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RECYCLED GLASS WASTE EXPANDED BY A MICROWAVE HEATING TECHNIQUE

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The authors present in this paper an original technology for the recovery of glass waste. From the glass waste by heating in the microwave oven, melting and transforming the initial load into a glass foam are performed. The heating method is based on microwave irradiation, a technique used by some industrial producers. Laboratory experiments have resulted in products with physical, mechanical and morphological characteristics, such as those manufactured industrially (compressive strength of 5.5 MPa, thermal conductivity of 0.06 W/m K, porosity of 86.8%, with homogeneous pores with dimensions between 1.0-1.6 mm). The specific energy consumption was very low (0.83 kWh/kg) compared to the industrial one, with the theoretical possibility of reduction with maximum 25%.

Keywords: foam glass gravel, packaging glass waste, colored glass, foaming process, compressive strength

1. Introduction

The recycling of plastic, metal, paper and glass waste has become a global concern since the last decades of the 20th century. A large amount of glass waste is generated annually in the world. In 2017, 14 million tons of glass waste materials were generated only in Europe [1]. Most of it is the post-consumer packaging glass. According to The European Container Glass Federation [2], in 2017 the recycling rate for glass packaging in UE was of 76%. Also, the flat glass cullet coming from demolition or rehabilitation of buildings is an important source of glass waste.

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The global recycled glass market in 2017 was evaluated at over $ 3500 million [3]. These types of waste are composed of soda-lime glass, the most common form of glass, which is also the most suitable for recycling and reuse, mainly in the glass industry as a raw material for the manufacture of new glass and, more recently, as a raw material for the production by a sintering-foaming process of some cellular glasses usable as substitutes for existing building materials.

Generally, processing the glass waste containing various colored materials that need to be separated for the manufacture of new glass involves high costs. In the case of the cellular glass manufacture, this separation is not necessary. This was the main reason for the orientation towards the industrial manufacture of the glass foam, as well as the unique physical and mechanical characteristics of this product which simultaneously incorporates light weight, low thermal conductivity, acceptable or even high mechanical strength for a porous material and other properties such as fire resistance, impermeability, non-toxicity, chemical stability, non-deformability, resistance to the attack of rodents, insects, bacteria, etc.

The cellular glass made of glass waste can cover a wide range of properties, depending on the manufacturing recipe, the nature of the foaming agent and the mineral additions, the process temperature and duration, the heating and cooling rate. These parameters can influence the physical, mechanical and morphological characteristics of the products at the end of the sintering-foaming process [4]. Thus, very light porous materials with an apparent density below 0.15-0.20 g/ cm3, high porosity and acceptable compressive strength up to 1.2-1.25 MPa, are suitable for their use, mainly as thermal and acoustic insulation panels, floors. and wall tiles, architectural panels, filters, absorbers, gas sensors, etc.

Also, relatively lightweight products with an apparent density of over 0.30-0.35 g/ cm3, fine and homogeneous porosity and much higher compressive strength (up to 6 MPa) are used as filling material in road and railway construction, foundation infrastructure, roof gardens, bridge abutments, insulating of underground pipelines and storage tanks, mechanical shock resistant- exterior panels, etc. [4]. This type of porous materials with high mechanical strength, produced in the form of pieces with dimensions up to 80 mm, are called foam glass gravel. There are industrial plants in several European countries for the production of foam glass gravels, different as the manufacturing recipe and possibly as an appearance, especially in the case of products made with glycerol as a foaming agent (which are generally, black), but very similar in physical and mechanical properties. The information provided by the literature is extremely limited, especially regarding the specific energy consumption.

What is well known is that all plants that manufacture cellular glass and particularly foam glass gravel use conventional heating techniques (burning fossil fuels or electrical resistances). The main industrial manufacturers of foam glass gravel are Geocell Schaumglas (Austria), Misapor Switzerland (Switzerland), Glapor Werk Mitterteich (Germany), Hasopor (Sweden), Foamit (Finland).

Geocell Schaumglas is one of Europe's leading producers of foam glass gravel. The company owns four plants, two in Austria and two in Germany [5]. The recycled glass represents the basic raw material, of which 90 wt.% is colored post-consumer packaging glass and 10 wt.% is colorless flat glass waste. The literature does not specify the foaming agent. The process temperature is below 900 ºC, achieved in a conveyor belt tunnel furnace. The main characteristics of the foamed product are bulk density of 0.15 g/ cm3, thermal conductivity of 0.08 W/m·K and compressive strength of 5.7 MPa.

