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PRODUCTION OF POROUS GLASS-CRYSTALLINE MATERIALS USING DIFFERENT TYPES OF NATURAL AND RECYCLED RESOURCES

S. Nikitin, Y. Kol'tsova, Y. Beliy

Ukrainian State Chemical–Technological University,
8, Gagarina str., 49000, Dnepropetrovsk, Ukraine

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ABSTRACT

In this article the results of experimental studies aimed at obtaining glass-crystalline porous materials based on various types of natural and recycled materials are presented. The possibility of using cheap water-containing natural and recycled materials as bloating agents was established. The influence of the content of such additives on the structure and properties of porous glass-crystalline materials at different firing temperatures was evaluated. Prospects of using the developed materials as insulation products in the construction industry are shown.

Keywords: pores, heat insulation, foam glass, glass-crystalline material, bloating agent, breakage glass, slag.

INTRODUCTION

Currently, the question of insulation of residential buildings in order to keep them warm and reduce energy costs on their heating is very acute. In today's building materials market, there are many insulating materials of organic and inorganic origin. Due to the relatively low cost, the most common ones are mineral wool and plastic foam. However, these materials have significant exploitation drawbacks.

Plastic foam is characterized by low rates against high temperatures. Most of them are smoldering or burning well, emitting toxic products of combustion. Under prolonged influence of high temperature, oxygen, light, etc., certain physical and chemical processes (thermal, mechanical, photochemical degradation) occur in polymers, leading to a significant loss of properties of the foam due to a decrease in the molecular mass of the polymer, the formation of free radicals and reduced length of chain macromolecules [1].

On the contrary mineral wool is frost-resistant, does not burn, and has a temperature range up to 873K. However, its use as an independent heat insulator in most cases is impractical because it shows self-packing and shrinkage, leading to reduction in durability. In addition, mineral wool is a collaterally-hydrophilic material with water absorption up to 600 %. Directly moisture does not cause the destruction of wool, but it can be a habitat for the development of house fungis, which form organic acids (oxalic, citric acid), that ruin it [2].

In our opinion, among the existing insulation materials the best exploitation characteristics has foam glass. It belongs to the cellular inorganic heat insulators with closed or open pores having size of 0.1 - 5 mm. Foam glass is obtained with heat treatment of a mixture of thin ground glass powder with a bloating agent. The material properties depend on the way of production, the composition of the glass and the type of bloating agent. Foam glass has a unique set of properties: small water absorption, resistance to decay, microorganisms, insects and rodents; fire resistance, chemical resistance to most environments; it can be cut, drilled, ground [3]. Foam glass is the insulation material that is suitable for long-term use in building structures.

However, in the manufacture of foam glasses the use of special welded glass, relatively expensive bloating agent and high firing temperatures cause its high cost. Thus, there is a need to study the possibility of involvement in the synthesis of porous material of new raw materials, mostly recycled.

Among the various industrial wastes, concentrated in Ukraine, and non-deficient natural raw materials, of particular interest for the synthesis of porous glass-crystalline materials as bloating agent is the slag of blast-furnace and open-hearth furnace production, asbestos cement products breakage (building slate) and loam. This is possible, because all of these materials include certain components (carbonates, hydrates, calcium and magnesium silicate hydrates, etc.) that can form enough of the gas phase at temperature intervals close to the softening temperature of glass breakage. However, we believe that a significant effect on the bloating of the mass of glass, except carbonate may have water including compounds.

Bezborodiy M. [4] found that a "fluxing" nature of OH-ions is shown by the fact that the continuous silicate network is broken into individual silicate islands that are as much smaller in size and as much bigger in number, as there are more OH-ions in the melt. It was found that the decrease in glass viscosity with increasing of water content was most evident in the low-temperature region. The authors explain this fact by breaking of connections due to penetration of OH groups into the structure of the glass. In this regard, we believe that the use of natural and recycled water including materials as bloating agent, is of particular interest. Moreover, the role of bound water contained in natural rocks, wastes from industry and other bloating agents in the preparation of porous materials is not fully studied.

In studies of the influence of the gas phase composition on the kinetics of foam forming in foam glass, conducted by E. Schultz [5], it was found that the first condition for foaming is the presence of water vapor in the glass. Its presence reduces viscosity and surface tension of the glass, which greatly facilitates the development of pyroplastic foams. It can be concluded that for lowering of the temperature for obtaining porous materials, it is preferable to have bound water, which can enter the bloating agent mixture with the help of adding in it substances, containing hydration water.

The method of producing foam glass without using of traditional bloating agent has been developed by the authors [6]. By milled (to specific surface 5000 g/cm^2) bottle or widowpane glass breakage by semi-dry pressing, shaped samples are produced with their next thermal and humidity treatment. This treatment is carried out in order to watering and the formation of silanes - water (hydroxyl groups, connected directly with silicon [7]). Obtained silanes - water acted as a bloating agent under the foaming temperature of 1073K.

