



Foam Glass Gravel Experimentally Made in a 10 kW-Microwave Oven

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Abstract

The experimental manufacture of foam glass gravel from glass waste has been quantitatively extended by increasing the power of the microwave oven from 0.8 to 10 kW, the authors' interest being focused on the quality of the foamed product. The work equipment was rather improvised, the existing used oven not being adequate except to small extent for the requirements of the experiment, but it allowed obtaining a product similar to those industrially manufactured by conventional techniques. Using a recipe previously tested on the 0.8 kW-microwave oven composed of 1 wt.% glycerol as a liquid foaming agent together with 8 wt.% water glass as an enveloping agent and 8 wt.% water as a binder, the main features of the foam glass gravel lumps were: bulk density of 0.22 g/cm³, porosity of 88.9%, thermal conductivity of 0.057 W/m·K, compressive strength of 5.9 MPa and pore size between 0.10-0.30 mm. The specific energy consumption was negatively influenced by the excessive internal volume of the oven, but even under these conditions its value was relatively low (between 1.53-1.69 kWh/kg).

Introduction

In the last 50 years, the waste recycling including also glass waste has become a common practice, especially in Europe. According to an EU study achieved in 2020 (Packaging, 2020), 14 million tons of glass waste are generating annually, the highest proportion of this quantity being the post-consumer packaging glass. The recycling rate of packaging glass in EU member states has reached 76% (Record, 2019). So, the glass waste is a very large reserve available for use as a raw material in manufacturing processes of products with newly created value. Also, the valorization of these reserves has major ecological effects by releasing large areas of land occupied by waste storage.

In recent decades, it has been found the solution of using glass waste to obtain materials whose properties are suitable for replacing some building materials existing on the market. Industrial manufacturers from Europe and the United States (Misapor Switzerland, Pittsburgh Corning, Geocell Schaumglas, Glapor Werk Mitterteich, etc.) have already developed manufacturing technologies for materials (named "glass foams") obtained by foaming the glass waste and their physical, thermal and mechanical characteristics, uniquely combine the properties of a porous material with the mechanical strength and resistance to various external agents specific to the parental glass.

The traditional thermal insulation materials used in the buildings construction are: mineral wool, expanded polystyrene, extruded polystyrene, polyurethane, etc. According to (Jelle, 2011), none of these materials includes all the requirements of an optimal thermal insulation material (light weight, high mechanical strength, low thermal conductivity, chemical and physical stability, robustness, resistance to fire, water, attack of rodents, insects, bacteria, acids, resistance to freeze-thaw cycle, low cost). By comparison, the glass foam simultaneously includes all the features mentioned above (Scarinci et al., 2005; Hurley, 2003).

The type of glass foam tested in this paper is the so-called "foam glass gravel". Unlike the glass foams generally used in the form of panels for thermal insulation of interior or exterior walls of buildings with lower compressive strength (up to 2.8 MPa), the foam glass gravel is a thermal insulation material in the form of lumps with dimensions between 15-80 mm, with durability and high compressive strength (4-6 MPa or more). This product can be used as a material for foundation in light constructions, floor tiles for industrial constructions, road and railway constructions, arrangements around buildings or on their roofs, drainages, sports fields, airport runways, etc. The main foam glass gravel industrial producers are: Geocell Schaumglas (Austria), Misapor Switzerland (Switzerland), Glapor Werk Mitterteich (Germany), Veriso (Germany), Technopor Handels (Austria), Hasopor (Sweden), Glasopor (Norway), Foamit (Finland), Vetropor (Switzerland), with facilities in several European countries (Austria, Germany, Italy, Switzerland and the Scandinavian countries). In the Nordic countries, the manufacture of foam glass gravel is focused on the road construction, due to the difficult climatic conditions of this geographical area and the special requirements imposed on this material by the freeze-thaw cycle (Cosmulescu et al., 2020; Geocell, 2016; Misapor, 2016; Declaration, 2017; Glapor, 2017; Glamaco, 2018; Hibbert, 2016).

