. The granular fillers can be natural mineral materials (perlite, vermiculite, tripoli) [3–5], and granules based on liquid glass [6–10]. The production technology consists of preparing the binder, preparing the moulding mass of granules and the binder, making products and creating conditions for rapid obtaining of the binder [11–16]. However, the large-scale industrial production of composite insulation materials based on liquid glass in the form of plates and blocks has not been established yet. This is caused by complexity of heating inner layers of the liquid glass composition using traditional convective heating, which makes it impossible to obtain high-quality insulation materials.

During production of these materials, it is possible to use microwave irradiation as an alternative source of heat treatment. This method is promising in terms of such important properties as high efficiency coefficient, automation and high quality of the obtained products. The use of microwave irradiation will provide volumetric heating of all the layers of the liquid glass composition, and bloating of both granules and the binder efficiently (quickly and thoroughly) to remove moisture at lower temperatures and to obtain strong bloated material [17].

The purpose of this work is to research the properties of thermal insulation materials based on liquid glass obtained by volume and contact grouting under the action of microwave irradiation, and to choose the most effective method of obtaining composite materials.

2. Experimental

The paper proposes two methods of obtaining composite insulation materials in the form of blocks: volume grouting and contact grouting. The volume grouting is conducted by mixing unbloated granular filler with the liquid glass binder, and then bloating them by microwave irradiation. During the contact grouting, the bloated granular filler is used; the voids between granules are subsequently filled with the bloated liquid glass binder hardened by microwave irradiation.

The choice of the grouting method of the liquid glass granulate by the binder was carried out by determining the basic physical and mechanical properties of thermal insulation materials depending on the ratio of the granular filler to the binder. In order to determine the quality of the obtained samples, the following important properties of thermal insulation materials have been determined: apparent density, water absorption and moisture sorption, strength, and mass loss during bloating. The phase composition of the material was also determined to evaluate the transformation of its structure during bloating.

Determination of these values has been carried out on samples of the correct form with dimensions of 100×100×35 mm^3 using the following methods:

Mass loss is calculated by the formula:

$$W = (m - m_1) \cdot 100 / m_1, \quad (1)$$

where W is the mass loss,%; m is the mass of the sample before foaming, (g); m_1 is the mass of the foamed sample, (g).

The apparent density of the TIM is determined as the ratio of the sample mass to the volume occupied by it, including the volume of the gas phase; it is calculated by the formula:

$$\rho = \frac{m}{V}, \quad (2)$$

where m is the sample mass; V is the sample volume.

Moisture sorption and water absorption were determined on samples of rectangular shape with dimensions of 100×100×35 mm. The samples were dried to constant mass at a temperature of 50–60°C, and then weighed with the accuracy up to 0.01 g. While determining moisture sorption, the samples were placed over water poured into a desiccator placed in a thermostat with a constant temperature of 20±3°C. After 72 h, the samples were removed from the desiccator and weighed. Moisture sorption was calculated by the formula:

$$W_c = \frac{m_2 - m_1}{m_2} \cdot 100\%, \quad (3)$$

where m_1 is the mass of the sample dried to a constant mass, (g); m_2 is the mass of the sample after water vapour saturation, (g).

While determining water absorption, the sample was immersed in water; during the first 3 h, the sample was immersed in water to half and completely immersed in water for the remainder of the test. After 24 h, the sample was removed from water, excess water was removed from its surface, and the sample was weighed. The mass of water poured onto the weighing cup from the sample voids during weighing was included in the mass of the water-saturated sample. Water absorption was calculated by the formula:

$$W_n = \frac{m_3 - m_1}{m_1} \cdot 100\%, \quad (4)$$

where m_3 is the sample mass after saturation with water, (g).

The strength properties of the materials were determined using a P-5 test machine, which allows determining the breaking load with an accuracy of not less than 0.5 kgf. For materials that do not demonstrate brittle fracture, the breaking stress has been determined at 10 % compression strain. In order to determine it, the sample must have the shape of a cube with an edge length equal to the product thickness. The sample is placed on the press base plate so that the compression force is directed parallel to the vertical axis of the sample, and the axis of the sample passes through the centre of the base plate press. The load on the sample should be increased gradually without shocks at a speed of 10 mm/min. The sensors of the press measure the sample strength properties during the test. The breaking stress at 10 % compression strain was calculated by the formula:

$$\sigma_{10} = \frac{P}{l \cdot b}, MPa, \quad (5)$$

where P is the load at 10 % linear strain, H, (kgf); l is the sample length, cm; b is the sample width, cm.

