EXPERIMENTAL MANUFACTURE OF THE FOAM GLASS GRAVEL FROM GLASS WASTE AND SILICON CARBIDE ON A 10 kW-MICROWAVE OVEN

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Abstract: The work presents the results of the experimental manufacturing process on a 10 kW-microwave oven of foam glass gravel (FGG) from glass waste (98 wt. %) and silicon carbide (2 wt. %), the sintering/foaming temperature being varied between 910-930 ºC. The paper originality is the significant increase (about 8 times) of the amount of glass-based raw material foamed by the unconventional heating compared to the previous experiments. The main features of the FGG experimental samples were: bulk density between 0.26-0.31 g·cm^{-3}, compressive strength between 8.5-9.7 MPa and thermal conductivity between 0.068-0.077 W/mK, suitable for their use as thermal insulation material in several applications in construction.

Keywords: foam glass gravel, glass waste, microwave heating, silicon carbide

1. INTRODUCTION

Materials recycling (plastic, metal, glass, paper etc.) has become a universal concern in the last decades. In particular, glass waste recycling aims at the manufacture of new products for the construction sector, rather than the reuse of this waste in glass industry for the production of new glass. By an adequate heat treatment at high temperature, the glass waste can be turned into a porous material, mainly a good thermal insulator having also other excellent characteristics that recommend it as a possible replacement for existing building materials.

A new type of thermal insulation material made of glass waste with drainage properties having typical lump size of 10-75 mm [1] is the foam glass gravel (FGG). The industrial manufacture of FGG began in the last decades of the 20th century. According to [2], currently over 600,000 m^3 are produced annually in Europe, the main manufacturers being from Germany, Austria, Switzerland and Scandinavian countries. Unlike other types of polymeric or fiberglass insulating materials, FGG has important advantages due to its characteristics: heat insulator, fire resistance, chemical resistance, light weight, pest-proof, frost resistance, high compressive strength, electrical insulator, etc. The application domains of this material are: insulation floor slab, roof gardens, backfill around pipelines and swimming pools, road and railway construction, bridge abutments, sports fields etc. In Scandinavia, the FGG production is focalized to the use as sub-base for road construction. The absence of capillarity, the structural stability and drainage prevent the road surface degradation by the freeze-thaw succession.

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The raw material used in the manufacture of FGG is recycled glass waste (post-consumer packaging bottle or its combination with flat glass waste) ground to a grain size between 75-150 μm. The proportion of glass waste in the starting powder mixture is usually 98 wt. %, the remaining 2 wt. % being represented by a foaming agent. According to [2, 3], the industrially used foaming agent is either solid (silicon carbide-SiC, limestone-$CaCO_3$ or gypsum-$CaSO_4$) or liquid (glycerol-$C_3H_8O_3$ associated with sodium silicate-Na_2SiO_3). The manufacturers Geocell Schaumglas (Germany) [2-4] and Misapor Switzerland (Switzerland) [3, 5] use solid foaming agents, while Glapor Werk Mitterteich (Germany) uses glycerol [3, 6].

The principle of the FGG manufacturing process by foaming glass waste is similar to that of producing any glass foam. The foaming agent releases a gaseous compound by a decomposition or reduction reaction at high temperature up to 900-930 ºC forming gas bubbles in a viscous glass melt. The adequate viscosity of the molten glass facilitates the capture and trapping the bubbles, which then by cooling turn into a pores network characteristic of foamed products [7]. The industrial process of FGG manufacturing occurs in conveyor belt-tunnel ovens powered by burning a fuel or by electricity consumption [2, 8]. In the case of FGG, the cooling of the foamed material is forced by blowing cold air causing its breaking into lumps [2]. The size of the lumps is variable being accepted between 10-75 mm.

According to the literature [2], the bulk density of FGG is between 0.13-0.21 $g \cdot cm^{-3}$, the values being higher in the case of products made for road base. The thermal conductivity has values between 0.08-0.09 W/m·K reaching even 0.13 W/m·K in the manufacture of FGG for road construction [9] and the compressive strength is in the range 3-9 MPa. According to the technical books of Geocell, Misapor and Glapor companies [3-6], the maximum values of the compressive strength reach only 5.7-6.0 MPa.

