HIGH MECHANICAL STRENGTH POROUS MATERIAL USED AS A FOAM GLASS GRAVEL EXPERIMENTALLY MANUFACTURED FROM GLASS WASTE BY AN UNCONVENTIONAL TECHNIQUE

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ABSTRACT

The paper presents a new technique for making foam glass gravel by expanding at high temperature recycled glass waste with a liquid carbonic foaming agent (glycerol) in combination with sodium silicate (also called "water glass"). Unlike the conventional methods known worldwide in similar technological processes, the paper proposes a fast, economical and clean unconventional technique (microwave irradiation), applied to a very small extent in the industrial sector, although in the household it is a widely used process. The experiments carried out by the Daily Sourcing & Research Company on a 0.8 kW-microwave oven aimed at the manufacture of foam glass gravels with physical and mechanical characteristics almost similar to those industrially obtained by conventional methods, simultaneously demonstrating the superior energy efficiency of the technique of using microwave energy as an unconventional source. The products experimentally made had low thermal conductivity (between 0.057-0.063 W/ m·K), high compressive strength for this material type (up to 5.9 MPa) and very low pore size (below 0.9 mm). The specific energy consumption had values between 0.83-0.88 kWh/ kg comparable to those industrially achieved, but it should be considered that the use of a high power microwave equipment could improve the process efficiency by up to 25%.

Keywords: foam glass gravel; glass waste; glycerol; microwave; compressive strength.

REZUMAT

Lucrarea prezintă o nouă tehnică de fabricare a pietrișului de sticlă celulară prin expandare la temperatură înaltă a deșeurilor de sticlă reciclată cu un agent de spumare carbonic lichid (glicerină) în combinație cu silicat de sodiu (denumit și "apă de sticlă"). Spre deosebire de metodele convenționale cunoscute pe plan mondial în procese tehnologice similare, lucrarea propune o tehnică neconvențională rapidă, economică și curată (iradierea cu microunde), aplicată în extrem de mică măsură în sectorul industrial, deși în domeniul casnic constituie un procedeu foarte utilizat. Experimentele efectuate de societatea Daily Sourcing & Research pe un cuptor cu microunde de 0,8 kW au avut ca obiectiv fabricarea unor agregate din sticlă celulară cu caracteristici fizice și mecanice aproape similare cu cele obținute industrial prin metode convenționale, demonstrând în același timp eficiența energetică superioară a tehnicii utilizării energiei microundelor ca sursă neconvențională. Produsele realizate experimental au avut conductivitate termică redusă (între 0,057-0,063 W/ m·K), rezistență mare la compresiune pentru acest tip de material (până la 5,9 MPa) și dimensiunea porilor foarte mică (sub 0,9 mm). Consumul specific de energie a avut valori între 0,83-0,88 kWh/ kg, comparabile cu cele realizate industrial, dar ar trebui avut in vedere că utilizarea unui echipament cu microunde de putere mare ar putea îmbunătăți eficiența energetică a procesului cu până la 25%.

Cuvinte cheie: pietriș de sticlă celulară; deșeu de sticlă; glicerină; microundă; rezistență la compresiune.

