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CELLULAR GLASS AS INSULATION MATERIAL IN CONSTRUCTION UNDER CONDITIONS OF MECHANICAL STRESS

BY

LUCIAN PĂUNESCU[1,*], SORIN MIRCEA AXINTE[2,3] and ALEXANDRU FITI[1]

[1]Cosfel Actual SRL Bucharest, Romania
[2]Daily Sourcing & Research SRL Bucharest, Romania
[3]Department of Applied Chemistry and Materials Science,
University "Politehnica" of Bucharest, Romania

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Abstract. Cellular glass with excellent thermal insulation properties (low density of 0.26 $g \cdot cm^{-3}$, very low heat conductivity of 0.064 $W \cdot m^{-1} \cdot K^{-1}$, and high porosity of 87.6%), and in the same time, relatively high compressive strength of 6.0 MPa was prepared. The mixture was composed of finely ground solid materials (glass waste, borax, and Na_2CO_3) and liquids (Na_2SiO_3 solution and water addition), separately processed and then mixed together. The obtained dense paste was poured into a mold, pressed, removed from the mold, to be freely loaded into the microwave oven. Sintered at 825ºC, the expanded material resulted as a cellular product adequate for using as insulation material in construction under the conditions of mechanical stress.

Keywords: cellular glass, recycled glass waste, sodium carbonate, borax, sodium silicate solution.

*Corresponding author; *e-mail*: lucianpaunescu16@gmail.com

1. Introduction

In the current conditions of the global energy and environmental crisis, recycling different types of waste (metals, plastics, glasses, paper and cardboard, etc.) has become a necessity, even more so in countries with developed economies also affected by high annual generation rates of these wastes.

Glass recycling for the glass industry (re-introducing it as a raw material in the new glass manufacturing circuit) is making still more than 50 years ago, having an important role in reducing the energy consumption of the industrial process and reducing carbon dioxide CO_2 emissions. However, the high cost of separating glass by colour (that differs in terms of quality) influences the degree of residual glass recycling from 90% in the case of green glass production, to 70% in the case of amber glass, reaching only 60% in the case of colourless glass (Harder, 2018).

Currently, around 130 million tons of glass is manufacturing in the world, of which 48% represent containers or hollow glass, 42% represent flat glass products, and 5% constitute tableware. The total amount of recycled glass worldwide does not exceed 27 million tons (21%), of which the container glass represents 32%, having the highest recycling rate, while the flat glass represents only 11%, having the lowest recycling rate (Harder, 2018). According to the data provided in 2015 by The European Container Glass Federation (FEVE) (FEVE, 2015), the main countries in the EU that reach the highest recycling rates of over 95% (Sweden, Belgium and Slovenia) have efficient separate collection systems for glass waste.

In the last decades, the interest of the construction sector towards cellular glass products manufactured by the expansion of finely ground glass waste mixed with an appropriate pore-forming agent, following a sintering process at high temperature (750-1150ºC) depending on the agent nature significantly increased (Scarinci *et al.*, 2005). Cellular glass is mostly used as a thermal or acoustic insulator in construction due to its remarkable properties (light weight, low thermal conductivity, high porosity, physical and chemical stability, non-flammability, impermeability, non-toxicity, corrosion resistance, resistance to different external attacks of rodents, insects, bacteria, etc.) (Bian *et al.*, 2018; Sari *et al.*, 2022; Scarinci *et al.*, 2005).

Except glass waste as a silicate material used for the manufacture of lightweight cellular glass, a large number of other types of silicate waste such as coal fly ash as a by-product of coal burning in the energy industry, metallurgical slag as a by-product of pig iron or steel production, sludge captured from zinc hydrometallurgy, dust and fly ash captured in the filters of waste incinerators, etc., mixed with glass waste and a blowing agent and subjected to heat treatment at high temperatures, even over 1100ºC, ensure the premises for the production of cellular glass-ceramics. These are cellular products with a predominantly crystalline structure (within the limits of 50-95%), the rest being amorphous

residual glass, obtained through the controlled crystallization of glass. According to the literature (Rawlings *et al.,* 2006), only typical compositions of glass can be precursors of glass-ceramics. Crystallization of glass in the form of cellular glass-ceramics is a heterogeneous transformation process composed of two stages: nucleation, in which stable volumes of the crystalline phase are formed, and growing, which involves the movement of glass atoms or molecules across the glass/crystal interface as well as in the crystal (Rawlings *et al.*, 2006).

