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FOAM GLASS GRAVEL FROM RECYCLED GLASS WASTE PRODUCED WITH THE MICROWAVE ENERGY

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ABSTRACT: The results of the research in the field of microwave energy application in the process of manufacturing foam glass gravel from colored glass waste, calcium carbonate as a foaming agent, borax as a fluxing agent and sodium silicate solution as a binder are presented in the paper. The thermal process of sintering/ foaming the powder mixture was carried out in a 0.8 kW-domestic microwave oven adapted for high temperature operation. The foamed product at 845 ºC, with 1.5% calcium carbonate, 7% borax and 8% sodium silicate, reached the highest value of compressive strength (9.5 MPa), thermal conductivity 0.105 W/ m·k, apparent density 0.80 g/ cm^3 and a homogeneous distribution of pores in the material structure with dimensions below 1 mm.

KEYWORDS: foam glass gravel, microwave, calcium carbonate, borax, sodium silicate, compressive strength.

1. INTRODUCTION

Foam glass gravel is a cellular material with low density and high compressive strength made from recycled glass waste, being suitable for load-bearing thermal insulation under foundation or floor as well as for insulation around the perimeter of the building. It is also applied in the structure of road and rail constructions, insulation of heating pipelines, insulation of underground storage tanks, etc. It is used in the form of pieces with dimensions between 10-90 mm obtained from the heat treatment at the high temperature of the powder mixture formed from recycled glass waste and low mass ratios of mineral additives with foaming or facilitating foaming role of the glass. The industrial manufacture of foam glass gravel was started in the mid-80s of the last century in several European countries, mainly in Switzerland, Germany and Austria [1, 2]. The great industrial manufacturers use different recipes and produce glass foams with particular characteristics. Thus, the companies of the Misapor concern use about 98% recycled glass waste and 2% different foaming agents according to their own recipe (gypsum, limestone or silicon carbide). Bulk density of the foam glass gravel is very low (0.13-0.21 g/ cm^3), high mechanical strength (4.9-6.0 MPa) and the foaming temperature is between 700-900 ºC [2-4]. Geocell Schaumglas GmbH with manufacturing facilities in Austria and Germany, one of the European leaders of foam glass gravel, uses as raw material 90% colored container glass waste and 10% colorless flat glass waste. The thermal conductivity of the product is about 0.08 W/ m·K, the bulk density is extremely low (below 0.15 g/ cm^3) and the compressive strength exceeds 5 MPa [5, 6]. Glapor Werk Mitterteich GmbH uses a basic recipe composed of 87% recycled glass waste, 12% sodium silicate ("water glass"), 1% glycerin as a foaming agent and minor mass ratios of kaolin. By combining the physical characteristics of the glass with the insulating properties of a closed cellular structure, Glapor cellular glass products have high compressive strength (over 5 MPa), are lightweight (0.11-0.14 g/ cm^3), fire resistant and rodent resistant. The thermal conductivity has values between 0.052-0.058 W/ m·K [7]. Glamaco company based in Coswig (Germany) also produces foam glass gravel using recycled glass waste (about 95%), to which is added sodium silicate, glycerol, calcium carbonate, water and kaolin. The raw material comes from its own glass melting facilities, and as an alternative source from treated recycling flat or container glass. The final product, with dimensions between 10-60 mm, has a bulk density between 0.14-0.20 g/ cm^3, thermal conductivity between 0.06-0.08 W/ m·K and high compressive strength (4-6 MPa). The physical, mechanical and morphological characteristics of the foam glass gravels produced by Glamaco are similar to those made in the other factories mentioned above.