While determining the flexural breaking stress, the sample is in the shape of a parallelepiped with dimensions of 250×10×10 mm^3. The sample is placed on two pillars with rounding points in the joints. The distance between the axes of the pillars is 200 mm. The load on the sample is transmitted through a roller with a diameter of 10 mm laid across the width of the sample at an equal distance from the pillars. The load is considered destructive if the sample is destroyed. The flexural breaking stress was calculated by the formula:

$$\sigma_i = \frac{3P \cdot l}{2b \cdot h^2}, MPa, \quad (6)$$

where P is the destructive load at flexion, (kgf); l is the distance between the axes of the pillars, cm; b is the sample width, (cm); h is the sample thickness, (cm).

Determination of thermal conductivity was carried out on an ITS-1 device made according to the asymmetric design; it is equipped with a thermometer, which is located between the test sample and the cold plate of the device. The method aims to create a constant heat flux passing through the flat sample of a certain thickness and directed perpendicularly to the front (largest) face of the sample, in order to measure the density of this heat flux, the tempera-

ture of the opposite face sides and the sample's thickness. The sample is in the shape of a rectangular parallelepiped, the largest (front) face of which is a square with a side equal to the side of the working surfaces of the device plates (150×150 mm^2). The sample thickness is less than the length of the edge of the front face and within the limit of 10–25 mm. The test sample is placed in the device to determine its thermal resistance. The location of the sample is horizontal; the direction of the heat flux is from top to bottom. During the test, the temperature difference of the front faces of the sample ΔT_u is 10–30 K. There are setpoint temperatures of the working surfaces of the device plates. Signals of the thermometer e_u and the temperature sensors of the front faces of the sample are checked every 300 s. After reaching the steady temperature condition, the thickness of the sample d_u is measured.

The effective thermal conductivity of the material (thermal conductivity coefficient) is calculated by the formula:

$$\lambda_{effu} = \frac{d_u}{\frac{\Delta T_u}{q_u} - 2R_k}, \quad (7)$$

where R_k is the thermal resistance, m^2·K/W (it is taken equal to zero for thermal insulation materials and products).

The density of the constant heat flux q_u through the sample is calculated by the formula

$$q_u = f_u \cdot e_u. \quad (8)$$

To control the restructuring of the thermal insulation material during bloating, it is necessary to know the composition and properties of the initial and final structures, as well as to obtain such evaluation parameters that allow evaluating the restructuring regardless of their composition and properties. These requirements are satisfied by the volume phase properties of structures and, first of all, the volume fraction of the solid phase. The main advantage of using the volume phase properties is that the total volume of solid particles, liquid and gaseous phases of the dispersed system or structure is equal to one regardless of the structure type and the type of external energy deposition on the system.

$$K_s + K_l + K_g = 1. \quad (9)$$

If the wet material is used, then the apparent density of the wet material is determined first $\rho_{vol} = m_{vol}/V$, and then the apparent density of the dry material is calculated:

$$\rho_c = \rho_u/(1 + W_a), {}^{kg}/_{m^3}, \quad (10)$$

where W_a is the absolute moisture of the material relative unit.

The volume fraction of the solid phase is determined by the ratio of the apparent density of the material to its actual density:

$$K_s = \rho_c/\rho_a, \text{ r.u.}, \quad (11)$$

The volume moisture W_{vol} or the volume fraction of the liquid phase is determined by the formula:

$$W_v = K_l = W_a \cdot \rho_c, \text{ r.u.} \quad (12)$$

The volume fraction of the gaseous phase or the sample porosity is determined by the difference:

$$K_g = 1 - (K_s + K_l), \text{ r.u.} \quad (13)$$

The volume particles' balance for dry, two-phase material has the following form:

$$K_s + K_g = 1. \quad (14)$$

Based on this equation, the volume fraction of the gaseous phase K_g is determined at the known value of K_s.