As mentioned above, the heating technique used in the industrial production of FGG is conventional. The Romanian company Daily Sourcing & Research has performed in recent years several small-scale experiments for the production of FGGs using the unconventional microwave heating technique. Various manufacturing recipes were tested using as a raw material either a mixture of colored container glass waste [10-12] or colorless flat glass [13] and as a foaming agent calcium carbonate together with sodium borate (borax) and sodium silicate [10], glycerol together with sodium silicate and water [11, 13] or SiC and water [12].

The experiment presented in [10] using calcium carbonate (1.5 wt. %), borax (3 wt. %) and sodium silicate (8 wt. %) had in its optimal variant the sintering temperature of 855 ºC, the density of a compact lump of 0.62 $g \cdot cm^{-3}$, porosity of 71.8 %, thermal conductivity of 0.087 W/m·K , compressive strength of 7.4 MPa and pore size between 1-1.6 mm. The specific energy consumption was 1.07 kWh/kg.

According to [13], the optimal FGG manufactured of flat glass waste, glycerol (1.3 wt. %), sodium silicate (5.9 wt. %) and water had the sintering temperature of 818 ºC, bulk density of 0.24 $g \cdot cm^{-3}$, porosity of 86 %, thermal conductivity of 0.063 W/m·K, compressive strength of 5.3 MPa and pore size between 0.5-0.9 mm. The specific energy consumption was 0.86 kWh/kg.

A product [11] made of a mixture of colored container glass waste, glycerol (1 wt. %), sodium silicate (8 wt. %) and water was sintered at 823 ºC having bulk density of 0.24 $g \cdot cm^{-3}$, 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. The specific energy consumption was 0.88 kWh/kg.

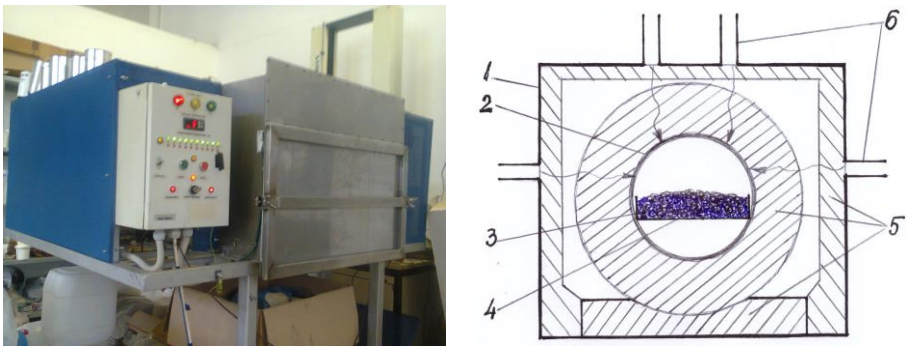
According to the paper [12], the optimal FGG made of a mixture of colored container glass waste, SiC (1.7 wt. %) and water had the sintering temperature of 922 ºC, bulk density of 0.27 $g \cdot cm^{-3}$, porosity of 80.9 %, thermal conductivity of 0.075 W/m·K, compressive strength of 7.5 MPa and pore size between 0.5-1 mm. The specific energy consumption was 1.00 kWh/kg.

The four experiments presented in the literature [10-13] were performed on a 0.8 kW-microwave oven frequently used by the Daily Sourcing & Research Company. In the current paper, the authors tested the sintering/foaming process of glass waste on a 10 kW-microwave oven existing in the company's experimental base and used for other technological purposes. The much higher capacity of the oven allowed the loading of the raw material on a large surface compared to previous experiments and the creation of working conditions much closer to the characteristics of an industrial microwave oven.

