### DENSE GLASS FOAM PRODUCED IN MICROWAVE FIELD

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**Abstract:** Experimental results obtained in the process of manufacturing dense glass foam using the microwave energy are presented in the work. The glass foam is produced from bottle glass waste, calcium carbonate as foaming agent and borax as fluxing agent. The high compressive strength (2.5 - 6.2 MPa) is the main mechanical feature of this product, which together with other physical and morphological features (apparent density 0.60 - 0.90 g/cm<sup>3</sup>, porosity 59.1 - 72.7%, thermal conductivity 0.081 - 0.105 W m K, water absorption 0.5 - 1.0%, pore size 0.5 - 3 mm), are appropriate for using as a substitute for similar building materials existing on the market.

Keywords: dense glass foam, expanded glass foam, compressive strength, glass waste, microwave field

### **1. INTRODUCTION**

The dense glass foam is a porous material obtained by sintering at high temperature of silicate wastes, which in presence of a foaming agent incorporated in the powder mixture of raw material generates numerous bubbles. The material has a structure with low size pores, which ensures it both a low apparent density and thermal conductivity and a very high mechanical strength. These are characteristics required of an insulating material or a concrete aggregate in construction [1].

Several types of glass foam are industrially manufactured currently. The main are "Technopor" made by Misapor Switzerland Company with subsidiaries in some European countries (Germany, France and Austria) and "Foamglas" made by Pittsburgh Corning Company with subsidiaries in Belgium, Czech Republic, China and United States.

The "Technopor" products are glass foams with high compressive strength (up to 6.0 MPa), fire and rodent aggression resistance and non-absorber of humidity. Due to these features, they are used in commercial and industrial buildings as outside insulating materials, road construction, infrastructures foundation, sports grounds and other fields where a high mechanical strength is required. According to the literature [2], the manufacturing process of the "Technopor" products requires a specific consumption of electricity of about 100 kWh/m<sup>3</sup>, at which is added about 25 kWh/m<sup>3</sup> consumed for processing the waste material before entering into the furnace.

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The "Foamglas" products are resistant to compression and humidity being made as blocks for the thermal insulations of masonry of buildings, as well as large size plate shape, used broken as concrete aggregates. The main characteristics of "Foamglas" products are: low thermal conductivity, absolute tightness against water and vapour, compressive strength (up to 2.75 MPa) without deformation. The specific consumption of fossil fuel to make "Foamglas" reaches 4.24 kWh/kg [2].

Excepting the industrial production, the experimental research carried out in the world on dense glass foam manufacturing is presented in the literature. The use of coal ash and glass waste as raw material in proportions of 40 and respectively 60 wt.% and addition of 0.5 wt.% calcium carbonate as foaming agent and 30 wt.% borax as fluxing agent led to obtain a product with apparent density 0.46 g/ cm<sup>3</sup>, high compressive strength (over 5 MPa) and low thermal conductivity (0.36 W/ m K) [3].

An innovative technique of producing glass foam for tiles destined to the building front with risk of terrorist attack is described in the US patent [4]. The product is made from siliceous materials and a foaming agent. The apparent density of this material has values between 1 - 1.28 g/ cm<sup>3</sup>. Its characteristic is the absorption of the thermal shock of the blast.

To improve the mechanical strength of the foamed glass, sodium silicate in a weight proportion of maximum 40% in mixture with glass waste and calcium carbonate as foaming agent was used [5]. The mechanical strength of the product reached 6 - 8 MPa.

Experimental research on the glass foam manufacturing with the microwave energy, using two types of industrial glass waste from soda-lime silicate glasses and respectively cathodic ray tubes and different foaming agents (C, SiC, AlN) has been carried out [6]. The glass foams obtained with carbon as foaming agent have high dielectric loss and are suitable as green electromagnetic absorbent in construction.

Excepting the experiments in microwave field mentioned above, all manufacturing processes of expanded glass foam used currently in the world apply the conventional heating techniques. The advantage of the microwave using consists in directing the heating only on the material subjected the thermal process. The heating is fast, "clean" and economical. Though the microwave energy is currently used in the household, this advanced heating system is not yet industrially applied than in an extremely low scale [7].

### 2. METHODS AND MATERIALS

#### 2.1. Methods

The latest research of the Romanian company Daily Sourcing & Research has been focused on conducting the experiments in working conditions similar to those in an industrial furnace with conveyor belt, where the raw material is loaded. To operate with the microwave energy, the industrial furnace should have the sidewalls and the flat vault coated with a layer of a ceramic microwave susceptible material (e. g.: silicon carbide). The microwave field, generated by magnetrons placed on the outer furnace housing, is propagated through this layer. The thickness of the layer should ensure an optimal ratio between the waves absorbed in the layer and those that penetrate it coming in contact with the powder mixture. In the first case, the indirect heating of the material is produced by thermal radiation of the inner surface of the layer and, in the second case, the direct heating occurs, generated in the material core and developed to the peripheral areas. Previous tests have shown that a silicon carbide wall with thickness of 3.5 mm is optimal to obtain the double effect of heating.