The International Group of Companies Misapor Switzerland manufactures foam glass gravel in several plants, two in Switzerland, one in Italy and one in Germany [6]. Manufacturing recipes include 98 wt.% recycled glass (mixed glass waste and green packaging glass waste) and 2 wt.% calcium carbonate, silicon carbide or gypsum as foaming agents separately used [7]. The technological process is carried out in conveyor belt tunnel furnaces at temperatures between 800-1000 ºC depending on the nature of the foaming agent. The Misapor foam glass gravels have bulk density between 0.16-0.20 g/ cm3 (gross density between 0.24-0.37 g/cm3), thermal conductivity in the range 0.075-0.095 W/m·K and compressive strength between 4.9-6.0 MPa [8-10].

Another manufacturer of foam glass gravel is the German company Glapor Werk Mitterteich [11]. The basic raw material is the recycled glass composed of post-consumer packaging glass or flat glass waste in weight ratio of 87%. A liquid carbonic foaming agent (glycerol) is used in a weight ratio of 1%, associated with 12% sodium silicate aqueous solution and a very low addition of kaolin.

The main features of Glapor foam glass gravel are the bulk density between 0.13-0.21 g/cm3, the thermal conductivity below 0.12 W/m·K and the compressive strength up to 6.0 MPa. The porosity of the pieces is very fine, the pore size being below 300 μm. The Swedish company Hasopor [12] manufactures foam glass gravel especially as filling material for road construction. The recycled glass from post-consumer packaging glass is the raw material. The nature of the foaming agent is not specified in literature.

The heating process is performed in conveyor belt tunnel furnaces at about 1000 ºC. During the process the glass-based material expands growing in volume 4-5 times. The bulk density of the foam glass gravel is about 0.18 g/cm3.

In the Nordic countries (Scandinavia), the main market for foam glass gravel is road construction due to harsh weather conditions. The foam glass gravel should prevent the road degradation caused by the freeze-thaw sequence. The

Finnish company Foamit, like Hasopor, industrially produces materials for this purpose using recycled glass. Pieces with dimensions between 3-60 mm, bulk density between 0.18-0.21g/cm3 and thermal conductivity in the range 0.08-0.13 W/m K [13] are produced by the Finnish company. As noted above, all global production of cellular glass and particularly foam glass gravel is based on conventional heating technique.

An advanced heating method, known since the mid-20th century, is the use of the microwave energy. This unconventional technique was applied in the household for the food preparation, but its industrial use was done extremely limited.

According to the literature [14], it was experimentally found in the last 10-15 years that several material types are suitable for the microwave heating (organics, ceramics, metals, polymers, glass, etc.), but the industrial application of this technique is still in an inconclusive stage. The microwave irradiation of glass was considered inadequate in 1997 [15] due to the high content of some chemical compounds (SiO_2, Al_2O_3) that are not microwave susceptible and negatively influences the efficiency of the microwave heating starting at room temperature up to about 500 ºC. Above this temperature, the dielectric characteristics of the glass (e.g. electrical conductivity) are greatly improved and the heating becomes very efficient. For this reason, the paper [16] considered that applying this technique in the UK to existing industrial furnaces for the manufacture of glass foams would not be cost effective, because it would involve the use of two heating systems on the same furnace, a conventional one in the first zone (up to 500 ºC) and the other unconventional in the second zone (from 500 ºC to the final temperature of 800-1050 ºC). This idea, theoretically correct, is practically incorrect, due to the inherent presence of some contaminants (Fe_2O_3, Cr_2O_3, etc.) in the composition of commercial glass, which makes up most of the glass waste used as raw material. These contaminants are microwave susceptible and allow the heating of the glass with maximum efficiency starting from room temperature [17]. Experimental demonstration of this theory has been performed by some authors of this paper [18]. In the last three years, they performed several tests on the experimental basis of the Romanian company Daily Sourcing & Research Bucharest, aiming sintering-foaming processes in the microwave field on laboratory equipment adapted to this purpose.

The aim of the research whose results are presented in the paper was experimentally determined the influence of the composition of the glass-based raw material on the characteristics of foam glass gravel produced in microwave field.

2. Experimental

The principle of producing a cellular glass consists in the thermal treatment at high temperature (800-1050 ºC) of a mixture with fine granulation composed of glass waste, a foaming agent and possibly other mineral additions with fluxing role. The foaming agent has the role to release a gaseous compound following a decomposition or oxidation reaction at a temperature correlated with the softening temperature of the raw material. Thus, the gas in the form of bubbles remains trapped in the viscous mass of the material and then by cooling it forms a homogeneous porous structure. The foaming agent adopted in this work was calcium carbonate. By the thermal decomposition at over 800 ºC, it releases CO_2 that contributes to the foaming process and CaO that enters the composition of the glass [4].

