



## Cellular Glass Manufactured by Microwave Irradiation of Residual Glass, Eggshell and Borax

Lucian Paunescu<sup>1,2</sup>, Sorin Mircea Axinte<sup>2,3</sup>, Felicia Cosmulescu<sup>4</sup>

<sup>1</sup> Bilmetal Industries SRL Popesti Leordeni-Ilfov, Romania

<sup>2</sup> Daily Sourcing & Research SRL Bucharest, Romania

<sup>3</sup> Department of Applied Chemistry and Materials Science, University "Politehnica" of Bucharest, Romania

<sup>4</sup> Cosfel Actual SRL Bucharest, Romania



\*Corresponding Author Lucian Paunescu

Email: [lucianpaunescu16@gmail.com](mailto:lucianpaunescu16@gmail.com)

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### Abstract

The paper presents an improved method of manufacturing cellular glass using residual glass (91 %), sodium borate (5 %), eggshell waste (4 %) and added water (9 %). Compared to methods using eggshell as an expanding agent producing cellular glass with low compression strength, the technique adopted by the authors is original by the addition of sodium borate, which contributes to increasing the compression strength and the use of the unconventional electromagnetic wave heating method, which ensures very economical specific energy consumption. The optimal variant of cellular glass had the following characteristics: density of 0.40 g/cm<sup>3</sup>, porosity of 81 %, heat conductivity of 0.086 W/m·K, compression strength of 4.3 MPa and the cell dimension between 0.3-0.9 mm. The specific energy consumption of the process was 0.80 kWh/kg. The product has adequate features for using as a heat insulation material under conditions of quite high mechanical loading.

## Introduction

In the last decades, large amounts of residual glass coming mainly from consumed drinking bottle and flat glass residue from building demolition are being generated around the world. Partly, the residual glass is reused as a raw material in the glass industry. However, the selection of residue by color involves high costs. A modern solution for using recycled residual glass without selecting by color is the manufacture of cellular glass. There are several companies worldwide (Pittsburgh Corning, Geocell, Misapor, Glapor, etc.) that produce different types of cellular glass using recycled residual glass, the application area being in civil and industrial construction as heat insulation materials (Scarinci et al., 2005; Dragoescu & Paunescu, 2020).

All the characteristics of cellular glasses are remarkable: light weight, low heat conductivity, relatively high compression strength, resistance to fire, humidity and freeze-thaw cycles, resistance to rodents and insects aggression, chemical resistance, are non-toxic, non-deformable and durable. Therefore, these materials represent viable solutions to replace existing building materials based on polystyrene. Due to these characteristics, the cellular glasses are suitable both for the inner and outer walls of buildings as well as for applications involving difficult mechanical and environmental stresses (road and railway construction,

bridge construction elements, underground thermal insulation of thermal pipes and storage tanks, airport runways, drains, sports fields, etc.) (Scarinci et al., 2005).

The theoretical principle of expanding the residual glass is the release of a gas or gaseous compound by a material embedded in the starting powder mixture called expanding agent. Several types of solid materials (coal, carbon black, calcium carbonate, sodium carbonate, dolomite, silicon carbide, silicon nitride, etc.) and liquid (glycerol, petroleum oil, etc.) are commonly used. The release of the gas takes place at relatively high temperatures (750-1100 °C), which should be in correlation with the softening point of the glass, so that its viscosity be adequate for spreading the gas as bubbles in the softened residual material, but not to allow to come out from this mass. By cooling after stopping the heating process, the gas bubbles turn into pores forming a structure specific to the cellular glass (Scarinci et al., 2005).

The global trend is to use cheap expanding agents or their existing residual substitutes. One such example is the eggshell with a content of over 96 % calcium carbonate.

The works (Fernandes et al., 2013 and 2014) present experimental results obtained in cellular glass manufacturing processes from residual cathode ray tube (CRT) glass using eggshells as an expanding agent. The temperature of the thermal process was between 650-750 °C and the weight ratio of the expanding agent was variable. The best characteristics of the cellular glass were obtained using 3 % eggshell residue, the optimal temperature being 700 °C for 15 min. The obtained product had a density of 0.29 g/cm<sup>3</sup> and a compression strength of 2.34 MPa.