Excepting the industrial facilities producing foam glass gravel, experimental results of some research teams are also known from the literature. Thus, the physical properties and the microstructure of a new type of cellular glass-ceramic obtained from borosilicate glass with different Sb_2O_3 contents were investigated in the paper [8]. The powder mixture used for the production of the cellular glass-ceramic with high mechanical strength was constituted by a borosilicate glass waste, different mass proportions of Sb_2O_3 between 0 - 1.2%, the optimum value being experimentally 0.9%, black carbon (1 %) as a foaming agent and 6% disodium phosphate (Na_2HPO_4) as an additive. The heating was carried out in a conventional oven, in which a graphite crucible containing the processed powder was inserted. The heating speed was very low, only 5 ºC/ min, the optimum sintering temperature being 775 ºC. According to these parameters, the apparent density was 0.408 g/ cm^3, the porosity 84.6%, and the water absorption was 1.57%. The compressive strength of the product reached the value of 4.4 MPa. According to the literature, a technique to improve the compressive strength of foamed glass products is the use in the raw material mixture of a sodium silicate solution. The work [9] presents experimental results obtained by heat treatment at 750 ºC of a mixture composed of 80% industrial glass waste (from the manufacture of flat glass), 20% carbon fly ash, calcium carbonate (1%) and sodium silicate solution (10%) as a binder. The compressive strength of the product thus obtained reached 6 MPa, the apparent density was 0.42 g/ cm^3, and the water absorption was 2.1%. According to [10], the sodium silicate introduced into the glass waste homogenizes the chemical composition and the most important technological properties of the material. Due to the chemical reaction between the glass water and the surface of the glass waste particles, silicates are formed containing chemically bound water. It is released at temperatures of 600-620 ºC and facilitates the formation of the cellular product. The glass water present in the powder mixture increases the amount of glass phase and decreases the tendency of the glass towards crystallization. Another paper [11] presents an experimental technique for the manufacture of glass-ceramic foams with high mechanical strength using 60% recycled glass waste, 40% fly ash, and supplementary 30% borax and 0.5% calcium carbonate. The sintering and foaming temperature was 800 ºC for 45 min. The obtained product had a bulk density below 0.46 g/ cm^3, the compressive strength over 5 MPa and the thermal conductivity about 0.36 W/ m·K.

All the manufacturing techniques presented above, both industrial and experimental ones, were based on conventional heating methods using the energy released by burning fossil fuels or that of electrical resistances. A fast, economical and clean technique using microwave energy, too few industrially applied so far, has recently been tested [12] by the Romanian company Daily Sourcing & Research. A manufacturing recipe comprising 90.5-91.7% colorless container glass waste, 3-5% calcium carbonate as a foaming agent and 4.5-5.3% borax as a fluxing agent was used. From the point of view of the mechanical strength of the foamed material, the best combination of raw material and additives was the one that contained 91.7% glass waste, 3.0% calcium carbonate and 5.3% borax. The sintering/ foaming temperature was 837 ºC, the heating rate 19.3 ºC/ min and the specific energy consumption 1.02 kWh/ kg. The following product characteristics corresponded to these functional parameters of the microwave oven: apparent density 0.89 g/ cm^3, porosity 59.5%, thermal conductivity 0.106 W/ m·K, compressive strength 6.0 MPa, water absorption 0% and the pore size 0.5-0.7 mm, largely similar to those of materials of this type produced by conventional methods.

The current paper presents new experimental results of the Daily Sourcing & Research company in the field of foam glass gravel production with high mechanical resistance using microwaves as an energy source.

2. METHODS AND MATERIALS

2.1 Methods

According to the literature [1], the conventional technique used for the industrial manufacture of foam glass gravel consists of loading the pressed powder mixture onto the metal conveyor belt of a tunnel furnace, powered with fossil fuel or electricity, in the case of the use of electrical resistances. By ensuring inside the oven of a suitable high temperature, depending on the nature of the foaming agent and the influence of some additives, the sintering and foaming process of mixture is achieved. The free cooling on the conveyor belt causes internal stresses in the material, facilitating its cracking and breaking in mechanically stabilized pieces smaller than 80 mm, which fall at the end of the belt.