In the mathematical form, for porisation of the liquid glass composition, the law is written as follows:

$$K_{s_1} + K_{l_1} = K_{s_i} + K_{l_i} + K_{g_i} = \\ = \ldots = K_{s_2} + K_{g_2}, \quad (15)$$

where K_s, K_l, and K_g are, respectively, the volume fractions of the solid, liquid and gaseous phases in the system at the corresponding technological stage. For the material being analyzed, the following can be written:

$$K_{s_1} + K_{l_1} = K_{s_2} + K_{g_2} = 1. \quad (16)$$

Here one can get the value (n), which characterizes the intensity of the structure formation processes in dynamic systems. It shows the relative change in the ratio of the volume concentration of the solid phase and the free pore space during transition of the dispersed system from one state into another under the influence of external energy deposition (chemical, mechanical or thermal). The value of n can be determined from the ratio:

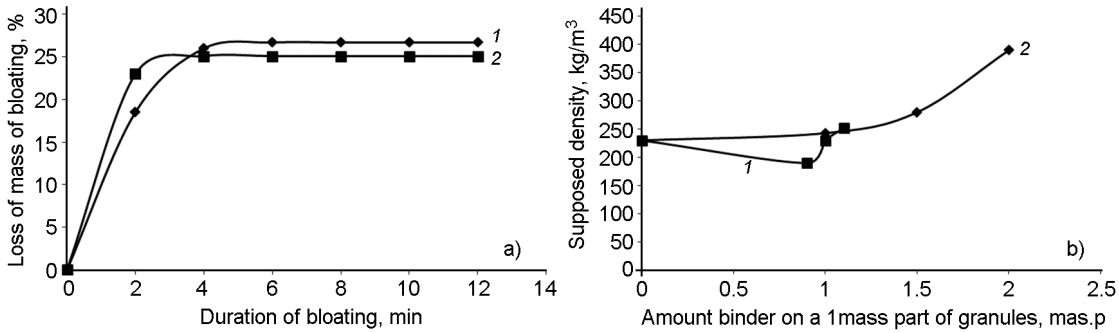

Fig. 1 Dependences of the material mass loss on duration of bloating (a) and apparent density of materials on binder-to-granular filler ratio (b): *1*- volume grouting, *2* - contact grouting.

$$K_{s_2}/(1 - K_{s_2}) = n \cdot K_{s_1}/(1 - K_{s_1}), \quad (17)$$

where K_{s_1}, K_{s_2} is the volume fraction of the solid phase at the beginning and the end of the system porization, respectively.

If no changes occur in the system, then $n = 1$. The decrease in the system volume is characterized by $n > 1$, and its increase (porization) — by $n < 1$. If the change in value n within (0 to 1) is normalized, then the degree of the material restructuring (α_n) can be determined by the ratio:

$$\alpha_n = (\frac{1}{n_i} - 1)\frac{1}{n_i}, \quad (18)$$

where $1/n_i$ is the material bloating during or at the end of deposition of the dispersed system; $1/n_i = V_2/V_1$, where V_2 and V_1 is the system volume in the final and initial states, respectively.

The latter dependence shows that the larger the value of α_n, which characterizes the transition of the system from the initial state to the final one, the more intensively the system becomes porized and the material is restructured. In addition, the above formulas lead to an important technological conclusion: the higher the volume concentration of the solid phase in the source material, the more intense the porization.

The parameter α_n can be used as a dependent variable in kinetic studies. Data processing has been performed using B.Yerofeyev's equation:

$$\alpha_n = 1 - e^{-k\ln\tau} \cdot e^h \quad (19)$$
$$\text{or } \ln(1 - \alpha_n) = -k \cdot \ln\tau + b,$$

where τ is the restructuring duration; k is the porization rate constant of the structure; b is the constant coefficient at the given heating rate of 3.5.

Hence the reaction rate constant is calculated by the formula:

$$k = -\left(\frac{\ln(1 - \alpha_n) - b}{\ln\tau}\right), s^{-1}. \quad (20)$$

The activation energy of apparent porization of the LGC has been determined by the formula:

$$E_n = R\frac{\ln\tau_1 - \ln\tau_2}{\frac{1}{T_1} - \frac{1}{T_2}}, \quad (21)$$

where E_n is the activation energy of apparent porization, kJ/mole; R is the universal gas constant, $R = 8.314$ J/(mole·K); τ_1, τ_2 is the time of reaching the parameter α_{ni} at temperatures T_1, T_2 [18].

3. Results and discussion

To compare the effectiveness of methods for producing block material, the data of mass loss during bloating for contact and volume grouting are presented in Fig. 1.