2. METHODS AND MATERIALS

2.1. Methods

The method adopted by the authors took into account the significant increase of the surface occupied by the raw material powder mixture, the increase of the total power of the microwave generators and their number as well as the change of their position on the oven walls. The experimental microwave equipment (overall image and constructive scheme of the equipment) is presented in Figure 1. The oven (b1) with an internal volume of about 0.4 m^3 is equipped with 10 magnetrons (b6), three on each of the side walls and four in the vault. Therefore, the powder mixture (b3) was loaded and pressed into a rectangular mold (b4) with the sides of the base of 250 and 360 mm and the height of the walls of 40 mm made of 1.5 mm-stainless steel sheet. A SiC ceramic crucible (b2) with an outer diameter of 300 mm, a height of 450 mm and a wall thickness of 10 mm was placed in a horizontal position inside the oven. Through the crucible opening, the mold with the material subjected to the heat treatment was inserted with the smaller side in front being supported in a horizontal position on the inner wall of the crucible. The role of the ceramic crucible was to protect the glass waste from the full direct impact with the microwave flow, which previously proved [14] to be unsuitable for the commercial glass (soda-lime glass) foaming. In this way, the direct microwave heating was partial, while the microwave energy absorbed in the crucible wall was converted into heat and ensured an indirect heating of the material by thermal radiation. The relationship between the direct and indirect microwave heating was not determined during this experiment. As in all previous small-scale tests of glass foaming, the ceramic crucible was protected on the outside with several thick ceramic fiber mattresses (b5) to avoid the heat loss outside the system. The crucible opening was also covered with ceramic fiber mattresses. The temperature control was performed with a thermocouple whose head was fixed on the side wall of the mold.



a b

Fig. 1. Experimental microwave equipment:
a – overall image of the 10 kW-microwave oven; b – constructive scheme of the equipment: 1 – microwave oven; 2 – SiC crucible; 3 – pressed powder mixture; 4 – metal rectangular mold; 5 – ceramic fiber-thermal insulation; 6 – waveguide.

The basic principle of the process of glass foaming with silicon carbide as a foaming agent is the release of carbon dioxide (CO_2) through the oxidation reaction of SiC (1) in the oxidizing atmosphere of the oven having the most favorable conditions in thermodynamic terms at temperatures around 900 ºC [7].

$$SiC + 2O_2 = SiO_2 + CO_2 \quad (1)$$

CO_2 (gas) is captured in the viscous melt of the glass and directly contributes to the material foaming, while SiO_2 (solid) is incorporated into the molten mass of the glass [7].

2.2. Materials

The manufacturing recipe included a mixture of colorless, green and amber post-consumer packaging bottle (in the 50/20/30 weight ratio) (98 wt. %), SiC as a foaming agent (2 wt. %) and water addition as a binder (13 wt. %).

The post-consumer packaging bottle was selected by color, broken, thermally washed at 250 ºC (to remove the organic contaminants), ground and sieved at the grain size below 100 μm. These operations were performed in the company Bilmetal Industries SRL Popesti-Leordeni. The chemical composition of the glass waste types is shown in Table 1 based on the X-ray fluorescent spectrometry analysis carried out in the Metallurgical Research Institute Bucharest.

Table 1. Chemical composition of the glass waste types

Chemical composition (wt, %)	Glass type		
	Colorless	Green	Amber
SiO_2	71.5	71.2	71.4
Al_2O_3	1.9	1.8	1.9
CaO	12.0	10.2	10.3
Fe_2O_3	0.1	0.4	0.3
MgO	1.0	2.2	2.3
Na_2O	13.3	13.0	13.2
K_2O	0.1	0.5	0.6
Cr_2O_3	0.1	0.2	0.1
SO_3	0.2	0.3	0.3

The SiC purchased from the market was used at the initial grain size below 40 μm.

2.3. Characterization of the FGG samples

The FGG lumps were subjected to the usual methods of determining the physical, thermal, mechanical and morphological characteristics. The bulk density was measured by weighing a batch of lumps fully loaded into a container of known volume and dividing the total mass of the batch by the inner volume of the container [15]. The porosity was calculated by the method of comparing the true and bulk density [16]. The thermal conductivity was measured by the heat-flow meter method (ASTM E1225-04) and the compressive strength was determined using a TA.XTplus Texture Analyzer (ASTM C552-17). The water absorption for 24 hours was determined by the water immersion method (ASTM D570) and the microstructural configuration of the FGG samples was investigated with an ASONA 100X Zoom Smartphone Digital Microscope.

