FOAM GLASS GRAVEL MADE FROM GLASS WASTE BY MICROWAVE IRRADIATION

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ABSTRACT

The experimental results of the manufacturing process of foam glass gravel using recycled glass waste (in very high ratio) are presented in the paper. The technique used for heating at 815-837 °C of the raw material, composed of a powder mixture of glass, calcium carbonate (between 3-5%) as a foaming agent and borax (between 4.5-5.3%) as a fluxing agent, is unconventional based on microwave irradiation. The main physical, mechanical and morphological characteristics of the glass foam (low thermal conductivity between 0.087-0.110 W/ m·K, very high compressive strength up to 6.3 MPa, practically zero water absorption, pore size between 0.7-1.7 mm), similar to those industrially manufactured by conventional methods, are suitable for load-bearing thermal insulation under foundations or floorings, for insulation around the perimeter of buildings, underground heating pipes, underground tanks, as well as a filling material in the structure of road and railway constructions.

Keywords: foam glass gravel; microwave irradiation; calcium carbonate; borax; compressive strength.

1. INTRODUCTION

A significant amount of container waste is represented by the glass waste, especially, from post-consumed drinking glass (about 70%) [1]. The most common method to recovery this waste is recycling the glass in a closed circuit for the industrial manufacture of the new glass, but this requires high costs. For this reason, in recent decades, several solutions for re-using the glass waste as a building material have been tested worldwide and adopted for the industrial manufacture of these products.

REZUMAT

În lucrare sunt prezentate rezultatele experimentale ale procesului de fabricare a pietrişului de sticlă celulară utilizând deşeuri de sticlă reciclată (în proporţie foarte înaltă). Tehnica utilizată pentru încălzirea la 815-837 °C a materiei prime, compusă dintr-un amestec pulbere de sticlă, carbonat de calciu (între 3-5%) ca agent de spumare şi borax (între 4.5-5.3%) ca agent de fluidizare, este neconvenţională, bazându-se pe iradierea cu microunde. Principalele caracteristici fizice, mecanice și morfologice ale sticlei celulare (conductivitate termică redusă între 0,087-0,110 W/m·K, foarte înaltă rezistenţă la compresiune până la 6,3 MPa, absorbtia apei practic nulă, dimensiunea porilor între 0,7-1,7 mm), similare cu cele fabricate industrial prin metode convenţionale, sunt adecvate pentru izolarea termică portantă sub fundaţii sau pardoseli, pentru izolarea în jurul perimetrului clădirii, a conductelor de încălzire subterane, a rezervoarelor subterane, precum și ca material de umplutură în structura construcţiilor rutiere și feroviare.

Cuvinte cheie: pietriş de sticlă celulară; iradiere cu microunde; carbonat de calciu; borax; rezistenţă la compresiune.

The cellular glass is made by a sintering-foaming process at high temperature of a powder mixture consisting of glass waste, a foaming agent and possibly other mineral additions that facilitate the process. At the appropriate temperature, the foaming agent releases a gas through a chemical decomposition or oxidation reaction, which should remain trapped in the viscous mass of the raw material, and by cooling forms a characteristic porous structure [2].

Depending on the type of the glass waste, but especially, on the nature of the foaming agent and mineral additions, the physical,
mechanical and morphological characteristics of the foamed products can greatly differ. Thus, it can be manufactured lightweight cellular glass, with high porosity, very low thermal conductivity and acceptable mechanical resistance, mainly used as thermal insulating material [2, 3], but also dense cellular glasses with very high mechanical resistance, used for load-bearing thermal insulation under the foundations, underground heating pipes and underground tanks or as a filling material in the structure of road and railway constructions [4, 5]. In the second category of products is the foam glass gravel, whose achievement represented the purpose of the research presented in the paper.

Several European countries (Switzerland, Austria and Germany) started to manufacture industrially the foam glass gravel after 1980. Geocell Schaumglas GmbH, with plants in Germany and Austria, is one of the main manufacturers of this product using as a raw material colored container glass waste (90%) and colorless flat glass waste (10%) [5]. Also, the Misapor Switzerland corporation with branches in Germany, France and Austria produces dense cellular glasses from glass waste with high compressive strength and low thermal conductivity for applications requiring high mechanical stresses [6]. The facilities used to manufacture the foam glass gravel are tunnel furnaces with conveyor belt heated by conventional methods (burning of fossil fuel or electrical resistances). Although the manufacturing recipes differ from company to company, being used several variants of foaming agent (limestone, silicon carbide, gypsum, glycerin) and mineral additions, all the products have compressive strength of over 5-6 MPa and low bulk density (below 0.25 g/ cm³) [5, 6].

