High-strength cellular glass made by an effective unconventional technique

Sticlă celulară de mare rezistență realizată printr-o tehnică neconvențională eficientă

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Abstract. *The manufacture of cellular glass using recycled residual glass as the basic raw material had the objective of obtaining a porous material with excellent thermal insulation properties and, at the same time, achieving a relatively high compression strength for construction applications. The work is original due to the type of heating adopted (unconventional) and the own solution chosen to obtain the maximum heating efficiency without affecting the quality of the final product (partially direct heating with microwaves and partially indirect heating by thermal radiation). The energy efficiency of the process (specific consumption below 1 kWh/kg) was remarkable.*

Key words: *cellular glass, recycled residual glass, borax, calcium carbonate, microwave.*

Rezumat. *Fabricarea sticlei celulare utilizând sticlă reziduală reciclată ca materie primă de bază a avut ca obiectiv obținerea unui material poros cu excelente proprietăți termoizolante și, în același timp, realizarea unei rezistențe la compresiune relativ înaltă pentru aplicații in construcții. Lucrarea este originală datorită tipului de încălzire adoptată (neconvențională) și propriei soluții alese pentru a obține eficiemța maximă de încalzire fără afectarea calității produsului final*

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(parțial, încalzire directă cu microunde și parțial, încălzire indirectă prin radiație termică). Eficiența energetică a procesului (consum specific sub 1 kWh/kg) a fost remarcabilă.

Cuvinte cheie: sticlă celulară, sticlă reziduală reciclată, borax, carbonat de calciu, microundă.

1. Introduction

Important climate changes due to the destruction of the protective ozone layer of the planet through the excessive emissions of greenhouse gases in the atmosphere have seriously sensitized specialists from all over the world in the last decades. Sources emitting CO_2 have been drastically reduced especially in large fossil fuel-consuming industries and thus, a new global policy of intensive recycling the waste, whose annual generation is in an alarming increase has received special attention [1]. Waste recycling is important due to the energy saving required to produce these materials and implicitly, the reduction of CO_2 emissions during their manufacture. Recycling aims both at the reintroduction of waste into the production circuit as well as their use for the manufacture of new material types.

The construction materials industry is one of the main industries affected by the current international policy and especially, the manufacture of cement as the basic material for making the construction concrete [2].

Among the recyclable wastes (plastics, metals, glasses, rubber, paper and cardboard, textiles, etc.), the residual glass plays an important role in the production of construction materials [3].

Recently, several techniques for manufacturing porous materials with high mechanical strength for construction applications have been experimentally tested.

Assefi et al. [4] used residual alkaline battery and sodium carbonate as expanding agents to sinter/expand LCD waste screen from TV device. The porous ceramic material had high compression strength (18.7 MPa), high flexural strength (6 MPa), but relatively low heat conductivity (0.22 $W \cdot m^{-1} \cdot K^{-1}$) and an acceptable value of apparent density (0.85 $g \cdot cm^{-3}$).

Cellular glass with high compression strength was experimentally made [5] from photovoltaic module waste (80 wt. %), clay (10 wt. %), and 10 wt. % eggshell (with high calcium carbonate content). The temperature of the process was 900 °C. The compression strength reached 12.8 MPa that corresponded to a heat conductivity value of 0.121 $W \cdot m^{-1} \cdot K^{-1}$.

A modern raw hollow sphere technique was applied by Qu et al. [6] in the process of sintering at 680-800 °C and foaming in several stages of recycled residual glass. Specimens of cellular glass with fine porosity and homogeneous structure, apparent density within the limits of 0.129-0.229 $g \cdot cm^{-3}$, porosity between 91-95 %, heat conductivity in the range of 0.055-0.077 $W \cdot m^{-1} \cdot K^{-1}$, and compression strength up to 5.92 MPa were obtained.

König et al. [7] tested making the cellular glass from cathode-ray-tube (CRT) panel glass utilizing carbon and MnO_2 as pore-forming agents. The sintering

temperature range was between 700-800 °C. Apparent density had very low values (up to 0.131 $g \cdot cm^{-3}$) and also the heat conductivity (up to 0.042 $W \cdot m^{-1} \cdot K^{-1}$).