To simulate the working conditions of a tunnel furnace, where the material is exposed to microwave radiation during the displacement of the conveyor belt along its length, were used a domestic microwave oven adapted to the high temperature requirements (Figure 1) and a cylindrical silicon carbide crucible with wall thickness of 3.5 mm placed with the opening down on ceramic fiber mats. The oven has a single 0.8 kW magnetron positioned on one of the sidewalls. Thick layers of ceramic fiber mats are fixed around the sidewall and bottom of the crucible to ensure the thermal protection. The powder mixture previously pressed is deposited on a metal plate with the diameter  $\emptyset$  107 mm placed on the bottom ceramic fiber layers. Over the plate containing the pressed mixture is placed the silicon carbide crucible with the diameter  $\emptyset$  125 mm and the height 100 mm. The expansion of the mixture is free under the influence of microwave irradiation and respectively the heat transferred by the walls and vault through thermal radiation. Unlike the domestic microwave oven, the rotation mechanism of the support containing the material is not functional. So, the heating process takes place statically.

The measurement of the powder mixture temperature during the process is made with a Pyrovar type radiation pyrometer placed above the oven. The pyrometer visualizes a small surface of the silicon carbide crucible bottom unprotected with ceramic fiber, which allows its visualization with the pyrometer. The correlation of the temperature measured on the crucible surface with the temperature of the material from inside was experimentally achieved.



Fig. 1. The adapted 0.8 kW microwave oven.

It was adopted a methodology of experimentation consisting in testing the six variants of the mixture composition formed by the waste of consumed bottle colorless glass, calcium carbonate as foaming agent and borax as fluxing agent. The mixture was wetted with 8.5% water addition as binder for the pressing process. The powder mixture composition corresponding to the tested variants are shown in Table 1. The weight ratio of glass waste had values between 90.0 - 94.0%, the weight ratio of calcium carbonate was between 1.5 - 5.0% and the weight ratio of borax was kept constant at 5%. These weight proportion ranges are the result of previous experiments conducted in various other conditions in the company Daily Sourcing & Research. Therefore, the ranges adopted for the test were limited to the values specified above.

Variant	Weight composition, wt.%					
	Bottle glass waste	Borax	Calcium carbonate	Water absorption		
1	90.0		5.0			
2	90.7		4.0			
3	92.0	5.0	3.0	8.5		
4	93.0		2.5			
5	93.5		2.0			
6	94.0		1.5			

Table 1. The powder mixture composition

At the end of the sintering/ foaming process, the glass foam samples were tested in laboratory to determine the physical, mechanical and morphological features (apparent density, porosity, thermal conductivity, compressive strength, hydrolytic stability and water absorption). The current methods [8 - 11] were used.

### 2.2. Materials

Consumed bottle colorless glass waste was the raw material used in experiments. According to [12] the chemical composition of this waste is the following: 71.8% SiO<sub>2</sub>, 1.9% Al<sub>2</sub>O<sub>3</sub>, 12.0% CaO, 1.0% MgO and 13.3% Na<sub>2</sub>O. The glass waste was broken and ground in a ball mill, its granulation being less than 100  $\mu$ m. Calcium carbonate as foaming agent had a very fine granulation (under 40  $\mu$ m), being used without other mechanical processing such as it was purchased from the market.

Due to the high Na<sub>2</sub>O content, which is by far the most dominant fluxing material [13], especially, for the glass industry, borax is used in experiments as fluxing agent. Purchased from the market at a grain size under 400  $\mu$ m, it was used in experiments under 130  $\mu$ m after the grinding in an usual electric device.

The powder mixture prepared for the each experimental variant was dry homogenized and wetted with water (8.5% over the amount of raw material) in a small laboratory device.

Then, the wet mixture was loaded into a demountable mold and was pressed by mechanical methods up to about 2 - 3 MPa.

#### 3. RESULTS AND DISCUSSION

The main functional parameters of the sintering/ foaming process (quantities of raw material and glass foam, sintering/ foaming temperature, average heating and cooling speeds, index of volume growth and specific consumption of electricity) are shown in Table 2.