Except the industrial manufacturers mentioned above, several experimental results obtained in the manufacture field of dense porous materials are presented in the literature. The bibliographic source [7] mentions a porous material with the apparent density of 0.46 g/ cm³, the thermal conductivity of 0.36 W/ m·K and the compressive strength over 5 MPa made from glass waste and fly ash in the mass ratio 60/ 40, calcium carbonate (0.5%) as a foaming agent and borax (30%) as a fluxing agent. Another source [8] highlights a new type of cellular glass-ceramic made from borosilicate glass waste, up to 1.2% Sb₂O₃, 1% black carbon as a foaming agent and 6% disodium phosphate (Na₂HPO₄) as an additive. The apparent density of the foamed material was 0.408 g/cm³, the porosity 84.6% and the compressive strength reached 4.4 MPa. According to the work [9], a porous material with the compressive strength up to 6 MPa and the apparent density of 0.42 g/ cm³ was obtained by the sintering-foaming process from a powder mixture containing industrial flat glass waste and coal fly ash waste in the mass ratio 80/ 20, being used calcium carbonate (1%) as a foaming agent and sodium silicate solution (10%) as a binder. As in the case of the industrial production, all of the experimental tests described above were performed using conventional heating methods.

Although a source of economic, fast and “clean” energy (the microwave energy) has been known since the middle of the last century, it is very few used in industrial heating processes [10]. In recent years, the Romanian company Daily Sourcing & Research Bucharest has successfully tested on experimental scale numerous sintering-foaming processes of recycled glass waste [3, 11, 12]. The current paper presents results obtained in the process of manufacturing the foam glass gravel using the unconventional technique of heating the raw material by microwave irradiation.

2. METHODS AND MATERIALS

2.1. Methods

The technique industrially used for the manufacture of foam glass gravel consists of depositing a layer of pressed powder mixture of raw material on the metal conveyor belt of a tunnel furnace powered by burning a fossil fuel or by thermal radiation of electrical resistances. The temperature required for the sintering-foaming process of the raw material
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is thus reached on the length of the heating zone of the furnace and the cooling of the material is achieved at a moderate speed in the area following the active zone of the furnace. At the cold end of the conveyor belt, the foamed material breaks into pieces due to the cracks created by the internal stresses.

The method adopted for the experimental scale manufacture of a material with similar characteristics by heating due to microwave irradiation consists of the use of a 0.8 kW domestic microwave oven adapted for high temperature operation (Fig. 1), in which the pressed powder material is placed on a metal plate, on the surface of the bed of ceramic fiber mattresses, deposited at the base of the oven on the rotating device. The material is protected by exclusively direct microwave irradiation with a microwave susceptible ceramic tube made of silicon carbide and silicon nitride in the mass ratio 80/20, with a wall thickness of 3.5 mm. Also, a lid of the same material is placed above the tube. This ceramic construction is very well thermally insulated on the outer walls with ceramic fiber mattresses, having the role of avoiding heat loss to the non-insulated sheet of the oven. The temperature control of the material subjected to heat is done with a radiation pyrometer, positioned above the oven in its central axis, which visualizes the sample through holes provided in the ceramic lid, the ceramic fiber mat and the upper metal wall of the oven. A single microwave generator of 0.8 kW placed on one of the side walls of the oven ensures the thermal needs of the process.

Partially, the ceramic tube and lid are penetrated by electromagnetic waves and come into contact with the microwave-susceptible raw material. On the other hand, to a lesser extent, they absorb these waves, warming up quickly and transferring the heat through thermal radiation.

The adopted experimentation methodology includes four compositional variants containing colorless glass waste (between 90.5-91.7%) as raw material, calcium carbonate (between 3-5%) as a foaming agent, borax (between 4.5-5.3%) as a fluxing agent and, in addition, a constant water addition of 10% for all tested variants. Table 1 shows the mass ratios of each component that composes the powder mixture for the manufacture of the foam glass gravel corresponding to the four variants.