According to the technical data provided by the manufacturers, the general manufacturing method of foam glass gravel is the use of a finely ground mixture containing glass waste (predominantly post-consumer packaging glass and, to a lesser extent, flat glass) as a raw material and the foaming agent deposited pressed on a conveyor belt. The powder mixture is introduced by slowly moving of the conveyor belt into a heating oven powered by either gaseous fuel or electricity. The low movement speed of the material allows its heating to the optimal foaming temperature (800-1000 °C) while it is in the heating zone. Next, the material is moved through the cooling zone (free or forced), some technologies applying cold air blowing that causes internal stresses into the sintered material. Thus, at the exit end of the conveyor belt, the material is received in the form of lumps with dimensions below 80 mm, without the need to break the compact mass of glass foam. The main difference between the methods applied by the producers is the manufacturing recipe and especially, the type of the used foaming agent. Misapor Switzerland uses gypsum (CaSO_4), limestone (CaCO_3) or silicon carbide (SiC), Glapor Werk Mitterteich opts for a liquid foaming agent (glycerol) associated with sodium silicate (Na_2SiO_3) and very low kaolin ratio and Glamaco, a very important supplier of industrial ovens for manufacturing the foam glass gravel, recommends SiC, manganese oxide (MnO_2), glycerol associated with Na_2SiO_3 , coal powder, CaCO_3 or CaSO_4 (Cosmulescu et al., 2020).

As noted above, the heating techniques used in the industrial manufacturing processes of foam glass gravel are exclusively conventional (gaseous fuels burning or electric resistances). In the last four years, the Romanian company Daily Sourcing & Research has experimentally produced various types of glass foam (including also foam glass gravel) using the microwave energy. This unconventional heating technique is fast and economical due to its peculiarities that make it completely different from the known conventional techniques. Although the advantages of microwave heating are recognized in the literature (Kharissova et al., 2010), its

industrial application has reached an insignificant energy level being used only in drying and heating processes at low temperatures. Currently, the use of microwaves as a method of heating solids worldwide is still in various stages of experimental research.

Recent experiments conducted by the Daily Sourcing & Research company aimed to manufacture foam glass gravels using a 0.8 kW-adapted microwave oven. Several variants of foaming agents and additives were tested: CaCO_3 together with borax ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) and Na_2SiO_3 , glycerol together with Na_2SiO_3 and water as well as SiC with water addition. The experimental results have shown that foam glass gravels with characteristics similar to those industrially manufactured and with very low specific energy consumption (0.86-1.07 kWh/kg) (Cosmulescu et al., 2020; Paunescu et al., 2020a; Paunescu et al., 2020b; Paunescu et al., 2019; Dragoescu et al., 2020; Paunescu et al., 2020c) can be obtained. The current paper is an important step forward aiming to test the manufacture of foam glass gravels in an improvised microwave oven with a much higher power (10 kW) compared to the oven used in previous experiments (0.8 kW). Also, the loading capacity with raw material of the 10 kW-oven and implicitly, the surface occupied by it, increased significantly allowing the determination of the influence of microwave field on the foaming process.

Methods

The existing microwave oven in the experimental base of the company Daily Sourcing & Research, designed for other technological processes and with a total installed power of 10 kW magnetrons, has been adapted to carry out experiments on the manufacture of foam glass gravel. According to the constructive scheme in Figure 1B, the distribution of the microwave field was made through ten waveguides (6), three on the each side wall and four on the flat vault of the oven equidistantly placed. For this purpose, a silicon carbide-cylindrical crucible (2) with an outer diameter of 300 mm, a height of 450 mm and a wall thickness of 10 mm was inserted in a horizontal position in the inner space of the oven (1), being protected with thick layers of ceramic fiber mattresses (5) for conserving the thermal energy of the ceramic crucible. The powder mixture was loaded and pressed into a rectangular mold (4) with the base side dimensions of 250 and 360 mm and the walls height of 40 mm made of 1.5 mm-stainless steel sheet. A thermocouple whose hot welding was fixed on the side wall of the mold allowed the control of the process temperature. The overall image of the microwave oven and its constructive scheme are shown in Figures 1A and 1B.

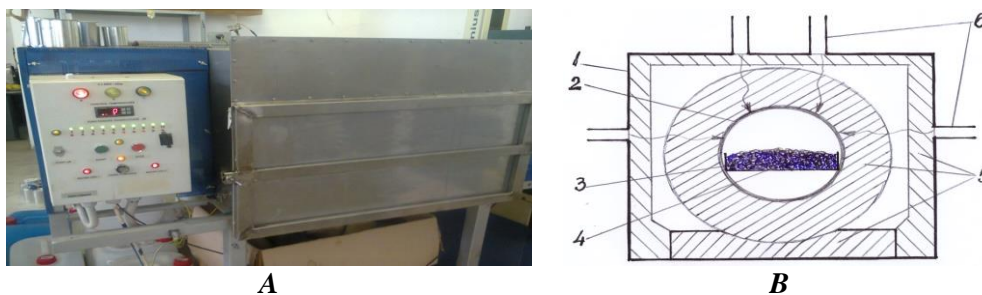


Figure 1. 10 kW-microwave oven used for manufacturing the foam glass gravel
A – overall image of the oven; B – constructive scheme: 1 – microwave oven; 2 – SiC-ceramic crucible; 3 – pressed powder mixture; 4 – metal mold; 5 – ceramic fiber-thermal insulation; 6 – waveguide.

