

SIMULTANEOUS USE OF LIQUID AND SOLID FOAMING AGENTS BY A NONCONVENTIONAL TECHNIQUE TO OBTAIN A HIGH-STRENGTH GLASS FOAM WITH FINE POROSITY

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ABSTRACT: A glass foam with typical characteristics for a foam glass gravel usable as a thermal insulating material in difficult climatic conditions and with mechanical stress was made by sintering at 834 °C a powder mixture composed of colorless glass waste (93.1 wt.%), glycerol (1.0 wt.%), sodium silicate (4.8 wt.%), calcium carbonate (0.8 wt.%), kaolin (0.2 wt.%) and water addition (14.5 wt.%). The physical, thermal, mechanical and morphological characteristics of the product were: apparent density of 0.28 g/cm³, porosity of 86.67%, thermal conductivity of 0.063 W/m·K, compressive strength of 7.3 MPa, water absorption of 4.3% and pore size between 0.10-0.35 mm. The specific energy consumption of the manufacturing process was 0.78 kWh/kg. The originality of the work is the use of a nonconventional heating technique based on the microwave energy.

KEYWORDS: glass foam, microwave heating, glass waste, glycerol, sodium silicate, calcium carbonate

1. INTRODUCTION

In recent decades, there has been a strong trend worldwide to recycle all forms of waste (plastic, glass, metal, paper, textiles, etc.) on the one hand, to avoid occupying large areas of land with materials landfills considered unusable and on the other hand, to find new uses with newly created value of some waste following important economic and energy effects. This global trend of protecting the environment both at the ground level and in the atmosphere and saving energy in energy-consuming industrial processes has emerged in close dependence on the global energy crisis of the 1970s and the danger of overheating the Earth's climate due to uncontrolled emissions into the atmosphere of greenhouse gases.

The glass is industrially produced in different qualities and destinations, the most widespread assortment being packaging glass as bottles and other containers (for packaging the drinks, food, pharmaceuticals, cosmetics, etc.) which represents about 56% (21 Mt) of the total glass production in the EU [1]. The main advantage of glass for the environment and health is the possibility of recycling it without loss of quality. The energy required for the industrial manufacture of soda-lime glass (the most common type of glass) is 2670 GJ/ton, while the full use of recycled glass waste as

a raw material leads to a reduction of energy consumption up to 1880 GJ/ton [1]. However, the recycling of glass waste in the new glass production circuit involves high costs related to the color selection of waste. It was found that it is more profitable to use glass waste in the manufacturing process of glass foam, a product with very good thermal insulation characteristics (low density and low thermal conductivity) and quite high mechanical strength that can be used as a replacer for existing building materials on the market [2, 3].

Different types of glass foam have been industrially manufactured in the world since the middle of the 20th century by several manufacturers, of which the most important are: Misapor Switzerland with facilities in Germany, Austria, France and Switzerland, Pittsburgh Corning with facilities in the United States, Belgium, Czech Republic, United Kingdom, Germany and China, Geocell Schaumglas with facilities in Austria, Germany and Hungary, Glapor Werk Mitterteich in Germany and others. The main products of the Misapor company known as "TECHNOpor" have high compressive strength (4.9-6.0 MPa), low thermal conductivity (0.075-0.095 W/m·K), low density (0.21-0.40 g/cm³), are non-deformable, fire-retardant, non-absorbent of water and steam, resistant to freeze-thaw, resistant to the aggression of salts, acids, bacteria, rodents and insects. "TECHNOpor" products are used as thermal

insulation materials in conditions of mechanical stress and difficult environmental conditions [4-6]. Pittsburgh Corning products known as "Foamglas" are manufactured as blocks for the thermal insulation of building walls as well as aggregates. The products have the compressive strength between 1.6-2.75 MPa, ensure a constant thermal insulation and are completely sealed against water and steam [7]. More companies especially from northern Europe (Scandinavia) are focused on making foam products applicable in the field of road construction adapted to the harsh and very variable climate conditions between day and night specific to the Nordic countries [8]. This type of foam products with fine porosity, low density and high compressive strength, manufactured in conveyor belt tunnel ovens in the form of pieces (gravels) with irregular dimensions between 15-85 mm are called foam glass gravel. All the industrially manufactured products in the world mentioned above are obtained by conventional heating techniques (with electric heating elements or burning fossil fuels).