![Fig. 1. The experimental microwave equipment](image)

<table>
<thead>
<tr>
<th>Variant</th>
<th>Component</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Colorless glass waste</td>
<td>91.7</td>
<td>91.3</td>
<td>90.8</td>
<td>90.5</td>
</tr>
<tr>
<td>2</td>
<td>Calcium carbonate</td>
<td>3.0</td>
<td>3.7</td>
<td>4.4</td>
<td>5.0</td>
</tr>
<tr>
<td>3</td>
<td>Borax</td>
<td>5.3</td>
<td>5.1</td>
<td>4.8</td>
<td>4.5</td>
</tr>
<tr>
<td>4</td>
<td>Water addition</td>
<td>10.0</td>
<td>10.0</td>
<td>10.0</td>
<td>10.0</td>
</tr>
</tbody>
</table>

Table 1. Composition of the powder mixture

2.2. Materials

Colorless glass waste from post-consumed drinking glass is the raw material used in experiments. The chemical composition of this waste contains: 71.7% \( \text{SiO}_2 \), 1.9% \( \text{Al}_2\text{O}_3 \), 12.0% \( \text{CaO} \), 1.0% \( \text{MgO} \), 13.3% \( \text{Na}_2\text{O} \), 0.05% \( \text{Cr}_2\text{O}_3 \) and 0.05% other oxides. The glass was broken and ground in a laboratory electrical device with a grain size below 250 \( \mu \text{m} \). Calcium carbonate was purchased from the market, being used in experiments without further processing, with the granulation below 40 \( \mu \text{m} \). Borax was purchased from the market at the grain size below 400 \( \mu \text{m} \). After grinding
in the laboratory device, its granulation was reduced below 130 μm.

2.3. Characterization of the samples
The physical, mechanical and morphological characteristics of the foam glass gravel samples experimentally carried out by microwave irradiation were determined in the laboratory using common methods of analysis. The apparent density was measured by the gravimetric method [13] and the porosity was determined by the method of comparing the true and apparent densities of the material [14]. The compressive strength was determined by tests performed on an uniaxial press. Using the guarded-comparative-longitudinal heat flow technique, according to ASTM E 1225-04, the thermal conductivity of the samples was measured. The hydrolytic stability of the foamed material was determined using the standard procedure ISO 719: 1985 [15, 16] and the water absorption was measured by the traditional method of the sample immersion in water.

3. RESULTS AND DISCUSSION

3.1. Results
The main functional parameters of the sintering-foaming process for the manufacture of foam glass gravel are shown in Table 2 and the physical, mechanical and morphological characteristics of the foamed products are presented in Table 3.

According to the data in Table 2, the amount of the material loaded in the oven and heated to 815-837 ºC was kept constant at the value of 570 g, the amount of the foamed product being 551-553 g. The sintering-foaming process was finished after 38-46 min, resulting a specific energy consumption between 0.92-1.11 kWh/ kg, when the power of the oven microwave generator was 0.8 kW. The average heating rate varied between 17.7-20.9 ºC/ min, confirming that the heating rate has much higher values in the lower temperature range (up to 650-700 ºC) and increasingly lower values in the high temperature range.

Under the conditions in which the experimental oven does not have the possibility to vary the power of the microwave generator, the amount of the loaded material and the level of the heat loss outside the system determine the heating rate. Previous own experimental results have shown that the optimum range of the heating rate value in the sintering-foaming processes of glass waste, so that a homogeneous porous structure be obtained, is between 14-20 ºC/ min, with the observation that lower values are really beneficial, but the specific energy consumption would increase beyond the profitability limit of the process.

Generally, data on the specific energy consumption of industrial processes for the manufacture of glass foams are not provided in the literature. According to a market study [17], the average conventional energy consumption in Misapor is about 0.83 kWh/ kg. On the other hand, the paper [10] considers that the energy advantages of a microwave equipment of high capacity, as compared to an oven of the type used experimentally, would allow an efficiency of up to 25%. In conclusion, the unconventional manufacturing process would be more efficient than the conventional one currently used worldwide.

Table 3 confirms that a mass ratio of 5.3% borax in the powder mixture, together with a lower weight of the foaming agent (3.0%), corresponding to variant 1, strongly influences the compression strength value of the foam glass gravel. The apparent density of the product as well as the thermal conductivity have relatively low values (0.89 g/ cm³ and 0.110 W/ m·K respectively), although they are the highest of the four samples experimentally performed.