Previous experiments (Paunescu et al., 2017) have indicated that the soda-lime glass, from which the usual commercial glass is made, is not suitable for fully direct microwave heating, its internal structure being severely affected during the foaming process. For this reason, the authors' team adopted the solution of placing a ceramic screen (crucible or tube) made of a high

microwave susceptible material between the source generating electromagnetic waves and the irradiated material. This technical solution was successfully tested in all experiments performed on the 0.8 kW-microwave oven, the optimal thickness of the crucible/tube wall being between 2.5-3.5 mm. The ceramic crucible available for the tests performed on a much larger scale had a wall thickness of 10 mm and this certainly reduced the optimal ratio between the direct and indirect microwave heating.

The experiments performed on the 10 kW-microwave oven aimed at the manufacture of foam glass gravels by using a liquid carbonic foaming agent (glycerol) together with Na_2SiO_3 and water. Generally, a liquid foaming agent has the advantage of easily dissipating among the fine particles of glass waste, as unlike to a solid agent. The glycerol ($\text{C}_3\text{H}_8\text{O}_3$) embedded in the powder mixture decomposes in the oxidizing atmosphere of the oven releasing a wide spectrum of compounds between CO_2 and pure carbon and also hydroxyl compounds (Karandashova et al., 2017). The thermal decomposition of glycerol is a complex process, which is initiated at low temperatures (below $200\text{ }^\circ\text{C}$) and occurs in several stages up to about $850\text{ }^\circ\text{C}$ (Dou et al., 2008). Due to the high affinity of carbon for oxygen that causes its premature combustion in oven conditions, enveloping of the glass particles with aqueous solution of sodium silicate (also called "water glass") was experimentally found. Initially added to the solid powder mixture together with the liquid foaming agent and water, the "water glass" slows down the decomposition of glycerol and intensifies the glass particles sintering. The water addition favors the formation at about $800\text{ }^\circ\text{C}$ of the so-called "water gas" containing hydrogen and carbon monoxide, which contributes to the foaming process of the glass (Karandashova et al., 2017).

The materials used in experiments were: a mixture of post-consumer packaging glass containing 50 wt.% colorless glass, 20 wt.% green glass and 30 wt.% amber glass, glycerol as a liquid carbonic foaming agent, a 30% aqueous solution of sodium silicate ("water glass") as an enveloping agent and water as a binder. The chemical composition of the three types of glass waste is shown in Table 1.

Table 1. Chemical Composition of the Glass Waste Types

Glass type	SiO_2	Al_2O_3	CaO	Fe_2O_3	MgO	Na_2O	K_2O	Cr_2O_3	SO_3	Other oxides
Colorless	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
Amber	71.1	2.0	12.1	0.2	1.1	13.3	0.1	-	0.05	0.05

The glass waste was selected by color, broken, thermally washed at 250°C to remove the organic contaminants, ground in a ball mill and sieved at the grain size below $100\text{ }\mu\text{m}$. The glass waste processing operations were performed at Bilmetal Industries SRL Popesti-Leordeni (Romania).

The dosage and preparation of the liquid mixture (glycerol, aqueous solution of sodium silicate and water) were performed in a separate vessel. The mixing was done with an electrically operated device for 10 min. Then, the liquid mixture was poured over the powder material and the final mixing was performed.

The foam glass gravel lumps were characterized using methods of determining the physical, thermal, mechanical and morphological features. 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 (Scorgins, 2015). The thermal conductivity was determined by the heat-flow meter method (ASTM E1225-04) and the porosity was calculated

by the method of comparing the true and bulk density (Anovitz & Cole, 2005). The compressive strength was measured using a TA.XTplus Texture Analyzer (ASTM C552-17). The water absorption (during 24 hours) of the lumps was determined by the water immersion method (ASTM D570). The microstructure configuration of the foam glass gravel lumps was investigated with an ASONA 100X Zoom Smartphone Digital Microscope.

