# REVIEW: MAKING STRENGTH CELLULAR GLASS

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ABSTRACT: The original non-conventional technique of preparing expanded bottle utilizing recycled bottle waste and a combination between glycerin in association with $Na_2SiO_3$ and $Na_2CO_3$, as liquid and respectively, solid expanding agents, was experimentally tested on an 800 W-electromagnetic wave oven and shown in this article. The work objective was obtaining a cellular glass with physio-mechanical and thermal features almost identical to those achieved by conventional techniques at industrial-scale. Thus, the heat conductivity was in the range of 0.060-0.072 $W \cdot m^{-1} \cdot K^{-1}$, while the compression resistance increased at 6.5-7.2 MPa. Also, the experimental results confirmed the remarkable power efficiency of the non-conventional process, the power use value being diminished to an extremely reduced measure (0.74-0.83 $kWh \cdot kg^{-1}$). In conclusion, the cellular product made by the unused technique worldwide proved to be suitable for application in special constructions.
KEYWORDS: microwave heating, cellular glass, residual glass, glycerin, sodium carbonate.

## 1. INTRODUCTION

The need to protect the environment exhibited more and more intensively in the last decades due to the serious damage to the ozone layer of the planet, having implications on its overheating, has reoriented humanity's concerns towards the significant reduction of the excessive consumption of fossil fuel that generates greenhouse gases. The optimal way to solve these ecological problems has already been identified at the global level and involves the sharp increase in recycling degree of reusable waste capable of providing products with newly created value.

Glass, which has been found to practically not lose its valuable characteristics through aging and is not affected during the reconditioning processes, is the material waste type that can be recovered with less power effort (approximately 500 kJ/kg) compared to that necessary for its initial production (4500 kJ/kg) [1]. Several researches carried out in the world in the last decades have laid the foundations for the industrial scale manufacturing of various cellular glass types adequate for using mainly as a heat-insulating product in building [2], but also as a filter, absorber, catalyst support, gas sensor, etc. On the other hand, denser cellular bottles could be made as adequate materials for drainage, foundation infrastructure, filling in road and railway construction, sports grounds, underground heat pipes and storage tanks, bridge abutments, etc. [3].

Several facilities are in operation in North America, Europe (Austria, Belgium, Germany, the Scandinavian countries, the United Kingdom, Hungary, the Czech Republic, Russia, etc.) as well as in China [4]. According to this report, the world glass foam market is estimated to increase to 618 million USD up to 2025.

Considering the wide availability of glass waste throughout the world as well as its high generation rate, making the cellular glass by sintering using glass waste and an adequate expanding agent has become very interesting and economically profitable for manufacturers in the construction materials industry [5].

Cellular glass products constitute remarkable combination of physical, thermal and mechanical features (combining lightness and reduced heat conductivity with satisfactory values of compression resistance) as well as resistance to water, fire, attack of some external aggressors (insects, rodents, bacteria) [5].

According to [6], the most frequently utilized heat-insulating materials are polystyrene types, phenolic cell, and wool from bottle. Unlike these classic materials, cellular glass has higher mechanical resistance as well as resistance to the aggression of external agents. On the other hand, the energy requirement for the production of cellular glass is about 10 times lower by comparison with the requirement for the manufacture of expanded polystyrene [7].

The main products known on the cellular glass market are ”Technopor” of the Swiss company Misapor and ”Foamglas” of the North American company Pittsburgh Corning.

The first products have great durableness, great compression resistance (above 5 MPa), reduced heat conductance (under 0.095 $W \cdot m^{-1} \cdot K^{-1}$), being used especially in areas that require mechanical stress and at the same time, fireproof, frostproof, resistance to humidity and other external aggressors, like foundations, floors and roofs in industrial constructions, road and railway structures, airport runways, drainage, etc. [8, 9].

"Foamglas" products are manufactured as pieces for construction insulation, or lumps separated from the flat mass sintered on the belt of the tunnel kiln used as insulating filling material. The compression strength of these products falls between 1.6-2.75 MPa, the tightness against water and humidity is extremely low and they are non-deformable [10].