However, this method is time consuming. Firstly, you need to grind the glass breakage to a large specific surface area (5000 g/cm^2), and secondly - to carry out energy-intensive thermal and humidity treatment in complex, non-standard units.

The aim of this study was to obtain and study porous glass-crystalline materials with high exploitation characteristics, based on rationally selected composite mixtures of different types of non-deficient natural and recycled materials, containing hydration water.

EXPERIMENTAL

For porous glass-crystalline materials we used flat glass breakage with the additions of metallurgical slag (blast furnace, produced by the EVRAZ DMZ Petrovskogo and open-hearth of Interpipe NTRP), breakage of asbestos cement products (BACP) and loam of the Sursko-Pokrovskiy field in the Dnepropetrovsk region.

Charges were prepared by separate dry grinding of the raw materials in a ball mill to a particle size, which is characterized by a specific surface of 3000 g/cm^2. From the obtained raw mixtures with the use of the method of semi-dry pressing (w = 10 %) were formed cylindrically shaped samples with a diameter of 24 mm and a height of 9 - 10 mm. After drying at indoor temperature for 24 hours, the samples were fired in a laboratory furnace in the temperature range of 973 - 1173K with keeping at the maximum temperature for 0.5 - 1 hour, and that was followed by slow cooling. Formation of the samples was carried out in order to simplify the subsequent measurements. From the proposed mass composition porous materials can be obtained with the use of conventional powder technology of foam glass production [5, 8].

The sample volume of irregular geometrical shape was determined using a sand volumeter [9]. The specific gravity was determined in accordance with the state standard of Ukraine (SSU) № B V.2.7-42-97, the compressive strength was measured using a universal durability testing machine FP-10, according to the all-Union State Standard (AUSS) №8462-85. Heat conductivity of the obtained porous materials was measured on the instrument according to AUSS 7076-99.

X-ray phase analysis of the raw materials and the samples was carried out according to the method of powder on a DRON-3.0 diffractometer and Cu-Kα ra-

Table 1. Chemical analysis of experimental samples of raw materials, mass %.

Name of the material	Name of oxides											
	SiO_2	TiO_2	Al_2O_3	Cr_2O_3	Fe_2O_3	FeO	CaO	MgO	MnO	K_2O	Na_2O	LOI
Breakage glass [12]	72.60	–	1.60	–	0.10	–	8.10	3.60	–	0.50	13.50	–
Blast furnace slag	38.79	0.20	7.00	–	0.01	0.20	45.70	5.50	–	0.30	0.30	2.00
Open-hearth slag	28.00	–	6.50	2.00	20.36		16.10	15.00	3.00	0.24	tr.	0.30
Loam	70.02	0.11	8.50	–	2.29	–	6.67	1.57	–	2.02	0.73	8.09

diation. The identification of the crystalline phases was carried out [10].

Differential thermal and thermogravimetric analyzes of the raw materials were performed on a derivatograph of the Paulic-Paulic-Erdey system. The raw materials have been analyzed in air at the speed of heating of 10°C/min.

Scanning electron microscopic analysis was performed on the microscope REM-106Y. Images were obtained when working in secondary electrons. The maximum residual pressure in the microscope column (in the gun) was not more than 6.7 x 10^{-4} Pa (5 x 10^{-6} mm Hg. Cent.) and gun current - 111 mA.

RESULTS AND DISCUSSION

We conducted a preliminary analysis of the possibility for using experimental recycled and natural raw materials, as bloating agents. It was determined that the experimental blast furnace slag was mainly represented by four oxides - SiO_2, Al_2O_3, CaO and MgO, with their total content of about 97 mass % (Table 1). This slag belongs to basic slags, because it has a base module greater than 1. The phase composition of the blast furnace slag is represented mainly by a glass phase (95-97 %), in which crystalline formations are distributed; their identification has complications due to their small number (Fig. 1) [11].

According to the results of the differential thermal analysis (Fig. 2), an endothermic effect at 933K corresponds to the softening of the furnace slag glass phase, and intensive ekzo-effects at temperatures 1133 and 1253K, for its in-step crystallization. An exothermic effect at 1253K, which is accompanied by a mass gain (curves TG and DTG), may be partly connected with oxidation of the sulfide sulfur, which is part of the slag glass.

In addition, the thermogravimetric curve recorded sample mass loss of about 3.3 % in the temperature range 753 - 1053K, which is caused by the occurrence of processes of dehydration and decarbonization of compounds that are probably formed in the process of interaction of residual free lime with water during the wet granulation of the slag melt, and also with the ambient air, during the stay of already solid slag waste in the dumps.