The Romanian company Daily Sourcing & Research, which in the last four years has experimentally tested on a small scale different manufacturing recipes of glass foam using the nonconventional microwave heating technique, analyzed comparatively based on the literature data the procedures used and the results obtained in this field by the industrial manufacturers [8]. Based on this analysis, the Romanian company adopted two manufacturing variants using glycerol as a liquid carbonic foaming agent and two variants using solid foaming agents (calcium carbonate with addition of borax and sodium silicate as well as silicon carbide, respectively). The product made with solid agents had very high compressive strength (7.4-7.5 MPa), but too high apparent density (0.35 and 0.65 g/cm³). The product made with glycerol had low density (0.24 g/cm³) and sufficiently high compressive strength (5.3-5.9 MPa). The conclusion was that the best foamed product was manufactured with 1% glycerol, 8% sodium silicate and 8% water by sintering at 823 °C with a low specific energy consumption of 0.88 kWh/kg. The characteristics of the product were: apparent density of 0.24 g/cm³, porosity of 89.1%, thermal conductivity of 0.063 W/m·K, compressive strength of 5.9 MPa and pore size between 0.3-0.6 mm.

According to the literature [9], an addition of sodium carbonate (Na₂CO₃) to the powder mixture containing 92% glass waste, 3.5% glycerol and 3.5% sodium silicate could lead by sintering at 850 °C to a foam product with a very high average compressive strength of 16 MPa, but also an average bulk density

of 0.67 g/cm³, obviously too high for a material that should have thermal insulation characteristics.

According to the paper [8], the German company Glamaco, suppliers of ovens and equipment for manufacturing the glass foam, recommends a manufacturing recipe of a material such as foam glass gravel containing 95% glass waste, 5% glycerol, sodium silicate and CaCO₃ as well as water addition and a very small amount of kaolin. The recommended sintering temperature should be below 900 °C, resulting a foamed product with an apparent density between 0.15-0.20 g/cm³, bulk density between 0.10-0.15 g/cm³, thermal conductivity in the range 0.06-0.08 W/m·K and compressive strength between 4-6 MPa.

The solution theoretically proposed by Glamaco company with the possibility of choosing the value of weight ratios of glycerol, sodium silicate and CaCO₃ in the range of maximum 5% and the addition proportions of water and kaolin was adopted by the authors for the experiment presented further. The heating technique was nonconventional by using the microwave energy.

2. METHODS AND MATERIALS

2.1 Methods

The foaming process of the glass waste having incorporated in the powder mixture a liquid carbonic foaming agent (glycerol) and a solid foaming agent (calcium carbonate) occurs by the thermal decomposition of the two agents. By decomposing in the oxidizing atmosphere of the oven, glycerol forms several compounds between carbon dioxide and pure carbon also including hydroxyl compounds [10]. Due to the high oxidation ability of carbon, in order to prevent its premature combustion with oxygen from the oven atmosphere, the fine glass particles must be envelope with the aqueous sodium silicate solution, which is initially added to the powder mixture. Thus, the glycerol decomposition is slowed down and the glass sintering is accelerated. The water addition in the mixture favors the formation of "water gas" containing hydrogen and carbon monoxide by the reaction of water vapor with carbon at about 800 °C. Thus, the "water gas" contributes to foaming the glass [10]. Simultaneously, the decomposition of calcium carbonate, the other foaming agent incorporated in the starting mixture occurs. The decomposition reaction takes place at temperatures above 750 °C [11] with the formation of calcium oxide (CaO) and carbon dioxide according to reaction (1).



