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STRENGTH AND POROUS BUILDING MATERIAL PREPARED FROM RECYCLED RESIDUAL GLASS THROUGH MICROWAVE RADIATION

BY

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Abstract. New method of producing strength and porous cellular glass with load-bearing properties from recycled flat glass using simultaneously two expanding agents: glycerol (liquid) and eggshell (solid) recycled as a by-product from the food industry was designed and tested by microwave heating. Eggshell was used as a substitute for the usual calcium carbonate and the microwave heating was made by the original method of predominantly direct and partially indirect heating leading to energy saving. Results showed that cellular glass with low bulk density (0.16-0.19 g·cm^{-3}) and high compression strength (up to 7.6 MPa) were obtained.

Keywords: cellular glass, flat glass waste, microwave heating, glycerol, eggshell.

1. Introduction

The last decades of the 20th century and the beginning of the new millennium meant a great challenge for humanity faced with an energy crisis caused by the reduction of global hydrocarbon reserves and an ecological crisis

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generated by excessively high emissions of greenhouse gases (mainly CO_2) in the atmosphere causing damage to the protective ozone layer and the danger of overheating the planet (Axinte *et al.*, 2021).

The consequence of this unprecedented situation was the set of restrictive measures at the international level involving activities that consume more fossil fuel and that generate important CO_2 emissions. Scientific research was and is involved in finding solutions for the effective use of recycled waste and manufacturing new products with added value to replace the more energy-consuming and implicitly highly polluting traditional products.

Some of the industrial activities that consume high fuel quantities and that seriously influence the environment quality are those involved in the manufacture of construction materials. Since the mid-1980s, it has been found that recycled glass waste can be an excellent cheap raw material for the production of materials suitable for the construction sector. In addition, this waste has a wide availability in the world (Scarinci *et al.*, 2005).

Several international companies from the United States and Europe have developed numerous facilities for the production of so-called cellular glass, especially in European countries, but also China has become an important manufacturer of products based on recycled glass.

The current work refers especially to porous products with high mechanical strength and load-bearing features, usable as fillers in numerous applications in the construction field such as: light foundations, insulation of the building perimeter, drainage, underground insulation of heat pipes and tanks, road and railway construction works, etc. Fillers in the form of lumps with sizes between 20-80 mm, known as cellular glass gravels, are predominantly made from recycled glass waste (about 98%) and different mineral expansion agents depending on the manufacturing recipe of each manufacturer (Hibbert, 2016; Zegowitz, 2010).

According to Cosmulescu *et al.*, (2020), the Swiss concern Misapor uses mixed glass waste or green container glass (98%) and silicon carbide, gypsum or limestone as expanding agents. The process temperature is within the limits of 800-1000°C. The product has the bulk density between 0.16-0.19 $g{\cdot}cm^{-3}$, thermal conductivity of 0.12 $W{\cdot}m^{-1}{\cdot}K^{-1}$, and compression strength in the range of 4.9-6,0 MPa. The Austrian company Geocell Schaumglas uses 90% coloured post-consumer drinking bottle and 10 % colourless flat glass waste. The temperature of the sintering-foaming process is almost 900°C and the products have the bulk density of 0.15 $g{\cdot}cm^{-3}$, heat conductivity of 0.08 $W{\cdot}m^{-1}{\cdot}K^{-1}$, and compression strength of 5.7 MPa. Glapor Werk Mitterteich Company (Germany) produces cellular glass gravel having the manufacturing recipe composed from 87% recycled glass (flat glass or post-consumer drinking bottle), 1 % glycerol as a liquid expanding agent, 12% sodium silicate, and under 0.5% kaolin. The bulk density of this gravel is within the limits of 0.13-0.21 $g{\cdot}cm^{-3}$, and the compression strength is in the range of 4.9-6.0 MPa. Porosity of lumps is very fine with pore dimensions under 300 µm.

The current industrial production of cellular glass gravel is based on the use of conventional heating methods (thermal energy produced by burning fuels or electricity).

An ecological and very energy efficient method of heating is the unconventional technique of using microwave radiation. Discovered in the middle of the 20th century, electromagnetic waves were applied especially in transmissions and radars and less in thermal processes of drying and heating at low temperatures, without reaching high temperatures.