We also studied an open-hearth slag that was preliminary magnetically enriched. It should be noted that the studied slag is heterogeneous at its corn composition: it contains solid, hard milling inclusions of different nature, and their content reaches 35.52 mass %. Mainly, such inclusions are in large fractions of open-hearth slag (larger than 1 mm), which is suggesting their metallic nature. For further research the well milling non-magnetic part of the research open-hearth slag was chosen.

According to the X-ray phase analysis (Fig. 3) it was found that the main crystalline phase of the slag is β-quartz, which corresponds to the data of the chemical

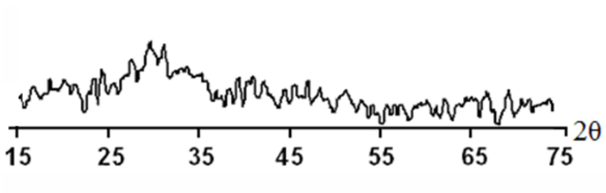


Fig. 1. XRD analysis of sample of blast furnace slag.

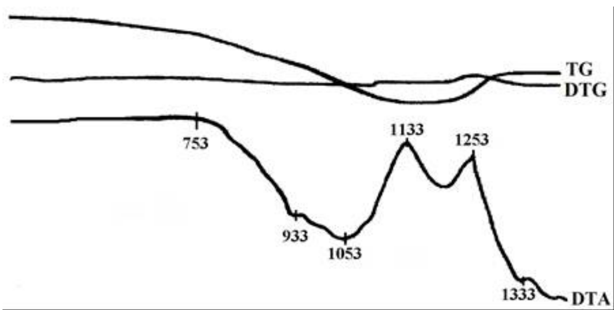
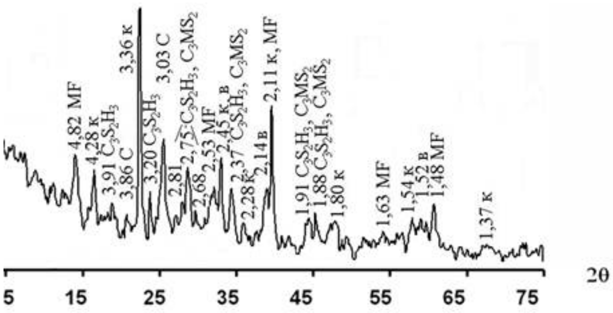


Fig. 2. Differential thermal analysis of sample of blast furnace slag.



к – β-quartz; в – RO- phase; MF – $MgFe_2O_4$; $C_3S_2H_3$ – $3CaO{\cdot}2SiO_2{\cdot}3H_2O$; C_3MS_2 – $3CaO{\cdot}MgO{\cdot}2SiO_2$; C – $CaCO_3$

Fig. 3. XRD analysis of a sample of open-hearth slag.

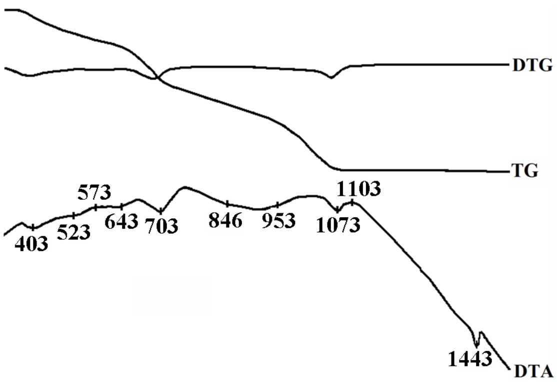


Fig. 4. Differential thermal analysis of the sample of open-hearth slag.

analysis (Table 1). In addition, in the open-hearth slag there is a fairly high number of calcium carbonate in the form of calcite ($CaCO_3$), with his characteristic diffraction peaks and calcium hydrosilicate, which is likely to be the result of the interaction of free lime from the ambient air and the quartz component.

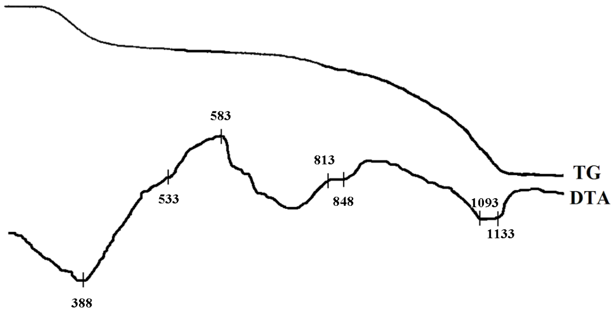


Fig. 5. Differential thermal analysis of the sample of loam.

