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High-porosity cellular glass made by microwave heating with a liquid expanding agent

Sticlă celulară de porozitate ridicată fabricată prin încălzire cu microunde cu un agent de expansiune lichid

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Abstract. *The work aimed at producing a high-porosity cellular glass, using a recipe composed of soda-lime-silica glass waste, borax as a fluxing agent, and aqueous KOH solution as an expanding agent. The heating process for sintering/foaming was based on microwave irradiation, a known unconventional technique, but practically not used in high-temperature applications until now. The results showed that density of 0.26 g·cm^{-3}, thermal conductivity of 0.069 W·m^{-1}·K^{-1}, and compressive strength of 1.6 MPa were obtained, corresponding to the optimal variant. The work originality consists in the use of the liquid foaming agent and applying the own microwave heating technique.*

Key words: *cellular glass, microwave, KOH solution, glass waste, high-porosity.*

Rezumat. *Lucrarea a vizat producerea unei sticle celulare cu porozitate ridicată, utilizând o rețetă compusă din deșeuri de sticlă sodă-calciu-silice, borax ca agent de flux și soluție apoasă de KOH ca agent de expansiune. Procesul de încălzire pentru sinterizare/spumare s-a bazat pe iradierea cu microunde, o tehnică neconvențională cunoscută, dar practic neutilizată în aplicații la temperaturi înalte până în prezent. Rezultatele au arătat că s-a obținut densitatea de 0,26 g·cm^{-3}, conductivitatea termică de 0,069 W·m^{-1}·K^{-1} și rezistența la compresiune de 1,6 MPa, corespunzătoare variantei optime. Originalitatea lucrării constă în utilizarea agentului de spumare lichid și aplicarea tehnicii proprii de încălzire cu microunde.*

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Cuvinte cheie: sticlă celulară, microundă, soluție de KOH, deșeu de sticlă, porozitate înaltă.

1. Introduction

According to the latest literature data [1], in 2022, the total packaging waste per inhabitant in EU countries reached 186.5 kg, of which 36.1 kg of glass packaging waste, representing 19 % of the entire amount of packaging waste. Thus, the total annual quantity of glass packaging waste generated in the EU reached 16 million tonnes, i.e. an impressive value. An increasing trend in the amount of glass waste is exhibited in the EU and also in the world, taking into account that the EU reported 15.2 million tonnes in 2019 [2].

Partially, recycled glass waste, either from post-consumer drinking bottles or from broken and replaced window glass in buildings, is already industrially reused in the traditional production of new glass, the operation being cost-effective in energy terms. However, a significant financial disadvantage is the technological necessity of colour sorting of recycled glass waste. This processing involves quite high costs [3].

A new trend regarding the use of this waste type has begun to be felt in the world in the last 3-4 decades, the waste being directed mainly towards the manufacture of alternative construction materials. In this application field, the glass quality imposed by its chemical composition (closely related to the colour of the initial material) is not a major requirement, what's important being the removal of contaminants (plastics, heavy metals, organic materials, etc.), washing and advanced grinding.

The production of materials with thermal insulation qualities (low weight, low heat conductivity, and high porosity) requires foaming the residual glass with an adequate expansion agent, incorporated into the waste matrix, by applying a heat process at high temperatures between 750-1150 °C [4].

Several companies from European countries (Austria, Belgium, Germany, the Czech Republic, Scandinavia), and the United States, joined by China due to the assimilation of current technologies, are at the forefront of the global market for cellular glass made from residual glass [5].

Various types of expanding agents are used in the industrial processes of manufacturing cellular glass: carbon products including coal, black carbon, glycerol, etc., carbonates/sulfates such as calcium carbonate, sodium carbonate, dolomite, gypsum, etc., carbides/nitrides including silicon carbide and silicon nitride [4].

The new millennium has promoted numerous small-scale experiments on the manufacture of cellular glass presented in the world literature. Various types of recycled glass waste have been tried, including also other species of silicate materials suitable for the production of foams intended for thermal insulation of buildings, such as: cathode ray tube [6], borosilicate glass [7], bituminous shale waste [8], metallurgical slag [9], zinc hydrometallurgy waste [10], quartz sand and coal gangue [11], and coal fly ash [12]. Also, different types of expanding agent have been tested, such as: agents of vegetable origin [13, 14], eggshell [15], and agents of organic origin [16].