Based on the data shown in Fig. 1, bloating is faster during contact grouting. A constant value of material mass loss of 25.1 % is reached during the 4th minute of the process. During volume grouting, that is, simultaneous bloating of the liquid glass binder and the granular filler, the constant value of the material mass is reached during the 6th minute, and the loss amount is 26.7 %. The higher mass loss value in the latter case is explained by the conversion of the bound moisture into vapour in both the binder and granules; while during contact grouting, pre-bloated granules are used, and the formation of voids is observed only in the liquid glass binder. For the same reason the material takes less time to reach the constant mass value.

While obtaining block insulation materials by contact grouting, the mass ratio of

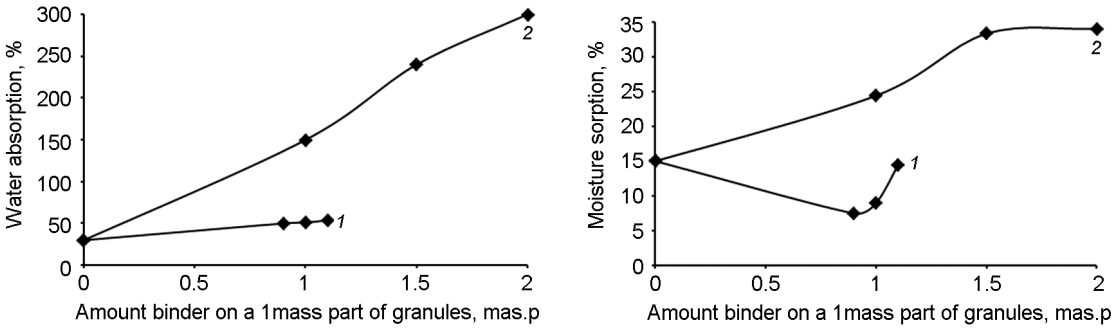

Fig. 2. Dependences of water absorption (a) and moisture sorption (b) of block materials on binder-to-granular filler ratio: *1* — volume grouting, *2* — contact grouting.

the granular filler to the binder is of great importance, since density will also change significantly in the case of changing this ratio. This dependence is shown in Fig. 1(b). In the graph, the zero point corresponds to the density of the material obtained without the binder, i.e., sintering only granules. During this method of obtaining a thermal insulation material, in the intergranular space a large number of big voids are formed, which adversely affects the properties of the blocks. Adding the binder into the material allows achieving the filling of the inter-granular space, thereby strengthening the material and increasing its water and moisture resistance. As it can be seen from the figure, as the amount of the binder increases with respect to the granular material, the block density also increases. During volume grouting, to get a homogeneous material structure, a small amount of the binder is required, which evenly fills the voids in the bloated granules. At the highest binder-to-granular filler ratio of 1.1:1, the sample density is 252 kg/m^3. During contact grouting, at the highest binder-to-granular filler ratio of 2:1, the material density reaches a value of 390 kg/m^3. That is, as the binder amount increases, the packing density of the granules decreases, as the distance between the frame-forming elements (granules) increases; this leads to the formation of big voids during the binder bloating and granules collapsing, so the material density increases.

When the binder-to-granular filler ratio is 1:1 during contact grouting, the material density is the lowest: 243 kg/m^3, but the obtained sample has an unsatisfactory appearance because the binder does not fully cover the upper layer of granules. A relatively low density 280 kg/m^3 combined with a satisfactory appearance is observed in the material obtained at the binder-to-granular filler ratio of 1.5:1. Based on these data, it is better to obtain block materials by volume grouting, since it reduces the binder loss (the binder-to-granules ratio is 1:1), and the material is characterized by low density, a satisfactory appearance and high strength due to sintering of granules with each other and uniform filling of the voids between the granules with the bloated binder.

One of the defining properties of thermal insulation materials is water absorption and moisture sorption (hygroscopicity), since excessive moisture absorption by the material leads to deterioration of its thermal insulation properties. Fig. 2 shows the water absorption and moisture sorption values for materials obtained by contact and volume grouting.

Although the material obtained by sintering only the granules (zero point) is characterized by lower values of water absorption and moisture sorption, it has low strength and an unsatisfactory appearance. It is seen from Fig. 2, in the case of contact grouting, with increasing the binder amount per 1 pts·wt. of the granular filler, water absorption and moisture sorption increase respectively to 300 % and 34 %. The low adhesion of LGB to granules causes the formation of a large number of voids in the intergranular space; this leads to excessive absorption of water and its vapour from the environment. For materials obtained by volume grouting, water absorption and moisture sorption values are much lower: when the binder amount is increased to 1.1 per 1 pts·wt, the values increase to 53.7 % and 14.5 %. Due to the simultaneous bloating of granules and the binder, a dense packing of granules filled with a fine-porous binder is observed; this prevents active absorption of water and its vapour.