3. RESULTS AND DISCUSSION

3.1. Results

Using a similar composition of starting materials (98 % glass waste, 2 % SiC and 13 % water addition) the experiments were performed in three variants of functional parameters. The amount of dry raw material was kept constant at 4.00 kg, the wet amount of material reaching 4.52 kg. The FGG amount determined after the release of the foamed material from the mold was between 3.88-3.90 kg indicating a material loss of 2.5-3 wt. %. According to Table 2, the sintering/foaming temperature had values between 910-930 ºC with heating times between 59-67 min.

Table 2. The main functional parameters of the manufacturing process of FGG

Parameter	Variant 1	Variant 2	Variant 3
Dry raw material/foam glass gravel amount (kg)	4.00/3.88	4.00/3.91	4.00/3.90
Sintering/foaming temperature (ºC)	910	920	930
Heating time (min)	59	62	67
Average rate (ºC/min) · heating · cooling	 15.1 7.5	 14.2 7.4	 13.6 7.6
Index of volume growth	1.50	1.60	1.75
Specific energy consumption (kWh/kg)	1.98	2.06	2.23

As a result of the heating process of raw material in the three variants of the heat treatment, a porous high-strength material was obtained, which after cooling was broken into lumps suitable for use as FGG (less than 70 mm). The appearance of the products according to Figure 2 is similar to those obtained by conventional industrial manufacturing techniques.

a b c

Fig. 2. Appearance images of FGG lumps:
a – sample 1 heated at 910 ºC; b – sample 2 heated at 920 ºC; c – sample 3 heated at 930 ºC.

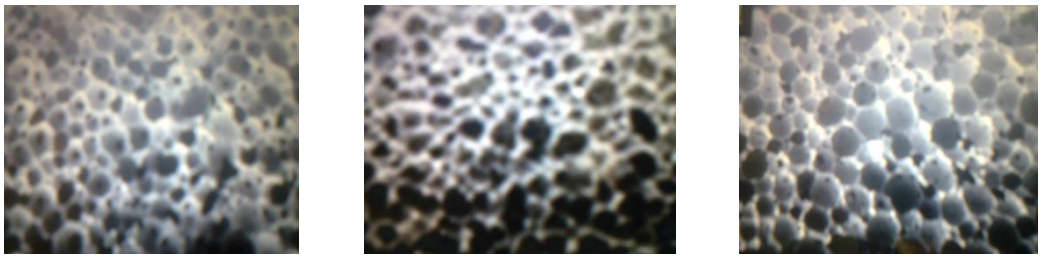
According to Table 2, the heating rate varied between 13.6-15.1 ºC/min being much lower compared to the similar experiments performed on the 0.8 kW-microwave oven (16.6-20.9 ºC/min). Given that both the manufacturing process of FGG on the 10 kW-oven and that performed on the 0.8 kW-oven did not have the ability to adjust the power of the magnetrons during the process, both microwave equipment operating at maximum heating capacity, results that the energy efficiency of the equipment with lower power is higher due to the nature of the material of the ceramic crucible and its wall thickness. These constructive characteristics influenced the proportion of the direct microwave heating to the detriment of the indirect heating, increasing the heating rate and reducing the value of the specific energy consumption. Another parameter that influences the energy efficiency of the heating process is the level of heat loss outside the system and which is more difficult to control in the case of ovens with larger powers and dimensions.

The main physical, thermal, mechanical and morphological characteristics of FGG samples are presented in Table 3.