Another category of cellular glass with good mechanical properties includes cellular glass gravel. The important peculiarity of this product is the correlation between heat conductivity and compression resistance. According to some articles [11], qualitatively, cellular glass gravels have the thermal conductivity about twice as great by comparison with insulations with free filling available in commerce corresponding to the same value of compression resistance. Also, low denseness, extremely reduced water-absorbing, fine porosity, fireproof, and resistance to various external agents, chemical stability, and excellent load-bearing properties complete their excellent characteristics [12].

Making process of cellular glass gravel involves glass waste without quality claims (post-consumer drinking bottle regardless of colour and flat glass). Among the known expanding agents, solid products (such as: silicon carbide, calcium carbonate, coal powder, gypsum, sodium silicate, manganese oxide) or liquids (glycerin) are usually used [13]. Compression strength of this cellular glass type is relatively high of 5-6 MPa or more, while the heat conductivity value is quite low (under 0.09 $W \cdot m^{-1} \cdot K^{-1}$). The principal manufacturers of cell bottle gravel are the Austrian Geocell, the Swiss Misapor, and the German Glapor.

A cell bottle gravel manufacturing recipe used by Glapor Company including 87 % residual glass, 1 % glycerin, and 12 % $Na_2SiO_3$ allowed to obtain a material with bulk denseness under 0.21 $g \cdot cm^{-3}$ and compression resistance of above 5 MPa. Glamaco Co. from Jena, Germany [14], manufacturer of furnaces for making cellular bottle gravel recommended a recipe composed of 95 % residual glass, glycerin, $CaCO_3$, and $Na_2SiO_3$ summing 5 %, which ensures the production of cellular bottle gravel with denseness below 0.20 $g \cdot cm^{-3}$, heat conductance between 0.06-0.08 $W \cdot m^{-1} \cdot K^{-1}$, and compression resistance of over 4 MPa.

Except cellular glass with low bulk density, low heat conductivity, and moderate mechanical resistance, other cellular glass types are made with high mechanical properties adequate for load-bearing abilities and heat-insulating under foundations, underground heat pipes and storage tanks, road construction, etc. [15].

The manufacturing process of cellular ceramic glassware using fly ash together with residual glass, expanding agent ($CaCO_3$), and flux agent (borax) [16] allowed obtaining products with weightiness of 0.46 $g \cdot cm^{-3}$, heat conductance of 0.36 $W \cdot m^{-1} \cdot K^{-1}$, and compressive resistance of above 5 MPa.

Until now, all cellular glass gravel manufacturing techniques are produced by traditional warming procedures. Unlike these, the non-conventional heating with electromagnetic waves adopted by authors of the current work has proved that manufacturing the cellular glass gravel is viable in technical terms and, in addition, it is environmentally friendly and more energy efficient compared to traditional methods. Electromagnetic waves have been discovered since the 1930s, being used especially in the field of communications and radars. Despite the remarkable energy efficiency in solids heating processes [17], microwaves have only been applied in the case of drying or heating them at low temperatures [18, 19].

The adoption by authors' team of the non-conventional heating technique required in manufacturing processes of cellular glass by sintering at temperatures between 800-1150 °C constituted the main originality of processes and the scientific contribution of Romanian authors lately, starting from 2018.

The testing consequences [17] demonstrated that the use of the mix including 93 % uncoloured bottle, 1 % glycerin, 4.8 % sodium silicate, low quantities of calcium carbonate and kaolin (0.8 and respectively, 0.2 %) as well as water, subjected to sintering process at over 830 ºC, a cell bottle gravel with very good features can be produced (denseness of 0.28 $g \cdot cm^{-3}$, heat conductance of 0.063 $W \cdot m^{-1} \cdot K^{-1}$, compression resistance of over 7 MPa, water, and low pore dimension). The power use had low values (0.78 $kWh \cdot kg^{-1}$).