Experiments performed through factorial designs were presented in the paper [8]. Cellular glass specimens with high porosity (up to 92 %) and bulk density within the limits of 0.16-0.79 $g \cdot cm^{-3}$ were obtained using sodium hydroxide (NaOH) as an effective expanding agent. In this experiment, NaOH and borax contents as well as the sintering temperature value were the variable elements. The role of NaOH was to reduce the density value of the cellular product and slightly increase the proportion of open porosity.

The methods of making the cellular glass mentioned above included conventional heating techniques. Heating through converting the electromagnetic wave power into heat is recognized in the scientific world as a fast, economical and environmentally friendly process [9]. Although electromagnetic waves were discovered 80 years ago, their main area of application included communications and radars. The advantages in terms of energy have been highlighted so far only in industrial and domestic processes involving low drying and heating temperatures. Although experimental research on the mechanism of heating through this unconventional method is revealed more often in the literature lately [10-12], the industrial application in processes that require high temperatures is still not known.

Under these conditions, the Romanian companies Daily Sourcing & Research SRL and Cosfel Actual SRL have jointly initiated small-scale research on making different types of cellular products using recycled residual glass or alumino-silicate waste subjected to partially or completely direct heating at high temperatures (800-1150 °C). One of the papers of the authors' team presented in the literature refers to results of the experiment aimed at making porous cellular glass with relatively high mechanical strength [13]. Mixing recycled post-consumer container glass (90-94 wt. %), borax (5 wt. %) as a fluxing agent due to its relatively high Na_2O content, $CaCO_3$ (1.5-5 wt. %) as a pore-forming agent, and water (8.5 wt. %) as a binder, they were obtained by sintering/expanding at 820-851 °C cellular glass specimens with apparent density between 0.60-0.90 $g \cdot cm^{-3}$, heat conductivity in the range of 0.081-0.105 $W \cdot m^{-1} \cdot K^{-1}$, and compression strength within the limits of 2.5-6.2 MPa. The partial direct microwave heating technique using a ceramic tube of SiC and Si_3N_4 with the wall thickness of 3.5 mm for protecting the material against the destructive effect of microwave flow through completely direct contact with it led to obtaining heating rates between 15.9-17.0 °C/min and homogeneous structures of the foamed product with pore size between 0.5-3 mm depending on the mixture composition.

Maintaining the unconventional microwave heating system of the mixture based on residual glass, the current paper aimed at testing the correlation between the amounts of borax and $CaCO_3$ as fluxing and respectively, expanding agents introduced into the starting mixture. Except for its role as a flux material due to its rich-Na_2O content, borax significantly contributes to improving the mechanical strength of the foamed product due to the high level of boron in its composition. Thus, by increasing the proportion of $CaCO_3$ addition that improves the pore size, a higher borax content can compensate for the decrease in mechanical strength. The aim of the work was the

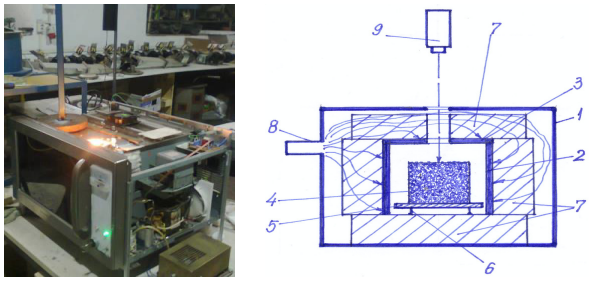
experimental determination of the optimal ratio of borax and $CaCO_3$ so that the cellular glass to have heat-insulating and mechanical properties suitable for its use as a construction material.

2. Methods and materials

The method adopted for the manufacture of cellular glass is based on the sintering and expansion of the material mixture in powder state by heating to temperatures at which the pore-supplying agent releases a gas that is trapped in the form of bubbles in the thermally softened material. The heating method is unconventional using the microwave field emitted by a single emitting source (magnetron) placed in one of the side walls of the microwave oven. Since initial tests showed that soda-lime-silica type glass powder is unsuitable for the microwave foaming process by direct heating due to the serious destruction of the structural configuration, it was necessary to adopt an original solution of positioning a protective screen made of microwave susceptible materials between the source and the material. The thickness of the screen wall (2.5 mm) proved optimal to provide a predominant microwave field that penetrates the screen and comes into direct contact with the material. Partially absorbed in the wall mass, heats it up quickly. The hot wall transmits the heat through thermal radiation and thus, the material is heated by a double effect (partially direct and partially indirect).