The content of hydrosilicate and calcium carbonate in the phase composition of studied open-hearth slag is also confirmed by differential thermal analysis (Fig. 4), namely, the presence of significant mass loss in the analyzed samples. The highest loss (7.5 %) is observed in the temperature range 573 - 1073K. In the curve of the differential-thermal analysis there is an endoeffect with a minimum in the temperature range 693 - 703K, related to the dehydration of the calcium hydrosilicate, called afvillit ($3CaO{\bullet}2SiO_2{\bullet}3H_2O$), contained in the slag (Fig. 3), which, moreover, can be partially attributed to the decomposition of $Mg(OH)_2$. The endothermic effect at 1053-1073K and the corresponding mass loss are associated with calcium decarbonization [13].

In this work, research of clay materials of different deposits of Dnipropetrovsk and Zaporizhzhya regions was carried out [14]. It was found out that the most suitable for use as a bloating agent is loam.

Loam, used in this work, is a polymineral kind of clay material of hydromicakaolinite composition with inclusions of montmorillonite. Approximate quantitative

Table 2. Quantitative mineralogical composition of the research loam.

Component	The content of the phases in the clay,%
Kaolinite	10 - 20
Hydromicas	2 - 3
Montmorillonite	3 - 5
Quartz	50 - 55
Carbonates (calcite, dolomite)	17 - 20
Feldspars	2 - 3
Glauconite	1 - 2
Accessory minerals	4 - 6
Carbonaceous shales	to 1

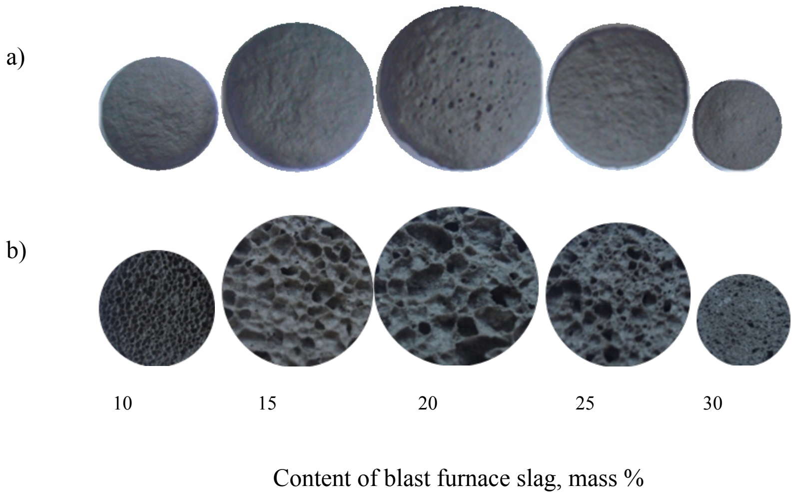


Fig. 6. Appearance of test specimens (a) and the distribution of pores (b), fired at 1173K.

relationships among the mineral phases in its structure are shown in Table 2.

In order to study the processes occurring during the loam heat treatment, its thermogravimetric analysis was carried out (Fig. 5). In the thermogram in the temperature region 380 - 500K, an interlayer and free water removal was fixed. In the temperature range 800 - 873K a series of endo-effect was visible, which indicated modification transition of quartz and removal of chemically bound water. The double endo-effect in the temperature range 993 - 1223K is associated with the processes of magnesite and calcite decarbonization. Taking into account the above mentioned, we can predict that the use of loam in a composition mixture with brakeage glass will ensure sufficient gas phase discharge during the softening of the glass mass and will contribute to the formation of pores.

Asbestos-cement products (slate) breakage, produced using 15 mass % Chrysotile asbestos and 85 mass % Portland cement were also used in our work. According to data from [15], chrysotile asbestos ($Mg_6[Si_4O_{10}](OH)_8$), which is magnesium hydro silicate, is dehydrated when heated to temperatures close to the temperature of liquid phase formation of the breakage glass, which may contribute to the formation of porous structure. Besides, calcium hydro silicates that are the main structure-building elements of the cement stone, dehydrate at temperatures 393 - 973K (depending on the type of calcium hydrosilicate) [16], which coincides with the temperatures of glass breakage softening.

We have investigated the raw mixtures, that contained breakage glass and various concentrations of the above mentioned additives, that can act as a bloating agent. Breakage glass served as the main component, which due to the high content of alkali oxides (15 %) helped to reduce the temperature of the material foaming.

Samples containing from 10 to 45 mass % of blast furnace slag were initially fired at a temperature of 1173K. The analysis of their properties allowed to define a clear tendency for lowering of the specific gravity of the material with reduction of the mass of blast furnace slag in the experimental charge from 45 to 20 mass %. Further reduction of the slag from 20 to 10 mass % led to a gradual increase of the specific gravity. The appearance of samples and pore distribution are shown in Fig. 6.