The experimental use of soda-lime-silica glass waste as raw material, borax as a fluxing agent and aqueous $NaOH \cdot H_2O$ solution as an expanding agent, in order to manufacture cellular glass, has been recently presented in the literature [14, 17]. The variation of the porosity aspect (from predominantly closed to predominantly open) was obtained by using different ratios between fluxing agent and expanding agent, which modified the density of the foamed material within very wide limits (between 0.16-0.79 $g \cdot cm^{-3}$), while the porosity reached very high values (up to 92 %). The role of $NaOH \cdot H_2O$ consisted in reducing the density value as well as decreasing the proportion of closed porosity. The addition of borax associated with small proportions of $NaOH \cdot H_2O$ favoured the densification process and the creation of a closed-pore structure. Foaming the mixture based on glass waste was achieved due to the water vapor released by the thermal decomposition of the foaming agent. Corresponding to the glass transition temperature of 587 ℃, a crystalline phase of hydrated sodium and calcium silicate ($Na_2CaSi_2O_6 \cdot 2H_2O$) is formed. Upon reaching the softening point of the mixture containing the glass of over 700 ℃, the release of water vapour from the crystalline phase mentioned above generates bubbles inside the viscous mass, which later turn into cells.

All industrial processes for manufacturing cellular glass, according to data provided in the literature, provide the thermal energy necessary for sintering the glass-based mixture using well-known conventional processes based on burning traditional fuels or electrical resistance heating.

The technique based on the application of the peculiarities of electromagnetic waves to convert their power as energy carriers into heat and its propagation in the mass of the material subjected to heating from the inside to the outside, in the opposite direction to conventional thermal processes, has been known since the middle of the 20th century. However, the interest of researchers was focused on other fields of application, in telecommunications and radar. The energetic properties, despite the remarkable efficiency of microwaves, did not arouse the interest of specialists. Practically, the only applications in this area were limited to industrial drying processes and heating processes at low temperatures.

However, researchers from the Romanian company Daily Sourcing & Research SRL have used the energy peculiarities of microwaves since 2016, achieving significant levels of energy efficiency in small-scale cellular glass manufacturing processes [18] compared to those of industrial processes known in the world.

The current paper includes a method for manufacturing microwave-assisted high-porosity cellular glass designed and tested on a small scale by the Romanian authors, which may contribute to the development of the mentioned technique. The original elements introduced in this work are the use of the foaming agent in the form of an alkaline KOH-based solution as well as the achievement of the thermal sintering/foaming process by unconventional microwave heating.

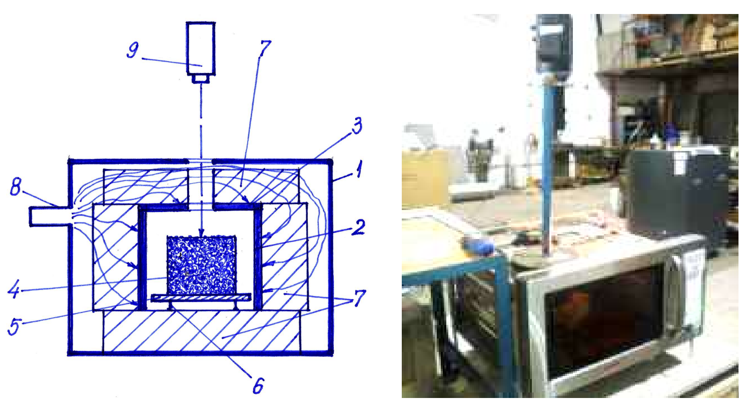
2. Methods and materials

2.1 Methods

As mentioned above, two important elements of originality characterized the current work: microwave heating in an own version and the choice of a liquid foaming agent not used in the cellular glass manufacturing process.

The sintering/foaming process of glass waste was based on heating the raw material under the influence of microwave irradiation as a particularly energy-efficient unconventional technique, applied by the authors in an original version consisting of predominantly direct and partially indirect heating by placing a protective screen made of microwave-susceptible materials in the form of a ceramic tube. This tube was positioned between the source emitting electromagnetic waves and the material to be heated. The method has the role of tempering the intensity of direct contact of waves with the material surface, which, in the case of glass, causes destruction of its internal structure. The wall thickness of the ceramic tube (of 2.5 mm) is essential for the irradiation process efficiency, being experimentally determined. The microwave flux is partially absorbed in the mass of the vertical tube wall, heating it quickly and efficiently. Predominantly, the flux penetrates the tube wall and comes into direct contact with the glass sample to be heated. In this way, the heating process is mixed: predominantly direct and partially indirect. The partially indirect heating is of the conventional type, being achieved by thermal radiation from the inner surface of the tube to the glass material.