Other research described in the literature (Souza et al., 2017) has focused on the expanding process of soda-lime silica glass residue, i.e. the main commercial glasses, the expanding agent being also eggshell in weight proportions between 1-30 %. The residual glass together with the eggshell residue was homogenized and axially pressed at 20 MPa. The firing temperature was 900 °C for 30 min. The expanded products had the porosity between 60-95 %, heat conductivity within the limits 0.055-0.177 W/m·K and compression strength between 0.15-1.50 MPa, being suitable for applications where heat and sonic insulation and the fire resistance are their main requirements, not also the mechanical strength.

Approximately similar results were obtained in experiments described in the paper (Saparaddin et al., 2020). The starting material was composed of the same types of residual materials: soda-lime silica glass and eggshell residues, the weight proportion of eggshell being between 1-9 %. The temperature of the manufacturing process was around 800 °C and the heating speed was 10 °C/min. The main characteristics of the expanded products (apparent specific gravity and compression strength) varied, the lowest value of density of 0.326 g/cm<sup>3</sup> and the lowest compression strength of only 0.04 MPa corresponding to the specimen made with 3% eggshell residue and the highest values of these characteristics (0.822 g/cm<sup>3</sup> and respectively, 0.8 MPa) corresponding to the specimen made with 9 % eggshell.

Therefore, the main disadvantage of the manufacture of cellular glass from residual glass and eggshell residue is the very low level of mechanical strength of the products.

The team of authors of this paper has conducted numerous experimental tests in the last five years on the manufacture of cellular glasses, the original character of research being the application of unconventional electromagnetic wave (microwave) heating method. To date, the eggshell residue has not been used as an expanding agent instead of calcium carbonate, but calcium carbonate has been directly used. In the paper (Paunescu et al., 2018) is presented a solution for the manufacture of a denser cellular glass with compression strength reaching to 6.2 MPa under the conditions of addition (5 wt.%) in the starting mixture of sodium borate (borax) as a fluxing agent. The boron content of over 11 % of borax contributed to a significant

increase of the material compression strength compared to cellular glasses made of residual glass and calcium carbonate with mechanical strength below 1.3 MPa.

In the present paper, the authors adopted the solution of expanding the residual glass using eggshell as a residual expanding agent and borax as a fluxing agent. The heat treatment technique of the powder mixture was unconventional. The aim of the work was to improve the mechanical strength of the porous material.

## Methods

The chemical reaction that characterizes the expanding process of residual glass using eggshell residue as a substitute for calcium carbonate is the carbonate decomposition reaction. According to (Karunadasa et al., 2019), the thermal transformation of calcium carbonate ( $\text{CaCO}_3$ ) (1) into calcium oxide ( $\text{CaO}$ ) with the release of carbon dioxide ( $\text{CO}_2$ ) is begun slowly and then is developed rapidly at temperatures over 750 °C.



Unlike the conventional heating ovens used in cellular glass manufacturing processes with eggshell as an expanding agent presented above, the manufacture of cellular glass with eggshell and borax described below took place in an electromagnetic wave oven of 0.8 kW in the Romanian company Daily Sourcing & Research Bucharest, commonly used in household for preparation of food. The oven was adapted for operations at high temperature (over 1000 °C). The technical solution adopted by the authors was similar to that used in the last own experiments of expanding the residual glass. It was experimentally determined the need to use a protective screen made of a high electromagnetic wave susceptible material ( $\text{SiC}$  and  $\text{Si}_3\text{N}_4$  in 80/20 ratio) placed between the irradiation source and the heated material. The optimal thickness of the screen (ceramic tube or crucible) was determined at 2.5 mm, so that the heating process takes place predominantly direct, by penetrating the ceramic wall and the direct contact with the material subjected to heating and partially indirect, by absorbing waves in the mass of ceramic wall, which heats up quickly and transfers the heat by thermal radiation. The direct microwave heating is started in the middle of the material, the energy of electromagnetic waves being converted into heat. This type of heating is completely opposite compared to the conventional heating, the heat being volumetrically transmitted from the inside to the outside areas, the process taking place very quickly (Kitchen et al., 2014; Jones et al., 2002). Another feature of the direct microwave heating is the process selectivity (Kitchen et al., 2014; Jones et al., 2002), which involves the heating only of the target material, not also the massive components of the oven, contributing to a high energy saving.