The experimental research carried out by Daily Sourcing & Research from Romania [20] regarding making the cellular glass gravel in conditions of utilizing the non-conventional electromagnetic wave

warming method tested several technological versions. In one of them, glycerin was the expanding agent (liquid) and in other versions, $CaCO_3$ with borax and $Na_2SiO_3$ adding and also SiC respectively, were adopted as solid agents. The cellular material produced with solid factors were characterized by great compression resistance (7.4-7.5 MPa), but also quite high weightiness measures (up to 0.65 $g \cdot cm^{-3}$) compared to industrial cellular glass gravel. Instead, the cellular product manufactured with glycerin had reduced denseness (0.24 $g \cdot cm^{-3}$) and satisfactory values of compression resistance (5.3-5.9 MPa). Concluding the mentioned study was that manufacturing recipe including 1 % glycerin, 8 % $Na_2SiO_3$, and 8 % water, under the conditions of sintering the mixture at 823 ℃ with reduced power consumption of 0.88 $kWh \cdot kg^{-1}$, is the optimal solution, the product having good physical, thermal, and mechanical characteristics.

Taking into account the previous testing conclusions acquired by the writers of this work, the last test aimed at improving the performance of cellular products. For this purpose, it was decided to use as expanding agents the combination of glycerin as an aqueous material and sodium carbonate ($Na_2CO_3$) as a solid material. According to [21, 22], $Na_2CO_3$ is considered as a common foaming factor used in preparing procedure of glass foam. Normally, the weight proportion between 2-6 % is adequate for performing this operation. By comparison with $CaCO_3$, which facilitates expansions of residual glass of over 450 % by adding 2 % $CaCO_3$, foaming with $Na_2CO_3$ is significantly reduced (only 90 % corresponding to the addition of 5 % $Na_2CO_3$ [5]. A lower expansion is suitable for the material type manufactured in this experiment.

## 2. METHODS AND MATERIALS

### 2.1 Methods

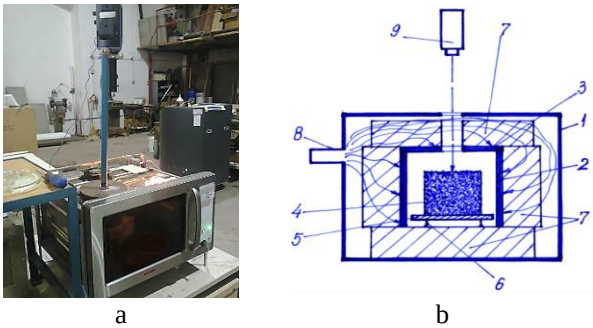
Glycerin ($C_3H_8O_3$) as a liquid carbonaceous material has the capacity to disperse easily betwixt the small-sized particles of residual glass and this physical property was the main reason for its adoption as an expanding agent. The glycerin role in foaming process is based on its decomposition in oxidizing atmosphere of the furnace setting at liberty more gaseous components (such as $CO_2$, CO, etc.) up to the pure solid C as well as hydroxyl groups [23]. The decomposition initiation occurs at small temperatures (almost 200 ℃) and develops in successive steps up to approximately 850 ℃ [24]. It is known from previous experiments that a carbonic material like glycerine exhibits great harmony for atmospheric oxygen, potentially causing the early combustion of carbon and thus disrupting the expanding procedure. For this reason, the aqueous product of $Na_2SiO_3$ is usually utilized in association with glycerin to cover the bottle grains. In this way, the glycerin breaking up procedure is diminished and the glass sintering is accelerated.

As mentioned above, the solid expanding agent used in this experiment together the glycerin was $Na_2CO_3$. The fine powder of $Na_2CO_3$ from the starting mixture thermally decomposes above 750 ℃, releasing gaseous $CO_2$ that participates directly in the expanding procedure of residual bottle forming gas bubbles as well as $Na_2O$ (solid) that penetrates the matrix of the melt bottle. Developing the thermal process leads to growing the internal pressure of gas bubbles resulted both from the glycerin decomposing and that of $Na_2CO_3$.

This phenomenon influences the increase in volume of the thermally softened glass-based material, leading to its foaming. Finishing the sintering/foaming technology, bubbles transform into pores as a result of cooling the material.

The experimental microwave oven utilized during this experiment was an 800 W-oven usually used in the household for food preparation (Figure 1). Several constructive and operational adaptations were carried out for allowing to reach high temperatures of up to 1150 ℃. Also, the initial rotating system of material subject to heating was abolished due to the higher volume and mass of the assembly including the crucible made of susceptible material to microwaves and the heated sample.