The microwave equipment used in this experiment (also in other own previous experiments) was designed and made in Daily Sourcing & Research Company using a 800 W-microwave oven applied in the household for food preparation, constructively and functionally adapted for operation at high temperatures (above 1100 °C). The constructive and functional scheme of the oven (A) as well as the overall image of the equipment (B) are shown in Fig. 1.



A B

Fig. 1. Microwave equipment for preparing cellular glass
A – constructive and functional scheme: 1 – microwave oven; 2 – ceramic tube; 3 – ceramic lid; 4 – pressed sample; 5 – metal plate; 6 – support; 7 – ceramic protection; 8 – waveguide; 9 – radiation pyrometer; B – overall image of the equipment.

An important ability of potassium hydroxide (KOH) dissolved in water is the complete decomposition into ions of sodium (Na^+) and OH^- group. At the ambient temperature, the fine glass powder slowly reacts with the aqueous KOH solution. Silica (SiO_2) available in high ratios in the glass composition (over 70 % in the case of soda-lime-silica glass) reacts with hydrated KOH forming potassium silicate (K_2SiO_3) according to the following reaction:

$$SiO_2 + 2KOH \rightarrow K_2SiO_3 + H_2O \qquad (1)$$

By growing the process temperature to above 700 °C, the intensity of reaction (1) increases also. Upon reaching the glass transition temperature, the crystalline phase $K_2CaSi_2O_6 \cdot H_2O$ is formed, whose water of crystallization is released around 700 °C. Water vapour dissociates into oxygen and hydrogen, constituting the gases that contribute to the formation of bubbles in the softened mass of raw material, remaining blocked, to subsequently form cells.

It is known that the ability of melts containing silica to form glass is explained by the presence into these melts of three-dimensional silicon-oxygen polymeric structures, whose interaction leads to a significant growing of the melt viscosity upon cooling and the disorganization of the structure. Usually, the dissolution of SiO_2 particles in alkaline solutions involves the cleavage of siloxane bonds according to the equations (1) and (2) and the silica transfers in liquid phase in form of silicic acid ions with different levels of polymerization [19].

$$\text{Si-O-Si} + OH^- \rightarrow SiOH + SiO^- \qquad (2)$$

$$H_2O + SiO^- \rightarrow \text{Si-OH} + OH^- \qquad (3)$$

2.2 Materials

The materials that constituted the starting mix for preparing cellular glass were: post-consumer drinking bottle as raw material, sodium tetraborate decahydrate ($Na_2B_4O_7 \cdot 10H_2O$) as a flux agent, and aqueous potassium hydroxide (KOH) solution as an expanding agent.

The post-consumer drinking bottle was composed of colourless glass (40 wt. %), green glass (30 wt. %), and amber glass (30 wt. %), with their chemical composition exhibited in Table 1. The recycled waste was cleaned of any possible contaminants, washed, broken in a crusher, ground in a ball mill, and sieved, the grain size being selected under 83 μm.

Table 1

Chemical composition of glass waste

Chemical composition	Colourless glass (%)	Green glass (%)	Amber glass (%)
SiO_2	71.5	71.7	71.3
Al_2O_3	2.1	1.8	2.0
CaO	12.1	11.9	12.0
Fe_2O_3	0.1	-	0.2
MgO	1.1	1.2	1.0
Na_2O	13.3	13.1	13.0
K_2O	-	0.1	0.1
Cr_2O_3	0.1	0.1	-
SO_3	-	-	0.1

Sodium tetraborate decahydrate was chosen as a fluxing agent due to its high content of Na_2O, the best known chemical compound with flux properties, particularly for the glass industry. On the other hand, the boron from its composition plays an important role in growing the mechanical strength of cellular glass prepared with borax added.

An aqueous potassium hydroxide (KOH) solution was adopted as an expanding agent in this experiment. This choice has never been made until now. KOH is a strong alkali, very water-soluble, and highly corrosive. Other properties of KOH are: ability to degrade various materials, strong chemical base, high thermal stability, and reactivity toward acid. It is commercially available in flakes form, obtained by concentrating a purified electrolytic KOH solution [20].

2.3 Investigating methods to determine the cellular glass features

The apparent density was measured by the gravimetric method [21]. The porosity was calculated by the method of comparing the true and apparent density [22]. The thermal conductivity was measured by the guarded-comparative-longitudinal heat flow meter method (ASTM E1225-04) and the compressive strength was determined using a TA.XTplus Texture Analyzer (ASTM C552-17). The water absorption was determined based on the instructions provided in ASTM C240 standard and the microstructural configuration of the cellular glass samples was investigated with an ASONA 100X.