The powder mixture of the starting material (residual glass, sodium borate and eggshell residue) was homogenized and pressed into a metal mold, then removed from the mold and deposited freely on a steel sheet plate placed on a thick bed of ceramic fiber mattresses at the base of oven. The cylindrical pressed material with a diameter of about 85 mm and a height of about 60 mm was protected with a  $\text{SiC}/\text{Si}_3\text{N}_4$  ceramic tube with the outer diameter of 125 mm, the height of 100 mm and the wall thickness of 2.5 mm purchased from China, provided with a  $\text{SiC}/\text{Si}_3\text{N}_4$  lid having a 30 mm hole in its central area for viewing the mixture during the heating. Because the wave flux heating is initiated in the middle of material, the outer surface of the tube and lid has been efficiently thermal protected with ceramic fiber mattresses to avoid the heat loss to the unprotected metal walls of the oven. The oven has a single microwave generator, the waveguide for the flux emission being placed in one of the side walls of the oven. The temperature control of the material was carried out with a Pyrovar type radiation pyrometer (measuring range between 600-2000 °C) mounted above the oven on the central axis at about 400 mm. The upper metal wall of the oven was provided with a 30 mm hole on the

same vertical axis as the hole in the ceramic tube and lid. Figure 1 shows a picture of the electromagnetic wave oven (A) and a principle scheme of the experimental plant (B).

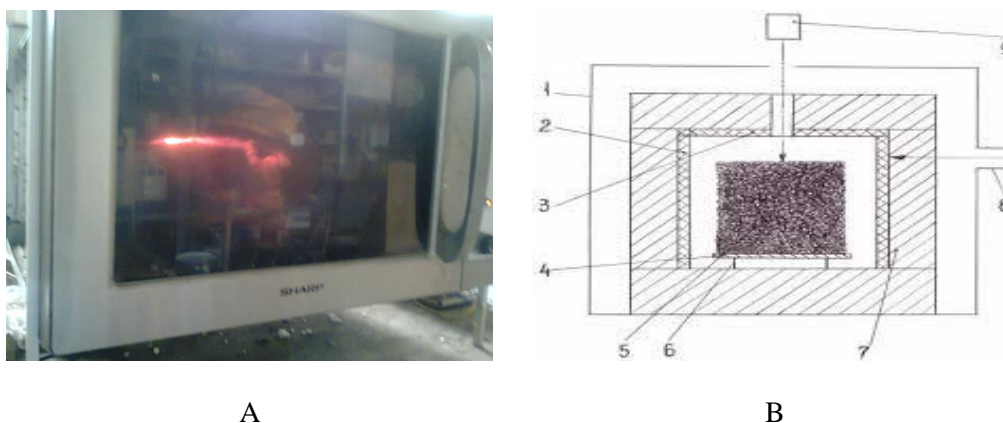


Figure 1. The experimental electromagnetic wave plant

A – picture of the electromagnetic wave oven; B – principle scheme of the plant: 1 – electromagnetic wave oven; 2 – SiC/Si<sub>3</sub>N<sub>4</sub> tube; 3 – SiC/Si<sub>3</sub>N<sub>4</sub> lid; 4 – steel sheet plate; 5 – pressed material for heating; 6 – rising support; 7 – heat insulation; 8 – emission wave source; 9 – pyrometer.

As mentioned above, the materials used in the experiment were: recycled consumed drinking bottle as starting based material, sodium borate (borax) as a fluxing agent and eggshell residue as an expanding agent containing predominantly CaCO<sub>3</sub>.

The residual glass was recycled consumed drinking bottle composed from colorless glass (about 70 %), green and amber glass ((15 % each assortment). The residue was broken, ground in a ball mill and sieved at grain size less than 100 μm. The residual glass processing operations were performed in the Romanian company Bilmetal Industries Popesti-Leordeni, Ilfov. The oxide composition of the consumed drinking bottle types is presented in Table 1.

