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COMBINED EFFECT OF GLYCEROL AND CALCIUM CARBONATE AS EXPANDING AGENTS FOR MANUFACTURING FOAM GLASS GRAVEL ON A PILOT MICROWAVE OVEN

BY

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Abstract. Foam glass gravel, a building material with excellent load-bearing and insulation properties, has become very attractive in recent decades due to its low production costs and its environmental friendliness. Used in construction, especially in harsher climates, this product uses recycled residual glass as a raw material. The authors of current paper have recently approached this field using electromagnetic waves as energy carriers converting microwave power into heat. Raw material was recycled post-consumer glass and the combination of a liquid agent (glycerol) and a solid agent (calcium carbonate) was used as the glass powder foaming agents. The experiment took place in a modified 10 kW-oven, much larger than the 800 W-microwave oven used previously. Glass foam gravel had excellent thermal insulation properties and very good compressive strength being at the qualitative level of industrial products manufactured by conventional methods.

Keywords: foam glass gravel, glass waste, glycerol, calcium carbonate, compressive strength.

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1. Introduction

The last 3-4 decades have highlighted major energy and ecological problems facing the planet involving the global hydrocarbon crisis and the danger of overheating the earth's atmosphere due to the destruction of the protective ozone layer under the influence of large emissions of greenhouse gases (mainly carbon dioxide).

These extreme problems have led to the initiation on an international level of programs to reduce and even block some technologies characterized by very high CO_2 emissions and high consumption of fossil fuels (de Bruyn *et al.*, 2020).

A solution of great interest in the world is the extensive recycling of material waste (metals, glass, plastics, paper and cardboard, textiles, etc.) available on a large scale and whose annual generation rate is worrying. In principle, the recycling of a waste by re-introducing it into the own production circuit as a cheap raw material contributes to the significant reduction of production costs. Thus, Scarinci *et al.* (2000) mention that new glass requires 4500 kJ/kg, while by using recycled glass waste the new glass requires only 500 $kJ \cdot kg^{-1}$. Also, the orientation of some recycled waste towards other types of materials with newly created value (mostly in construction) has a special importance for the environment and the rational use of energy resources.

In the field of glass waste recycling, the literature mentions numerous techniques for obtaining porous products by adding together with finely ground glass waste some mineral additives that at temperatures of 800 - 1100ºC release a gas or gaseous compound in the softened glass mass generating pores (Scarinci *et al.*, 2005). Excellent physical, thermal, and mechanical properties (light weight, low heat conductivity, resistance to fire, water, frost, acids, bacteria, insects, rodents, and acceptable or high compressive strength) are incorporated in these products. Their properties are suitable both for use as thermal insulation materials in buildings, as well as insulation in the perimeter of buildings, drainage, road and railway construction, sports fields, insulation of underground pipes for energy fluids and storage tanks, bridge abutments, etc.

The most well-known thermal insulation materials currently used in construction are expanded or extruded polystyrene and phenolic foam (used as boards), glass mineral wool (used as rolls), hempcrete (as blocks), etc. (Insulation Materials, 2017). But the energy consumption for their manufacture is much higher (about ten times) compared to that required for the production of glass foam.

The compressive strength starts from minimum values of 0.7 - 2 MPa and heat conductivity can have values between 0.052 - 0.12 $W \cdot m^{-1} \cdot K^{-1}$, the average value being around 0.08 $W \cdot m^{-1} \cdot K^{-1}$ (Hibbert, 2016; Zegowitz, 2010).

Large amounts of foam glass gravel are annually produced in Europe, especially in countries with a harsher climate in the northern areas (Nordic countries, Austria, Germany, and Switzerland) and the main manufacturers are Geocell (Austria), Misapor (Switzerland), Glapor (Germany), Technopor (Austria) (Cosmulescu *et al.*, 2020).

The material mixture mainly composed of finely ground recycled residual glass to which are added various mineral additives as foaming agents that vary from manufacturer to manufacturer (limestone, gypsum, silicon carbide, glycerol together with sodium silicate) is loaded onto the belt of a furnace with a conveyor belt and heated by conventional techniques. After reaching the foaming temperature, the mixture is freely cooled and then forced cooled with blowing air to create internal stresses in the material. Thus, at the cold end of the strip, the product falls in the form of lumps, which easily detach from the mass of sintered and cooled material.