Under these conditions, since 2017 the Romanian company Daily Sourcing and Research Bucharest carried out a small-scale experimental program for the use of microwaves in heating processes at temperatures between 800-1150°C for sintering/expanding the glass waste and making different types of cellular glass. This program has also included the production of cellular glass gravel in different compositional variants (Cosmulescu *et al.*, 2020). In the first version, post-consumer green drinking bottle, calcium carbonate ($CaCO_3$), borax ($Na_2B_4O_7$), and sodium silicate (Na_2SiO_3) were used. The process temperature was 855°C and the specific energy consumption was 1.07 $kWh \cdot kg^{-1}$. The product characteristics were: bulk density of 0.34 $g \cdot cm^{-3}$, heat conductivity of 0.087 $W \cdot m^{-1} \cdot K^{-1}$, and compression strength of 7.4 MPa. In the second version, colour flat glass, glycerol ($C_3H_8O_3$), Na_2SiO_3, and water were used. The process temperature was the lowest (818°C) and the specific energy consumption had the lowest value (0.86 $kWh \cdot kg^{-1}$). The results showed a product with bulk density of 0.14 $g \cdot cm^{-3}$, heat conductivity of 0.063 $W \cdot m^{-1} \cdot K^{-1}$, and compression strength of 5.3 MPa. The third version used a mixture of post-consumer drinking bottle, $C_3H_8O_3$, Na_2SiO_3, and water. The process temperature reached 823°C and the specific energy consumption was 0.88 $kWh \cdot kg^{-1}$. The product had a bulk density of 0.14 $g \cdot cm^{-3}$, heat conductivity of 0.063 $W \cdot m^{-1} \cdot K^{-1}$, and compression strength of 5.9 MPa. The last version has also included a mixture of post-consumer drinking bottle, silicon carbide (SiC), and water. The temperature required for the foaming process was the highest (922°C) and the energy consumption reached 1.00 $kWh \cdot kg^{-1}$. The product had the bulk density of 0.20 $g \cdot cm^{-3}$, heat conductivity of 0.075 $W \cdot m^{-1} \cdot K^{-1}$, and compression strength of 7.5 MPa. The comparative analysis of the four cellular glass gravel variants indicated the third version as being the optimal variant.

In a more recent paper (Păunescu *et al.*, 2022a) of the current work team, results of the experiment regarding the manufacture of cellular glass gravel through a recipe combining the liquid foaming agent (glycerol) with a solid agent ($CaCO_3$) are presented. The heating technique was also based on the microwave radiation. The basic raw material was the combination of the three types of recycled glass waste (colourless, green, and amber). Glycerol was combined with sodium silicate solution in an increasing ratio from 1:4 to 1:8 and $CaCO_3$ was associated with borax in a slightly decreasing ratio from 0.41 to 0.36. The required temperature of manufacturing process was between 830-840°C and the value of energy consumption varied between 0.99-1.20 $kWh\cdot kg^{-1}$. The experimental results showed a material with bulk density slightly increasing from 0.15 to 0.19 $g\cdot cm^{-3}$, heat conductivity increasing from 0.074 to 0.090 $W\cdot m^{-1}\cdot K^{-1}$, and compression strength also increasing from 6.5 to 7.6 MPa. The conclusion of this experiment showed that the optimal version was that with 0.8% $CaCO_3$, 2% borax, 1% glycerol, 6% sodium silicate, and 9% water, characteristics of the optimal specimen being 0.16 $g\cdot cm^{-3}$ bulk density, 0.073 $W\cdot m^{-1}\cdot K^{-1}$ heat conductivity, and 6.9 MPa compression strength. The energy consumption had the value of 1.03 $kWh\cdot kg^{-1}$.

The current work presented below involved the use of a manufacturing recipe changed from the team's previous work, including the use of recycled flat glass waste and the replacement of $CaCO_3$ as the usual solid foaming agent with a recycled food industry waste (chicken eggshell). Obviously, the technique of heating by microwave radiation in the own adapted oven was preserved, being the main element of the work originality compared to the methods commonly used in the world.