Table 3. Main physical, thermal, mechanical and morphological characteristics of FGG

Variant	Bulk density ($g \cdot cm^{-3}$)	Porosity (%)	Thermal conductivity (W/m·K)	Compressive strength (MPa)	Water absorption (vol. %)	Pore size (μm)
1	0.31	85.4	0.077	9.7	3.8	50-250
2	0.29	86.3	0.074	9.4	4.0	100-400
3	0.26	87.7	0.068	8.5	3.4	150-450

Analyzing the data in Table 3, the high values of the compressive strength (8.5-9.7 MPa) of the three obtained materials are noticed. The physical and thermal characteristics of the FGG samples that compete to determine the thermal insulation character of the material (bulk density, porosity and thermal conductivity) have adequate values that define this property. The bulk density has values between 0.26-0.31 $g \cdot cm^{-3}$, the porosity is between 85.4-87.7 % and the thermal conductivity is within the limits of 0.068-0.077 W/m·K. The pore size is very low (below 450 μm) in all three experimental variants. The analysis of the microstructural configuration of the three FGG samples was carried out based on the pictures in Figure 3.



a b c

Fig. 3. Microstructural configuration of the FGG samples:
a – sample 1; b – sample 2; c – sample 3.

The uniformity of the pore distribution contributes to the microstructural homogeneity of the samples. The pore size is within tight limits: 50-250 μm (sample 1), 100-400 μm (sample 2) and 150-450 μm (sample 3).

3.2. Discussion

The experiment described in this paper was the first significant increase (about 8 times) of the amount of glass-based raw material subjected to the process of manufacturing a glass foam by the unconventional technique of using the microwave energy and its conversion into heat. The purpose of the experiment was primarily to test the obtaining a foaming material similar in terms of quality to those industrially manufactured by current conventional methods.

Considering information from the literature on the characteristics of some industrially manufactured FGGs [3] it is found that in general, the physical, thermal, mechanical and morphological characteristics are similar. The compressive strength of the experimentally produced materials was higher (8.5-9.7 MPa) than that of the currently made materials (4.9-6.0 MPa), this difference being compensated by the slightly higher values of bulk density (0.26-0.31 $g \cdot cm^{-3}$) compared to the usual bulk density values (0.13-0.21 $g \cdot cm^{-3}$). Also, the values of thermal conductivity of experimental products are lower and consequently more advantageous for a thermal insulator (between 0.068-0.077 W/m·K) compared to industrially manufactured materials (0.08-0.09 W/m·K). The reference data presented as terms of comparison belong to the companies Geocell (Austria), Misapor (Switzerland) and Glapor (Germania), the world's leading manufacturers of FGGs.

It should be taken into account that, as mentioned above, the main purpose of the experiment described in this paper was investigating the influence of the significant surface extension of raw material loaded into the oven and its microwave irradiation from top to bottom on the quality of the foaming process. All the previous tests carried out by authors under the conditions of unconventional microwave heating performed in a low capacity oven (0.8 kW), in which the dimensions of the material mixture were much smaller. The results of the current experiment were excellent in terms of quality.

The use in this stage of an improvised equipment for experimental purposes influenced the efficiency of the heat transfer in the oven and the specific energy consumption values were obviously higher (1.98-2.23 kWh/kg) compared to the values usually achieved in the tests performed on the 0.8 kW-oven (0.8-1 kWh/kg). Worldwide, the specific energy consumption of the conventional industrial production of FGG is approximately similar to that obtained by microwave heating in the experiments on the 0.8 kW-oven, although the information in literature regarding this parameter is very poor.

The design of a microwave oven suitable for achieving an optimal conversion of microwave energy into heat for heating and foaming the glass-based raw material is the aim of authors for the next stages of finalizing the manufacturing technology.

4. CONCLUSIONS

The results of the experimental manufacturing process of foam glass gravel (FGG) from glass waste and silicon carbide are presented in this work. The paper originality was the significant increase (about 8 times) of the amount of glass-based raw material foamed by the unconventional microwave heating. The used experimental microwave equipment was an existing 10 kW-microwave oven constructively adapted by inserting a SiC-ceramic crucible in a horizontal position including a mold with the pressed powder mixture. The manufacturing recipe included 98 wt. % glass waste, 2 wt. % SiC as a foaming agent and 13 wt. % water addition as a binder. Three experimental variants were used, the sintering/foaming temperature varying between 910-930 ºC. The main characteristics of the FGG experimental samples were: bulk density between 0.26-0.31 $g \cdot cm^{-3}$, porosity between 85.4-87.7 %, compressive strength in the range 8.5-9.7 MPa and thermal conductivity between 0.068-0.077 W/m·K. The pore size was very low (below 450 μm). Generally, these characteristics are similar to those of the industrially made FGGs.