The fields of application of cellular glass-ceramics overlap with the fields of application of light cellular glass, with the difference that the mechanical strength of these materials is usually higher. The manufacture of cellular glass or cellular glass-ceramics has also focused on areas where the mechanical stress is significantly higher (such as road construction, foundation infrastructure, lightweight concrete aggregates, sports fields, etc. (Zegowitz, 2010; Zhu *et al.*, 2016).

The main assortments of industrially manufactured cellular glass in the world are "TECHNOpor", under the license of the Misapor Switzerland Company, with branches in Germany, France and Austria and "Foamglas", under the license of the Pittsburgh Corning Company, with branches in the USA, Europe (Belgium, Great Britain, Czech Republic, Germany), and China.

Industrially manufactured "TECHNOpor" products are characterized by excellent durability, high compressive strength (4.9-6.0 MPa), low thermal conductivity (0.075-0.095 $W \cdot m^{-1} \cdot K^{-1}$), low density (1.21-1.40 $g \cdot cm^{-3}$), physical rigidity, resistance to frost, impermeability, non-flammability, and external aggression of acids, salts, rodents, insects and bacteria. Products of this type are suitable for foundations in light weight constructions, floor slabs of industrial halls, road construction, railway embankments, bridge abutments, airport runways, industrial roofs, drainage, sports fields, etc. (Technical Information, 2016).

The typical scheme of the technological flow for producing the "Foamglas" product includes making the molten glass in a controlled chemical composition using raw materials and ingredients predominantly based on waste glass. The molten glass is unloaded from the furnace on metal conveyor belts, where it is cooled before being introduced into the ball mill where carbon black is added as a blowing agent. After very fine grinding, the powder is loaded into metal molds that are transported along a heating furnace with metal conveyor belt or roller hearth at 850ºC resulting in the typical cellular product. A controlled cooling follows, avoiding thermal shocks, after which the molds are unloaded by turning over and the porous material is cut to the required dimensions. "Foamglas" products are industrially manufactured in the form of blocks for the thermal insulation of building masonry, being compressive resistant and waterproof as well as in the form of broken pieces of flat mass sintered on the metal conveyor belt being used as porous aggregates. According to the technology applied at the Tessenderlo plant (Belgium), the starting mixture

consists of ground glass waste and carbon black as an expanding agent to obtain cellular glass-ceramics suitable for seals, coatings, linings, adhesives, etc. The main characteristics of "Foamglas" products are: uniform (constant) thermal insulation, absolute tightness against water and water vapour, moderate compression strength (1.6-2.75 MPa) without deformations (Foamglas, 2016).

The manufacture of cellular glass in the form of gravel or aggregate is the range of cellular products made by Geocell Schaumglas from Gaspoltshofen (Austria) (Geocell, 2015) with two facilities in Germany and one in Austria. The Austrian company is listed as one of the most important manufacturers of cellular glass in Europe. Its products, characterized by low density, compressive strength, heat-insulating properties, drainage, and low weight loading, represent durable substitutes for traditional construction materials. Fields of using include underground thermal insulation, insulation of new floors in old buildings, insulation for roofs, filling material for landscaping, roof gardens, filling material for road and railway construction structures. The cellular glass aggregate is produced in the form of pieces with sizes within the limits of 10-90 mm, easily separated as a result of the internal tension created into the mass of sintered material through a more intense cooling. The basic raw material is recycled glass waste, to which low proportions of mineral additives with the role of expanding or facilitating this process are added (Geocell, 2015).

Except for the industrial manufacturing results of the cellular glass products shown above, different experiments aimed at this field have been carried out and presented in the literature.