1. INTRODUCTION

Foam glass gravel is an assortment of building material industrially manufactured from recycled glass waste by sintering and foaming at high temperature. The material simultaneously embeds several characteristics (high mechanical strength, fine porosity, low apparent density, low thermal conductivity, non-absorbent of water or humidity, fire and rodent resistant), which recommend it for different construction types: insulation fill in foundations of buildings, parking decks, roofs of car park or perimeter insulation, substructure in road and railway constructions, insulation for long-distance heating pipe lines, insulation of underground storage tanks. The manufacture of foam glass gravel began in the last decades of the 20th century, after the waste recycling (metal, paper, glass, etc.) became a priority activity in the world, especially in developed and developing countries, both for ecological and economic reasons. The main global foam glass gravel manufacturers are Geocell Schaumglas GmbH [1], with facilities in Austria and Germany, as well as Misapor Switzerland Corporation [2], with branches in Germany, France and Austria. The technological process is carried out in tunnel ovens with conveyor belt heated by conventional methods (burning of fossil fuels or electrical resistances). Generally, the raw material is container glass waste (mixture of colorless, green and amber glass) and / or colorless flat glass waste. The foaming agents used by the manufacturers are very different (limestone, silicon carbide, gypsum, glycerol etc.), as well as the mineral additions. In all cases, the foamed product has the compressive strength of 5-6 MPa (or more) and the bulk density below 0.25 g/ cm^3 [1 - 3]. A particular case is the foam glass gravel produced with glycerol as a liquid carbonic foaming agent. It is known from the literature [4] that liquid foaming agents can contribute to obtaining very fine porous structures due to a wider contact between the liquid agent and the fine glass particles compared to a solid foaming agent. German companies Glapor Werk Mitterteich GmbH and Glamaco GmbH commonly use recipes that include glycerol. Thus, Glapor uses 87% glass waste, 1% glycerol, 12% sodium silicate (also called “water glass”) and below 0.5% kaolin. The foamed product has the bulk density of 0.13-0.21 g/ cm^3 and the compressive strength of 4.9-6.0 MPa [5, 6]. Glamaco uses 95% glass waste, glycerol and calcium carbonate as foaming agents as well as “water glass” as an enveloping material, accounting for 5%, water addition and very low kaolin ratio. The final product has the bulk density between 0.15 and 0.20 g / cm^3, the thermal conductivity between 0.06 and 0.08 W/m·K and the compressive strength in the range 4-6 MPa [7]. Results of some experimental tests are presented in the work [8] carried out under the conditions of manufacturing a foam glass gravel with a very fine porosity (pore size below 300 μm). The sintering / foaming temperature was varied between 800 and 850ºC. The heating rate of the mixture composed from glass waste, glycerol, water glass and water in different mass ratios, had a low value (8ºC / min). After the sintering temperature was reached, it was kept constant for 30 min. The thermal process was carried out in a laboratory oven heated with electrical resistances. The experiments highlighted the optimum composition of the mixture to obtain a product with very fine porosity: 90% colorless glass waste, 4% glycerol, 3% water glass and 3% water. In the work [9] the influence of the glycerol/ water glass mass ratio used in the sintering/ foaming process of a container glass waste on the morphological characteristics of the sample were experimentally analyzed. Mass ratios of glycerol as a foaming agent, between 1.0 and 1.5%, mixed with water glass as an enveloping material, between 2.0 and 5.0%, were used in the process. The sintering temperature obtained by a conventional heating technique was varied between 800 and 850ºC. The heating rate had very high values between 26 and 166ºC / min. The obtained products had a fine porosity, with pore sizes between 0.25 mm and 1.5-2.0 mm depending on the heating rate and on the final value of the sintering temperature. The experimental results showed that the combination of 1.5% glycerol and 5% water glass, the process temperature of 850 ºC and the average heating rate in the range 50-80 ºC / min are the optimal functional parameters for obtaining foam glass gravel with physical and mechanical characteristics similar to industrially manufactured products.

As mentioned above, the industrial or experimental manufacturing techniques of foam glass gravel used in the world have been conventional. In recent years, the Romanian company Daily Sourcing & Research Bucharest has carried out numerous experiments regarding the production of glass foams from glass waste, using a fast and economical unconventional method by microwave irradiation of the material. Obviously, the tests recently made for manufacturing foam glass gravel were based on the same heating technique. Experiments using colored (green and amber) container glass waste, a solid foaming agent (calcium carbonate), borax as a fluxing agent and water glass have led to the manufacture of a foam glass gravel with compressive strength up to 9.5 MPa, apparent density of 0.8 g / cm^3, thermal conductivity of 0.105 W / m·K and pore sizes below 1 mm. The sintering / foaming temperature required to obtain this product was 845ºC. Other experiments using glycerol (between 1.0 and 1.8 wt.%), water glass (5.3-7.5 wt.%) and water addition (7.7-10.0 wt.%) allowed to obtain a foam glass gravel with an apparent density between 0.20 and 0.26 g / cm^3, porosity between 85.5 and 88.2%, thermal conductivity between 0.056 and 0.070 W / m·K, compressive strength in the range 4.6-5.8 MPa and water absorption between 0.8 and 1.7%. The porosity of the material was fine, the pore size varying from 0.8-1.1 mm to 0.3-0.8 mm, which correspond to the glycerol consumption of 1%, water glass 7.5% and water 7.7%. The experiments results shown above are presented in two papers that are being published in Romanian journals.