CaO is treated in the molten glass, while the CO₂ is released into the viscous mass of the glass. Both CO₂ resulting from the decomposition of CaCO₃ and the gaseous products released by the decomposition of glycerol and respectively, by the generation of "water gas", form gas bubbles spread in the viscous mass of the glass. By increasing the internal pressure of the bubbles due to the increase of temperature, the viscous mass significantly increases its volume by expansion and foaming of the material occurs. By its cooling at the end of the thermal process, the bubbles turn into pores forming a porous structure typical for the glass foam [2].

The nonconventional heat treatment technique of the pressed powder mixture based on glass waste with mineral additives embedded in a 0.8 kW-microwave oven of the household type, but adapted to high temperature operation, was also used in these experiments as in numerous other tests performed in the company Daily Sourcing & Research and published in the literature [8, 12-14]. Previous experiments [15] have unequivocally shown that the commercial soda-lime glass is not suitable for the direct microwave heating under the normal operating conditions of microwave ovens (domestic or industrial) at a frequency of 2.45 GHz, corresponding to a wavelength of 12.2 cm. By microwave heating to 800-900 °C, serious destructive effects on the structure of the glass in its core were found. This effect is not valid in the case of silicoaluminous materials (with Al₂O₃ content over 10 wt.%), several heat treatments of such materials being performed by the direct heating [16]. However, in the case of waste from the commercial glass, the optimal solution was found to place a ceramic tube or crucible made of SiC and Si₃N₄ (materials with high microwave susceptibility) to protect the sample subjected to heating against the too active microwave field. A thickness of the tube (crucible) of 2.5-3 mm ensures both the penetration of the ceramic wall and the direct contact of the microwave with the sample (direct heating), as well as the partial absorption of microwaves in the wall mass, its rapid heating and then the transfer of thermal energy by radiation to the sample (indirect heating). So, a very efficient mixed microwave heating is achieved and at the same time non-destructive to the structure of the foamed glass. It should be noted that so far the ratio value of the two heating types has not been determined, but the energy effect of the mixed heating indicates the predominance of the direct heating.

The direct heating has some special features compared to the conventional heating. The

microwave field penetrates the heated material to its core and the microwave energy is converted into heat. Thus, the heating is initiated in the core of the material and propagates volumetrically to its peripheral areas. So, it can be said that the irradiated material itself generates heat. This way of heating is very fast and economical [17]. Another characteristic of the direct microwave heating that fundamentally differentiates it from the conventional heating is selectivity [18], which assumes that only the irradiated material (which has microwave-susceptible components) is intensely heated, while other components of the oven remain practically cold.

Figure 1a presents a picture of the microwave oven during the heating of the material protected with ceramic tube and lid as well as the ceramic fiber mattresses on the outside and Figure 1b shows the construction scheme of the experimental equipment. As shown in the scheme, the microwave irradiated material (3) is placed freely in a pressed state in the inner space of the ceramic tube (5) closed above with the ceramic lid (2). The thermal protection with ceramic fiber (7) of the space between the ceramic tube wall and the oven walls (1) has a special importance for minimizing the heat loss to the outside and implicitly, for obtaining a high energy efficiency. The radiation pyrometer (9), mounted above the oven on a vertical central axis, allows the visualization of the upper surface of the irradiated material through holes provided in the upper metal wall of the oven, the ceramic lid and the upper layer of ceramic fiber.

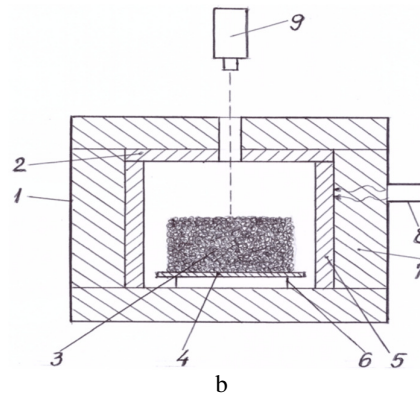
2.2 Materials

The materials used in the production of glass foam according to the recipe applied in this experiment were: colorless post-consumer packaging glass as raw material, glycerol as a liquid carbonic foaming agent, calcium carbonate as a solid foaming agent, sodium silicate as an enveloping agent for carbon particles, kaolin as an additive with the role of improving the mechanical strength of glass foam.