The direct heating has a high energy efficiency and is based on the conversion of microwave power into heat through the contact of the waves with the material. The start of heating takes place in the central area of the irradiated material and the heat propagation occurs volumetrically in its entire mass from the inside to the outside [11, 12]. The process is fast and economical and is completely different from the conventional heating.

The microwave equipment is an 800 W-oven currently used in domestic applications, constructively and functionally modified for high temperature operations. The rotating mechanism at the base of the oven was abolished, an insulating bed made of ceramic fiber mattresses (resistant up to 1200 °C) being deposed in this space. The material subjected to heating and expansion was freely placed on this bed and was protected on the sides and on top by a ceramic tube made of SiC (80 %) and Si_3N_4 (20 %) with the outer diameter of 125 mm, height of 100 mm and wall thickness of 2.5 mm (purchased from China) as well as a lid made of the same material with the thickness of 6 mm provided with 30 mm-central hole. The outer surfaces of the ceramic tube and lid were thermally insulated with the same type of ceramic fiber mattresses. A radiation pyrometer installed above the oven on a metal rod at 400 mm allowed monitoring the temperature evolution of the sample (over 680 °C, when the ceramic material began to emit thermal radiation detectable by the pyrometer). The upper metal wall of the oven was also provided with an axial hole of 30 mm. As a whole, the microwave equipment (Fig. 1) was designed according to an original own concept within the two Romanian companies mentioned above and was presented in several previously published works [14-16].



a b

Fig. 1. Microwave equipment
a – overall image of the equipment; b – functional scheme: 1 – microwave oven; 2 – ceramic tube; 3 – ceramic lid; 4 – pressed material; 5 – metal plate; 6 – metal support; 7 – ceramic protection; 8 – waveguide; 9 – pyrometer.

The materials that composed the mixture prepared for making the cellular glass were: residual glass recycled from post-consumer commercial colourless containers as the main raw material, borax as a fluxing agent, $CaCO_3$ as a pore-supplier agent, and water addition as a binder.

Residual glass processing meant selection by colour, washing, drying, breaking, grinding in a ball mill, and sieving, the selected grain sizes being below 100 μm. The oxide composition of residual glass included 71.7 % SiO_2, 2.0 % Al_2O_3, 11.8 % CaO, 1.1 % MgO, and 13.2 % Na_2O, according to determinations performed in the Metallurgical Research Institute Bucharest with AXIOS-sequential type X-ray fluorescence spectrometer.

Sodium tetraborate (borax) in powder form is a mineral from the borate class with the chemical formula $Na_2[B_4O_5(OH)_4]\cdot 8H_2O$ [17]. The crystallization water is removed by heating to 100 °C and at over 400 °C the anhydrous product is obtained. Theoretically, the composition of borax contains 30.8 % sodium oxide (Na_2O) as one of the most effective fluxing materials and 69.2 % boric oxide (B_2O_3), boron significantly influencing the mechanical strength of the product that includes boron in its composition.

Probably, one of the cheapest and frequently used pore-forming agents in the processes of making the glass foam [18], $CaCO_3$ was chosen for this experiment. By decomposing this carbonate slowly starting at about 750 °C and rapidly continuing up to about 900 °C [19] carbon dioxide (CO_2) is released and contributes to the pore-formation process and calcium oxide (CaO) enters the composition of the glass molten. $CaCO_3$ had very fine grain size (under 20 μm) commercially purchased at these dimensions.

A number of four experimental versions were adopted to test the variation influence of the mixture component weight proportions on cellular glass specimen features. Table 1 centralizes the four manufacturing recipes, in which residual glass had values within the limits of 88.9-92.4 wt. %, borax represented between 5.9-9.5 wt. %, $CaCO_3$ had increasing values (like also the borax) in the range of 1.7-2.6 wt. %, and water addition as a binder (to facilitate pressing the dry powder mixture) was used in a constant weight proportion of 8 wt. %.