Using calcium carbonate (1-3%) and ornamental stone processing waste (5-10%) as pore-forming agents and recycled glass bottle (85-95%), glass foam was experimentally produced by conventional heating and sintering at 900ºC, the heating rates having various values within the limits of 3-20ºC/min. The stone processing waste was chosen due to its dolomite content of about 15%. The investigation of the physical, thermal and mechanical characteristics of specimens showed that the porosity was between 75-90%, thermal conductivity in the range of 0.04-0.07 $W \cdot m^{-1} \cdot K^{-1}$, and compressive strength reached the maximum value of 4.3 MPa (Souza *et al.*, 2022).

Cellular glass-ceramic was experimentally manufactured using blast furnace slag and waste glass as raw materials contributing with TiO_2, ZrO_2, and CaF_2 as nucleating agents in the making process (Ding *et al*., 2015). $CaCO_3$ as a blowing agent, trisodium phosphate ($Na_3PO_4 \cdot 12H_2O$) as a foaming stabilizer, and pentahydrate borax ($Na_2B_4O_7 \cdot 5H_2O$) as a fluxing agent were also added. Results indicated that cellular glass-ceramic with 50% blast furnace slag led to excellent comprehensive properties (bulk density of 0.79 $g \cdot cm^{-3}$, water absorption of 2.71 vol. %, and flexural strength of 14.34 MPa.

All the manufacturing processes (industrial or experimental) mentioned above were carried out by conventional heating methods. The experiment carried out by authors of the current work cited further (Păunescu *et al.*, 2020) was

carried out using the unconventional technique of microwave heating. The starting mixture included coloured glass waste (83.5-89.5%), $CaCO_3$ as a blowing agent (1.5%), borax (3-8%), and sodium silicate solution (3-8%). The thermal process took place at temperatures between 835-855ºC, the heating rate varying between 19-21.4ºC/min. The results obtained in the four experimental options were variable, the optimal option being that using 3% borax and 8% sodium silicate. The characteristic values of the optimal specimen were: apparent density of 0.62 $g \cdot cm^{-3}$, porosity of 71.8%, heat conductivity of 0.087 $W \cdot m^{-1} \cdot K^{-1}$, compressive strength of 7.4 MPa, water absorption of 7.4 vol. %, and pore size between 1-1.6 mm.

Another work that adopted the unconventional method of thermal treatment of the starting mixture by microwave irradiation (Păunescu *et al.*, 2021) has used in the optimal option eggshell waste (4%) as a rich $CaCO_3$-expanding agent for glass waste (91%) as well as borax (5%) as a fluxing agent, and 9% water addition as a binder. The temperature of the sintering process was 823ºC, with a high average heating rate of 22.3ºC/min. Cellular glass characteristics were: apparent density of 0.40 $g \cdot cm^{-3}$, porosity of 81%, heat conductivity of 0.086 $W \cdot m^{-1} \cdot K^{-1}$, compressive strength of 4.3 MPa, and cell dimensions in the range of 0.3-0.9 mm. Specific energy consumption of the making process was extremely low of only 0.8 $kWh \cdot kg^{-1}$.

Cellular glass-ceramic from blast furnace slag and container glass waste in 40/60 weight ratio with high compressive strength (14.1 MPa) was prepared applying the microwave heating technique (Grigoras *et al*., 2020). The finely ground material mixture also included $CaCO_3$ (6.5%) as a blowing agent, borax (7.8%) as a fluxing agent, TiO_2 (5%) as a nucleating agent, $Na_3PO_4 \cdot 12H_2O$ as a foam stabilizer, and water (8%) as a binder. The sintering temperature was 900ºC. Characteristics of optimal product specimen were: density of 0.82 $g \cdot cm^{-3}$, porosity of 75.9%, heat conductivity of 0.135 $W \cdot m^{-1} \cdot K^{-1}$, water absorption of 3.4 vol. %, and pore size between 0.3-0.6 mm. Specific energy consumption was very low of 0.9 $kWh \cdot kg^{-1}$.