In the present paper, new experimental results obtained in the manufacturing process in microwave field of foam glass gravel using glycerol as a liquid carbonic foaming agent are presented. Except for the intention to produce a foamed material with very fine porosity and high mechanical strength, the economic criterion was taken into account, the market price of glycerol of about 1 USD / kg [10] being significantly lower (3-4 times) than the prices of calcium carbonate and silicon carbide, the main materials used as solid foaming agents.

2. 2. METHODS AND MATERIALS

2.1. Methods

The principle of producing glass foam consists in the release of a gas in the thermally softened mass of the material. Due to its suitable viscosity, the gas remains blocked in the form of bubbles and after cooling it forms pores that constitute the porous structure of the glass foam [11]. In the case of carbonic foaming agents, the gas results either from the reaction of carbon with oxygen in the atmosphere between the glass particles or from the carbon reduction reaction of some components of the glass. According to [1], the general form of the carbon reduction reaction in the mass of glass powder is:

$$SO_4^{2-} + 2C = S^{2-} + CO + CO_2 \qquad (1)$$

were SO_4^{2-} comes from the glass composition and S^{2-} returns to the glass composition.

The method of using glycerol as a liquid carbonic foaming agent in association with water glass and water is a method known in the literature [4, 8] and it has been adopted by the authors of this paper. Glycerol ($C_3H_8O_3$), that is an organic material [12], decomposes in the oxidizing atmosphere of the oven to form carbon dioxide, carbon monoxide, pure carbon and hydroxyl compounds that enhance the foaming process of glass powder. Water glass is used as an enveloping material for fine carbon particles resulting from the glycerol decomposition for slow downing its decomposition process, thus avoiding the premature carbon oxidation. The water addition contributes to reducing the viscosity of the liquid mixture and favors the formation of "water gas", whose components (hydrogen and carbon monoxide) intensify the foaming. The use of this liquid mixture together with the glass powder allows obtaining a foamed product characterized by a very fine porosity, impossible to obtain under the conditions of using a solid foaming agent [4].

The originality of the method adopted for the manufacturing of foam glass gravel with liquid carbonic foaming agent consists in the use of microwave energy, unlike the conventional techniques applied in the world.

The used experimental equipment was the 0.8 kW-microwave oven of the type used in household for the food preparation, adapted for the operation conditions at high temperature (up to 1200 ºC) (Fig. 1). Since 2017, all tests carried out in the company Daily Sourcing & Research for glass foam production occur on this microwave equipment, its constructive and functional principle being described in several published papers [13-15]. The main component of the experimental equipment is a ceramic tube made of a high microwave susceptible material based on silicon carbide, with the outer diameter of 125 mm, the wall thickness of 3.5 mm and the height of 100 mm, provided with a lid of the same material type. The role of the ceramic tube and lid which protect the pressed powder material placed in the free inner space is to moderate the aggressive effect of the direct microwave heating on the glass-based material, the microwave field being partially absorbed in the ceramic wall, which is intensely heated transferring heat through thermal radiation and partially penetrates this wall coming in direct contact with the material. This mixed microwave heating type constitutes the optimal solution for a fast and energy efficiency process, confirmed in previous experiments.

a b

Fig. 1. The experimental microwave equipment (a – microwave oven; b – ceramic tube)

The control of the thermal process was provided by a radiation pyrometer mounted above the oven in the central axis. Thus, the temperature evolution of the upper surface of heated material was determined. Stopping the increase of its temperature represented the indication of the intense foaming of the sample head, i.e. finishing the process.

2.2. Materials

The raw material was represented by container glass waste composed of 50% colorless glass, 20% green glass and 30% amber glass. According to previous determinations of the three types of glass waste [16], the average chemical composition of the glass mixture was calculated, the results being shown in Table 1. The glass waste was thermally washed at 250ºC, to remove the organic contaminants, ground in a ball mill and sieved at the grain size below 150 μm.