According to the work of Cosmulescu *et al*., (2020), several variants for the production of foam glass gravel were tested in an 800 W-microwave oven adapted for high temperature operation. Heating by microwave irradiation of the mixture has replaced the traditional method of conventional heating. In terms of quality, the products had characteristics similar to those of industrially manufactured foam glass gravel (apparent density of 0.24 - 0.35 $g \cdot cm^{-3}$, heat conductivity within the limits of 0.063 - 0.075 $W \cdot m^{-1} \cdot K^{-1}$, and compressive strength between 5.3 - 7.5 MPa) and, in addition, the energy efficiency of the process was higher (0.86 - 1.00 $kWh \cdot kg^{-1}$).

The current paper aimed at the development of research in the production of foam glass gravel in the microwave field by enlarging the surface of the material subjected to microwave heating and also changing the way of producing the contact of electromagnetic waves with the extended horizontal surface of the material mixture based on glass waste. The manufacturing recipe used recycled container glass waste as raw material and a mixture of glycerol (liquid) and calcium carbonate (solid) as foaming agents.

2. Methods and Materials

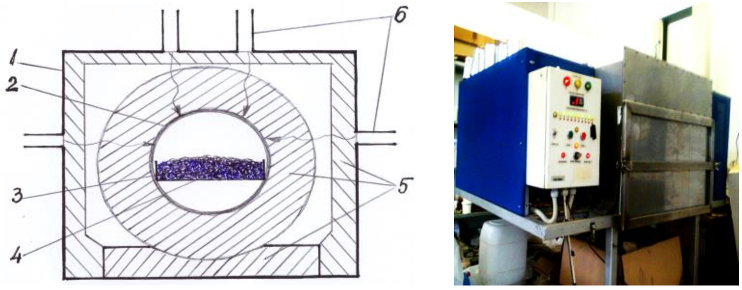
In this experiment, the combination of two foaming agents was adopted, one organic liquid (glycerol) and the other solid (calcium carbonate). Glycerol ($C_3H_8O_3$) was chosen due to the facile dispersion of the liquid agent among the fine particles of the residual glass and the creation of an intimate contact between the two material types. According to the literature (Karandashova *et al*., 2017), glycerol decomposes in the oxidizing atmosphere of the furnace into various reaction products from CO_2 to pure carbon and hydroxyl compounds. This chemical process is initiated at low temperature (below 200ºC) and takes place up to about 850ºC (Dou *et al*., 2008). Because glycerol as a carbonic agent has a strong affinity for oxygen, conditions can be created for the premature burning of carbon, affecting the foaming process by loss of CO_2 or CO outside the furnace. A known variant for enveloping the particles is the addition of sodium silicate aqueous solution, which slows down the decomposition process of $C_3H_8O_3$ and intensifies the glass sintering. The use of calcium carbonate ($CaCO_3$) as an expanding agent is well known. Its decomposition (eq. 1) initiated at over 750ºC and continued until about 900ºC (Karunadasa *et al*., 2019; Paunescu *et al*., 2022) provides the CO_2 necessary for the formation of bubbles in the softened glass mass.

$$CaCO_3 = CaO + CO_2 \qquad (1)$$

The combined use of glycerol and $CaCO_3$ as foaming agents for residual glass in the cellular glass manufacturing process was previously tested by some of the authors of the current paper in an adapted 800 W-microwave oven (Paunescu *et al.*, 2021). The manufacturing recipe included colorless container glass waste (93.1 - 94.7 wt. %) as raw material, glycerol (1.0 - 1.1 wt. %) as liquid foaming agent, calcium silicate (3.0 - 4.8 wt. %) as a liquid enveloping material to delay the decomposition of glycerol, calcium carbonate (0.8 - 1.1 wt. %) as a solid foaming agent, very low addition of kaolin powder (0.2 wt. %) as a thermal protection agent for ceramic materials, and water addition as a binder. The sintering/expanding process temperature was within the limits of 834 - 841ºC.

The experiment results showed that the apparent density was between 0.26 - 0.30 $g·cm^{-3}$, heat conductivity had values between 0.06 - 0.069 $W·m^{-1}·K^{-1}$, compressive strength was within the limits of 7.0 - 7.5 MPa, and water absorption in the range of 4.3 - 4.9 vol. %. The specific energy consumption of the experimental process was very low (0.78 - 0.85 $kWh·kg^{-1}$). Cellular glass lumps had excellent characteristics specific to foam glass gravels produced on an industrial scale by conventional heating techniques.