The glass waste was selected by color, broken, ground in a ball mill and sieved, the grain size being less than 100 µm. Its chemical composition [19] is shown in Table 1.



a



b

Figure 1. The experimental microwave equipment

a – 0.8 kW-microwave oven; b – constructive scheme of the equipment: 1 – microwave oven; 2 – ceramic lid; 3 – pressed mixture; 4 – metal plate; 5 – ceramic tube; 6 – metal support; 7 – ceramic fiber thermal insulation; 8 – waveguide; 9 – radiation pyrometer.

Table 1. Chemical composition of glass waste and kaolin

Chemical composition	Colorless glass waste wt. %	Kaolin powder wt. %
SiO ₂	71.7	57.63
Al ₂ O ₃	1.9	37.77
CaO	12.0	0.35
Fe ₂ O ₃	-	0.86
MgO	1.0	0.60
Na ₂ O	13.3	-
K ₂ O	-	1.80
Cr ₂ O ₃	0.05	-
P ₂ O ₅	-	0.31
All other oxides	0.05	0.68

CaCO₃ was purchased from the market with a grain size below 40 μm and was used in the experiment without other mechanical processing. The kaolin powder was purchased from the market with a fine grain size (below 10 μm). Its chemical composition [20] is shown in Table 1. Glycerol (C₃H₈O₃) and sodium silicate (Na₂SiO₃) in the liquid state were also purchased from the market and used in this physical state. The aqueous solution of Na₂SiO₃ had a concentration of 36.8%.

3. RESULTS AND DISCUSSION

3.1 Results

As mentioned above, the authors' team adopted for experiments a general theoretical recipe recommended by the German company Glamaco for the industrial manufacture of foam glass gravel containing soda-lime glass waste, two foaming agents (glycerol and calcium carbonate) simultaneously used, sodium silicate, water and kaolin powder. The weight proportion of each component of the recipe was varied by the authors. Four experimental variants were established and the heating technique adopted was nonconventional by using the microwave energy, unlike the conventional technique proposed by the German company even

by the constructive solution of the industrial oven designed for this purpose. The four variants are shown in Table 2.

The main functional parameters of the manufacturing process of glass foam are presented in Table 3. The sintering/foaming temperature, heating time, average heating and cooling rate, index of volume growth and specific energy consumption were targeted.

According to the data in Table 3, the sintering/foaming temperature had values in the range 834-841 °C and the heating time varied between 34-37.5 min. Due to the constructively adopted system of mixed microwave heating, predominantly by direct heating, the average heating rate had relatively high values (between 21.9-23.9 °C/min) leading to an excellent specific energy consumption within the limits of 0.78-0.85 kWh/kg. The volume growth by the expansion of raw material was low (between 30-60%) determining the fine porosity of the products according to Figure 2.

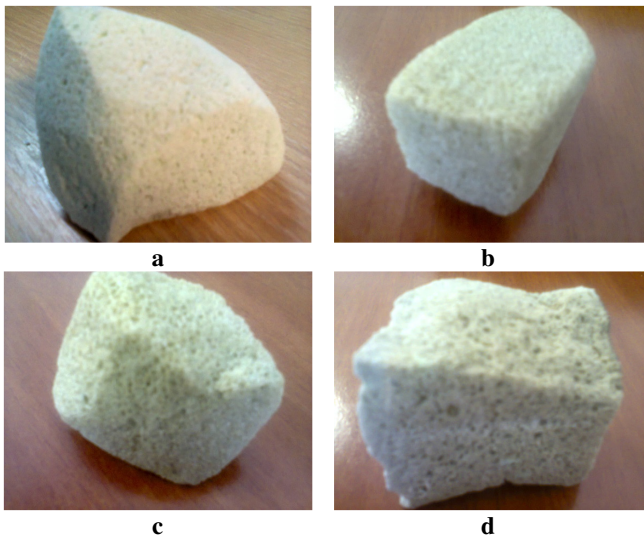
The investigation of the physical, thermal, mechanical and morphological characteristics of the four experimentally made glass foams was performed using common analysis techniques. The apparent density was measured by the gravimetric method [21] and the porosity was calculated by the method of comparing the true and apparent density [22]. The compressive strength was determined using a TA.XTplus Texture Analyzer (ASTM C552-17) and the thermal conductivity was measured by the heat-flow meter method (ASTM E1225-04). The water absorption was determined by the water immersion (for 24 hours) method (ASTM D570). The microstructural configuration of the samples was examined with an ASONA 100X Zoom Smartphone Digital Microscope. The main physical, thermal, mechanical and morphological features of glass foam samples are shown in Table 4.