3. Results and discussion

3.1 Results

For the purpose of experimental production of high-porosity cellular glass made with a liquid blowing agent, four versions of the manufacturing recipe containing the above-mentioned components were chosen. Table 2 indicates the values corresponding to each version.

Table 2

Composition of the experimental versions

Version	Coloured glass waste (%)	Sodium tetraborate hydroxide (%)	Aqueous KOH solution (%)
1	86.0	11.5	2.5
2	86.0	10.0	4.0
3	86.0	8.5	5.5
4	85.5	7.0	7.5

Physical-mechanical, heat, and morphological characteristics of samples corresponding to tried experimental versions are presented in Table 3.

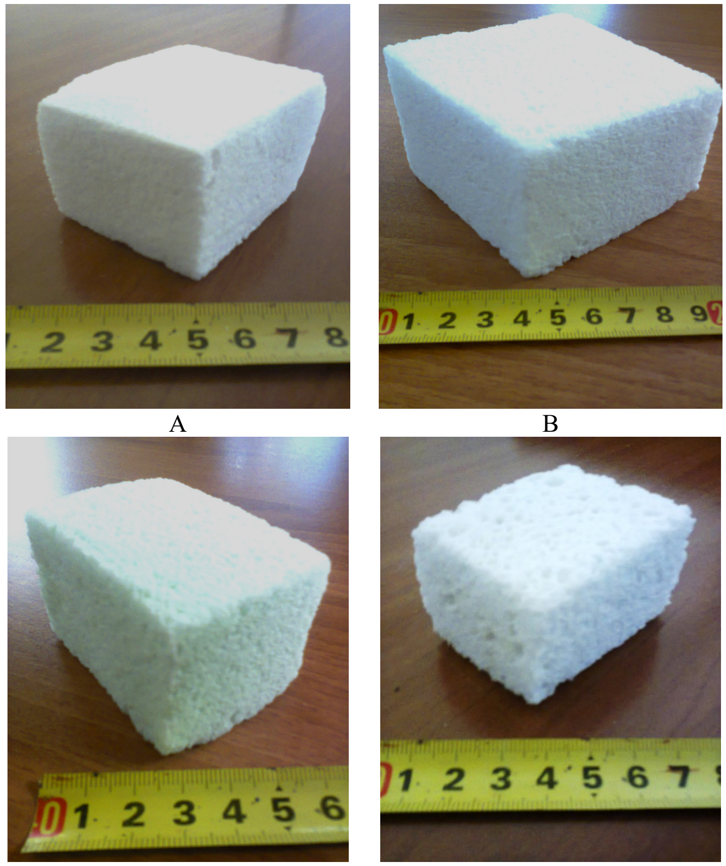
Table 3

Physical-mechanical, heat, and morphological features

Feature	Version A	Version B	Version C	Version D
Apparent density ($g \cdot cm^{-3}$)	0.43	0.34	0.25	0.14
Porosity (%)	78.7	83.1	87.5	92.0
Heat conductivity ($W \cdot m^{-1} \cdot K^{-1}$)	0.089	0.074	0.059	0.043
Compressive strength (MPa)	2.06	1.87	1.68	1.48
Water-absorbing (vol. %)	2.24	2.20	2.16	2.10
Cell size (mm)	0.1-0.3	0.1-0.4	0.3-0.6	0.7-0.9

Results of the main characteristics of cellular glass specimens presented in Table 3 show the decreasing values of apparent density (from 0.43 to 0.14 $g \cdot cm^{-3}$) with growing the aqueous KOH solution used in the experiment (from 2.5 to 7.5 %). Also, heat conductivity registered a significant reduction (from 0.089 to 0.043 $W \cdot m^{-1} \cdot K^{-1}$). The high porosity of cellular glass samples influenced the relatively low level of compressive strength, whose values were in the range of 1.48-2.06 MPa, without being an obstacle to applications in the field of thermal insulation in construction. The water absorption measured in the case of the four cellular glass samples was within normal limits for this material type, i. e. between 2.10-2.24 vol. %.

The appearance of the outer surface of the four cellular glass samples is exhibited in Fig. 2.