Previous experiments on the manufacture of foam glass gravel in the 0.8 kW-microwave oven have shown excellent physical, thermal and mechanical characteristics of products made with a liquid carbon foaming agent (Paunescu et al., 2020c). Taking into account these results, four experimental variants with compositions within limits close to the optimal composition resulting from testing on the 0.8 kW-oven were adopted. The four variants adopted for the experiments performed on the 10 kW-microwave oven are presented in Table 2.

Table 2. Composition of the Experimental Variants

Component	Variant 1	Variant 2	Variant 3	Variant 4
Glass waste	84.7	83.0	81.3	79.6
Glycerol	0.9	1.0	1.1	1.2
Water glass	7.2	8.0	8.8	9.6
Water	7.2	8.0	8.8	9.6

According to the data in Table 2, the glycerol/water glass ratio was kept constant at the value of 1/8 and the weight ratios of water glass and water were kept equal.

According to the adopted experimental methodology, the manufacturing process were conducted keeping unchanged the oven power, the followed parameter being the temperature indicated by the thermocouple fixed on the metal wall of the mold. The pre-established range of the process temperatures was similar to that experimentally obtained in previous tests performed on the 0.8 kW-microwave oven (Paunescu et al., 2020c). The wet raw material amount was 3.95 kg and this was kept constant in all four experiments.

Results and Discussion

The main functional parameters of the manufacturing process of foam glass gravel are shown in Table 3.

Table 3. The Main Functional Parameters of the Manufacturing Process of Foam Glass Gravel

Var.	Wet raw material/ foam glass gravel amount kg	Sintering/ foaming temperature °C	Heating time min	Average rate °C/min		Index of volume growth	Specific energy consumption kWh/kg
				Heating	Cooling		
1	3.95/3.85	819	45	17.8	5.6	1.50	1.53
2	3.95/3.85	820	46	17.4	5.4	1.60	1.56
3	3.95/3.89	822	47.5	16.9	5.7	1.70	1.60
4	3.95/3.87	825	50	16.1	5.5	1.85	1.69

According to the data in Table 3, the manufacturing process of foam glass gravel using colored glass waste, glycerol, water glass and water was performed at temperatures between 819-825 °C, the heating time being between 45-50 min. The heating rate varied in small limits (16.1-

17.8 °C/min) and the specific energy consumption had relatively low values between 1.53-1.69 kWh/kg. It should be noted that the values of specific energy consumption obtained in the experiments on the 0.8 kW-microwave oven were considerably lower (below 0.88 kWh/kg) (Paunescu et al., 2020c). This difference is explained by the fact that the existing 10 kW-oven was not suitable for the experiments described above, its internal volume being much too large. Under these conditions, the specific energy consumption was a secondary parameter, the authors' interest being focused on the quality of the foamed products.

Appearance images of the foam glass gravel lumps obtained in the experiments are shown in Figure 2. The superficial examination of the appearance of lumps shows that they all have a very fine and homogeneous porosity.