The particularities of the non-conventional electromagnetic wave warming process of solids, unrelated from the traditional process, characterized by the heating beginning into the specimen core, continued by the propagation in entire volume from the interior to the exterior [23, 24], did not require additional thermal protection measures for metal oven walls, but only protecting the outer surface of the crucible that absorbs electromagnetic waves (with ceramic fiber resistant to 1200 ℃).




a b

**Figure 1.** Testing apparatus
a – picture of the electromagnetic wave furnace; b – constructive and functional concept: 1 – oven casing; 2 –

cylinder; 3 – top; 4 – warmed specimen; 5 – metal board; 6 – stand; 7 – ceramic fiber protection; 8 – microwave guide; 9 – pyrometer.

### 2.2 Materials

A mixture consisting of uncoloured, green, and brown residual bottle in approximately similar ratios constituted the main part of the starting material composition. The oxide components of the three types of recycled bottle are indicated in Table 1.

**Table 1.** Components of residual glass types

| Component | Residual glass type (%) | | |
|---|---|---|---|
| | Uncoloured | Green | Brown |
| $SiO_2$ | 71.7 | 71.8 | 71.1 |
| $Al_2O_3$ | 1.9 | 1.9 | 2.0 |
| CaO | 12.0 | 11.8 | 12.1 |
| $Fe_2O_3$ | - | - | 0.2 |
| MgO | 1.0 | 1.2 | 1.1 |
| $Na_2O$ | 13.3 | 13.1 | 13.3 |
| $K_2O$ | - | 0.1 | 0.1 |
| $Cr_2O_3$ | 0.1 | 0.1 | - |
| $SO_3$ | - | - | 0.1 |

The bottle processing operations including selection by colour, washing, drying, cracking, crushing, and screening were performed at Bilmetal Industries SRL Popesti-Leordeni (Romania). The selected particle dimension for this experiment was under 80 μm.

Glycerin ($C_3H_8O_3$) was the liquid expanding agent chosen for this experiment, being commercially available in liquid state. Its use was done in association with the aqueous solution (38 %) of $Na_2SiO_3$ also known as "water glass", whose role is enveloping agent to avoid early burning of carbon from glycerin [18].

The solid foaming agent adopted by the authors was sodium carbonate ($Na_2CO_3$) known from the literature [5] as a supplier of gaseous compounds ($CO_2$ and/or CO) required in the expanding process, resulting from the thermal decomposition. Available on the market as a fine powder, $Na_2CO_3$ is an alkaline water-soluble salt [25].

Very low weight ratio (below 0.2 wt. %) of kaolin was introduced in the solid starting mixture as a very fine powder (under 10 μm). Mainly, the kaolin composition includes 57.6 % $SiO_2$, 37.8 % $Al_2O_3$ as well as reduced proportions of potassium, iron, magnesium, and calcium oxides [26]. The kaolin role consists in ability of kaolinite (existing in the kaolin composition) to offer heat protecting for ceramic materials (in this case, the glass).

Four experimental versions were designed and tested within the experiment. The structure of materials mixed to obtain these versions is presented in Table 2.

**Table 2.** Structure of testing variants (wt. %)

| Structure | Variant 1 | Variant 2 | Variant 3 | Variant 4 |
|---|---|---|---|---|
| Residual glass | 88.0 | 87.4 | 87.6 | 85.1 |
| Glycerin | 1.0 | 1.0 | 1.1 | 1.1 |
| Sodium silicate | 7.8 | 8.2 | 8.7 | 9.8 |
| Sodium carbonate | 3.0 | 3.2 | 3.4 | 3.8 |
| Kaolin | 0.2 | 0.2 | 0.2 | 0.2 |
| Distilled water addition | 14.0 | 14.3 | 14.5 | 14.8 |

In accordance with Table 2, except for the constant ratio of kaolin (0.2 %), the values of the other components of the mix composition were more or less varied. Residual glass proportion was reduced from 88.0 % (in the case of version 1) to 85.1 % (in the case of version 4). The liquid agent (glycerin) ratio was very low modified (within the limits of 1.0-1.1 %). Instead, sodium silicate increased in value from 7.8 to 9.8 %. The solid expanding agent proportion was changed within low limits (between 3.0-3.8 %). The values range was kept far from the recommended maximum limit (6 %) so that the foaming process to take place without intensity and the pore size to remain at a low level.