The current work aimed at increasing the weight proportion of Na_2O as a fluxing agent included in the composition of sodium carbonate (Na_2CO_3) used as a blowing agent replacing $CaCO_3$. On the other hand, borax as sodium borate contributes both to the supply of Na_2O in the starting mixture composition, as well as boron in the form of B_2O_3, a component with the role of significantly increasing the mechanical strength of the final product. Sodium silicate as a liquid solution has the advantage of easily dissipating among the fine particles of the solid mixture. Some experiments presented in the literature indicate the possible use of Na_2SiO_3 as the only expanding agent without another traditional agent (Uribe *et al*., 2020), although in general, its application in foaming processes is known in association with glycerol as the main agent.

The main originality element characteristic to most of works carried out in the field of thermal sintering/foaming of silicate waste in the last seven years

in the common experimental base of Romanian companies Daily Sourcing & Research SRL and Cosfel Actual SRL is using the unconventional technique of microwave heating, advantageous in economic and ecological terms, compared to the conventional techniques adopted in the other papers from similar fields presented in the literature. In particular, the originality of this paper consists in the choice of Na_2CO_3 as a blowing agent for waste glass powder due to the possibility of adding Na_2O as a fluxing agent thus introduced into the material mixture.

2. Methods and Materials

Like in the general case of mixed material mixtures (solid/liquid), the preparation of the two component types was separately carried out. Finely ground glass waste (below 80 μm), borax and Na_2CO_3 were processed by dry mixing in a blender for 5 min, Na_2SiO_3 solution diluted with water was stirred in a glass vessel at a rate of 400 rpm for 3 min. After pouring Na_2SiO_3/water over the solid powder, the stirring process continued for another 3-4 min until a dense paste was obtained, which then was poured into a bottomless cylindrical metal mold with an inner diameter of 65 mm and a height of 60 mm, being manually pressed. After a maximum of 10 min, the compact pressed material was removed from the mold to be freely loaded into the microwave oven.

The blowing agent adopted in this experiment (Na_2CO_3) releases Na_2O (as a fluxing agent) and CO_2 (as a blowing gas) through thermal decomposition, which is initiated around 550ºC, but can continue up to over 900ºC (Scarinci *et al*., 2005).

A heat-insulating layer of ceramic fiber mats was placed at the base of the oven, on which a metal plate and the pressed material were deposited. The material subjected to heating was protected from the microwave field emitted from the waveguide placed in the side wall of the oven by a ceramic tube made of silicon carbide and silicon nitride, excellent microwave absorbing materials. The outer diameter of the tube was 125 mm, the height was 100 mm, and the wall thickness was 2.5 mm, the tube being purchased from China. A lid made of the same material with the thickness of 5 mm covered the ceramic tube.

The peculiarity of direct microwave heating is completely different from the conventional heating, the process initiation being done in the middle of the irradiated material where the power of electromagnetic waves is converted into heat. The heat developed inside the material propagates volumetrically in its entire mass from the inside to the outside (Jones *et al*., 2002; Kitchen *et al*., 2014). That is why it is important to effectively protect the outer surface of the ceramic tube and lid.

The oven used in the experiment was an 800 W-household type microwave oven (Fig. 1) constructively adapted for operation at high temperature (up to 1200ºC). The monitoring of the temperature on the hot surface of the

material was carried out with a Pyrovar type-radiation pyrometer mounted above the oven, which visualized the material through holes provided in the metal ceiling of the oven and the ceramic lid (Păunescu *et al*., 2022).

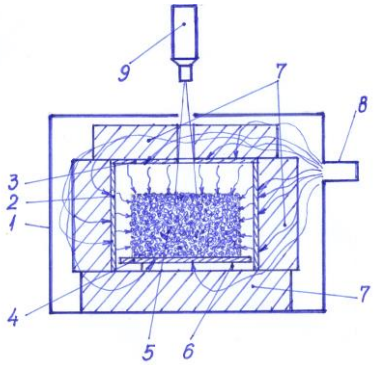

Fig. 1 – Composition of equipment for making the cellular glass
1 – microwave oven; 2 – ceramic tube; 3 – ceramic lid; 4 – metal plate; 5 – pressed material; 6 – metal support; 7 – ceramic protection; 8 – waveguide; 9 – pyrometer.