Table 1. Oxide composition of the consumed drinking bottle types

| Oxide composition<br>(wt, %)   | Drinking bottle type |       |       |
|--------------------------------|----------------------|-------|-------|
|                                | Colorless            | Green | Amber |
| SiO <sub>2</sub>               | 71.5                 | 71.2  | 71.4  |
| Al <sub>2</sub> O <sub>3</sub> | 1.9                  | 1.8   | 1.9   |
| CaO                            | 12.0                 | 10.2  | 10.3  |
| Fe <sub>2</sub> O <sub>3</sub> | 0.1                  | 0.4   | 0.3   |
| MgO                            | 1.0                  | 2.2   | 2.3   |
| Na <sub>2</sub> O              | 13.3                 | 13.0  | 13.2  |
| K <sub>2</sub> O               | 0.1                  | 0.5   | 0.6   |
| Cr <sub>2</sub> O <sub>3</sub> | 0.1                  | 0.2   | 0.1   |
| SO <sub>3</sub>                | 0.2                  | 0.3   | 0.3   |

Commercially purchased borax at a grain size below 400 μm was ground in a ball mill and sieved at a grain size of less than 130 μm. The oxide composition of sodium borate includes 30.8 % Na<sub>2</sub>O and 69.2 % B<sub>2</sub>O<sub>3</sub> (Borax, 2006).

The eggshell as a residue provided by a food laboratory was broken and ground in a laboratory device at a granulation below 40 μm. The oxide composition of the eggshell contains 96.8 % CaCO<sub>3</sub>, 0.4 % Na<sub>2</sub>O, 0.8 % MgO and 2 % other oxides (Saparaddin et al., 2020).

The cellular glass specimens were subjected to the usual methods of determining the main characteristics. The apparent specific gravity was measured by the gravimetric method (Manual, 1999). The porosity was calculated by the method of comparing the true and apparent specific gravity (Anovitz & Cole, 2015). The heat conductivity was measured by the heat-flow meter method (ASTM E1225-04) and the compression strength was determined using a TA.XTplus Texture Analyzer (ASTM C552-17). The water absorption was determined by the water immersion (for 24 hours) method (ASTM D570) and the microstructural configuration of the cellular glass specimens was investigated with an ASONA 100X Zoom Smartphone Digital Microscope.

## Results and Discussion

Four experimental recipes were adopted for testing the manufacture of cellular glass using borax and eggshell residue. The proportion of borax was kept constant at 5 wt. % as in the paper (Paunescu et al., 2018). The eggshell had successively increasing values between 2-8 wt. %, while the proportion of residual glass was accordingly reduced from 93 to 87 wt. %. The added water was used as a binder at a constant value (9 wt. %). The experimental manufacturing recipe composition is presented in Table 2.

Table 2. Experimental manufacturing recipe composition

| <b>Recipe</b> | <b>Residual glass (wt. %)</b> | <b>Sodium borate (wt. %)</b> | <b>Eggshell residue (wt. %)</b> | <b>Added water (wt. %)</b> |
|---------------|-------------------------------|------------------------------|---------------------------------|----------------------------|
| 1             | 93.0                          | 5.0                          | 2.0                             | 9.0                        |
| 2             | 91.0                          | 5.0                          | 4.0                             | 9.0                        |
| 3             | 89.0                          | 5.0                          | 6.0                             | 9.0                        |
| 4             | 87.0                          | 5.0                          | 8.0                             | 9.0                        |