2. Methods and Materials

In the case of adopting the procedure of expanding glass waste under the simultaneous effect of two agents, one solid (calcium carbonate) and the other liquid (glycerol), this process develops independently. According to (Karunadasa *et al.*, 2019), $CaCO_3$ decomposition occurs at over 750°C, CO_2 being released as a result of reaction (1).

$$CaCO_3 = CaO + CO_2 \qquad (1)$$

The reaction is initiated slowly, then develops rapidly. The work (Scarinci *et al.*, 2005) notes that in the case of commercial glass (soda-lime-silica glass) the expansion process with $CaCO_3$ reaches the optimal level within the limits of 800-900°C.

According to Moldoveanu, (2019), the decomposition process of glycerol begins at low temperature (less than 300°C) and intensifies around 750°C. Several gaseous products are released during the decomposition process (carbon monoxide, hydrogen, methane, ethane, carbon dioxide, ethylene, propylene, 1,3-butadiene, and isobutene).

Under the conditions of an adequate viscosity of the heated glass, the gases released in its softened mass remain blocked forming gas bubbles and the solid products resulting from the reaction (as in the case of calcium oxide) enter the composition of the melt. Later, by cooling, the bubbles turn into pores constituting the typical porous structure of the material (Scarinci *et al.*, 2005).

The method of heating the raw material using the ability of electromagnetic waves to carry energy and to convert its own power into heat through the contact with materials susceptible to microwaves has already been noted by researchers active in this field. The principle of converting the microwave power into heat was observed through the direct contact of the wave field with the material subject to heating. The initiation of the thermal process occurs in the central area of the material, where the mentioned conversion is producing. In this area, the thermal peak with the highest temperature develops, after which the heat propagates volumetrically in the entire volume of the material from inside to outside. Obviously, the heat propagation mode is completely opposite to conventional heating techniques. On the other hand, microwave heating applies the principle of selectivity, which allows heating only the targeted material, not other massive bodies in its vicinity. These particular characteristics allow obtaining very high heating rates and a remarkable energy efficiency, with the condition that the external surfaces of the material or its support to be properly protected (Jones *et al.*, 2002; Kitchen *et al.*, 2014).

Tests carried out 5 years ago in the Romanian company Daily Sourcing & Research showed that commercial glass cannot be fully heated directly with microwaves without affecting the internal structure of the foamed material. For this reason, the research team opted for the placement of a protective screen from SiC crucible with reduced wall thickness (2.5 mm) between the material and the microwave emitting source, which allows predominantly direct heating and partially indirect heating thus ensuring optimal conditions and excellent performance for the process development (Axinte *et al.*, 2019).

The oven utilized in this experiment was the same 800 W-microwave equipment described in the works of recent years related to the manufacture of cellular glass, being constructively and functionally adapted from the microwave oven usually used in the household for food preparation. A ceramic tube made of SiC and Si_3N_4 in 80/20 ratio with a wall thickness of 2.5 mm made in China, provided with a ceramic lid, represented the protective screen for tempering the too intense effect of the wave field on the pressed material mixture. Effective thermal protection to avoid heat loss outside was achieved with commercially available ceramic fiber mattresses resistant to 1200°C. Figure 1 presents images of the microwave oven (a), ceramic tube (b), and the thermal protection of tube and lid.

a b c

Fig. 1 – Experimental equipment
a – 800 W-microwave oven; b – ceramic tube; c – thermal protection.

Materials used in this experiment were: colourless flat glass coming from recycled window glass waste as basic raw material, glycerol as a liquid expanding agent, sodium silicate also known as water glass in solution with 38% concentration, chicken eggshell as food industry waste, rich in $CaCO_3$, and borax as a fluxing agent.

A standard composition of colourless flat glass contains 70-73% SiO_2, max. 1.5% Al_2O_3, 8.0-9.7% CaO, 3.5-4.5% MgO, 13.4-14.6% Na_2O, and max. 0.2% Fe_2O_3 (Collection, 2008). The recycled glass was washed, dried, broken, ground in a ball mill, and sieved, the selected grain size being below 100 µm.