Table 1. *Chemical composition of glass waste*

Compo-nent	Glass waste			
	Color-less	Green	Amber	Average
SiO_2	71.7	71.8	71.1	71.54
Al_2O_3	1.9	1.9	2.0	1.93
CaO	12.0	11.8	12.1	11.99
Fe_2O_3	-	-	0.2	0.06
MgO	1.0	1.2	1.1	1.07
Na_2O	13.3	13.1	13.3	13.26
K_2O	-	0.1	0.1	0.05
Cr_2O_3	0.05	0.09	-	0.043
SO_3	-	-	0.05	0.015
Other oxides	0.05	0.01	0.05	0.042

The liquid part of the load was composed of glycerol as a carbonic foaming agent, sodium silicate (“water glass”) as a 30% aqueous solution and water addition. It was prepared separately from the glass waste, each of the three components, previously dosed, being introduced in a collecting vessel and mixed for 15-20 minutes, following the complete dissolution in water of glycerol and water glass. Then, the liquid mixture was poured into the container containing the glass powder.

2.3. Characterization of the samples

The foam glass gravel samples produced by the sintering / foaming process of glass waste using glycerol and water glass were analyzed in the laboratory to determine their main features.

The apparent density was measured by the gravimetric method [17].

The porosity determining involved the comparison between the compact material density (melted and compactly cooled) and the density of the foamed product [18]. The thermal conductivity was determined by measuring the thermal flow which passes through the sample mass with a thickness of 50 mm placed between two metal plates, one heated and thermally protected and the other cooled [19]. The water absorption of the sample was measured by the water immersion method (ASTM D 570). To determine the compressive strength, a device built according to the own design was used, which develops an axial pressing force exerted by the hydraulically operated piston of maximum 20 tf, and can measure axial compressive strengths up to 40 MPa. The sample has a cylindrical shape with a diameter of 80 mm and a height of 70 mm. The test measures the value of the compressive strength of the sample before cracking. The microstructural configuration of the samples was performed with a Smartphone digital microscope.

3. RESULTS AND DISCUSSION

3.1. Results

As noted above, the materials used in the sintering/ foaming process for obtaining glass foam gravel were: container glass waste, glycerol, water glass and water. Four experimental variants were adopted, having the weight ratios shown in Table 2.

Table 2. *Composition of the experimental variants (wt.%)*

Component	Variant			
	1	2	3	4
Glass waste	82.3	82.7	82.8	83.0
Glycerol	1.6	1.4	1.2	1.0
Water glass	10.1	9.5	9.0	8.0
Water	6.0	6.4	7.0	8.0

The weight ratios of the components of the four tested variants were adopted based on previous own experimental results as well as some data provided by the literature. The glycerol/ water glass weight ratio was kept within a narrow range (0.125-0.158). Increasing the proportion of water addition from variant 1 to variant 4 has compensated for the reduction of the water glass ratio.

The main functional parameters of the sintering/ foaming process of glass waste and the physical and mechanical features of the foam glass gravel samples are presented in Tables 3 and 4.

Table 3. *Main functional parameters of the sintering/ foaming process*

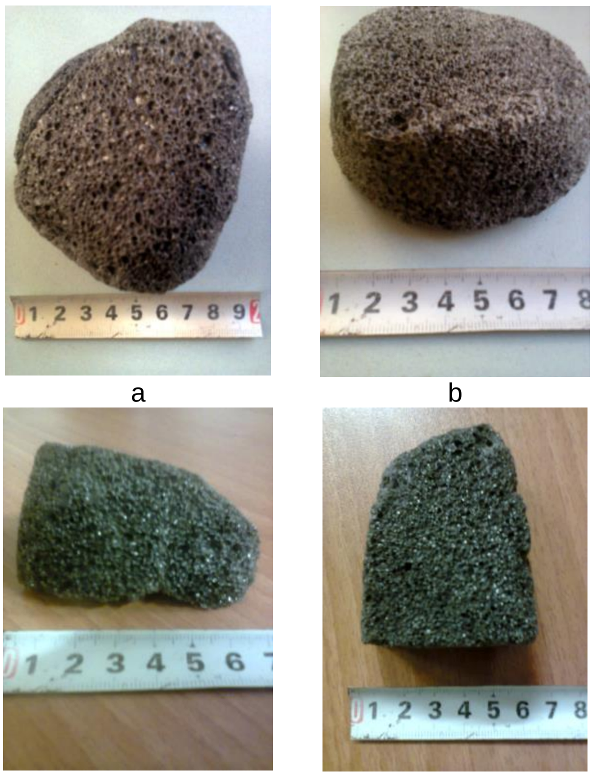
Variant	Raw material / foam glass gravel amount [g]	Sintering / foaming temperature [ºC]	Heating time [min]	Average rate, ºC / min		Index of volume growth	Specific energy consumption [kWh/ kg]
				Heating	Cooling		
1	625/ 562	815	40	19.9	5.8	1.80	0.83
2	625/ 560	817	40.5	19.7	5.7	1.80	0.84
3	625/ 558	820	41.5	19.3	5.9	1.70	0.87
4	625/ 558	823	42	19.1	6.1	1.65	0.88