Table 1

Composition of experimental versions

Composition	Version (g)			
	1	2	3	4
Residual glass	92.4	90.9	89.4	88.9
Borax	5.9	7.1	8.3	9.5
$CaCO_3$	1.7	2.0	2.3	2.6
Water addition	8.0	8.0	8.0	8.0

Common methods were used for investigating the cellular glass specimen features. Apparent density and porosity were measured by applying the Archimedes' method according to ASTM D792-20. Determining the heat conductivity was carried out by heat-flow method (ASTM E1225-04) [20]. The measure of compression strength was performed with TA.XTplus Texture analyzer. The common method of immersion of the sample under water was utilized to measure the water-absorbing ability (ASTM D570). Microstructural aspects of samples were examined with ASONA 100X Zoom Smartphone Microscope.

3. Results and discussion

The simultaneous increase in the amounts of borax and $CaCO_3$ with approximately opposite effects on the hardness and mechanical resistance of the foamed product and respectively, on its insulating properties (density, heat conductivity, and porosity) influenced the functional parameters of the manufacturing process (Table 2). Thus, the value of sintering/expanding temperature slightly increased from version 1, with the lowest contents of borax and $CaCO_3$ (834 °C), to version 4, with the highest contents of borax and expanding agent (842 °C). Implicitly, the heating duration increased within low limits from 31 to 40 min. Compared to the results obtained in an almost similar paper, the process being also carried out by the non-conventional method of microwave heating [13], the heating rates reached significantly higher values between 20.6-26.3 °C/min and influenced the considerably lower level of specific energy consumption (between 0.84-1.08 kWh/kg).

Table 2

Functional parameters of the process

Parameter	Version			
	1	2	3	4
Dry raw material/cellular glass amount (g)	430/ 386	430/ 385	430/ 387	430/ 386
Sintering/expanding temperature (°C)	834	837	839	842
Time heating process (min)	31	33	36	40
Average rate (°C/min) - heating - cooling	26.3 5.4	24.8 5.5	22.8 5.4	20.6 5.5
Specific energy consumption (kWh/kg)	0.84	0.89	0.97	1.08

Appearance images of cellular glass specimens made in the four versions are shown in Fig. 2.