Glycerol was used in the mixture as an effective liquid foaming agent of carbonic origin. According to (Dragoescu *et al*., 2020), its physical state facilitates easy penetration among the fine glass particles and as a result leads to obtaining the very fine porosity of cellular glass, because by decomposition it releases numerous gaseous compounds that help the expansion of the material.

Sodium silicate (water glass) also as an aqueous solution is used in general in association with glycerol in expansion processes. Also, it has ability to play the role of expanding agent even alone (Hribar *et al.*, 2023).

Chicken eggshell was adopted as a by-product of the food industry because it is very rich in $CaCO_3$ (between 86-92%) according to (Nimisha *et al.*, 2020). Eggshell was ground in an electrical laboratory device, the particle size being decreased under 40 µm. This replacement of $CaCO_3$, a frequently used foaming agent, but a relatively expensive product, with a waste represents another element of originality for this paper.

Borax (sodium borate) has the main role in this mixture as a fluxing agent due to its important content of Na_2O (30.8%). On the other hand, the contribution of boron (about 11%) in borax composition in increasing the mechanical strength of the material is well known. Available on the market with a grain size below 400 µm, the borax powder was supplementary ground, its grain dimension being reduced under 100 µm.

Four experimental manufacturing recipes presented in Table 1 were chosen and tested for the production of porous and strength material. The mixture included 82.4-92.3% colourless flat glass, 1.1% glycerol, 4.2-9.0%

Na_2SiO_3, 1.0-2.5% chicken eggshell, and 1.4-3.2% borax. Distilled water (8.5%) was added. An almost similar recipe from the point of view of the mixture composition, excepting the use of $CaCO_3$ instead of eggshell (Păunescu *et al.*, 2022) was previously experimented by authors' team of the current paper. The product had bulk density under 0.19 $g \cdot cm^{-3}$, and compression strength within the limits of 6.5-7.6 MPa.

Table 1

Composition of experimental versions (wt. %)

Composition	Version 1	Version 2	Version 3	Version 4
Colourless flat glass	92.3	89.0	85.7	82.4
Glycerol	1.1	1.1	1.1	1.1
Na_2SiO_3	4.2	5.8	7.4	9.0
Chicken eggshell	1.0	1.5	2.0	2.5
Borax	1.4	2.0	2.6	3.2
Distilled water	8.5	8.5	8.5	8.5

Known methods were applied to identify the main characteristics of cellular glass specimens. Bulk density was calculated as the ratio of the material mass to the volume of the container containing material lumps (Changmai and Kumar Purkait, 2021). Porosity was measured by mercury porosimetry and gas adsorption (ISO 15901-2: 2022). Heat conductivity was determined with HFM 466 Lambda apparatus using heat-flow method (SR EN 1946-3: 2004), while the compression strength was measured with 100 kN-hydraulic axial press equipment (EN 826: 2013). Water-absorbing of specimens was determined by the method of their immersion under the water (ASTM D570). Analyzing the microstructural appearance of cellular glass specimens was carried out with ASONA 100X Zoom Smartphone Microscope.

3. Results and Discussion

As mentioned above, the sintering-expanding process of the pressed mixtures corresponding to the four versions deposited freely in the inner space of the ceramic tube was performed in the 800 W-microwave oven. The functional parameters in the case of each version were identified, the results being shown in Table 2. The analysis of results in Table 2 shows that using a larger amount of wet raw material compared to that in the paper (Păunescu *et al.*, 2022a) the duration of the microwave heating process was kept almost within the same limits (34-40 min) due to the increase in the heating rate at significantly higher values (20.5-23.7°C/min).