Table 2. The main parameters of the sintering/ foaming process							
Variant	Raw	Glass	Sintering/	Average speed		Index of	Specific
	material	foam	foaming	(°C/ min)		volume	consumption
	quantity	quantity	temperature			growth	of electricity
	g	g	°C	Heating	Cooling		kWh/ kg
1		358.7	820	15.9	6.0	1.65	1.40
2		357.9	822	16.0	5.9	1.55	1.40
3	400	358.0	828	16.6	6.3	1.30	1.47
4		358.2	838	16.5	6.0	1.25	1.56
5		358.1	843	16.9	6.3	1.20	1.61
6		358.4	851	17.0	6.6	1.20	1.74

Table 2. The main parameters of the sintering/ foaming process

According to the data from the above table, the raw material quantity constituted the single parameter kept constant. The quantity loaded into the oven in all tested variants was 400 g. The glass foam quantity had values between 357.9 - 358.7 g, representing 89.5 - 89.7 %. The temperature of the sintering/ foaming process varied between 820 - 851 °C, the temperature value increasing with the decrease of the calcium carbonate proportion, i. e. the agent which has the role to favor the foaming process. Generally, the index of volume growth is insignificant, the maximum value of 1.65 being reached in the variant 1, characterized by the maximum proportion of calcium carbonate in the material mixture. The variants 5 and 6 with calcium carbonate proportions under 2 wt.% have volume increasing of only 1.20.

The specific consumption of electricity of the experimental manufacture process of glass foam has values between 1.40 - 1.74 kWh/ kg glass foam, being relatively high. The own experimental determinations on the heat losses outside and those through thermal accumulation at the end of the process represent over 70%. Thus, the real energy consumptions are: 0.4 - 0.5 kWh/ kg. The industrial furnaces operating with conventional heating systems, but in continuous operation regime, so with much lower heat losses, have energy consumptions of about 125 kWh/ m<sup>3</sup>, i. e. 0.11 - 0.18 kWh/ kg (at the values of dense glass foams), but they can reach 4.24 kWh/ kg [2]. Considering the working conditions significantly different, a comparison between the values of specific consumption of energy is not appropriate at this time.

The main characteristics of the glass foam samples are shown in Table 3.

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Variant	Apparent	Porosity	Compressive	Thermal	Water	Pore size	
	density		strength	conductivity	absorption		
	g/ cm <sup>3</sup>	%	MPa	W/ m K	%	mm	
1	0.60	72.7	2.5	0.081	1.0	1.0 - 3.0	
2	0.64	70.9	2.9	0.088	0.8	1.0 - 2.0	
3	0.71	67.7	3.6	0.093	0.7	0.9 – 1.5	
4	0.79	64.1	4.2	0.097	0.6	0.8 - 1.2	
5	0.84	61.8	5.6	0.101	0.7	0.6 - 1.0	
6	0.90	59.1	6.2	0.105	0.5	0.5 - 0.9	

Table 3. The main characteristics of the glass foam samples

The physical and mechanical characteristics of the glass foam samples experimentally obtained using the

microwave energy are very close to those of similar products made by conventional heating methods. The high compressive strength (2.5 - 6.2 MPa), low thermal conductivity (0.081 - 0.105 W/ m K), moderate apparent density  $(0.60 - 0.90 \text{ g/ cm}^3)$  and water absorption practically non-existent (0.5 - 1.0%) are appropriate characteristics for dense building materials involving a high mechanical strength. It is obviously that the weight proportion of the foaming agent (calcium carbonate) influences the features of the glass foam samples. Thus, the high proportions cause lower apparent density, thermal conductivity and compressive strength as well as higher porosities. Low proportions of calcium carbonate increase their density, thermal conductivity and compressive strength and decrease the porosity.

The pores size is also influenced by the weight proportion of calcium carbonate. The high proportions (variants 1 and 2) cause macrostructures with size pore up to 2 - 3 mm. Conversely, low proportions (variants 5 and 6) cause macrostructures with pore size under 1 mm (Figure 2).



Sample 4

Fig. 2. The macrostructure of glass foam samples.

Sample 6

Images of the pores microstructure of the samples 3 and 6 (arbitrarily selected) were obtained with a macroepidiascope with image analyzer, being shown in Figure 3.

Determining the hydrolytic stability of samples involved the use of 0.15 ml of 0.01M HCl solution to neutralize the extracted Na<sub>2</sub>O. The tests showed that the extracted Na<sub>2</sub>O equivalent is in the range 35 - 56  $\mu$ g, so that the stability joins in the hydrolytic class 2.



Sample 3

Sample 6

Fig. 3. Images of the pores microstructure of the samples 3 and 6.

### 4. CONCLUSIONS

The goal of research, whose results are presented in the work, is to obtain glass foam using the microwave energy with similar characteristics with those of materials manufactured currently by conventional heating methods.

Placing the raw material mixture under a silicon carbide crucible with opening down, introduced into the microwave oven created the working conditions of a tunnel furnace with conveyor belt powered with microwaves, the process being statically.

The main characteristics of the dense glass foam samples obtained experimentally in microwave field (apparent density 0.60 - 0.90 g/ cm<sup>3</sup>, thermal conductivity 0.081 - 0.105 W/ m K, compressive strength 2.5 - 6.2 MPa, water absorption under 1%, as well as a high macrostructural homogeneity) are similar to those made by conventional heating methods.

The products are appropriate for using as dense porous materials with high mechanical strength in construction. By comparison with the conventional heating methods of producing glass foam, the use of the microwave energy is at least theoretically more efficient.

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