In order to obtain a glass-crystalline material with a lower specific gravity and a more uniform porous

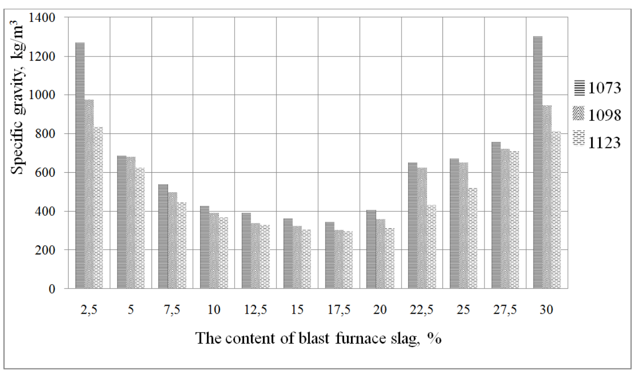


Fig. 7. Dependence of the specific gravity of the experimental mass in the content of blast furnace slag.

Fig. 8. Distribution of pores in the experimental samples, fired at 1123 K.

structure, we expanded the blast furnace slag concentration range in the experimental masses between 2.5 and 30 mass % with step of 2.5 mass %. These samples were fired at temperatures, lowered to the 1123 - 1073K range. This experiment revealed that at all experimental firing temperatures - 1123, 1098 and 1073K, there was a tendency of gradual decrease in the specific gravity of the samples with the increase of the content of the blast furnace slag in them from 2.5 to 17.5 mass % (Fig. 7). Further growth of this waste within the experimental charge led to a rise in their specific gravity due to increasing of the viscosity of the glass phase in them, because of growth of the content of silica, calcium and aluminum oxides. It should be noted, that the lowest values of the specific gravity had samples that were fired at a temperature of 1123K, though at the other two temperatures: 1098 and 1073K, samples swelling was significant. Masses, containing blast furnace slag from 7.5 to 12.5 mass % (Fig. 8), had a more uniform distribution of pores.

The analysis of the phase composition of the samples, containing glass breakage and blast furnace slag, fired at a temperature of 1123K, showed the formation and presence of such crystalline compounds as vollastanit, rankinite and doublecalcium sodium disilicate in the studied material (Fig. 9).

The obtained glass-crystalline samples were characterized by a fine porous (d = 1 - 2 mm) structure, similar to the foam glass. When the content of blast furnace slag is from 15 to 22.5 % we may obtain glass-crystalline porous materials with pores (d = 2 - 5 mm), similar to the keramzit, which is produced according to a well-known ceramic technology with firing at 1423 - 1523K [17].

The products from the glass-crystalline synthesized materials are characterized by a compressive strength of 12.4 MPa and specific gravity of 300 - 600 kg/m^3, and can be used as insulation materials and fillers in lightweight concrete.

It was found experimentally, that the optimal firing temperature of samples containing loam was 1023K. The

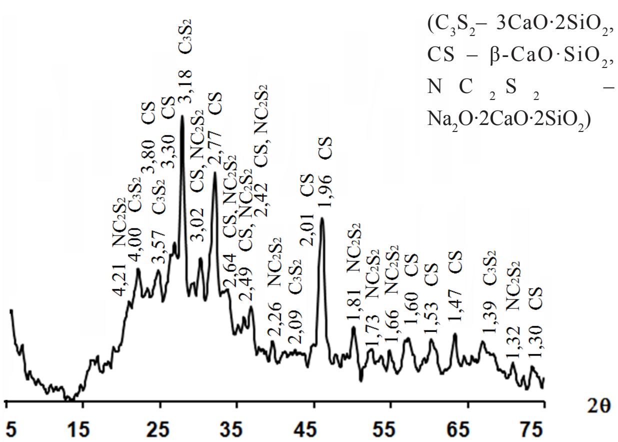


Fig. 9. XRD analysis of the glass-crystalline material containing 15 % of blast furnace slag.

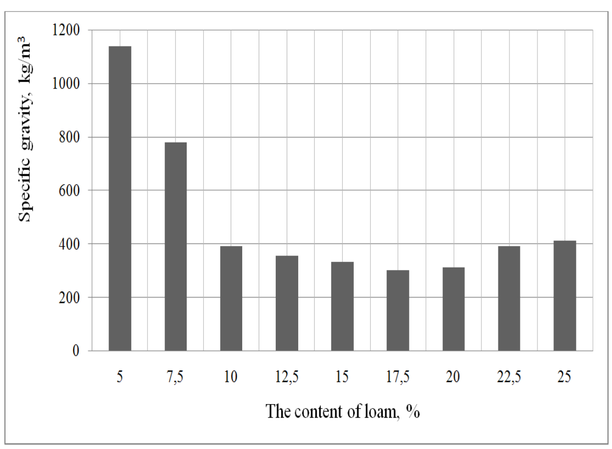


Fig. 10. Dependence of the specific gravity of the experimental mass on the content of loam.