Fig. 3 shows the dependences of the flexural breaking stress and at 10 % compression strain on the ratio of the liquid glass binder to the granular filler.

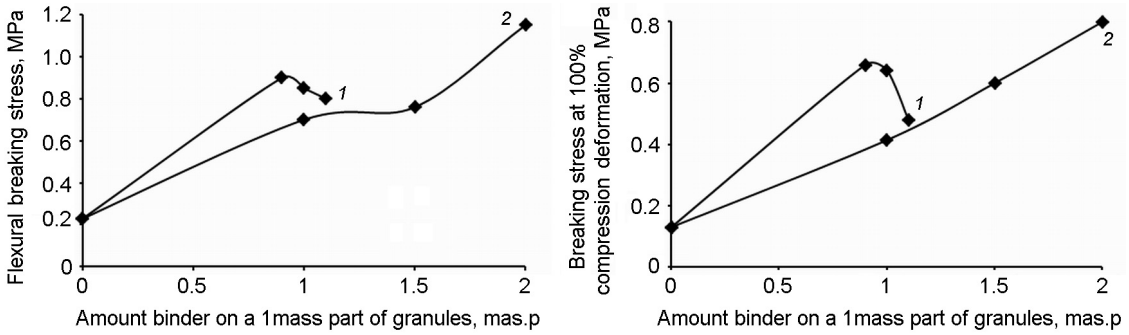

Fig. 3. Dependences of flexural breaking stress (a) and 10 % compression deformation (b) of block materials on binder-to-granular filler ratio: *1* — volume grouting, *2* — contact grouting.

The samples obtained at the same binder-to-granular filler ratio of 1:1 during both volume and contact grouting are compared (the densities of the samples are almost the same, respectively, 230 kg/m^3 and 243 kg/m^3). During volume grouting, the flexural breaking stress is 0.85 MPa, and during the contact one, it is 0.7 MPa. The use of unbloated, "raw" granules in the first case, leads to dense packing of granules since their volume during bloating increases, and the porous binder not only fills the intergranular voids but also improves thermal properties and strengthens the material. During contact grouting, when prebloated granules are used, strength of the insulation material is lower because the granules are unevenly distributed in the binder layer, while low adhesion between the mixture components causes sample breaking at the binder-granules contact points. The breaking stress at 10 % compression strain for materials produced at the granules-binder ratio of 1:1 by volume and contact grouting are 0.642 and 0.414 MPa, respectively. The formation of large voids, their collapsing, and inhomogeneous structure of the material obtained by contact grouting are the causes of the lower compression strain value.

To research the structure transformation of the composite insulation material depending on the grouting method, the phase composition of the system was determined. Table 1 shows the data revealing the influence of the granular filler-to-binder ratio per the volume fraction of the solid and gaseous phases of the block insulation material as well as the structure forming value n and the restructuring degree α_n.

As we can see from the above data, during contact grouting with an increase in the amount of the binder, the volume fraction of the solid phase also increases, and therefore the gaseous one decreases. The maximum value of the volume fraction of the gaseous phase, 0.469 r. u. is observed when the ratio of the binder to the granular filler is 1:1, and the degree of restructuring is 0.21. But at this ratio, the quality of the samples is poor because of the small amount of the binder, which is not enough to create a qualitative adhesive layer between the granules. The optimum binder-to-granular filler ratio is 1.5:1 in this grouting method, since the content of the gaseous phase and the restructuring degree in these samples

Table 1. The phase properties of composite thermal insulation material

Grouting method	Binder-to-granular filler ratio	Wet material						Foamed material						α_n	n
		W_a^u	ρ_{1c}^u	ρ_u^u	K_{s1}	K_{l1}	K_{g1}	W_a^f	ρ_{2c}^f	ρ_u^f	K_{s2}	K_{g2}	K_{l2}		
Without the binder		0.21	1.1	1.44	0.764	0.231	0.005	0.005	0.242	0.4	0.61	0.394	0.001	0.53	0.47
Volume	0.9:1	0.261	1.14	1.63	0.699	0.298	0.003	0.012	0.190	0.34	0.56	0.439	0.002	0.46	0.54
	1:1	0.267	1.18	1.73	0.682	0.315	0.002	0.016	0,23	0.52	0.44	0.554	0.004	0.63	0.37
	1.1:1	0.27	1.2	1.78	0.674	0.324	0.0018	0.018	0.252	0.59	0.43	0.568	0.005	0.64	0.36
Contact	1:1	0.246	0.72	1.23	0.585	0.177	0.24	0.011	0.243	0.46	0.53	0.469	0.003	0.21	0.79
	1.5:1	0.251	0.88	1.47	0.599	0.221	0.18	0.014	0.280	0.52	0.54	0.458	0.004	0.22	0.78
	2:1	0.262	0.93	1.52	0.612	0.244	0.15	0.018	0.390	0.71	0.55	0.444	0.007	0.23	0.77