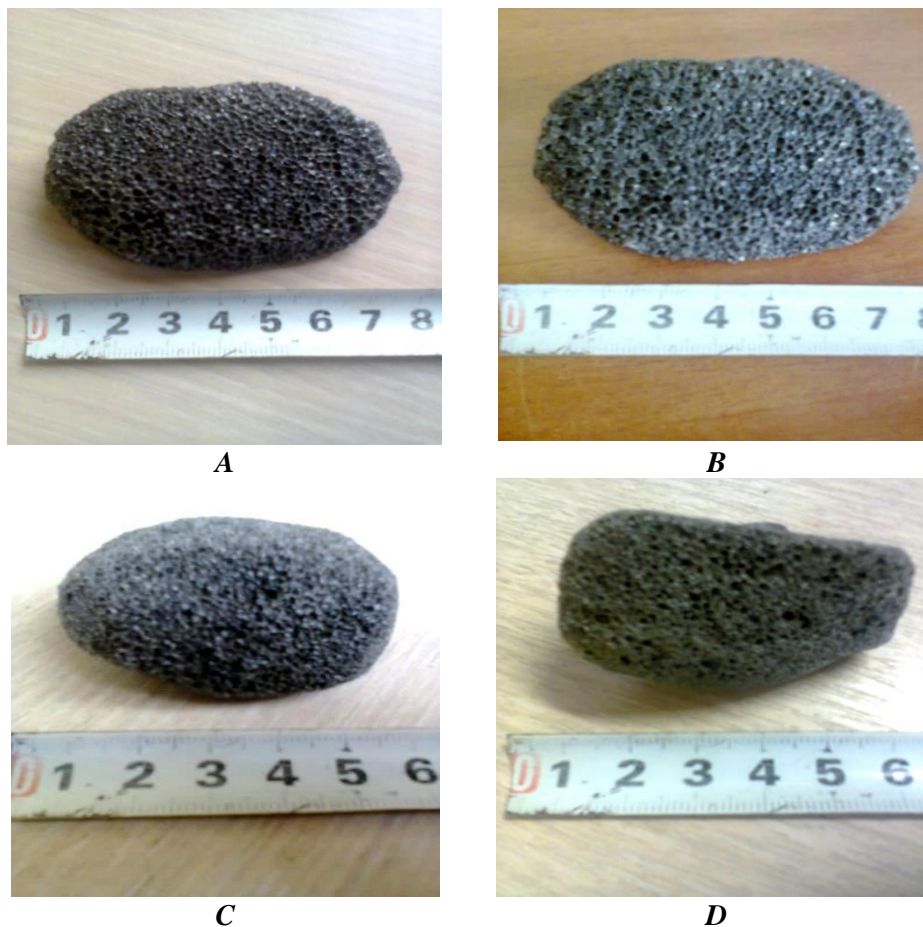


Figure 2. Appearance images of the foam glass gravel lumps
 A – sample 1 heated at 819 °C; B – sample 2 heated at 820 °C;
 C – sample 3 heated at 822 °C; D – sample 4 heated at 825 °C.

The main physical, thermal, mechanical and morphological characteristics of foam glass gravel lumps are presented in Table 4.

Table 4. Main Physical, Thermal, Mechanical and Morphological Characteristics of Foam Glass Gravel Lumps

Variant	Bulk density g/cm ³	Porosity %	Thermal conductivity W/m·K	Compressive strength MPa	Water absorption %	Pore size mm
1	0.22	89.0	0.059	5.8	1.2	0.05-0.25

2	0.22	88.9	0.057	5.9	0.9	0.10-0.30
3	0.20	89.5	0.053	5.4	0.8	0.10-0.40
4	0.18	90.0	0.049	5.0	0.8	0.15-0.55

Analyzing the data in Table 3, the low values of bulk density (0.18-0.22 g/cm³) and thermal conductivity (0.049-0.059 W/m·K) are noticed, which together lead to very good thermal insulation properties of the products. In addition, the compressive strength has high values (between 5.0-5.9 MPa), this requirement being very important in the characterization of a foam glass gravel. The porosity has also very high values between 88.9-90.0%. The water absorption determination has indicated very low values between 0.8-1.2%. The comparison of these data referring to the physical, thermal and mechanical features of the products indicates a great similarity with the characteristics of the foam glass gravel samples produced in favorable conditions in the 0.8 kW-microwave oven (Paunescu et al., 2020c), but also with those of industrially manufactured products (Glapor Werk Mitterteich) (Cosmulescu et al., 2020). The very fine porosity of foam glass gravel lumps can be observed in their microstructural configuration shown in Figure 3.