Table 2. Experimental variants of the manufacturing recipe

Variant	Colorless glass waste wt. %	Glycerol wt. %	Sodium silicate wt. %	Calcium carbonate wt. %	Kaolin powder wt. %	Water addition wt. %
1	93.1	1.0	4.8	0.8	0.2	14.5
2	94.5	1.1	3.2	0.9	0.2	14.5
3	94.7	1.0	3.0	1.0	0.2	14.5
4	93.4	1.0	4.2	1.1	0.2	14.5

Table 3. The main functional parameters of the manufacturing process of glass foam

Variant	Dry raw material/ glass foam amount g	Sintering/ foaming temperature °C	Heating time min	Average rate, °C/min		Index of volume growth	Specific energy consumption kWh/kg
				Heating	Cooling		
1	470/456	834	34	23.9	6.8	1.30	0.78
2	470/455	836	35	23.3	6.7	1.40	0.80
3	470/458	839	36	22.8	6.9	1.50	0.82
4	470/457	841	37.5	21.9	6.7	1.60	0.85

**Figure 2.** Appearance of the glass foam samples a – sample 1 heated at 834 °C; b – sample 2 heated at 836 °C; c – sample 3 heated at 839 °C; d – sample 4 heated at 841 °C.**Table 4.** The main physical, thermal, mechanical and microstructural features of the glass foam sample

Feature	Variant 1	Variant 2	Variant 3	Variant 4
Apparent density (g/cm ³)	0.28	0.26	0.28	0.30
Porosity (%)	86.67	87.62	86.70	85.71
Thermal conductivity (W/m·K)	0.063	0.060	0.064	0.069
Compressive strength (MPa)	7.3	7.0	7.3	7.5
Water absorption (%)	4.3	4.5	4.9	4.7
Pore size (mm)	0.10-0.35	0.25-0.45	0.30-0.55	0.40-0.65

Analyzing the features value of the samples made in the four experimental variants in Table 4, it can be observe the influences of the foaming agent type and content on these features. Thus, the lowest value of the apparent density was obtained in the case of using the highest content of glycerol (1.1 wt.%), a CaCO₃ content below 1 wt.% (0.9%) and a ratio between the liquid foaming agent (glycerol) and the enveloping agent (sodium silicate) of 1/2.9 (variant 2). The compressive strength reached the highest value in the case of variant 4, which had the highest CaCO₃ content (1.1 wt.%), an average glycerol content (1 wt.%) and a glycerol/sodium silicate ratio of 1/3. Variant 4 led to obtaining a sample with pore size between 0.40-0.65 mm, i.e. the highest, although the material apparent density reached the highest value.

However, examining the samples characteristics as a whole, it was observed that all experimentally manufactured porous products had low apparent densities (0.26-0.30 g/cm³) and high compressive strengths (7.0-7.5 MPa), which was also the research objective.

The microstructural configuration of the glass foam samples shown in Figure 3 indicated a very good microstructural homogeneity with fine porosity, the pore size being between 0.10-0.35 mm (variant 1) and reaching the highest dimensional range of 0.40-0.65 mm (variant 4).