The experimental microwave equipment with the power of 10 kW was adapted to act by the electromagnetic waves emission through the waveguide of ten magnetrons (three each on the two side walls and four in the flat vault of the oven) on a silicon carbide ceramic crucible (high microwave susceptible material) with the outer diameter of 300 mm, the length of 450 mm and the wall thickness of 10 mm. A 250x360x50 mm refractory steel mold containing the pressed material mixture subjected to sintering/expanding was inserted horizontally into the inner space of the ceramic piece. Electromagnetic waves with a frequency of 2.45 GHz acted on the SiC ceramic piece being absorbed in its wall, heating it quickly and effectively. Part of the microwave field penetrated the 10 mm-thick wall and came into direct contact with the surface of the mixture deposited in the mold. Thus, a mixed heating partially dielectric and partially by conventional thermal radiation was achieved. The constructive and functional scheme of the experimental equipment is shown in Fig. 1.



a b

Fig. 1 – Experimental microwave equipment
a – constructive and functional scheme: 1 – oven; 2 – SiC pressed crucible;
3 – metal mold; 4 – pressed mixture; 5 – thermal protection;
6 – waveguide; b – 10 kW-microwave oven.

Direct microwave heating has peculiarities that make it completely different from conventional heating. Thus, the initiation of the heating process takes place inside of the material subjected to irradiation and the heat propagation is carried out volumetrically in the entire mass of the material from the inside to the outside, i.e. in the opposite direction compared to conventional heating (Kitchen *et al.*, 2014). Therefore, the role of thermal protection of the material or the container in which it is stored is very important. On the other hand, the selectivity of microwave heating is a characteristic of it (Jones *et al.*, 2002), which means that only the targeted material is strongly heated, the other massive components of the furnace remaining relatively cold. Controlling the temperature of the heating process was facilitated by a thermocouple whose hot welding was fixed on the side wall of the refractory steel mold.

As mentioned above, the basic raw material used for the manufacture of foam glass gravel is recycled residual glass. In this experiment, colorless and green post-consumer container glass was recycled in a 70/30 weight ratio. The chemical composition of the two types of glass is shown in Table 1.

Table 1
Chemical composition of colourless and green glass

Glass type	SiO_2	Al_2O_3	CaO	MgO	Na_2O	K_2O	Cr_2O_3	Other oxides
Colourless	71.7	1.9	12.0	1.0	13.3	-	0.05	0.05
Green	71.8	1.9	11.8	1.2	13.1	0.1	0.09	0.01

Processing the residual glass was carried out in Bilmetal Industries SRL Popesti-Leordeni, Ilfov and consisted of selection by colour, washing, breaking, grinding in a ball mill, and sieving. The grain size of waste was chosen below 80 μm.

$CaCO_3$ was purchased from the market at a grain size below 36 μm and this size limit was used during the experiment.

Glycerol in liquid state (30% concentration) and sodium silicate (Na_2SiO_3) also known as "water glass" as an aqueous solution (36.8%) are commercially available being procured for the experiment.

Foam glass gravel lumps resulting from the experiment were analyzed to determine the physical, thermal, mechanical, and microstructural characteristics. The bulk density was measured by the usual method of weighing a batch of lumps completely loaded into a container with a known volume and then reporting the mass to the internal volume of the container (Scorgins, 2015). The calculation of the specimen porosity involved determining the density of the ceramic material after its melting and cooling, i.e. true density of the material without pores and its comparison with the bulk density (Anovitz and Cole, 2005). Heat conductivity was measured by the heat-flow meter method (SR EN 1946-3:2004) and compressive strength was determined using 10 kN-hydraulic axial press machine (EN 826:2013). Specimen immersing under water for 24 hours (BS 1881-122:2011) allowed identifying the water absorption volumetric proportion. Microstructural appearance of foam glass gravel specimens was observed with ASONA 100X Zoom Smartphone Digital Microscope.

Three experimental variants were adopted for making foam glass gravel by microwave sintering. The manufacturing recipe corresponding to each variant is presented in Table 2.