### 2.3 Methods for investigating the sample features

Determining the physio-mechanical, heat, and microstructural peculiarities of the cellular bottle was achieved utilizing usual procedures. Archimedes' method was chosen to measure the denseness and porousness (ASTM D792-20), in accordance with the literature recommendation [27]. The heat conductivity was verified using the "heat-flow" procedure (ASTM E1225-04), while the compressive resistance was measured with "TA.XTplus Texture" analyzer according to ASTM C552-17 standard. Volumetric proportion of water-absorbing was identified through the water-submersion procedure (ASTM D570). The appearance of cellular glass specimen microstructures was investigated with "ASONA 100X Zoom Smartphone Digital Microscope".

## 3. RESULTS AND DISCUSSION

### 3.1 Results

The principal operational frameworks of making procedure of cellular glass in 800 W-electromagnetic wave furnace are shown in Table 3.

**Table 3.** Principal operational frameworks

| Framework | Variant 1 | Variant 2 | Variant 3 | Variant 4 |
|---|---|---|---|---|
| Feedstock/cell | 450/ | 450/ | 450/ | 450/ |

| | | | | |
|---|---|---|---|---|
| bottle quantity (g) | 407 | 409 | 407 | 410 |
| Sintering temperature (°C) | 820 | 823 | 826 | 830 |
| Heating time (min) | 29 | 30 | 31 | 32.5 |
| Average speed (°C·min$^{-1}$)<br>- heating<br>- cooling | <br>28.3<br>5.0 | <br>27.4<br>5.1 | <br>26.6<br>4.9 | <br>25.5<br>5.1 |
| Specific power use (kWh·kg$^{-1}$) | 0.74 | 0.76 | 0.79 | 0.83 |

Analyzing the operational frameworks corresponding making technique of bottle foam in Table 3, the rapidity of the non-conventional warming is noted, the process speed exceeding 25 °C·min$^{-1}$. By default, duration of the warm-up was significantly diminished, falling within the range of 29-32.5 min. The specific power use was also diminished within the limits of 0.74-0.83 kWh·kg$^{-1}$, being under the usual level of industrial techniques of making cellular bottle.

The image of the aspect of testing manufactured bottle foam samples is shown in Figure 2. By increasing the proportion of sodium carbonate up to 3.8 % and the proportions of glycerin and sodium silicate up to 1.1 % and respectively 9.8 %, the conditions for intensifying the foaming process were improved and the specimens corresponding to versions 3 and 4 had a higher degree of growth in volume.

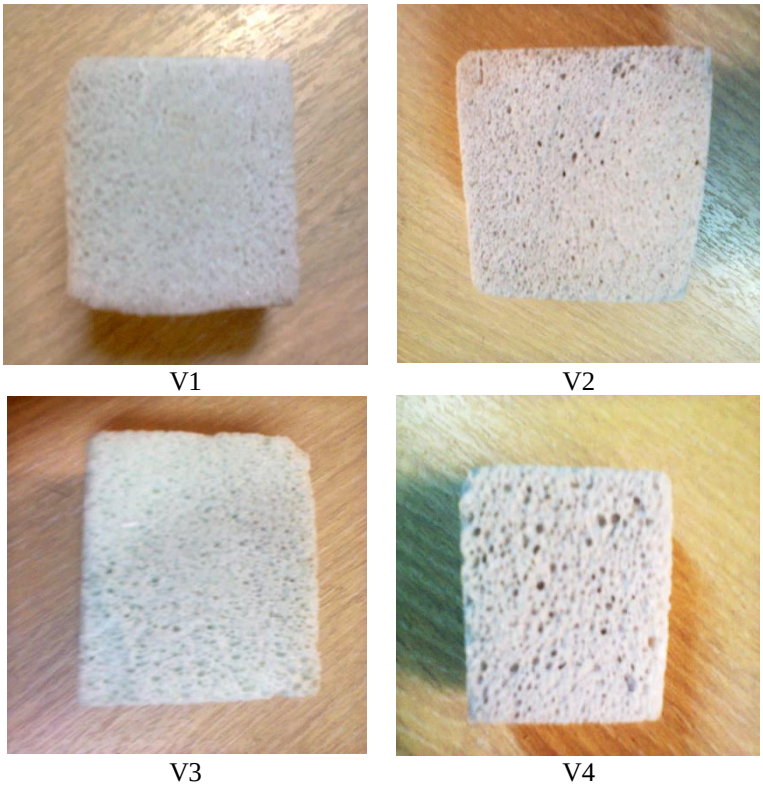



**Figure 2.** Image of cellular glass specimen appearance
V1 – variant 1; V2 – variant 2; V3 – variant 3; V4 – variant 4.