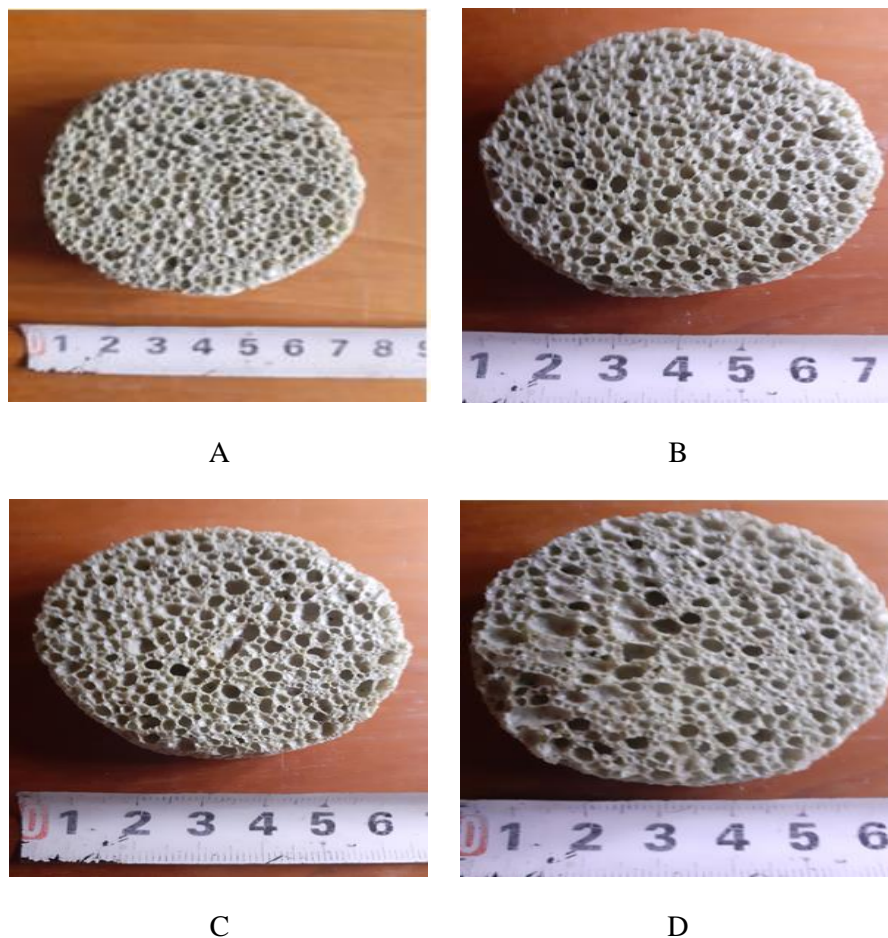
The experiment takes place on the 0,8 kW-electromagnetic wave oven described above. The dry material amount was kept constant for all the four specimens at 480 g, the wet amount of material being also constant at the value of 523.2 g. The main functional data of the manufacturing process are shown in Table 3.

Table 3. Main functional data of the heat treatment process

| <b>Functional date</b>                       | <b>Recipe 1</b> | <b>Recipe 2</b> | <b>Recipe 3</b> | <b>Recipe 4</b> |
|--|-----------------|-----------------|-----------------|-----------------|
| Dry basic material/cellular glass amount (g) | 480/<br>465     | 480/<br>466     | 480/<br>465     | 480/<br>467     |
| Sintering/expanding temperature (°C)         | 820             | 823             | 827             | 831             |
| Heating time (min)                           | 34              | 36              | 38              | 40              |
| Medium speed (°C/min)                        |                 |                 |                 |                 |
| · heating                                    | 23.5            | 22.3            | 21.2            | 20.3            |
| · cooling                                    | 5.5             | 5.7             | 5.4             | 5.7             |
| Index of increasing the material volume      | 1.40            | 1.55            | 1.70            | 1.90            |
| Specific consumption of energy (kWh/kg)      | 0.76            | 0.80            | 0.85            | 0.89            |

The functional data of the cellular glass manufacturing process varied in relatively low limits due to the variation of eggshell residue content. The process temperature increased from 820 °C (recipe 1) to 831 °C (recipe 4) and simultaneously, the heating time increased from 34 to 40 min. Obviously, the medium heating speed had decreasing values from 23.5 to 20.3 °C/min. The increase in volume of the specimens reached a maximum of 1.90 corresponding to recipe 4. The energy efficiency of the predominantly direct microwave heating process described above played a decisive role in obtaining these heating data. Consequently, the specific consumption of the process energy (taking into account the amount of expanded glass between 465-467 g) was very low (between 0.76-0.89 kWh/kg) compared to the values of the conventional industrially manufacturing processes [0.74-1.15 kWh/kg (Energocell, 2016)].