Post-consumer colourless container glass was the basic raw material used in the experiment. The recycled glass waste was selected by colour, washed, dried, broken, ground in a ball mill and sieved, so that the grain size below 80 μm was allowed. The chemical composition of the colourless glass included the following components: 71.7% SiO_2, 1.9% Al_2O_3, 12.0% CaO, 1.0% MgO, 13.3% Na_2O, 0.05% Cr_2O_3, and 0.05% other oxides.

Available in the market with a grain size below 400 μm, borax ($Na_2B_4O_7 \cdot 5H_2O$) has been mechanically processed by grinding to sizes below 100 μm.

Sodium carbonate (Na_2CO_3) with a concentration of 99% in the form of fine powder was also commercially purchased as well as the aqueous solution of sodium silicate (Na_2SiO_3) with a concentration of 38%.

The composition of the material mixture was dosed in four experimental versions shown in Table 1, in which borax varied between 5-11%, Na_2CO_3 within the limits of 3-4.5%, and Na_2SiO_3 in the range of 3-12%. The water addition as a binder was kept constant at 8%.

Table 1
Composition of materials used in experiment

Version	Glass waste (wt. %)	Borax (wt. %)	Na_2CO_3 (wt. %)	Na_2SiO_3 solution (wt. %)	Water addition (wt. %)
1	91.0	5	3.0	3	8
2	85.5	7	3.5	6	8
3	80.0	9	4.0	9	8
4	74.5	11	4.5	12	8

The apparent density of specimen was determined by its weighing with an electronic balance and calculation of volume based on geometrically dimensions. Reporting the mass to the volume gave the density value (Manual, 1999). Measuring the porosity involved the identification of true density of material after its melting and cooling (so of the material without pores). The percentage comparison of true density with apparent density allowed to evaluate the porosity value (Anovitz and Cole, 2005). Applying the heat-flow method (SR EN 1946-3:2004) led to identify heat conductivity. 10 kN-hydraulic axial press machine (EN 826:2013) was the facility used for measuring the compressive strength. The method of immersing the specimen under water was adopted for determining volumetrically proportion of water absorbed into the specimen mass in 24 hours (BS 1881-122:2011). Microstructural image of specimen section was obtained with Smartphone Digital Microscope ASONA type 100X Zoom.

3. Results and Discussion

The operation data of the sintering process for obtaining cellular glass products are presented in Table 2.

Table 2
Operation data of the sintering/expanding process

Option	Wet raw material/ cellular glass amount (g)	Sintering/ expanding temperature (ºC)	Heating time (min)	Average rate (ºC/min)		Index of volume increasing	Specific consumption of energy (kWh·kg^{-1})
				Heating	Cooling		
1	420/395.8	810	31	25.5	5.0	1.20	0.86
2	420/376.4	825	33	24.4	5.0	1.27	0.91
3	420/375.0	840	36	22.8	5.0	1.36	1.00
4	420/375.2	857	41.5	20.2	5.0	1.50	1.15

According to the data in Table 2, the thermal process of manufacturing the cellular glass was slightly influenced by the increase in the proportions of Na_2CO_3 from 3 to 4.5% and Na_2SiO_3 solution from 3 to 12%. As a result, growing in volume of the specimen mass followed an increasing slope. The optimal temperature for completing the expanding process was also increasing between 810-857ºC with the increase in the content of sodium carbonate and silicate, both with a foaming role.

The aspect of the four experimentally made specimens is shown in Fig. 2.