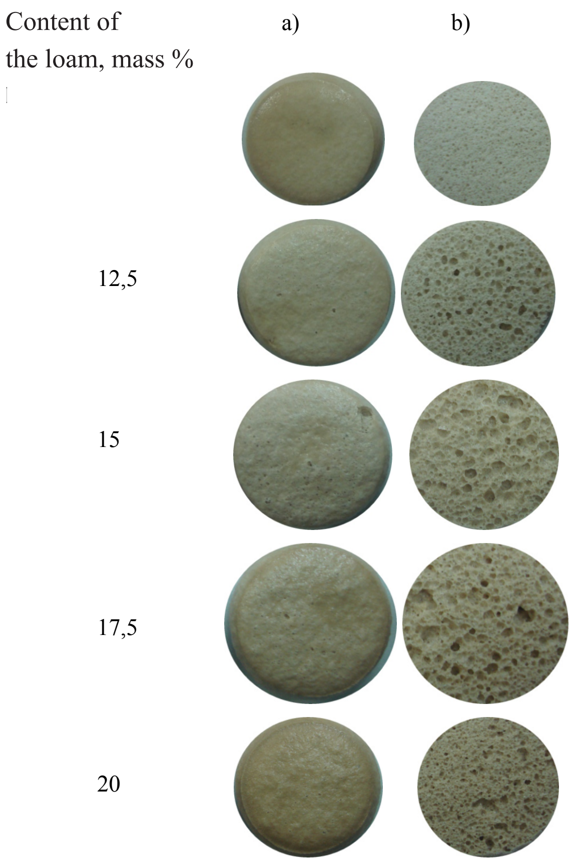


Fig. 11. Appearance of test specimens (a) and the distribution of pores (b), fired at 1023K.

analysis of the properties of the fired samples allowed us to determine a clear tendency for decreasing of the specific gravity with increasing of the loam content in the experimental charge from 5 to 17.5 mass %. Further increase of its content leads to a gradual increase in the specific gravity (Fig. 10).

As follows from Fig. 10, the lowest specific gravity can be reached with loam content of 17,5 mass %. However, such a sample has a somewhat heterogeneous structure with individual pores having diameter of 4 mm. The most homogeneous fine porous structure in the experimental samples is achieved when the content is 10 and 12.5 mass % (Fig. 11b).

Analysis of the phase composition of the sample containing 15 mass % of loam indicates the presence of a crystalline phase, which by its characteristic diffraction peaks can be attributed to devitryt (Fig. 12b). The pattern formed by the breakage glass without loam additives, fired at the same temperature and time is X-ray amorphous (Fig. 12a). Thus, loam contributes to the formation of the crystalline phase, which is usually present in devitrification of sheet glass.

We found out that for samples, containing BACP, the optimal firing temperature is 1023 K. These samples showed a tendency for reduction of their specific gravity with increasing of the number of BACP in the charge from 4.5 to 9.0 mass %. Further increase of its content leads to a gradual increase in the specific gravity (Fig. 13).

The samples, containing BACP, had an uniform porosity, specific gravity of 260 - 440 kg/m^3, compressive strength 1,8 - 3,0 MPa and thermal conductivity of not more than 0.06 W/m K.

As shown in Fig. 14, as a result of the use as a bloating agent of 4.5 to 6.0 mass % BACP, the charge forms an almost invisible microporous structure. However, the specific gravity of such samples does not exceed 460 kg/m^3, which is less than the specific gravity of the lightest samples of the previously developed porous materials, using fly ash (490 kg/m^3) [18]. This fact indirectly indicates that the samples, containing BACP, have lower thermal conductivity than the material, obtained from using a carbon bloating agent. This assumption is confirmed for all materials in which the bloating agent's

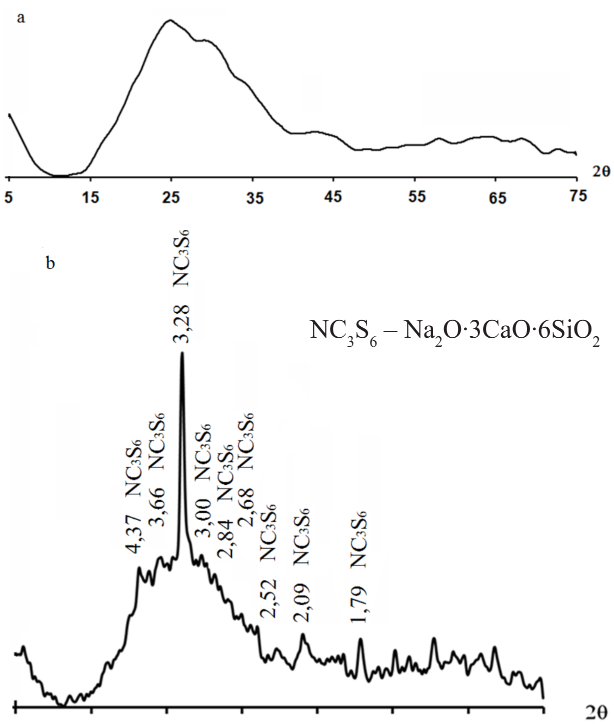


Fig. 12. XRD analysis of breakage glass, burnt at 1023K (a) and the test sample containing 15 mass % loam (b).