The cross section of the four cellular glass specimens made in the electromagnetic wave oven are shown in Figure 2.



*Figure 2. Cross section of cellular glass specimens*

*A – variant 1; B – variant 2; C – variant 3; D – variant 4.*

The identification of the cellular glass specimen characteristics by the methods mentioned above allowed to determine the influence of the variation of manufacturing processes data in Table 3 on the quality of the expanded products. Table 4 shows the main features of these products.

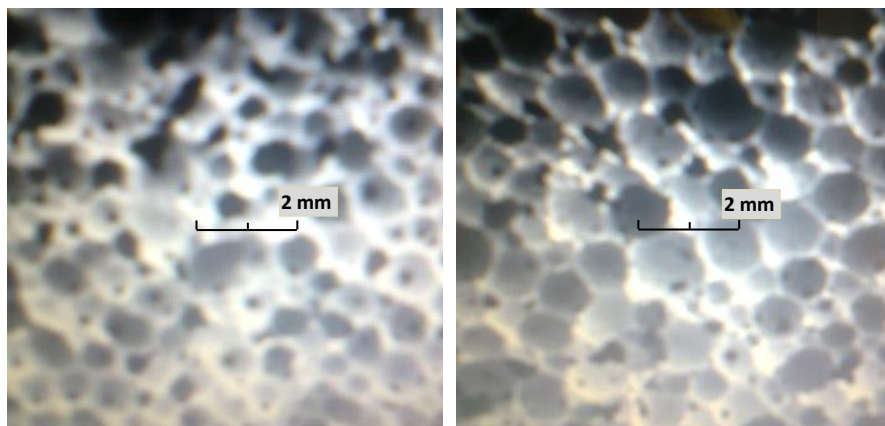
Table 4. Physical, thermal, mechanical and morphological features of cellular glass specimens

| Recipe | Apparent specific gravity (g/cm <sup>3</sup> ) | Porosity (%) | Heat conductivity (W/m·K) | Compression strength (MPa) | Water absorption (vol. %) | Cell size (mm) |
|--------|--|--------------|---------------------------|----------------------------|---------------------------|----------------|
| 1      | 0.45   | 78.6         | 0.091                     | 5.2                        | 1.1                       | 0.3-0.8        |
| 2      | 0.40   | 81.0         | 0.086                     | 4.3                        | 1.2                       | 0.3-0.9        |
| 3      | 0.36   | 82.9         | 0.080                     | 3.3                        | 0.9                       | 0.5-1.0        |
| 4      | 0.31   | 85.2         | 0.072                     | 2.4                        | 0.9                       | 0.4-1.8        |

Analyzing the data in Table 4, it is observed that the main objective of the experiment described in the paper, i.e. to increase the compression strength of the expanded products with eggshell and borax, was achieved. In recipe 1, the specimen made with 2 % eggshell and 5 % borax sintered and expanded at 820 °C reached the highest value of mechanical strength (5.2 MPa). Higher contents of eggshell as an expanding agent (4-8 %) and the same borax content (5 %) required higher process temperature (up to 831 °C) leading to the decrease of mechanical strength up to 2.4 MPa, but maintaining high values of this characteristic compared to the experimental results described in the literature.

The apparent specific gravity values of the products obtained in the four recipes are relatively low (between 0.31-0.45 g/cm<sup>3</sup>), the increasing proportion of eggshell contributing to the decrease of density to the lowest value corresponding to recipe 4. At the same time, the heat conductivity of the expanded products is reduced from 0.091 to 0.072 W/m·K and the porosity improves from 78.6 to 85.2 %. Therefore, higher eggshell contents as an expanding agent contribute to the improvement of the thermal insulation property of the expanded material. The moisture absorption is very low (0.9-1.2 vol. %).

For determining the microstructural homogeneity of cellular glass specimens the pore distribution configuration was investigated with a digital microscope (Figure 3).