Table 2

Functional parameters of the sintering-expanding process

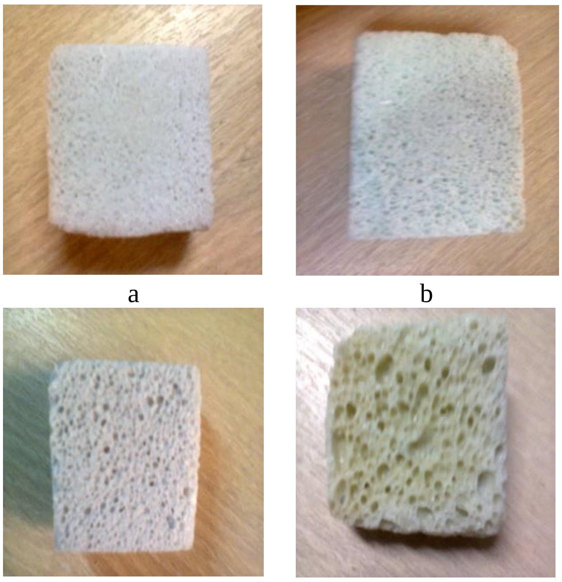
Parameter	Version 1	Version 2	Version 3	Version 4
Wet raw material/cellular glass product quantity (g)	470/ 423	470/ 422	470/ 424	470/ 424
Process temperature (°C)	825	830	835	840
Heating duration (min)	34	35	37	40
Average rate (°C/min)				
- heating	23.7	23.1	22.0	20.5
- cooling	5.8	5.9	5.7	5.8
Index of volume growing	1.40	1.60	1.80	2.40
Specific consumption of electricity ($kWh\cdot kg^{-1}$)	0.84	0.86	0.91	0.98

The growing in volume of specimens under the influence of expanding agents (glycerol and eggshell) was more pronounced (between 1.40-2.40), showing that the recycled waste (eggshell) is a very effective substitute for $CaCO_3$. A special mention is for the very low values of energy consumption (0.84-0.98 $kWh\cdot kg^{-1}$) of the cellular product manufacturing process, lower than that obtained in industrial processes using conventional heating.

Figure 2 presents appearance images of cellular glass products obtained in the four versions of this experiment.

Determining the physical, mechanical and microstructural characteristics of cellular glass specimens through the investigation methods mentioned above led to the results shown in Table 3.

Examining the results in Table 3 showed that cellular glass specimens have physical properties required by the purpose of their manufacture, i.e. bulk densities within the limits of 0.16-0.19 $g\cdot cm^{-3}$, heat conductivities between 0.069-0.079 $W\cdot m^{-1}\cdot K^{-1}$, and corresponding porosities (in the range of 84.7-85.8%). On the other hand, the compression strength values are high for specimens corresponding to versions 1 and 2 (7.6 MPa) and, although they decrease to 6.5 MPa (version 3) and 5.7 MPa (version 4), remain sufficiently high, being suitable for the purpose of manufacturing these construction materials. Water-absorbing falls within the normal level of these porous and strength products (1.6-2.4 vol. %).



a b

c d

Fig. 2 – Appearance of cellular glass specimens
a – version 1; b – version 2; c – version 3; d – version 4.

Table 3

Physical, mechanical, and microstructural features of cellular glass specimens

Feature	Version 1	Version 2	Version 3	Version 4
Bulk density (g·cm^{-3})	0.19	0.18	0.16	0.16
Porosity (%)	84.7	84.9	85.6	85.8
Heat conductivity (W·m^{-1}·K^{-1})	0.079	0.077	0.072	0.069
Compression strength (MPa)	7.6	7.6	6.5	5.7
Water-absorbing (vol. %)	2.4	2.2	1.9	1.6
Pore size (mm)	0.2-0.9	0.3-1.5	1.4-2.4	1.4-3.9

Microstructural appearance of cellular glass specimens is shown in Fig. 3. According to these pictures, the cell sizes that make up the specimen microstructure are increasing from 0.2-0.9 mm (version 1) to 1.4-3.9 mm (version 4). The microstructural aspect of specimens 1-3 is regular and homogeneous, while that of the specimen 4 is coarser compared to the others.