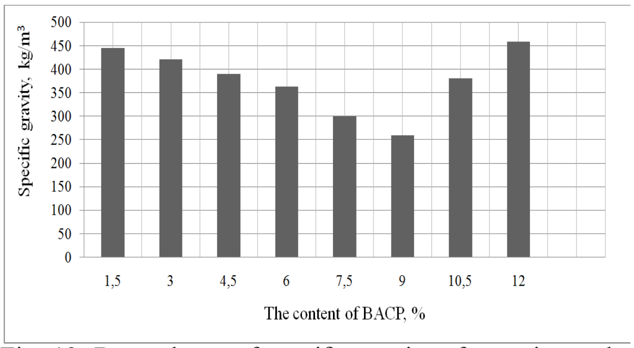


Fig. 13. Dependence of specific gravity of experimental mass on the content of BACP.

role is played by hydrates.

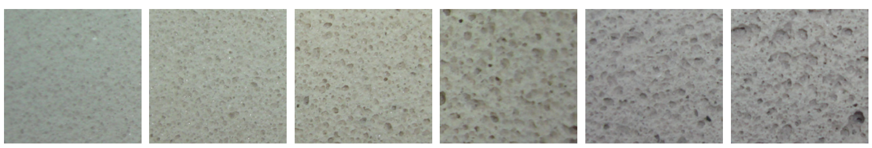
For example, samples containing loam have a lower thermal conductivity (0.05 W/m K) than industrial foam glass, produced by the JSC «Plant of building insulation materials» (Zaporozhye), for manufacturing of which carbon bloating agent is used. According to the technical conditions of Ukraine № 26.133011391-001:2006 «Insulation blocks from foam glass», the thermal conductivity of industrial foam glass is regulated to be no more than 0.066 W/m K, while the thermal conductivity of our research samples, with loam as a bloating agent is 0.05 W/m K . In addition, the temperature for obtaining of such porous material is 75K lower than the industrial one.

The same pattern is visible on the samples, obtained with open-hearth slag, as a bloating agent (Fig. 15), the previous studies of which indicated a high content of calcium hydrosilicate of afvillit in it ($3CaO{\bullet}2SiO_2{\bullet}3H_2O$).

Fig. 16 shows that the use of less than 5 mass % of open-hearth slag as the bloating agent does not give a porous material, and the addition of more than 15 mass % is not reasonable, as it results in compaction of the porous structure.

The obtained samples were characterized by homogeneous fine porous structure, specific gravity of 220 - 420 kg/m^3 and compressive strength of 1,5 - 6 MPa.

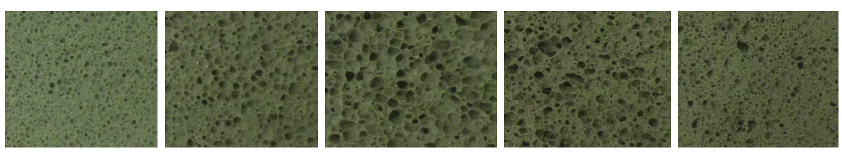
X-ray analysis proved the glass-crystalline structure of the obtained porous materials (Fig. 9, 12-b). Crystallization of foam glass in most papers is considered as a negative phenomenon. According to the authors of [19], in the synthesis of new compositions of foam glass, special attention should be paid to the search of compositions with a minimal tendency to crystallize at the foaming temperature, as crystallization deteriorates the foaming process or even prevents the obtaining of foam glass. To prevent crystallization in foam glass we needed to choose such foaming temperature conditions so that we could avoid the crystallization process at the time of foaming or before the start of foaming the formation of crystalline phase was found in the initial state. When considering foam glass crystallization processes, it should be noted that in the literature we can find little data on directional crystallization of pore partitions of vitreous material. With a prevailing idea that the crystal-



4,5 6,0 7,5 9,0 10,5 12,5

Content of BACP, mass %

Fig. 14. Distribution of pores in the experimental samples, containing BACP fired at 1023K.



5,0 7,5 10,0 12,5 15,0

Containing open-hearth slag, mass %

Fig. 15. Distribution of pores in the experimental samples containing open-hearth slag fired at 1023K.