Table 2

Composition of the experimental variants

Variant	Glass waste (wt. %)	Glycerol (wt. %)	Calcium carbonate (wt. %)	Sodium silicate solution (wt. %)	Water addition (wt. %)
1	93.2	1.2	0.9	4.7	15.0
2	93.7	1.1	1.0	4.2	14.0
3	94.3	0.9	1.1	3.7	13.0

According to the data in Table 2, in the experimental variants 1-3, the glycerol/calcium carbonate weight ratio decreased from 1.33 to 0.82 and the sodium silicate/glycerol ratio decreased from 5.22 to 3.36. The weight proportion of residual glass constituted from colourless and green glass was extremely high (between 93.2-94.3%) indicating an excellent waste recycling degree. Unlike the low amount of wet load processed in the 800 W-microwave oven (500 - 530 g) previously tested by the authors (Paunescu *et al.*, 2021), the 10 kW-oven described above allowed a significant increase in the amount of wet raw material mixture to about 4.40-4.55 kg by significant expansion of the surface of batch subjected to microwave heat treatment.

3. Results and Discussion

The manufacturing process of foam glass gravel in the adapted 10 kW-microwave oven had the following main functional parameters indicated in Table 3.

Table 3
Main functional parameters of the process

Variant	Wet raw material/ foam glass gravel amount (kg)	Sintering temperature (ºC)	Heating time (min)	Average heating/ cooling rate (ºC·min^{-1})	Specific energy consumption (kWh·kg^{-1})
1	4.48/4.03	835	47	17.3/7.9	1.71
2	4.52/4.06	838	49	16.7/7.8	1.77
3	4.55/4.10	840	55	14.9/8.0	1.96

According to the data in Table 3, wet raw material amounts between 4.48 - 4.55 kg were heated by microwave irradiation in the adapted oven (partially improvised). The material temperature determined by measuring with the thermocouple fixed on the surface of metal mold wall varied in low limits (from 835 to 840ºC). The average heating rates had sufficiently high values (in the range of 14.9 - 17.3ºC/min), the sintering process being completed in 47 - 55 min. The use of glycerol in higher weight proportion than that of calcium carbonate facilitated the foaming process of residual glass due to the more intimate contact made between the liquid agent and the glass particles allowing the reduction of the process duration and the final temperature. Cooling the sintered material was achieved by removing the mold outside the oven in the ambient air, the cooling rate being calculated at 7.8 - 8.0ºC/min to facilitate the development of internal tensions into the hot material mass. The specific energy consumption values were approximately double compared to the most efficient consumptions achieved in optimal heating conditions in the 800 W-microwave oven, but this is due to the less favorable energy conditions offered by the 10 kW-oven.

Foam glass gravel lump specimens corresponding to the three experimental variants tested in the 10 kW-microwave oven are shown in Fig. 2.

a b c

Fig. 2 – Aspect of foam glass gravel lumps
a – variant 1; b – variant 2; c – variant 3.

Lumps with dimensions of less than 60 mm were mechanically detached from the sintered and cooled material mass. Using the investigation methods to determine their physical, thermal, mechanical, and microstructural characteristics specified above, the results presented in Table 4 were obtained.

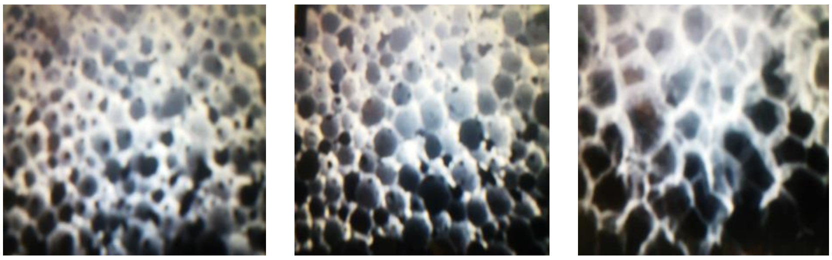
Table 4

Physical, thermal, mechanical, and microstructural features of specimens

Variant	Bulk density ($g \cdot cm^{-3}$)	Porosity (%)	Heat conductivity ($W \cdot m^{-1} \cdot K^{-1}$)	Compressive strength (MPa)	Water absorption (vol. %)	Pore size (mm)
1	0.29	86.2	0.067	7.6	3.5	0.10-0.24
2	0.27	87.1	0.063	7.4	3.3	0.20-0.41
3	0.25	88.1	0.059	7.1	3.0	0.38-0.64

The investigation of the results in Table 4 led to the conclusion that the association of glycerol with calcium carbonate in a higher weight ratio in favor of glycerol allows obtaining a finer microstructure, slightly higher bulk density, heat conductivity, and compressive strength as well as lower porosity. Bulk density had very low values (0.25 - 0.29 $g \cdot cm^{-3}$) indicating a light weight of lumps. Also, heat conductivity was within low limits (0.059 - 0.067 $W \cdot m^{-1} \cdot K^{-1}$) showing excellent thermal insulation properties. Compressive strength reached high values, all experimental variants facilitating resistances of 7.1 - 7.6 MPa considered excellent for this type of porous ceramic material.