The microwave heating is completely different compared to the conventional heating, being initiated in the core of the material, which absorbs electromagnetic energy and converts it into heat. Thus, the heating process is carried out volumetrically and is very fast [19]. The microwave heating eliminates the need to use energy for heating the massive components of the furnace (walls, vault, hearth, etc.) as in the case of the conventional heating with fuel or electrical resistances. Thus, a "selective" heating, characteristic of an unconventional heating, is performed, whereby only the targeted material receives the electromagnetic waves [19].

Previous tests [18] have shown that the direct microwave heating of the glass-based mixture causes the destruction of its internal structure due to the aggressive effect of this type of heating. The technical solution adopted was the placement between the material and the source of microwave emission of a ceramic screen from a highly microwave susceptible material, in the form of a crucible or tube with a lid, having an adequate wall thickness (3.5-5 mm).

The role of the screen is to take over a part of the microwave field through absorption (converted into thermal energy) and thus to decrease the intensity of the field that penetrates the ceramic screen and comes in direct contact with the material. Thus, a mixed heating is achieved, being composed of the direct heating described above and an indirect heating of the material by the radiation of the microwave-absorbent wall, heated quickly and intensely.

The experimental microwave equipment was a 0.8 kW-microwave oven commonly used in the household, adapted to operate at high temperatures (up to 1200 ^{o}C). The use of ceramic fiber mattresses for the outer protection of the ceramic tube and lid containing the pressed powder material was extremely important. The temperature control of the material was performed with a radiatiopyrometer, according to Fig. 1.

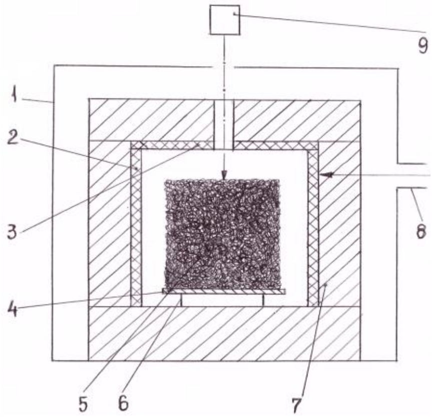


Fig.1 Constructive scheme of the experimental microwave equipment
1 – 0.8 kW microwave oven; 2 – ceramic tube; 3 – ceramic lid; 4 – metal plate;
5 – pressed glass-based raw material; 6 – metal support; 7 – ceramic fiber mattress;
8 – microwave generator waveguide; 9 – radiation pyrometer

The materials used in experiments were post-consumer packaging glass waste (containing various ratios of colorless, green and amber glass) calcium carbonate as a foaming agent, borax as a fluxing agent and a 30% aqueous solution of sodium silicate as a binder. Due to the high Na2O content, the most important chemical compound with fluidizing properties, especially for the glass industry, borax is used to reduce the softening point of the glass-based mixture.

On the other hand, the important boron content (about 11%) favors the mechanical properties of the product. In addition to the binder role, the sodium silicate solution has a strong adhesive role, also contributing to the increased mechanical strength of foam glass gravel. The chemical composition of the three glass waste types [21, 22] is shown in Table 1.

Table 1

Chemical composition of the glass waste types

Chemical composition	Packaging glass waste (wt.%)		
	Colorless	Green	Amber
SiO_2	71.7	70.5	71.2
Al_2O_3	1.9	1.8	1.9
CaO	12.0	10.15	10.35
Fe_2O_3	0.05	0.45	0.35
MgO	1.0	2.75	2.6
Na_2O	13.3	12.95	13.15
K_2O	-	0.45	0.6
Cr_2O_3	0.05	0.25	0.06
SO_3	-	0.25	0.3

The glass waste was crushed, ground in a ball mill and sieved at the grain size below 150 μm. Calcium carbonate had the grain size below 40 μm and the borax purchased from the market with a granulation below 400 μm was ground and sieved at dimensions below 150 μm. The foam glass gravel samples were characterized by traditional analysis methods. The apparent density was measured by the gravimetric method [23]. The porosity was calculated by the comparison method [24] of the density of the porous sample and the density of the same material type in compact state. The determination method of the thermal conductivity [25] consisted of measuring the thermal flow value that passes through a sample placed between two metal plates one heated and the other cooled. The compressive strength was determined by applying an axial force on the sample. The water absorption of the porous sample was measured by the traditional method of its water immersion (ASTM D 570). The microstructural configuration of the foam glass gravel samples was identified with a Smartphone digital microscope.