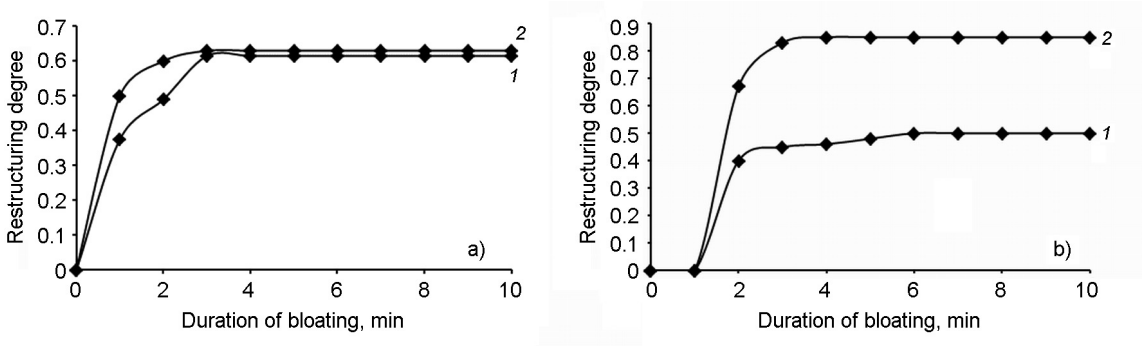

Fig. 4. Dependences of the restructuring degree parameter on the bloating duration: a) microwave oven: *1* — 500 W; *2* — 650 W; b) drying cabinet: *1* — 180°C; *2* — 280°C.

are slightly higher, K_g = 0,477 r. u., α_n = 0.24, respectively, and the quality of the samples is higher.

The studies have shown that during contact grouting the material restructuring is less intensive. It is explained by the fact that upon obtaining such samples only the binder is bloated. In this case, the restructuring degree is within 0.21–0.26, and the fraction of the gaseous phase is only 0.45–0.48 r. u. Whereas during volume grouting, these values are higher: α_n = 0.46–0.64 and K_g = 0.44–0.568 r. u.; and at the optimal binder-to-granular filler ratio of 1:1, these values are K_g = 0.554, and α_n = 0.63. When the binder-to-granular filler ratio is 0.9:1, the proportion of the gaseous phase and the value of the restructuring is less (K_g = 0.439, α_n = 0.46) due to the lower content of the binder, which takes an active part in bloating and the structure transformation.

The intensive material restructuring during volume grouting is explained by the presence of not bloated granules in the composition, and, consequently, a higher volume concentration of the solid phase in the source material. As it is known, the higher the volume concentration of the solid phase in the source material, the more intense the porization.

Therefore, from the obtained results it is clear that volume grouting provides obtaining thermal insulation materials with the best complex of operational characteristics. However, it is impossible to conduct volume grouting by traditional convective heating due to slow warming of inner layers of the liquid glass composition because of low thermal conductivity of the bloated outer layer of the sample. This problem can be solved by applying a relatively new technology of microwave bloating. The use of microwave technologies allows achieving uniform warming of all the layers of the sample, thus forming a uniform porous structure of the material. Besides, it helps to reduce the bloating temperature and the process duration due to heating of the liquid glass composition not only due to heat supply from the outside, but also due to internal acceleration of movement of water molecules, their friction, and thermal energy generation.

In order to determine the efficiency of a particular type of heating during obtaining thermal insulation materials, the kinetics of bloating has been studied under different conditions: at microwave heating and traditional convective heating.