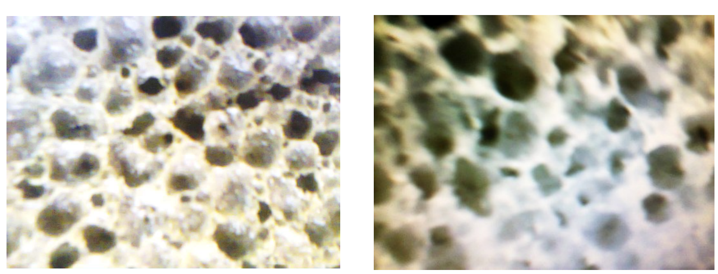


A B

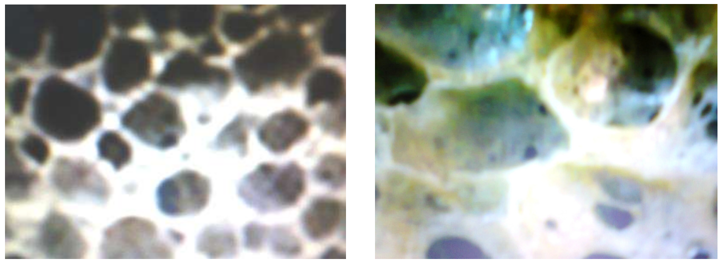
C D

Fig. 2. Appearance of outer surface of cellular glass samples
A – version 1; B – version 2; C – version 3; D – version 4.

The obvious increase of sample porosity obtained by growing the proportion of KOH solution is observable in images shown in Fig. 2. Sample D prepared by using the highest content of KOH solution is characterized by closed cells with dimensions up to about 1 mm. The analysis of the microstructural structures of the samples was performed based on the pictures in Fig. 3.



A B



C D |-----------| **1 mm**

Fig. 3. Microstructural aspect of high-porosity cellular glass samples
A – version 1; B – version 2; C – version 3; D – version 4.

The cell size ranges were determined for each of the four samples, the results being included in Table 3. Sample A had the lowest cell sizes in the range of 0.1-0.3 mm, while sample B had values between 0.1-0.4 mm. Sample C was characterized by higher cell dimensions within the limits of 0.3-0.6 mm, while sample D reached the highest size range (0.7-0.9 mm).

3.2 Discussion

The use of aqueous KOH solution as an expanding agent is a technological novelty promoted and tested by the authors' team in the manufacturing process of high-porosity cellular glass. Also, a fluxing agent from the borax category, namely sodium tetraborate decahydrate, was adopted in association with the mentioned expanding agent. In general, this material type is frequently used, especially in the glass industry being considered one of the most important fluxing agents due to its high Na_2O content.

According to the literature data [14, 17], the aqueous solution of NaOH as a foaming agent together with borax and soda-lime-silica residual glass is a recently used version. By varying the borax/foaming agent ratio, different values of the density of cellular glass were obtained (between 0.16-0.79 $g \cdot cm^{-3}$), the porosity reaching extremely high values (up to 92 %). Increasing the content of $NaOH \cdot H_2O$ solution contributed to the decrease of the density value and reducing the open porosity proportion. In contrast, borax favoured growing the cellular glass density and the generation of a closed-cell structure.

By comparison, cellular glass produced with KOH solution allowed lower values of apparent density (up to 0.14 $g \cdot cm^{-3}$) and heat conductivity (up to 0.043 $W \cdot m^{-1} \cdot K^{-1}$).

The application of electromagnetic waves as energy carriers, whose power is converted into heat through direct contact with the material to be heated, has proven to be a very fast heating procedure and at the same time very cost-effective in terms of energy. The paper [23] noted these energy performances of microwaves for glass processing compared to conventional processes (such as glass melting, sintering, devitrification, coating, reinforcing, etc.). However, commercial glass powder (soda-lime-silica glass type) subjected to direct microwave heating in 20 L-useful volume oven powered by 800 W suffered serious damage of its internal structure by heating to sintering/foaming temperatures of 850-950 °C [24], according to experimental observations of the current paper authors. The optimal resolving adopted by the authors was the own technical solution to temper the intensity effect of direct contact of the microwave flux with the glassy material by placing a microwave-absorbing protective screen between the wave emitting source and the heated material.

4. Conclusions

The work objective was to manufacture a cellular glass with excellent thermal insulation properties under the conditions of applying the unconventional microwave heating technique of the glass-based mixture. The originality of this technique applying consisted in choosing the own solution of mixed heating, preponderantly direct and partially indirect, by placing a microwave-absorbing protective screen between the wave emission source and the pressed material mixture. Another originality of the work in technological terms was the choice of aqueous KOH solution as a foaming agent instead of the NaOH solution successfully tried in previous recent experiments. The results were qualitatively similar, while the microwave applying led to energy advantages.

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