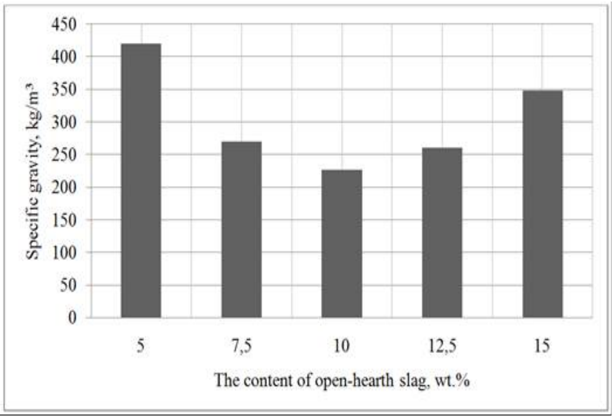


Fig. 16. Dependence of specific gravity of experimental mass on the content of open-hearth slag.

a)

b)

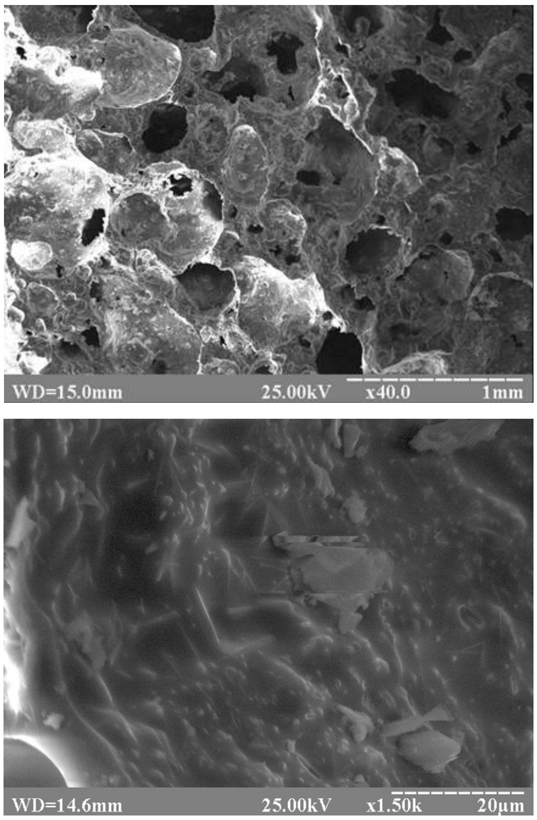


Fig. 17. SEM analysis of a sample containing 15 % blast furnace slag with magnification x40 (a) and x1500 (b).

lization in the foaming temperature region deteriorates the process of pores forming, all available information mainly refers to the exclusion of the crystallization process in order to improve the conditions for the development of a highly porous foam glass structure.

Thus, foam glass, despite a number of indisputable advantages, has a vitreous structure of pore partitions. In the classic foam glass, the content of non-melted particles or stable crystalline phases from raw materials is generally regulated at no more than 5%. This restriction is explained by the presence of large crystals in the glass that can cause the destruction between pore partitions and the growth of rate of water absorption and, as a result – reduce frost resistance and durability of the foam glass products.

On the other hand, the metastable state, which is characteristic for vitreous material under appropriate thermodynamic conditions, can cause uncontrolled crystallization, accompanied by destruction of the glass and its devitrification that leads to the loss of original properties of the foam glass products.

In connection with the above mentioned, in our opinion it is very important to search opportunities for obtaining of a porous material with a partially crystallized structure of pore partitions. The interest on this subject is due, firstly, to the fact that the crystallization to some extent deteriorates the foaming processes, resulting in the compacting and hardening of the foam glass structure, and secondly – the completeness of the crystallization processes in the porous material at the stage of technology is much better, than when the crystallization processes occur in exploitation.

To prove the assumption that fine porous crystallization between pore partitions increases the strength of the synthesized material, we carried out scanning electron microscopy of samples containing blast furnace slag (Fig. 17) with the highest compressive strength (4 - 12 MPa).

Fig. 17-a illustrates the partially crystallized structure of the synthesized porous material. In Fig. 17 many needle-shaped crystals of average length of 9 microns can be clearly seen. In our opinion they perform a reinforcing function, increasing the strength of the material.

CONCLUSIONS

The theoretical and experimental studies, confirmed by X-ray phase and differential thermal analyses, established the fundamental possibility for obtaining of glass-crystalline porous materials with non-deficient recycled and natural materials, containing hydration water in the structure. It was shown that the hydration water in the charge can significantly reduce the temperature of firing of such materials.

The rational concentration of bloating agents (natural and man-made) was established from the point of view of obtaining materials with desired properties.

Electronic microscopic analysis confirms the partial microcrystallization of the pore partitions of the developed porous material, which increases its strength.

The advantage of developed porous glass-crystalline materials is that they do not contain harmful to humans and the environment components. Their production can significantly reduce energy and material costs in comparison to the manufacturing of foam glass according to traditional technology [3, 20]. In addition, the use of waste of metallurgical and construction industries as a bloating agent will contribute to their partial utilization and improvement of the environment.

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