At microwave heating, the samples were manufactured at two power indices, 500 W and 650 W, since it was possible to reach low process temperatures — below 200°C (at the power less than 500 W bloating of the samples does not occur). Since bloating at traditional convective heating requires more time (heat is spent not only on heating of the liquid glass composition, but also on heating of the mold, cabinet walls), thus to compensate time, the maximum cabinet temperature of 280°C has been chosen, and the lowest temperature of 180°C at which bloating of composite materials in the oven is possible. Based on the obtained experimental data, a diagram of the dependence of the restructuring degree parameter α_n on the bloating duration has been made (Fig. 4).

The apparent porization activation energy and the porisation rate have been calculated by the restructuring degree α_n in two sections: *1* — corresponds to bloating from the beginning of heating till the material reaching the first indicator α_n; *2* — corresponds to the indicator α_n reaching its constant value and completion of the material restructuring. The calculated data for the bloating kinetics are given in Table 2.

The value of the process rate constant at 650 W is greater than at 500 W for the first

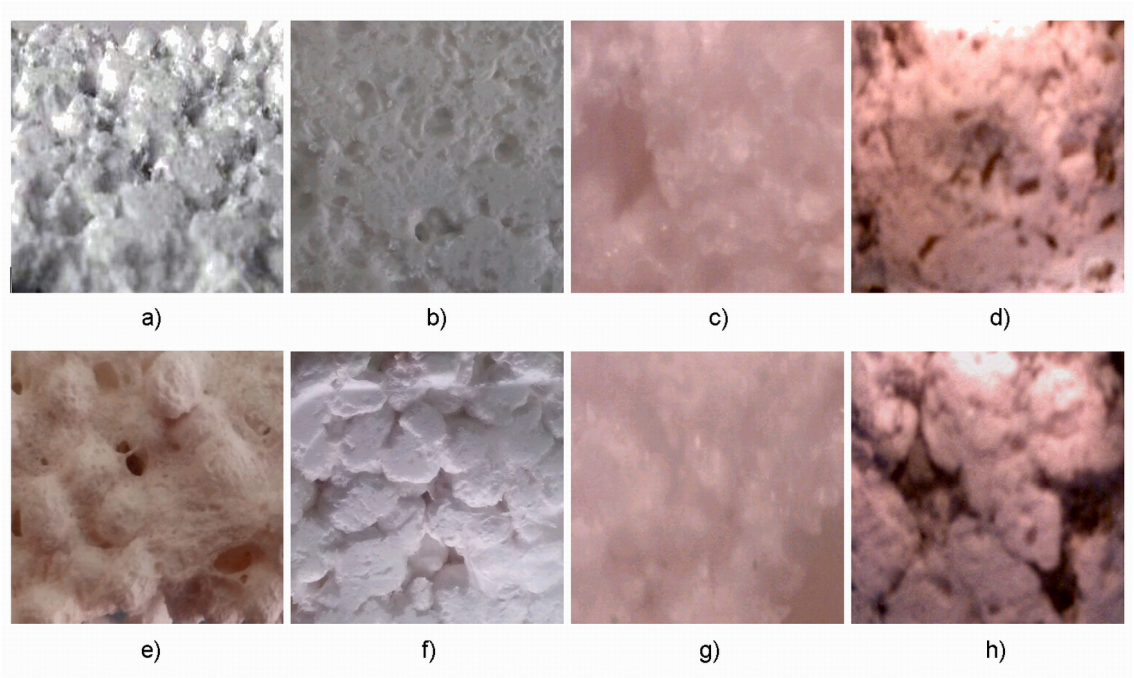

Fig. 5. Appearance and structure of insulation materials obtained by volume (a–d) and contact (e—h) grouting: a, e — upper surface of material; b, f — lower surface of material; c, g — structure of the granules (50× magnification); d, h — structure of the inter-granular space (10× magnification).

section; this fact indicates a higher process temperature. In the second section, the differences in rates at different power are not so noticeable indicating that the increased power gives a high rate of bloating only in the first stage of the process. Further bloating slows down. With the use of microwave irradiation during bloating of composite materials, the material restructuring is faster. At the power of 650 W (T = 117°C) α_n reaches its constant value of 0.615 during the 3rd min, while bloating during convective heating at T = 180°C gives the value of the restructuring value of 0.5, which is reached after 6 min. At 280°C, an initial sharp increase is observed in the bloating intensity, but after removing the sample from the oven, the bloated mass collapses and the restructuring degree reaches only 0.5. Bloating of samples in the microwave oven is characterized by higher activation energy and reaction rate, which indicates high intensity and efficiency of the process.