The method adopted for the experimental testing of the production of foam glass gravel in the microwave field, to ensure conditions as close as possible to the industrial ones, consists of using a 0.8 kW-domestic microwave oven, adapted to very high thermal stress (up to 1200 ºC) (Figure 1), in which the pressed powder mixture is freely placed on a metal plate positioned 20 mm above the ceramic fiber mattresses bed from the base of the oven, with a metal support. Outside the pressed mixture is placed concentrically a ceramic tube of a microwave susceptible material (silicon carbide and silicon nitride) with a wall thickness of 3.5 mm, allowing the microwave heating of the sample partial indirectly and partial directly. The ceramic tube has a lid of the same type of material, having a central hole of 30 mm to allow the control of the powder mixture temperature with a radiation pyrometer placed above the oven at about 400 mm in the central axis. The outer side wall of the ceramic tube and the lid are protected with several layers of ceramic fiber mattresses to avoid heat loss outside the system. A constructive scheme of the microwave equipment is shown in Figure 2. In these conditions, the additional thermal protection of the metal walls of the oven is not necessary, due to the particularities of the nonconventional heating as compared to those of the conventional heating. According to [13-16], the microwave heating is initiated in the core of the material subjected to heating, and the heat is then transferred to its peripheral areas. The presence of the 3.5 mm ceramic tube between the microwave-generating source of the oven and the pressed mixture allows, on the one hand, a partial direct heating of the material by waves penetrating the wall of the tube and, on the other hand, a partial indirect heating as a result of the thermal radiation of the heated tube surface through the absorption of electromagnetic waves. Thus, the hot zone consists of the ceramic tube and the sample inside it. The microwave field only acts on the microwave susceptible materials called dielectrics. The metal walls of the oven and the ceramic fiber layers are not heated from the waves. Therefore, an efficient thermal protection of the outer wall of the ceramic tube and its lid is required.

Figure 1 Overview of the microwave equipment

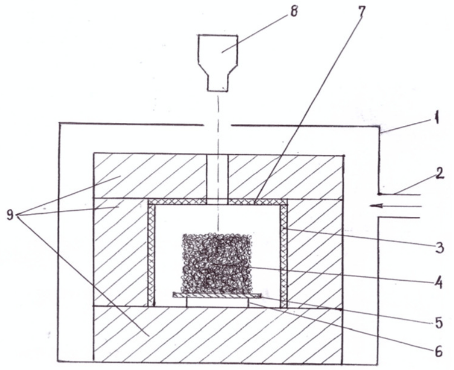


Figure 2 Constructive scheme of the microwave equipment

1 – 0.8 kW-microwave oven; 2 – waveguide; 3 – ceramic tube; 4 – pressed material; 5 – metal plate; 6 – metal support; 7 – ceramic lid; 8 – pyrometer; 9 – ceramic fiber mattress.

An experimentation methodology was adopted consisting of testing four variants of the mixture composition composed from the following materials: colored container glass waste (green and amber in equal mass ratios) as a raw material, calcium carbonate as a foaming agent, borax as a fluxing agent and aqueous solution of sodium silicate as a binder. The proportions of these materials corresponding to the four variants are presented in Table 1.

Table 1. Composition of the mixture components

Component wt.%	**Variant 1**	**Variant 2**	**Variant 3**	**Variant 4**
Colored glass waste	83.5	87.5	87.5	89.5
Calcium carbonate	1.5	1.5	1.5	1.5
Borax	7.0	3.0	8.0	5.0
Sodium silicate	8.0	8.0	3.0	4.0

The research aimed to identify the influence of borax and sodium silicate solution on the characteristics of foam glass gravel and on the functional parameters of the sintering/ foaming process, provided that the mass proportion of the foaming agent was kept constant. Therefore, the proportions of borax and sodium silicate varied between 3-8%, the share of these values alternating from variant to variant.

2.2 Materials

The chemical composition of the colored container glass (green and amber) [17] is shown in Table 2.

The recycled glass waste was broken and ground in a small laboratory grinding device, its grain size being below 250 μm. Calcium carbonate was used without other mechanical processing such as it was purchased from the market, having a very fine grain size below 40 μm. Borax has in its composition about 11% boron, which contributes to the increase of mechanical strength of the material that contains it. Also, Na_2O there is in high ratio in borax and it is considered the most active fluxing material, especially, for the glass industry. Purchased from the market at a grain size below 400 µm, it was used in experiments below 130 µm after grinding in the laboratory device. Sodium silicate, a liquid additive, is the most important of the soluble silicates. This material also called "water glass" is used as a binder. Its contribution at the increasing mechanical strength is remarkable [18].