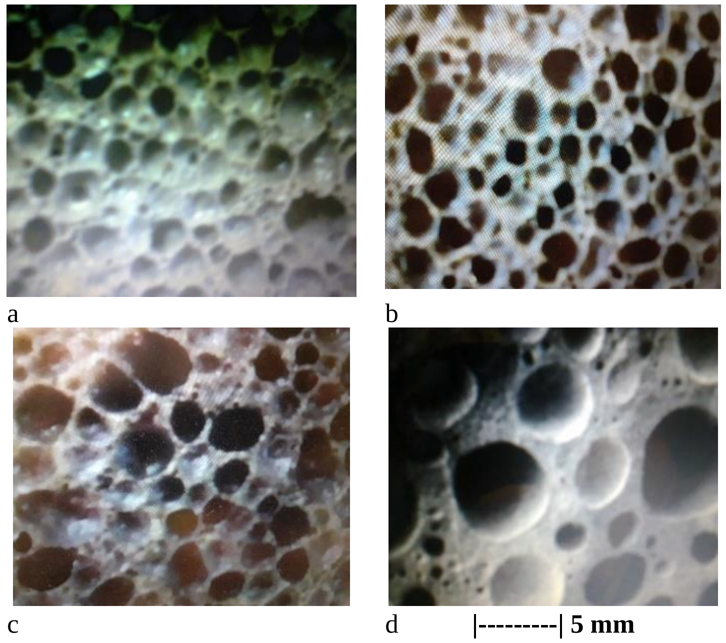


Fig. 3 – Microstructural appearance of cellular glass specimens
a – version 1; b – version 2; c – version 3; d – version 4.

The comparative analysis of cellular glass specimen features might point to version 2 as the best option. The mixture was composed of 89.0% flat glass, 1.1% glycerol, 5.8% water glass, 1.5% eggshell, 2.0% borax, and 8.5% distilled water. The sintering-expanding process temperature was 830℃ and the process duration was 35 min corresponding to the heating rate of 23.1℃/min and the specific consumption of electricity of 0.86 $kWh \cdot kg^{-1}$.

Both in qualitative terms and in economic terms producing the mentioned specimen using the microwave heating is recommended.

The manufacturing method that radically differentiates the experiment performed in Daily Sourcing & Research from the industrially used methods is the application of the unconventional microwave heating technique. This technique, unused in the world in processes at high temperatures, was adopted by authors of the current paper in expansion processes of residual glass, being tested on a very small scale (800 W). Experiments on a larger scale (10 kW) were carried out by the same team on an improvised microwave oven. The results were even in this situation promisingly good (Păunescu *et al*., 2021; Păunescu *et al*., 2022 b). Currently, efforts are being made to create a pilot plant with continuous operation for the production of cellular glass by microwave heating.

4. Conclusions

The paper aimed at testing a new method of manufacturing strength and porous cellular glass with load-bearing properties, in which the calcium carbonate usually used as an expanding agent was replaced with a very rich in $CaCO_3$ by-product of the food industry (chicken eggshell). The unconventional method of microwave heating through the original predominantly direct and partially indirect heating designed by authors of the paper was also applied in this experiment. The material mixture prepared for the manufacture of cellular glass from flat glass waste simultaneously included two types of expanding agent: glycerol (liquid) and chicken eggshell (solid), as well as water glass and borax as materials usually associated with the two agents. Products obtained in four experimental versions by heating in the range of 825-840℃, with high rates of over 20℃/min had bulk density between 0.16-0.19 $g \cdot cm^{-3}$ and compression strength up to 7.6 MPa. The specific consumption of energy was very low in the range of 0.84-0.98 $kWh \cdot kg^{-1}$, being more economic than in the industrial production. The authors' concern for the future is the realization of a continuous pilot plant for manufacturing the cellular glass by microwave heating.

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MATERIAL DE CONSTRUCȚIE REZISTENT ȘI POROS FABRICAT DIN STICLĂ REZIDUALĂ RECICLATĂ PRIN RADIAȚIA MICROUNDELOR

(Rezumat)

A fost concepută și testată prin încălzire cu microunde o nouă metodă de producere a sticlei celulare rezistente și poroasă cu proprietăți portante din sticlă plată reciclată, utilizând simultan doi agenți de spumare: glicerină (lichidă) și coajă de ou (solidă) reciclată ca produs secundar din industria alimentară. Coaja de ou a fost utilizată ca înlocuitor al carbonatului de calciu, iar încălzirea cu microunde s-a făcut prin încălzire predominant directă și parțial indirectă, conducând la economisirea energiei. Rezultatele au arătat că s-au obținut probe de sticlă celulară cu densitate redusă în vrac (0,16-0,19 g·cm^{-3}) și rezistență la compresie înaltă (până la 7,6 MPa).