Table 4. *Physical and mechanical features of the samples*

Variant	Apparent density [g/cm^3]	Porosity [%]	Thermal conductivity [W/m·K]	Compressive strength [MPa]	Water absorption [%]	Pore size [mm]
1	0.21	90.5	0.057	4.8	1.1	0.7 – 0.9
2	0.22	90.0	0.061	5.0	0.8	0.6 – 0.9
3	0.23	89.5	0.062	5.5	1.0	0.5 – 0.8
4	0.24	89.1	0.063	5.9	0.6	0.3 – 0.6

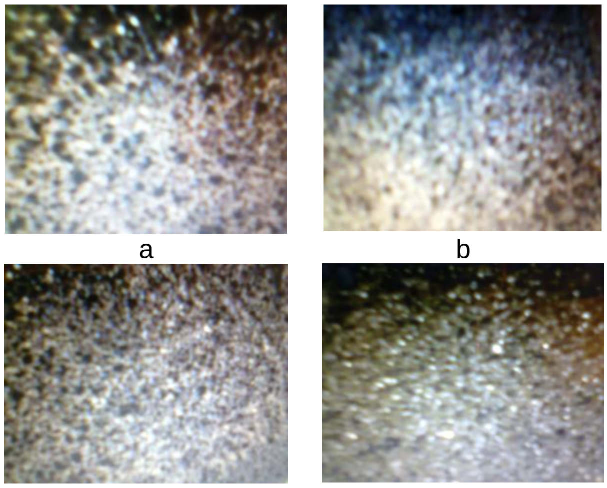
Analyzing the data in Table 3, firstly, the very low level of the specific energy consumption (0.83-0.88 kWh / kg) is remarkable, being comparable to the most efficient technological processes of manufacturing glass foam by conventional techniques. According to the work [20], a high power microwave equipment, equivalent to an industrial microwave oven, could have a higher energy efficiency of up to 25% because it allows a high-energy power source with long life, the use of an unique internal protection feature, uniform exposure in the microwave field and much lower process costs, comparing to the experimental oven used for tests. Also, the heating rate having values of over 19ºC is suitable for the sintering/ foaming process and, in the same time, is high enough so that the process to be energy efficient.

By examining the physical and mechanical characteristics of the obtained products, the high values of the compressive strength (between 4.8 and 5.9 MPa), the low apparent density (below 0.24 g/ cm^3) and the low thermal conductivity (between 0.057 and 0.063 W/m·K) should be highlighted. Following the performed tests, the foam glass gravel is practically not water-absorbent, the absorbed proportions being below 1.1%.



a b c d

Fig. 2. Images of the foam glass gravel samples (a – sample 1; b – sample 2; c – sample 3; d – sample 4)



a b c d

Fig. 3. Pictures of the microstructural configuration of samples a – sample 1; b – sample 2; c – sample 3; d – sample 4.

The porous structure of the products is very homogeneous, the pore size being very small (below 1 mm). Fig. 2 shows images of the four foam glass gravel samples. In Fig. 3 pictures of the microstructural configuration of samples are presented, confirming the fineness of the porosity of all the samples and especially of the samples with lower glycerol content.

Analyzing similar physical, mechanical and microstructural performances of foam glass gravels produced by microwave heating compared to those manufactured by the usual conventional techniques, it is obvious that they have the same application fields in the construction mentioned above. The main advantage of the process is the lower specific energy consumption.