Table 2. Chemical composition of the colored container glass

Glass type	SiO_2	Al_2O_3	CaO	Fe_2O_3	MgO	Na_2O	K_2O	Cr_2O_3	SO_3
Green	70.50	1.80	10.15	0.45	2.75	12.95	0.45	0.25	0.25
Amber	71.20	1.90	10.35	0.35	2.60	13.15	0.60	0.06	0.30

2.3 Characterization of the foam glass gravel samples

The foam glass gravel samples, resulted after the sintering/ foaming experimental process, were tested in laboratory to determine the physical, mechanical and morphological characteristics. Apparent density, porosity, thermal conductivity, compressive strength, hydrolytic stability and water absorption were determined by common methods of analysis. Thus, the apparent density was determined by the gravimetric method [19]. The porosity was calculated by the comparison method of the true and apparent density of the material, experimentally measured [20]. The compressive strength was determined with an uniaxial press and the thermal conductivity was measured by the guarded-comparative-longitudinal heat flow technique, according to ASTM E 1225-04. The hydrolytic stability of the samples was determined by the standard procedure ISO 719: 1985 [21, 22]. The water absorption of the sample was measured by the method of its immersion in water.

3. RESULTS AND DISCUSSION

3.1 Results

The functional parameters of the sintering/ foaming process are presented in Table 3.

Table 3. Parameters of the sintering/ foaming process

Parameter	Variant 1	Variant 2	Variant 3	Variant 4
Raw material/ foam glass gravel amount (g)	570/ 551	570/ 548	570/ 553	570/ 553
Sintering/ foaming temperature (ºC)	845	855	835	835
Heating duration (min)	41	44	38	39
Average rate (ºC/ min) - heating - cooling	 20.1 5.6	 19.0 5.4	 21.4 5.3	 20.9 5.5
Index of volume growth	1.25	1.30	1.35	1.50
Specific energy consumption (kWh/ kg)	0.99	1.07	0.92	0.94

Analyzing the data in Table 3, it is observed that the borax as a fluxing agent influenced the value of the process temperature, the highest proportions (7 and 8%) lowering the temperature level by at least 10-15 ºC. Implicitly, the heating duration was influenced by the proportion of borax in the materials mixture, the lowest (38 min) being reached at its maximum proportion of 8% (variant 2). The heating rate had high values between 19.0-21.4 ºC/ min, contributing to obtaining the specific energy consumption around 1 kWh/ kg. In general, the literature does not provide information regarding the energy consumption of the manufacturing processes of foam glass, neither industrial, nor experimental. According to a UK market study [23], an average consumption of the Misapor consortium relative to the volume unit of foam glass is 100 kWh/ m^3, which means up to 0.83 kWh/ kg. It should be mentioned that an continuous production flow is strongly energetically favored compared to an experimental process carried out on a small and discontinuous equipment. According to [24], an industrial microwave oven with a large capacity would be significantly more energy efficient (up to 25%) compared to a small oven of the type used in the household.

Table 4 presents the physical, mechanical and morphological characteristics of the four samples of foam glass gravel experimentally made.

Table 4. Physical, mechanical and morphological characteristics of the samples

Characteristic	Variant 1	Variant 2	Variant 3	Variant 4
Apparent density (g/ cm^3)	0.80	0.62	0.64	0.45
Porosity (%)	63.6	71.8	70.9	79.5
Thermal				

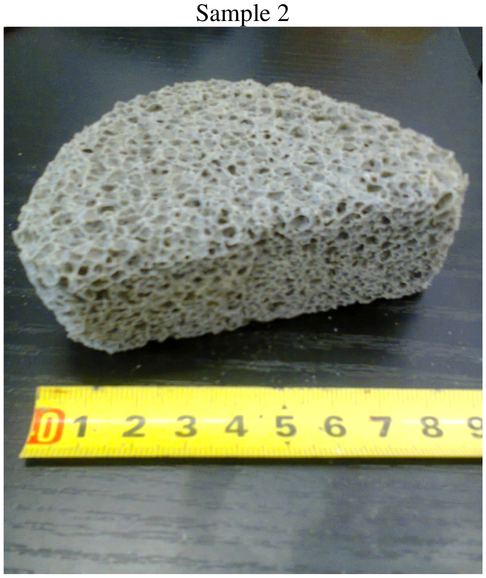
conductivity (W/ m·K)	0.105	0.087	0.090	0.071
Compressive strength (MPa)	9.5	7.4	5.6	3.5
Water absorption (%)	8.3	7.4	4.7	4.8
Pore size (mm)	0.8-1.0	1.0-1.6	1.2-2.0	2.0-3.5