3. Results and discussions

The experiments aimed to determine the influence of the composition of the glass waste on the characteristics of the foam glass gravel produced in the microwave field. Four experimental variants were adopted (Table 2), in which the weight ratio between colorless and green glass was successively 50/50, 75/25 and 85/15 and in the last variant it was used a ratio between the colorless, green and amber glass of 50/20/30, which is specific to the proportions of the recycled glass in Romania. The weight ratios of calcium carbonate, borax and sodium silicate were kept almost constant, with insignificant variations.

Table 2

Composition of the experimental variants

Variant	Packaging glass waste (wt.%)				Calcium carbonate (wt.%)	Borax (wt.%)	Sodium silicate (wt.%)
	Colorless	Green	Amber	Total			
1	44.00	44.00	-	88.0	2.0	3.5	6.5
2	65.97	22.03	-	88.1	2.0	3.4	6.5
3	74.63	13.17	-	87.8	2.0	3.4	6.8
4	43.90	17.56	26.34	87.8	2.0	3.5	6.7

Tables 3 and 4 present the functional parameters of the sintering-foaming process of the four variants and the physical, mechanical and morphological features of the samples 1-4, respectively.

Analyzing the data in Table 3, it can be observed that the highest heating rate (20.5 ºC/ min) corresponds to the manufacturing process of foam glass gravel using a mixture of colorless and green glass waste in the 85/15 ratio (variant 3), where the final temperature of the process was the lowest (820 ºC). Functional parameters quite close were obtained by variant 4, which used a mixture of colorless, green and amber glass waste in the 50/20/30 ratio, the process temperature being 825 ºC and the heating rate 20.1 ºC/ min. The index of volume growth of the raw material was very close in the case of the two variants above mentioned (1.70-1.75).

Table 3

Functional parameter of the sintering-foaming process

Variant	Dry raw material/ foam glass gravel quantity (g)	Sintering-foaming temperature (ºC)	Heating time (min)	Average rate (ºC/ min)		Index of volume growth	Specific energy consumption (kWh/ kg)
				Heating	Cooling		
1	580/ 564	840	43	19.1	5.6	1.50	0.89
2	580/ 563	835	41	19.9	5.8	1.65	0.85
3	580/ 563	820	39	20.5	5.8	1.75	0.81
4	580/ 565	825	40	20.1	5.7	1.70	0.83

What is remarkable in the data in Table 3 is the very low specific energy consumption, all four variants falling within the range 0.81-0.89 kWh/ kg, variant 3 having the minimum consumption and variant 4 having 0.83 kWh/ kg. The literature does not provide information regarding this functional parameter achieved by conventional heating techniques in industrial production. An approximate consumption of foam glass gravel manufacture can only be deduced from the work [16] at about 0.85 kWh/ kg. It would be considered the observation formulated in [14], according to which a high power industrial microwave equipment could lead to an increase of energy efficiency by up to 25% compared to a 0.8 kW-oven commonly used in household similar to the one in which the experiments described above were carried out.

Table 4 clearly shows that the sample representing variant 4 brings together the physical, mechanical and morphological features required for a piece of foam glass gravel produced with a solid foaming agent.

Table 4

The main physical, mechanical and morphological features of samples

Variant	Apparent density (g/ cm^3)	Porosity (%)	Thermal conductivity (W/ m·K)	Compressive strength (MPa)	Water absorption (%)	Pore size (mm)
1	0.42	80.9	0.080	6.0	1.2	1.0 – 1.5
2	0.32	85.5	0.069	5.3	1.4	1.2 – 1.8
3	0.40	81.2	0.077	4.7	1.5	1.5 – 2.5
4	0.29	86.8	0.060	5.5	1.4	1.0 – 1.6

The porosity of the sample is high (86.8%) having the pore size between 1.0-1.6 mm. The compressive strength is the highest with a value of 5.5 MPa like the best industrially manufactured foam glass gravel pieces (generally below 6

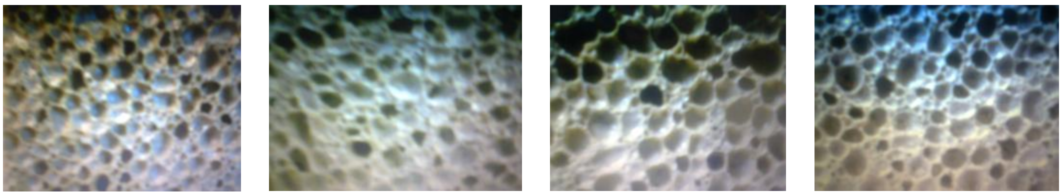
MPa). Also, the sample has a low thermal conductivity (0.06 W/ m·k), that indicates the quality of a very good insulating material.