A

B

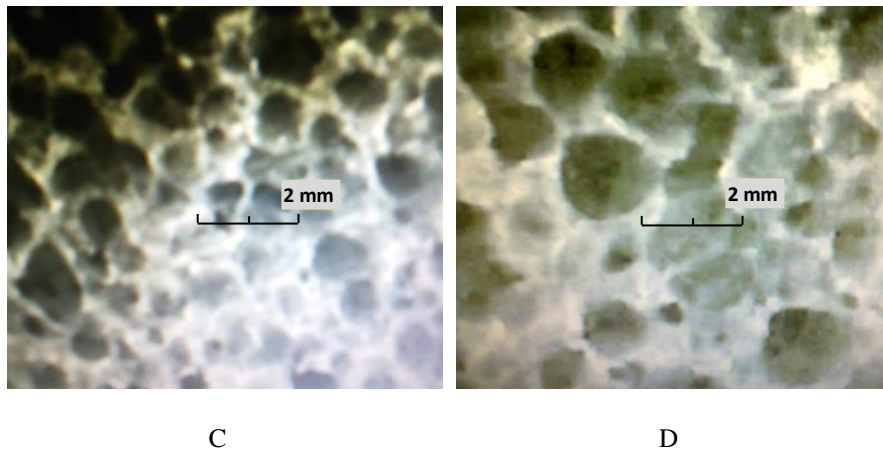


Figure 3. Microwave configuration of the glass foam samples  
 A – variant 1; B – variant 2; C – variant 3; D – variant 4.

As expected, the most homogeneous pore distributions were obtained for recipes 1 and 2 (between 0.3-0.8 mm and 0.3-0.9 mm, respectively). These microstructures correspond to the highest values of compression strength (4.3-5.2 MPa).

Taking into account the experimental results obtained in this experiment, it is observed that the property of heat insulation material of the products (determined mainly by apparent specific gravity and heat conductivity) changes in the opposite direction with the variation of their compression strength. Thus, a high mechanical strength is in accordance with a denser structure of the material implying higher values of apparent specific gravity and heat conductivity. Therefore, it is necessary to establish an optimal correlation between the physical, thermal and mechanical characteristics of cellular glass. Under these conditions, the authors' option for designating the optimal specimen of the experiment was oriented towards recipe 2 made from 91 % glass waste, 5 % borax, 4 % eggshell and 9 % added water by sintering/expanding at 823 °C with a specific energy consumption of 0.80 kWh/kg. The apparent specific gravity of the expanded product was 0.40 g/cm<sup>3</sup>, porosity was 81 %, the heat conductivity had the value of 0.086 W/m·K and the compression strength was 4.3 MPa. The water absorption was 1.2 vol. % and the cell dimension was between 0.3-0.9 mm. The product has suitable characteristics for using as a thermal insulation material under conditions of quite intense mechanical stress.

## Conclusion

The aim of the work was to improve the mechanical strength of the cellular glass under the conditions of using a food industry residue (eggshell) as a CaCO<sub>3</sub>-rich expanding agent. Previous experiments presented in the literature have shown that the heat treatment of residual glass together with eggshell allows obtaining a porous product with a low compression strength unsuitable for use also under conditions of mechanical stress. The current paper adopted a recipe previously applied in expanding processes of residual glass with CaCO<sub>3</sub>, using residual glass, eggshell and borax as a fluxing agent, which due to the boron content contributes to increasing the mechanical strength of cellular glass. The originality of the paper, as well as many other works of the Daily Sourcing & Research team, is the use of unconventional microwave heating technology, unlike the conventional cellular glass manufacturing techniques applied worldwide. The manufacturing recipe considered optimal included 91 % residual glass, 5 % borax, 4 % eggshell residue and 9 % added water, the process temperature being 823 °C. The main characteristics of the cellular glass were: apparent specific gravity of 0.40 g/cm<sup>3</sup>, porosity of 81 %, heat conductivity of 0.086 W/m·K, compression strength of 4.3



MPa and the cell dimension between 0.3-0.9 mm. The specific consumption of the process energy was 0.80 kWh/kg. The expanded product has adequate features for using as a thermal insulation material under conditions of quite intense mechanical stress.

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