Low activation energy at both stages of porization indicates that simultaneous bloating of the granular filler and the liquid glass binder in the microwave oven does not require high values of activation energy, since the sample layer warms up almost immediately. Bloating is characterized by two values of the apparent activation energy. In the temperature range of 102–117°C, the first period of bloating is characterized by E_P = 16.85 kJ/mole, which corresponds to the energy of breaking the hydrogen bonds and intense material porization due to evaporation of moisture and decomposition of the gas forming agent. The second period corresponds to the completion of porization and removal of residual moisture (E_p = 5.2 kJ/mole). With traditional convective heating, the both periods have values of E_P that differ slightly (10.61 and 14.4 kJ/mole, respectively), which indicates gradual release of moisture resulting mainly from the material drying rather than bloating. This is evidenced by the properties of the obtained materials given in Table 3.

The data presented show that the samples obtained in the microwave oven have 1.5 times less density than these obtained by convective heating, and the strength properties of such materials is 2 times higher, water absorption is 2 times lower, and hygroscopicity is almost 3 times lower. This shows formation of a cross-linked liquid glass structure with less defective low molecular weight fraction under the action of the microwave irradiation, whereas in the drying cabinet, curing of the liquid glass composition has not been completed and the binder has been only dried.

Electron microscopy studies on formation of the porous structure of thermal insula-

Table 2. Kinetic parameters of bloating composite insulation materials

Microwave oven power, W	Temperature in the drying cabinet, °C	Process temperature, °C	α_n		τ, x_B		E_n, kJ/mole		k, c^{-1}	
			1 section	2 section	1 section	2 section	1 section	2 section	1 section	2 section
500		102	0.375	0.615	1	3	16.85	5.2	0.96	0.86
650	–	117			0.8	2.8			1.03	0.869
–	180	180	0.4	0.5	2	3	10.61	14.4	0.84	0.81
–	280	280			1.2	1,5			0.94	0.93

Table 3. Comparison of the properties of block materials obtained by contact and volume grouting

Property	Indicator value	
	volume grouting (the binder-to-granular filler ratio is 1:1)	contact grouting (the binder-to-granular filler ratio is 1.5:1)
Apparent density, kg/m^3	230	280
Water absorption, %	51.2	241
Moisture sorption, %	9.0	33.3
Flexural breaking stress, MPa	0.85	0.76
Breaking stress at 10 % compression deformation, MPa	0.642	0.414
Thermal conductivity coefficient, W/m·K	0.0516	0.0548

tion materials obtained by volume and contact grouting were also carried out. Comparative photographs of the appearance and structure of the samples are shown in Fig. 5.

As can be seen from the photographs in Fig. 5, when thermal insulation materials are bloated by contact grouting, the obtained samples are characterized by an uneven distribution of bloated granules in the binder layer in both upper and lower surfaces. During bloating, granules settle at the bottom of the mold, foam is unstable and collapses forming large voids and areas of only the binder without the granular filler, which leads to a decrease in strength. In this case, the binder is unevenly distributed in the intergranular space (h); this leads to formation of pores of large diameter and, consequently, low strength, because the walls of large pores are thinner, and results in greater water absorption and hygroscopicity due to predominance of the open-porous structure. The structure of the bloated granule at contact and volume grouting (c, g) practically does not differ.

The samples obtained by volume grouting are characterized by more even surface, full granule coverage, close packing of granules, and uniform distribution of the binder in the inter-granular space. These samples have an ordered fine-porous structure and therefore high strength with the predominance of the closed-porous structure.

4. Conclusions

Obtaining block thermal insulation materials by volume grouting, that is, simultaneous bloating of the granular filler and the liquid glass binder prevents shrinkage, cracks, foam collapse and formation of large voids due to dense packing of the bloated binder that fills the intergranular space. As a consequence, the blocks are characterized by higher physical and mechanical properties (density of 230 kg/m^3, flexural breaking stress of 0.85 MPa, compression strain of 0.622 MPa) with higher absorption resistance to water and its vapours, and the lower thermal conductivity coefficient (0.0516 W/m·K compared to 0.0548 W/m·K during contact grouting). In addition, the volume grouting method uses crude granules, and, therefore, the volume concentration of the solid phase in the source material will be higher than when using bloated granules, which contributes to intensive porization.

Volume grouting and simultaneous bloating of both granules and the binder can be performed using microwave radiation, which allows for uniform heating of all the

layers of the sample, thus obtaining a uniform porous structure of the material and high operational characteristics reducing the bloating temperature and the process duration.

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