The experimental microwave equipment used in the experiments was not specially designed for this aim, it has other technological utilization. The improvisations necessary to be usable in this experimental process have influenced the energy efficiency of the adapted equipment leading to quite high values of the specific energy consumption (1.98-2.23 kWh/kg), much higher of the level reached in the experiments previously carried out on the 0.8 kW-microwave oven (0.8-1 kWh/kg).

REFERENCES

[1] Zegowitz A., Cellular glass aggregate serving as thermal insulation and a drainage layer, Buildings, vol. 11, 2010, p. 1-8.
[2] Hibbert, H., Understanding the production and use of Foam Glass Gravel across Europe and opportunities in the UK, Final Report, 2016.
[3] Cosmulescu, F., Paunescu, L., Dragoescu, M.F., Axinte, S.M., Comparative analysis of the foam glass gravel types experimentally produced by microwave irradiation, Journal of Engineering Studies and Research, vol. 26, no. 3, 2020, p. 58-68.
[4] https://www.mikewye.co.uk/wp-content/uploads/2014/09/GEOCELL-Brochure-UK-Sept-16.pdf (15.02.2021).
[5] https://www.misapor.ch/en/company (11.05.2021).
[6] http://www.glapor.de/en/produkte/cellular-glass-gravel (06.04.2021).
[7] Scarinci, G. Brusatin, G., Bernardo, E., Cellular ceramics: structure, manufacturing, Properties and Applications, Wiley-VCH GmbH & Co KGaA, Weinheim, Germany, 2005, p. 158-176.
[8] Hurley, J., Glass-Research and Development, Final report, A UK market survey for foam glass, Banbury-Oxon, Great Britain, The Waste and Resources Action Programme Publication, 2003.
[9] https://www.foamit.fi/en/foamit-products/ (22.06.2021).
[10] Paunescu, L., Dragoescu, M.F., Axinte, S.M., Foam glass gravel from recycled glass waste produced with the microwave energy, Nonconventional Technologies Review, vol. 24, no. 2, 2020, p. 22-28.
[11] Paunescu, L., Dragoescu, M.F., Axinte, S.M., High mechanical strength porous material used as a foam glass gravel experimentally manufactured from glass waste by an unconventional technique, Constructii, vol. 21, no. 1, 2020, p. 54-62.
[12] Paunescu, L., Dragoescu, M.F., Axinte, S.M., Foam glass gravel made of recycled glass waste and silicon carbide by microwave heating, Journal of Engineering Studies and Research, vol. 26, no. 3, 2020, p. 173-180.
[13] Dragoescu, M.F., Paunescu, L., Axinte, S.M., Nonconventional technique of sintering/foaming the glass waste using a liquid carbonic foaming agent, Nonconventional Technologies Review, vol. 24, no. 3, 2020, p. 4-12.
[14] Paunescu, L., Axinte, S.M., Grigoras, B.T., Dragoescu, M.F., Fiti, A., Testing the use of microwave energy to produce foam glass, European Journal of Engineering and Technology, vol. 5, no. 4, 2017, p. 8-17.
[15] Scorgins, A., Bulk density of industrial minerals: Reporting in accordance with the 2007 SME Guide, 2015. https://www.csaglobal.com/wp-content/uploads/2015/07/Bulk-density-of-industrial-minerals-Reporting-in-accordance-with-the-2007-SME-Guide.pdf (02.05.2021).
[16] Anovitz, L.M., Cole, D.R., Characterization and analysis of porosity and pore structures, Reviews in Mineralogy and Geochemistry, vol. 80, 2005, p. 61-164.