Microstructural appearance of foam glass gravel specimens are presented in Fig. 3.



a b c **1 mm**

Fig. 3 – Microstructural appearance of foam glass gravel specimens
a – variant 1; b – variant 2; c – variant 3.

According to images in Fig. 3, the minimum size of pores (0.10 - 0.24 mm) corresponded to variant 1 (with the highest glycerol / $CaCO_3$ ratio). The microstructure of this specimen is very fine. Variants 2 and 3 generated slightly larger microstructures with cell sizes between 0.20 - 0.41 mm and 0.38 - 0.64 mm, respectively.

The comparative analysis of the experimental results and the known performances of industrially manufactured foam glass gravels showed good similarity. All three specimens obtained by manufacturing recipes combining glycerol and calcium carbonate and the application of unconventional microwave heating method had physical, thermal, mechanical, and microstructural characteristics suitable for the purpose of their use in special constructions. The decrease of weight ratio of glycerol simultaneously with the increase of that of calcium carbonate in the mixture composition contributed to balancing the specimen properties leading to low bulk densities (0.25 - 0.29 $g \cdot cm^{-3}$) and at the same time very low heat conductivities (0.059 - 0.067 $W \cdot m^{-1} \cdot K^{-1}$), which ensures the required thermal insulation of this material type. Also, compressive strength in all variants reached high values (7.1 - 7.6 MPa) suitable for construction works for which they were designed.

4. Conclusions

The objective of the work was to experiment the manufacture of foam glass gravel from recycled glass waste using the combination of a liquid foaming agent (glycerol) with a solid agent (calcium carbonate) under the conditions of widening the surface of the pressed powder mixture in contact with electromagnetic waves. This experiment was previously preceded by testing the manufacture of the same product type on a microwave oven with much low power (12.5 times) compared to that tried in the current work. In terms of quality, foam glass gravel produced under the conditions of significant increasing the surface dimensions of the mold including starting material as well as the power of microwave equipment to 10 kW kept the product characteristics almost unchanged, i.e. excellent thermal insulation properties (bulk density of 0.25 - 0.29 $g \cdot cm^{-3}$ and heat conductivity of 0.059 - 0.067 $W \cdot m^{-1} \cdot K^{-1}$) and compressive strength of over 7 MPa. The results confirmed the viability of adopted technical solution. In principle, the application fields of foam glass gravel include: insulation in the perimeter of buildings, drainage, road and railway construction, sports fields, insulation of underground pipes for energy fluids and storage tanks, bridge abutments, etc.

For the future stages, the re-design of 10 kW-microwave oven is required to be adapted to the energy needs by reducing the oven dimensions.

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EFECTUL COMBINAT AL GLICERINEI ŞI CARBONATULUI DE CALCIU CA AGENŢI DE EXPANSIUNE PENTRU FABRICAREA PIETRIŞULUI DIN SPUMĂ DE STICLĂ ÎNTR-UN CUPTOR PILOT CU MICROUNDE

(Rezumat)

Pietrişul din spumă de sticlă, un material de construcţie cu excelente proprietăţi portante şi de izolaţie, a devenit foarte atractiv în ultimele decenii datorită cheltuielilor reduse de producţie şi prieteniei sale cu mediul. Utilizat în construcţii, în special în zonele cu climă mai aspră, acest produs consumă ca materie primă sticlă reziduală reciclată. Autorii lucrării curente au abordat recent acest domeniu utilizând undele electromagnetice ca purtătoare de energie convertind puterea microundelor în căldură. Materia primă a fost sticlă post-consum reciclată, iar ca agenţi de spumare a pulberii de sticlă a fost utilizată combinaţia între un agent lichid (glicerina) şi un agent solid (carbonatul de calciu). Experimentul a avut loc într-un cuptor adaptat de 10 kW, mult mai mare faţă de cuptorul cu microunde de 800 W utilizat anterior. Pietrişul din spumă de sticlă a avut excelente proprietăţi termoizolante şi o foarte bună rezistenţă la compresiune fiind la nivelul calitativ al produselor industriale fabricate prin metode convenţionale.