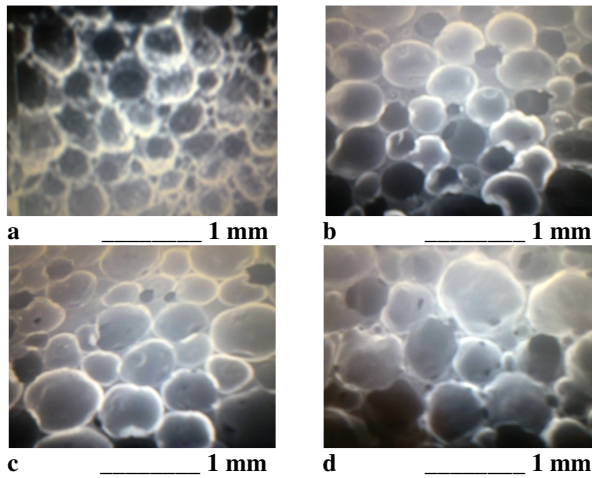


Figure 3. Microstructural configuration of glass foam samples a – sample 1; b – sample 2; c – sample 3; d – sample 4.

3.2 Discussion

In terms of ability of experimentally manufactured foam products to achieve quality thermal insulations, their thermal conductivity with low values between 0.060-0.069 W/m·K shows that this objective has been reached. The best thermal insulation properties were obtained in the case of sample 2 (thermal conductivity of 0.060 W/m·K combined with apparent density of 0.26 g/cm³ and porosity of 87.62%). These excellent thermal characteristics correspond to a high compressive strength (7.0 MPa), but the lowest of the four experimental variants. If it is considered that sample 4 has a relatively high apparent density for the purpose (0.30 g/cm³) and implicitly, the weakest thermal properties (thermal conductivity of 0.069 W/m·K and porosity of 85.71%) results that samples 1 and 3 could be the best products. Since the specific energy consumption of the manufacturing process of sample 1 has the most economical value (0.78 kWh/kg) it results that this sample can be considered as the optimal sample of the experiment.

Therefore, a glass foam with typical characteristics for a foam glass gravel usable as a thermal insulating material in difficult climatic conditions and with mechanical stress was made by sintering at 834 °C a powder mixture composed of colorless glass waste (93.1 wt.%), glycerol (1.0 wt.%), sodium silicate (4.8 wt.%), calcium carbonate (0.8 wt.%), kaolin (0.2 wt.%) and water addition (14.5 wt.%). The physical, thermal, mechanical and morphological characteristics of the product were: apparent density of 0.28 g/cm³, porosity of 86.67%, thermal conductivity of 0.063 W/m·K, compressive strength of 7.3 MPa, water absorption of 4.3% and pore size between 0.10-0.35 mm. The specific energy consumption of the manufacturing process was 0.78 kWh/kg.

In terms of quality, the product has characteristics almost similar to those of industrially manufactured products. In terms of energy, it is difficult to compare the results due to the lack of credible data in the literature on the industrial products.

4. CONCLUSION

The paper presents experimental results obtained in the manufacturing process of a high-strength glass foam with fine porosity usable as a thermal insulating material in difficult climatic conditions with large temperature variations and mechanical stress.

The originality of the work is the use of the nonconventional microwave heating technique, fast and economical, unlike the conventional techniques applied in the industrial manufacturing processes of this type of porous material (foam glass gravel).

Colorless soda-lime glass waste as raw material, glycerol and CaCO₃ as foaming agents (one liquid and the other solid), sodium silicate, kaolin and water, in varying proportions, were used constituting four experimental variants.

The optimal variant was made up of colorless glass waste (93.1 wt.%), glycerol (1.0 wt.%), sodium silicate (4.8 wt.%), calcium carbonate (0.8 wt.%), kaolin (0.2 wt.%) and water addition (14.5 wt.%) and was sintered at 834 °C.

The apparent density of 0.28 g/cm³, porosity of 86.67%, thermal conductivity of 0.063 W/m·K, compressive strength of 7.3 MPa, water absorption of 4.3% and pore size between 0.10-0.35 mm were the physical, thermal, mechanical and morphological characteristics of the optimal product.

The specific energy consumption of the manufacturing process had a very low value (0.78 kWh/kg).

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