3.2. Discussion

Various experimental processes in the microwave field for obtaining cellular products from silicate waste have revealed different behaviors of these materials in direct contact with the microwave radiation. It has been found that aluminosilicates that compose clay brick waste, construction concrete waste, coal ash, cement mortar waste, etc. are suitable for the direct microwave heating processes [21]. By comparison, the soda-lime glass waste used to make the commercial container glasses is not suitable for the microwave irradiation, which generates a structural hollow in the core of the heated sample [22].

The solution adopted by Daily Sourcing & Research Company was the use of a ceramic crucible or tube made of microwave susceptible materials based on silicon carbide placed between the microwave generating source and the heated material. The use of a crucible or tube with a wall thickness of 15-20 mm ensures the indirect heating of the sample, while a thickness of the wall of 3.5-5 mm creates the conditions of a partially direct and partially indirect heating. This heating mode was adopted in several previous experiments, including those presented in this paper, being significantly more energy efficient than the indirect heating mode.

Regarding the optimal weight proportions of glycerol as a foaming agent and water glass as an enveloping material as well as the weight ratio of these liquid additives, the literature provides contradictious data. In the most cases, the glycerol proportion in the starting mixture is 1-1.5%, excepting the experiments presented in the paper [8], where the optimal ratio is 4%. Instead, the water glass is used in a very wide range from 2% up to 12%, the maximum value being the one commonly used by Glapor Werk Mitterteich Company in industrial processes for manufacturing the foam glass gravel [5]. It must be considered that all the manufacturing processes mentioned above used conventional energy sources.

The experiments shown in the present paper were oriented towards relatively low proportions of glycerol (1-1.6%) and water glass (8-10.1%).

Except the published data, tests with glycerol up to 2.5% and water glass up to 12% were also carried out, but the best results were obtained with the proportions mentioned above, under the conditions in which the glycerol/ water glass weight ratio was between 1/ 6.3 – 1/ 8.

When using liquid foaming agents, such as glycerol, the role of the solid raw material granulation on the duration of the sintering/ foaming process is very important. It was experimentally found that the granulation of the glass waste < 150 μm compared to a granulation < 250 μm of the same waste type can lead to a reduction of the duration by about 20%.

4. CONCLUSION

High mechanical strength lightweight material as a foam glass gravel was experimentally manufactured from container glass waste, glycerol, sodium silicate (“glass water”) and water by a microwave heating technique at high temperature (815-823 ºC).

Unlike the conventional heating methods commonly used in industrial manufacturing processes and also in experimental tests presented in the literature, the authors applied the heat generation in the powder mixture based on glass waste by the microwave irradiation.

Although known since the middle of the 20^{th} century, the microwave heating process was not industrially applied only to an extremely small extent, despite the known energy and ecological advantages, being fast, “clean” and economical. The paper originality consists in the use of the microwave energy in a sintering / foaming process of glass waste exclusively powered with conventional energy sources.

The glycerol together with sodium silicate (“water glass”) and water in various weight proportions forms the homogeneous liquid component of the powder mixture. This liquid component has the property of homogeneously filling the volume occupied by the glass waste, so that the product will have a very fine porosity uniformly distributed.

The characteristics of the foam glass gravel produced with glycerol (between 1.0-1.6%) and water glass (between 8.0-10.1%) are: apparent density between 0.21 and 0.24 g/ cm^3, porosity over 89.1%, thermal conductivity in the range 0.057-0.063 W/ m·K, compressive strength between 4.8 and 5.9 MPa, water absorption below 1.1% and pore size between 0.3 and 0.9 mm. These characteristics are almost similar to those of industrially made foam glass gravel.

Although carried out under experimental conditions, on a very small microwave oven, the manufacturing process of foam glass gravel has recorded specific energy consumptions of 0.81-0.88 kWh/kg, comparable to those of the industrial manufacture of similar products on high capacity ovens in continuous regime. According to the literature, the use of high power microwave equipment (industrial type) could have higher - with up to 25% - energy efficiency.

Adopting the glycerol as a foaming agent was justified in economic terms by its very profitable market price compared to the price of the main materials commonly used as foaming agents (calcium carbonate and silicon carbide), considerably higher (3-4 times).

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