According to the literature, the thermal conductivity value of the best industrially manufactured foam glass gravel is in the range 0.06-0.08 W/m·K. The apparent density of the sample corresponding to variant 4 has the value 0.29 g/ cm3, being the lowest compared to that of the samples made by the other variants. Because there were not enough experimentally manufactured foam glass gravel pieces, the bulk density could not be determined to compare with similar data shown in the literature. Only in [8], it is mentioned the gross density value of the pieces of foam glass gravel manufactured by Misapor (0.24-0.37 g/ cm3) equivalent to a bulk density between 0.16-0.20 g/ cm3. Therefore, it can be concluded that variant 4 is like industrially manufactured products using conventional heating techniques.Pictures representing the foam glass gravel pieces experimentally produced by the unconventional heating technique are shown in Fig. 2.

Sample 1 Sample 2 Sample 3 Sample 4

Fig. 2. Pictures of the foam glass gravel samples

The microstructural configuration of the samples is presented in Fig. 3.



Sample 1 Sample 2 Sample 3 Sample 4

Fig. 3. Microstructural configuration of the samples

The homogeneity of the pore distribution and of their dimensions is obviously, contributing to the uniformity of the thermal insulation ensured by this samples. Some peculiarities of the powder mixtures used in the cellular glass manufacture favor the microwave absorption during the direct heating. Thus, important concentrations of alkali (Na2O, K2O) in these mixtures allow a very efficient heating in the microwave field due to the high electrical conductivity that favors the absorption of the waves [17]. The packaging glass used in experiments, regardless of its color, contains about 13% Na_2O (Table 1). Also, the use of borax as a fluxing agent with a content of about 11% Na_2O increases the efficiency of the microwave heating [26].

Higher heating rate and lower energy consumption are the main advantages of the unconventional method used in the experiments presented in the paper. These parameters have been highlighted in the last three years by the results of the own tests performed on an experimental scale.

4. Conclusions

A foam glass gravel was experimentally produced using different types of post-consumer packaging glass, calcium carbonate as a foaming agent, borax as a fluxing agent and an aqueous solution of sodium silicate by an unconventional heating technique at high temperature (between 820-840 ºC).

Quantitatively, the manufacturing recipe was kept almost constant containing 87.8-88.0 wt.% glass waste, 2 wt.% calcium carbonate, 3.4-3.5 wt.% borax and 6.5-6.8 wt.% sodium silicate. Qualitatively, the composition of the glass waste was varied and contained three weight ratios between colorless and green glass waste (50/50, 75/25 and 85/15, respectively) and a weight ratio between colorless, green and amber glass waste (50/20/30), constituting four experimental variants in this order. The determination of the physical, mechanical and morphological characteristics of the samples experimentally produced aimed their comparison with the characteristics of the similar products industrially manufactured in the world. The best of the four samples corresponding to the variant in which colorless, green and amber glass was used, had the apparent density 0.29 g/ cm^3, the porosity 86.8%, the thermal conductivity 0.06 W/ m·K, compressive strength 5.5 MPa, water absorption 1.4% and pore size between 1.0-1.6 mm evenly distributed.

Compared to the characteristics of the foam glass gravel industrially produced presented in the literature, the sample obtained by microwave heating is similar. The specific energy consumption of the unconventional heating process was very low (0.83 kWh/ kg), approximately at the level of the industrial energy consumption. According to the literature, a microwave equipment on an industrial scale would allow to reduce energy consumption by up to 25% compared to a low power oven, which would confirm the energy advantage of using the unconventional technique.

The originality of the paper consists in the new approach of the thermal process of sintering-foaming of the glass waste using the unconventional technique of microwave heating, unlike the conventional techniques applied in the industrial production of foam glass gravel. The industrial application of the unconventional technique involves several intermediate research stages using microwave equipment with significantly higher powers and higher production capacities. The results presented in the paper and those previously obtained are a guarantee of the possibility of application on an industrial scale.

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