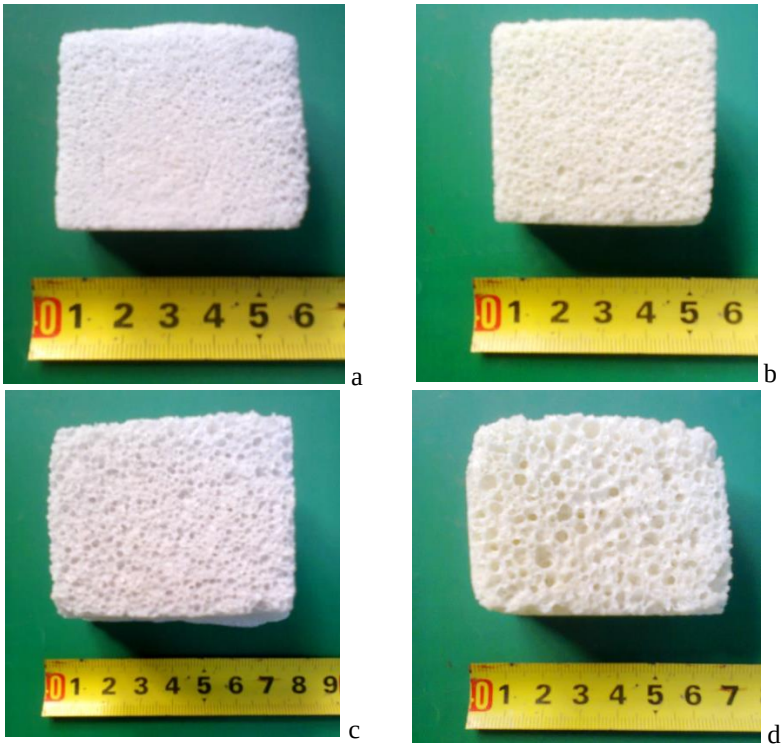

Fig. 2 – Aspect of cellular glass specimens
a – version 1; b – version 2; c – version 3; d – version 4.

Macrostructurally, the specimens shown in Fig. 2 have an appearance that changes with the increase of the content of Na_2CO_3 and Na_2SiO_3. Thus, the cell size is increasing from below 1 mm in the case of option 1, to much higher values up to about 3 mm in the case of option 4.

The application of the characterization methods of the cellular glass specimens allowed the identification of their main characteristics. Table 3 presents these results.

Table 3
Cellular glass characteristics

Characteristic	Version 1	Version 2	Version 3	Version 4
Apparent density ($g \cdot cm^{-3}$)	0.29	0.26	0.24	0.22
Porosity (%)	86.2	87.6	88.6	89.5
Heat conductivity ($W \cdot m^{-1} \cdot K^{-1}$)	0.068	0.064	0.060	0.057
Compressive strength (MPa)	6.5	6.0	5.4	4.9
Water absorption (vol. %)	1.3	1.0	1.1	0.9
Pore size (mm)	0.7-1.2	1.5-2.3	1.7-2.4	2.0-2.9

The characteristics of the specimens according to Table 3 meet remarkable values that show excellent thermal insulation properties (low density between 0.22-0.29 $g \cdot cm^{-3}$, very low heat conductivity within the limits of 0.057-0.068 $W \cdot m^{-1} \cdot K^{-1}$, and high porosity in the range of 86.2-89.5%), and in the same time, very good compressive strength of 4.9-6.5 MPa. Water absorption is at a very low level between 0.9-1.3 vol. %.

The microstructural organization of the specimens is shown in Fig. 3.

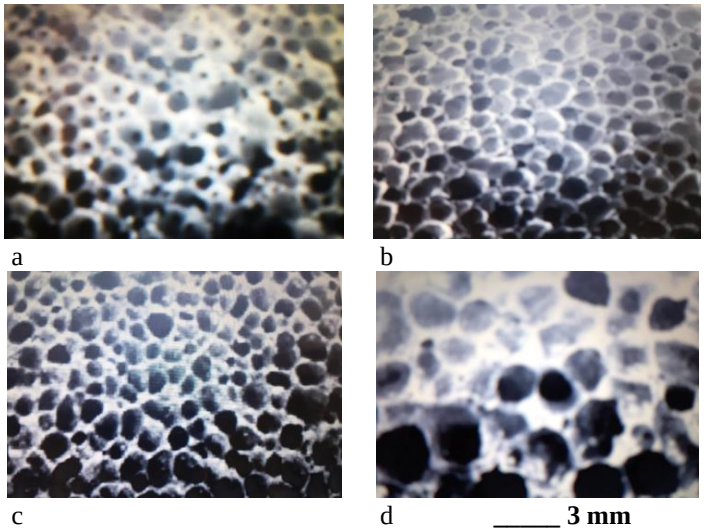

Fig. 3 – Microstructural aspect of cellular glass specimens
a – version 1; b – version 2; c – version 3; d – version 4.