The table clearly shows that the addition of the sodium silicate solution in the powder mixture allows a high compressive strength of the foamed product. It is also obvious that the cumulative addition of sodium silicate and borax contributes to the significant increase of this mechanical characteristic. Thus, if the use of 8% sodium silicate and 3% borax leads to a compressive strength of 7.4 MPa, cumulative additions of 8% sodium silicate and 7% borax allow a maximum resistance of 9.5 MPa. The apparent density of the samples has relatively high values (between 0.45-0.80 g/ cm^3), the maximum value corresponding to the maximum mechanical strength (variant 1). The thermal conductivity is in a range (0.071 - 0.105 W/ m·K) that characterizes a good insulating material. Water absorption is within moderate limits (4.7-8.3%), considering that high proportions of sodium silicate favor water absorption [18]. From the morphological point of view, the manufactured samples of foam glass gravel have a homogeneous cellular structure, with the size of pores measured at the level of the outer surface (due to the high hardness) below 2 mm (for variants 1-3) and between 2-3.5 mm (variant 4).

Images of foam glass gravel samples are shown in Figure 3.

Sample 1

Sample 2



Sample 3

Sample 4

Figure 3 Images of foam glass gravel samples

Morphologically, the foam glass gravel samples are similar to those industrially manufactured.

For determining the hydrolytic stability, 0.15 ml of 0.01 M HCl solution was used to neutralize the extracted Na_2O. The test showed that the stability joins in the second hydrolytic class.

3.2 Discussion

Theoretically, the microwave heating process of powder glass mixture should be obstructed at low temperatures due to the content of the transparent microwave materials (SiO_2, Al_2O_3). The electrical conductivity of the material, which decisively influences the formation of the dielectric, increases with temperature, but practically only at about 500 ºC reaches a value at which the microwave heating process becomes energy efficient. This theory developed by Knox and Copley [25] in 1997 completely blocked any initiative of manufacturers and researchers to use microwave energy in the process of foaming glass waste. In fact, in the composition of the commercial container glass, there are inherently small proportions of contaminants (Fe_2O_3, Cr_2O_3, etc.) with microwave susceptibility even at ambient temperature, which largely nullifies the effect of silica and alumina mentioned above [14, 26]. Daily Sourcing & Research has experimentally demonstrated that both bulk glass and powder mixture containing predominantly glass can be microwave heated with maximum efficiency starting at ambient temperature [26].

Some particularities of the glass based mixture used in the manufacture of glass foam, which favors the absorption of microwaves, should be mentioned. Thus, high concentrations of alkali (especially Na_2O) in the container glass waste (about 13%) allow efficient heating in the microwave field due to the correlation between its electrical conductivity and the microwave absorption [27]. Also, the use of borax (as a fluxing agent) with a high Na_2O content helps to increase the efficiency of microwave heating.

4. CONCLUSION

The objective of the research whose results are presented in the paper is the experimental manufacture of foam glass gravel from recycled glass waste using a nonconventional energy source (microwave energy).

The process of sintering/ foaming the powder mixture consisting of colored container glass waste, as a raw material, calcium carbonate as a foaming agent, borax as a fluxing agent and sodium silicate solution as a binder took place in a 0.8 kW-domestic microwave oven adapted for high temperature operation in Daily Sourcing & Research company.

Four variants of the manufacturing recipe were tested, in which the mass ratios of borax and sodium silicate solution varied between 3-8%, while the proportion of calcium carbonate was kept constant at 1.5%.

Of the four variants, the most interesting from the point of view of the maximum compressive strength of foam glass gravel samples was the sample corresponding to variant 1, which was foamed at 845 ºC, using 7% borax and 8% sodium silicate. The compressive strength reached the maximum value of 9.5 MPa, the thermal conductivity was 0.105 W/ m·K, the apparent density was 0.80 g/ cm^3, and the pore distribution in the material structure was homogeneous, their size being less than 1 mm.

The heating rate had high values between 19.0-21.4 ºC/ min, contributing to obtaining the specific energy consumption around 1 kWh/ kg. According to the literature, an industrial microwave oven with a large capacity would be significantly more energy efficient (up to 25%) compared to a small oven of the type used in the household.

Qualitatively, the foam glass gravel experimentally microwave made is relatively similar to those industrially manufactured.

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