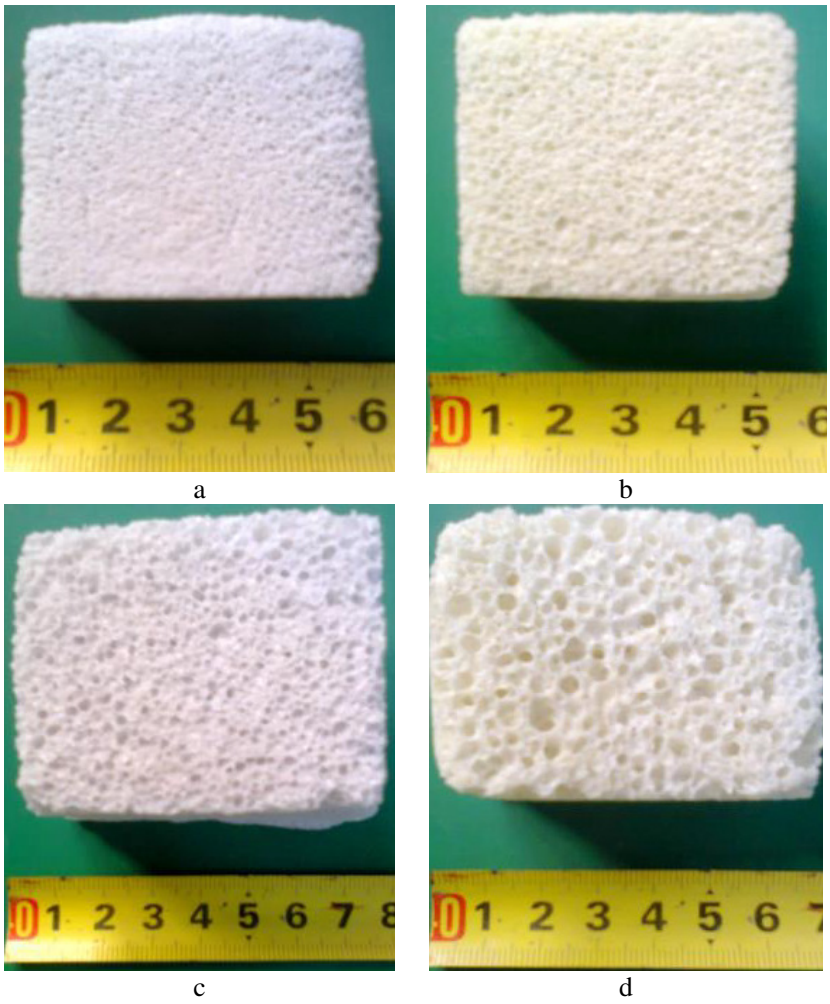


Fig. 2. Appearance of cellular glass specimens
a – specimen 1; b – specimen 2; c – specimen 3; d – specimen 4.

Examining the four pictures in Fig. 2, it can be easily observed that the use of increasing weight proportions of both borax and $CaCO_3$ (according to the data in Table 1) led to changing in the macrostructural aspect of specimens. A structure characterized by low pore sizes corresponded to version 1 (5.9 wt. % borax and 1.7 wt. % $CaCO_3$), while a more coarse structure including much wider and less uniform pores corresponded to version 4 (9.5 wt. % borax and 2.6 wt. % $CaCO_3$).

The application of physical, thermal, mechanical, and morphological (microstructural) investigation methods of each cellular glass specimen has allowed the determination of their characteristics. The results are shown in Table 3.

Table 3

Characteristics of cellular glass specimens

Characteristic	Version			
	1	2	3	4
Apparent density ($g \cdot cm^{-3}$)	0.55	0.51	0.46	0.41
Porosity (%)	75.0	78.1	80.6	82.9
Heat conductivity ($W \cdot m^{-1} \cdot K^{-1}$)	0.106	0.100	0.093	0.084
Compression strength (MPa)	4.2	6.0	4.3	4.0
Water-absorbing (vol. %)	0.7	0.9	1.1	1.1
Pore size (mm)	0.1-0.4	0.2-0.4	0.3-1.0	0.6-1.9

According to the data in Table 3, it was found that the characteristics indicating insulation properties (apparent density, heat conductivity, and porosity) have followed a relatively uniform downward slope, in the case of density (between 0.55-0.41 $g \cdot cm^{-3}$) and heat conductivity (between 0.106-0.084 $W \cdot m^{-1} \cdot K^{-1}$) and respectively, a uniformly increasing slope (between 75.0-82.9 %) in the case of porosity. An uneven evolution of the porous material property under the compression stress was noted. Thus, in the case of version 2 the highest compression strength value was reached (6.0 MPa), after which versions 3 and 4 recorded much lower values (4.3 and 4.0 MPa, respectively).

The explanation of this situation was investigated by also examining the microstructural aspect of cellular glass specimens shown in Fig. 3, in the context in which the content of borax has continued to increase as well as the content of $CaCO_3$.

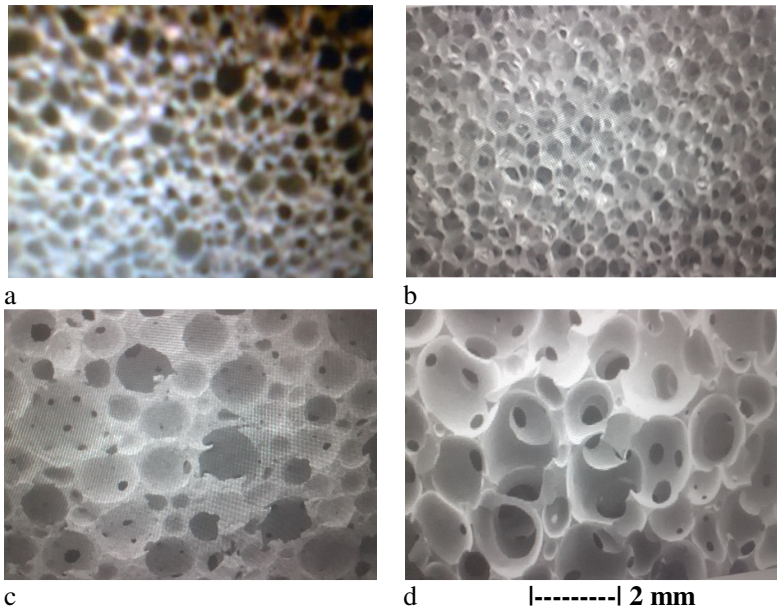


Fig. 3. Microstructural images of cellular glass specimen sections
a – specimen 1; b – specimen 2; c – specimen 3; d – specimen 4.