The very low level of the water absorption (below 0.8%), practically negligible, is noticeable, this feature being important for a porous product with insulating properties. The pore distribution in the samples structure of the foam glass gravel is very homogeneous at the variants 1-2, the pore size varying between 0.7-0.9 mm and 0.8-1.0 mm respectively.
Table 2. Parameters of the sintering/foaming process

<table>
<thead>
<tr>
<th>Variant</th>
<th>Raw material/foam glass gravel amount g</th>
<th>Sintering-foaming temperature °C</th>
<th>Heating duration min</th>
<th>Average rate °C/min</th>
<th>Index of volume growth</th>
<th>Specific energy consumption kWh/ kg</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>570/551</td>
<td>837</td>
<td>46</td>
<td>17.7</td>
<td>1.25</td>
<td>1.11</td>
</tr>
<tr>
<td>2</td>
<td>570/553</td>
<td>833</td>
<td>44</td>
<td>18.5</td>
<td>1.30</td>
<td>1.06</td>
</tr>
<tr>
<td>3</td>
<td>570/552</td>
<td>822</td>
<td>40</td>
<td>20.1</td>
<td>1.35</td>
<td>0.97</td>
</tr>
<tr>
<td>4</td>
<td>570/553</td>
<td>815</td>
<td>38</td>
<td>20.9</td>
<td>1.40</td>
<td>0.92</td>
</tr>
</tbody>
</table>

Table 3. Physical, mechanical and morphological features of the samples

<table>
<thead>
<tr>
<th>Variant</th>
<th>Apparent density g/cm³</th>
<th>Porosity %</th>
<th>Thermal conductivity W/m·K</th>
<th>Compressive strength MPa</th>
<th>Water absorption %</th>
<th>Pore size mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.89</td>
<td>59.5</td>
<td>0.110</td>
<td>6.3</td>
<td>-</td>
<td>0.7-0.9</td>
</tr>
<tr>
<td>2</td>
<td>0.80</td>
<td>63.6</td>
<td>0.105</td>
<td>5.2</td>
<td>0.2</td>
<td>0.8-1.0</td>
</tr>
<tr>
<td>3</td>
<td>0.70</td>
<td>68.2</td>
<td>0.093</td>
<td>4.0</td>
<td>0.5</td>
<td>0.9-1.5</td>
</tr>
<tr>
<td>4</td>
<td>0.62</td>
<td>71.8</td>
<td>0.087</td>
<td>2.7</td>
<td>0.8</td>
<td>1.0-1.7</td>
</tr>
</tbody>
</table>

Pictures of the foam glass gravel samples are shown in Fig. 2.

Images of the microstructural configuration of the samples viewed with a Smartphone Digital Microscope are shown in Fig. 3.

The test for determining the hydrolytic stability was carried out according to the standard procedure ISO 719: 1985 (confirmed in 2011) with 0.15 ml of 0.01 M HCl solution. The results indicated that the stability of the
foam glass gravel samples corresponds to the second hydrolytic class.

3.2. Discussion

Previous tests have allowed determining the optimum thickness of the ceramic tube wall made of silicon carbide and silicon nitride to the value of 3.5 mm. This thickness allows the partial penetration of the wall by the microwave field, so that the direct contact between the electromagnetic waves and the mixture containing glass waste does not affect the structural homogeneity of this material. The heating of the glass waste is performed both directly, from the core of the sample to its peripheral areas, and indirectly, by the heat transfer through thermal radiation from the inner surface of the ceramic tube (which has partially absorbed the microwave radiation) to the sample mass. This mode of unconventional heating allows obtaining remarkable energy efficiency, by comparison with the method of the totally indirect heating.

4. CONCLUSION

The research, whose results are presented in the paper, aimed the production of a porous foam glass gravel with high compressive strength in conditions of superior energy efficiency using microwave energy, compared to products industrially manufactured by conventional methods.

Using a recipe containing glass waste (91.7%), calcium carbonate (3%) as a foaming agent and borax (5.3%) as a fluxing agent, a porous product was obtained by heating at 837 °C with the compressive strength of 6.3 MPa, the thermal conductivity of 0.110 W/ m·K and the apparent density of 0.89 g/ cm³.

The specific energy consumption was very low (1.11 kWh/ kg) for a discontinuous experimental process on a very low capacity oven, compared to the continuous industrial processes made in high capacity tunnel furnaces.

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