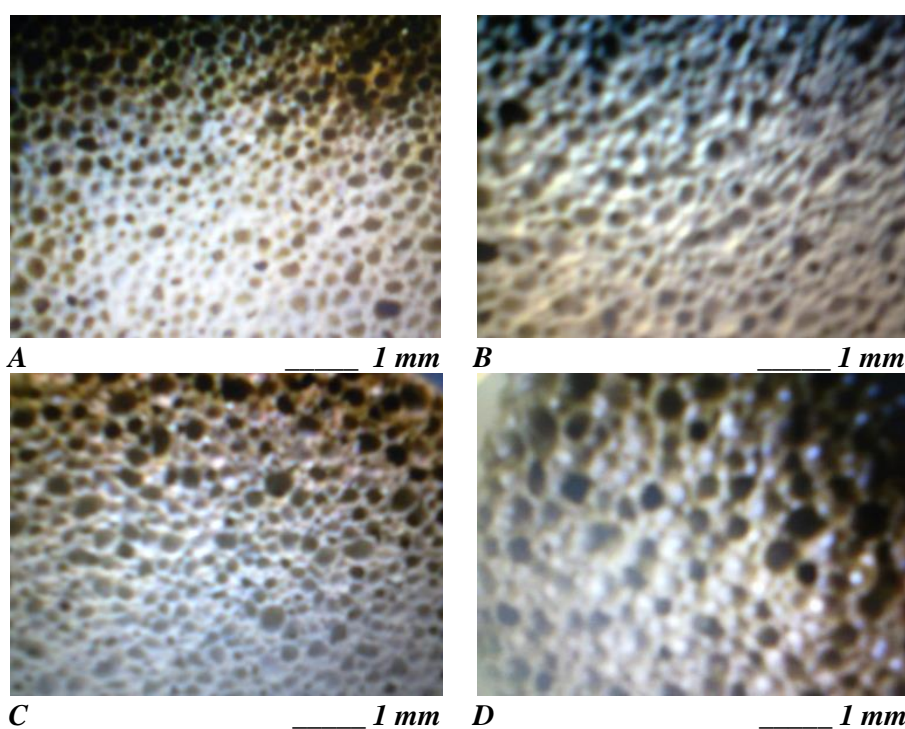


Figure 3. Microwave configuration of the foam glass gravel lumps
A – sample 1; B – sample 2; C – sample 3; D – sample 4.

The pore size is extremely low (below 550 μm in the case of sample 4, made with 1.2% glycerol, 9.6% water glass and 9.6% water). The sample 2 obtained using the optimal manufacturing recipe of the reference experiment (Paunescu et al., 2020c) with 1% glycerol, 8% water glass and 8% water had a pore size between 100-300 μm, i.e. at the level of industrially manufactured products by the Glapor company (Cosmulescu et al., 2020).

Considering the experimental results of the foam glass gravel manufacturing process, variant 2 was adopted as the optimal variant. Its manufacturing recipe coincided with the recipe used in the experiments performed on the 0.8 kW-microwave oven (Paunescu et al., 2020c) and which led to excellent physical, thermal, mechanical and morphological characteristics of the

product. The authors' interest in testing the manufacture of foam glass gravel using much larger quantities of starting material hastened the achievement of this goal in less appropriate conditions. The experimental microwave equipment was rather improvised.

The physical, thermal, mechanical and morphological characteristics of the optimal product (bulk density of 0.22 g/cm³, porosity of 88.9%, thermal conductivity of 0.057 W/m·K, compressive strength of 5.9 MPa and pore size between 0.10-0.30 mm) are specific for the foam glass gravels industrially manufactured with glycerol, water glass and water, having very good thermal insulation properties and simultaneously a high compressive strength.

Due to the fact that the 10 kW-microwave oven was not sufficiently suitable for the manufacturing process, the value of the specific energy consumption was significantly higher (1.56 kWh/kg) compared to the value reached in the reference experiment on the 0.8 kW-oven (0.88 kWh/kg) (Paunescu et al., 2020c). However, the energy efficiency of using the microwave energy has contributed to a relatively low consumption. In energy terms, comparison with similar industrial processes is not possible due to the lack of this information in the literature.

Conclusion

The paper presents the results of transition from the experimental manufacturing stage of foam glass gravel from glass waste on a 0.8 kW-microwave oven to a superior stage of manufacturing the same product on a 10 kW-oven with a much higher production capacity. Unlike the conventional heating technique used in the world in the industrial production of glass foam (including also the foam glass gravel), the research presented in the paper is based on the application of the unconventional microwave heating technique recognized in the literature as being faster and more economical. Based on the excellent results previously obtained in the experiments performed on the 0.8 kW-oven, four manufacturing recipes were adopted containing colored glass waste (colorless, green and amber) as raw material, glycerol as a liquid carbonic foaming agent (between 7.2- 9.6 wt.%) and water as a binder (between 7.2-9.6 wt.%). The sintering/foaming process was performed by heating at 819-825 °C with average rates between 16.1-17.8 °C/min. The physical, thermal, mechanical and morphological characteristics of the optimal product were: bulk density of 0.22 g/cm³, porosity of 88.9%, thermal conductivity of 0.057 W/m·K, compressive strength of 5.9 MPa and pore size between 0.10-0.30 mm, specific for the foam glass gravels industrially manufactured with glycerol, water glass and water, having very good thermal insulation properties and simultaneously a high compressive strength. Because the existing 10 kW-microwave oven was not sufficiently suitable for the process, the authors' interest was focused on the quality of the foamed product. The specific energy consumption had a relatively low value (1.56 kWh/kg), but it was affected by the too large internal volume of the oven, which makes it unsuitable for the foam glass gravel manufacturing process. The experimental results obtained so far regarding the manufacture of the foam glass gravel clearly indicate the need to design a new oven with a power of at least 10 kW suitable for this process with the prospect of maximizing the efficiency of the unconventional microwave heating technique.

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