The cross-section of the four cellular specimens revealed that except for the specimen corresponding to version 1, all other specimens were characterized by microstructures also containing open pores together with closed pores. This type of microstructure represents the case where a cell communicates with other neighbouring cells by perforating the neighbouring wall or through connecting bridges. Usually, a microstructure with partially open pores favours the decrease of porous material apparent density, but at the same time its compression strength should significantly decrease, as it happens in the case of experimental versions 3 and 4. Versions 1 and 2, due to the lower content of $CaCO_3$ as an expanding agent, had fine microstructures, generally with closed pores, in which the formation of open pores was hardly possible. The purpose of the experiment, as noted above, aimed to determine the correlation between the amounts of borax, that contribute to increasing the mechanical strength of the foamed material and $CaCO_3$, that contributes to the pore-formation. Both addition materials had increasing amounts in the four versions. Analyzing the data in Table 3, it can be concluded that the specimen manufactured in version 2 with 7.1 wt. % borax and 2.0 wt. % $CaCO_3$ by sintering at 837 °C using partially direct microwave heating is the optimal version.

The thermal insulation properties of the optimal product were excellent (apparent density of 0.51 $g \cdot cm^{-3}$, heat conductivity of 0.100 $W \cdot m^{-1} \cdot K^{-1}$, and porosity of 78.1 %), while the compression strength reached a high value (6.0 MPa) for this cellular glass type. Water-absorbing was very low (0.9 vol. %) and the material pores were homogeneous and low (pore size between 0.2-0.4 mm). The manufacturing method chosen by authors proved to be fast, "clean", and economical, the specific energy consumption being determined by counting at 0.89 kWh/kg. Cellular glass produced in this way is suitable for the use as thermal insulation material in construction and recycling the residual glass for its manufacture solves both the acute global problem of wastes, as well as increasing the ceramic material resistance to fire, water, and the attack of rodents, insects, bacteria.

4. Conclusions

The work concerned the non-conventional manufacturing of cellular glass from recycled residual glass, combining the heat-insulating and mechanical properties of cellular products. Usually, obtaining low values of the apparent density and heat conductivity also leads to low values, inappropriate for construction applications, of the product compression strength. In general, the pore-providing agent is responsible for obtaining a porous structure. In the case of the current experiment, $CaCO_3$ was chosen for this purpose. The correlation between the amount of the pore-providing agent and the amount of the additive used to improve the mechanical strength (borax) was the challenge of this work. In parallel, the effect of the use of electromagnetic waves leading to energy efficiency increasing for the heating process was a method successfully applied in the last 7-8 years by the Romanian companies Daily Sourcing & Research and Cosfel Actual, although the world industrial producers are not yet interested in this heating mode. The work tested the manufacture of cellular glass

under conditions of simultaneous increase in the content of borax and $CaCO_3$ within the limits of 5.9-9.5 wt. % and respectively, 1.7-2.6 wt. %. The fairly close results of the four experimental versions indicated that the optimal solution was that of using 7.1 % borax and 2 % $CaCO_3$, which provided the opportunity to reach the highest value of the compression strength (6.0 MPa) corresponding to the apparent density of 0.51 $g \cdot cm^{-3}$. Water-absorbing had very low values (0.9 vol. %) and also the pore size between 0.2-0.4 mm. The technical solution of using microwaves in the effective process of non-conventional heating for the manufacture of cellular glass has proven excellent and this fact has been recognized by specialists. The Romanian companies utilizing microwaves on a small scale intend to expand the capacity of the experimental equipment to values closer to those industrially required.

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