Examining the images in Fig. 3 shows the homogeneous aspect of the microstructural organization of specimens. The size of cells is uniformly increasing from low values characteristic of experimental version 1 (between 0.7-1.2 mm) to the larger values that characterize version 4 (between 2.0-2.9 mm).

The manufacturing process of cellular glass under the conditions of using the unconventional microwave heating technique led to very low energy consumption below 1 $kWh \cdot kg^{-1}$ (between 0.86-1 $kWh \cdot kg^{-1}$), except for version 4, whose heating duration was the highest.

The analysis of experimental results in their entirety determined the choice of version 2 as the optimal version. This version allowed obtaining excellent thermal insulation properties (density of 0.26 $g \cdot cm^{-3}$, heat conductivity of 0.064 $W \cdot m^{-1} \cdot K^{-1}$, and porosity of 87.6%), compressive strength reaching relatively high value of 6.0 MPa. The preparation of the optimal cellular product involved the mixing of 85.5% glass waste, 7% borax, 3.5% Na_2CO_3, 6% Na_2SiO_3 solution, and 8% water addition, the final temperature of the thermal process reaching 825ºC. The heating rate was high (24.4ºC/min) compared to the usual

values of conventional heating (5-15ºC/min) in similar manufacturing processes, without affecting the microstructural homogeneity of the final product.

The choice of Na_2CO_3 as a blowing agent had the advantage of bringing Na_2O into the material mixture with the role of fluxing, excluding the release of CO_2 in the form of foaming gas into the thermally softened material forming gas bubbles.

Known for 70-80 years ago and applied especially in the field of transmissions and radar as well as in some industrial processes of drying and heating solids at low temperature, microwaves applied in high temperature heating processes have shown their excellent energy efficiency by comparison with the conventional heating. The Romanian companies Daily Sourcing & Research SRL and Cosfel Actual SRL have developed in the last 7-8 years unconventional heating techniques applied in experimental works of cellular glass making, that confirmed their efficiency in terms of energy.

4. Conclusions

The present work concerned the experimental manufacture of dense cellular glass using a mixture composed of solid materials (recycled post-consumer container glass, borax, and Na_2CO_3) and liquids (Na_2SiO_3 solution and water addition), processed separately and then mixed together. The resulted paste was poured into a mold, pressed, removed from the mold, to be freely loaded into the microwave oven. The material was sintered and expanded at 825ºC resulting the cellular product with the following characteristics: apparent density of 0.26 $g \cdot cm^{-3}$, heat conductivity of 0.064 $W \cdot m^{-1} \cdot K^{-1}$, porosity of 87.6%, compressive strength of 6.0 MPa, water absorption of 1 vol. %, and pore size within the limits of 1.5-2.3 mm. The specific energy consumption of the producing process was at a low level of 0.91 $kWh \cdot kg^{-1}$. The cellular product has adequate properties for using as insulation material in construction under condition of mechanical stress.

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STICLĂ CELULARĂ CA MATERIAL IZOLATOR ÎN CONSTRUCȚII ÎN CONDIȚII DE SOLICITARE MECANICĂ

(Rezumat)

A fost preparată o sticlă celulară cu excelente proprietăți termoizolante (densitate redusă de 0,26 $g \cdot cm^{-3}$, conductivitate termică foarte mică de 0,064 $W \cdot m^{-1} \cdot K^{-1}$ și porozitate înaltă de 87,6%) și în același timp, rezistență la compresiune relativ înaltă de 6,0 MPa. Amestecul a fost compus din materiale solide fin măcinate (deșeu de sticlă, borax și Na_2CO_3) și lichide (soluție de Na_2SiO_3 și adaos de apă), procesate separat și apoi amestecate împreună. Pasta densă obținută a fost turnată într-o matriță, presată, scoasă din matriță, pentru a fi încărcată liber în cuptorul cu microunde. Materialul expandat sinterizat la 825ºC, a rezultat ca un produs celular adecvat utilizării ca material izolator în construcții în condiții de solicitare mecanică.