Investigating characteristics of the cellular glass samples using the methods mentioned above allowed their identification. Table 4 shows the measured values.

**Table 4.** Physio-mechanical, heat, and microstructural peculiarities of cell bottle specimens

| **Variant** | **Apparent denseness (g·cm$^{-3}$)** | **Porousness (%)** | **Heat conductance (W·m$^{-1}$·K$^{-1}$)** | **Compression resistance (MPa)** | **Water-absorbing (%)** | **Pore dimension (mm)** |
|---|---|---|---|---|---|---|
| 1 | 0.27 | 87.1 | 0.072 | 7.2 | 2.4 | 0.1-0.4 |
| 2 | 0.25 | 88.0 | 0.069 | 7.0 | 2.2 | 0.2-0.5 |
| 3 | 0.22 | 89.5 | 0.064 | 6.7 | 2.3 | 0.3-1.0 |
| 4 | 0.20 | 90.5 | 0.060 | 6.5 | 2.5 | 0.3-1.1 |

Values containing in Table 4 confirm that making recipe based on combining liquid and solid expanding agents is viable leading to obtaining cellular products with valuable thermal insulation features (reduced levels of denseness and heat conductivity) as well as, in the same time, high resistance to compression. Pore size is low (with values between 0.1-1.1 mm) on the entire domain of the made specimens.

Images of the microstructure corresponding to the foam products are presented in Figure 3.

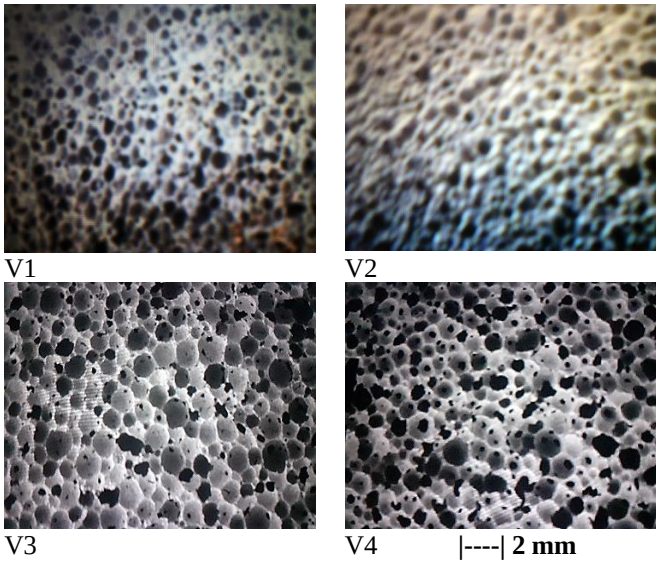



**Figure 3.** Microstructure pictures of cell samples
V1 – variant 1; V2 – variant 2; V3 – variant 3; V4 – variant 4.

3.2 Discussion

The experiment presented in this paper proved the remarkable effectiveness of applying the microwave heating procedure in glass sintering and foaming processes. Great warming speeds of above 25 $°C \cdot min^{-1}$ were easily reached without disturbing the macrostructure of the silicate product. Utilizing microwave irradiation at fairly great temperatures (above 800 °C) is a technical novelty, although these have been known since the 1930s.

## 4. CONCLUSION

The work followed the production of strength spongy glass with insulation particularity and simultaneously, with excellent resistance to compression. The original contribution of the article derives from adopting the non-conventional warming technique of the mixture subjected to the sintering process, unlike the conventional methods used in present at industrial-scale. The effectiveness of microwave application was proven in this experiment, being obtained characteristics of cell glass at the level of known industrial products (heat conductance below 0.072 $W \cdot m^{-1} \cdot K^{-1}$, resistance to compression in the range of 6.5-7.2 MPa) and in addition, under the conditions of extremely reduced power use (under 0.83 $kWh \cdot kg^{-1}$).

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