Fine porosity-cellular glass obtained by microwave-assisted heat treatment using the expansion ability of glycerol together with water glass

Sticlă celulară cu porozitate fină obținută prin tratament termic asistat de microunde folosind capacitatea de expansiune a glicerolului împreună cu apă de sticlă

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Abstract. *Results of microwave-assisted manufacturing fine porosity-cellular glass using a liquid carbonic foaming agent (glycerol) together with water glass are presented in this paper. Under the conditions of developing the process in oxidizing atmosphere of the oven, the water glass role was to avoid the premature burning of carbon resulted after glycerol decomposing .The use of authors' own version of microwave heating applied in the last experiments reconfirmed the solution viability and its remarkable energy efficiency. Also, as an original technique measure, water glass proportion in the mix was increased to 8-12 % leading to growing the fineness of specimen porosity*

Key words: *high porosity, cellular glass, microwave, glycerol, water glass.*

Rezumat. *Rezultatele fabricării cu asistența microundelor a sticlei celulare cu porozitate fină utilizând un agent de spumare carbonic lichid (glicerol) împreună cu apă de sticlă sunt prezentate în această lucrare. În condițiile desfășurării procesului în atmosfera oxidantă a cuptorului, rolul apei de sticlă a fost evitarea arderii premature a carbonului rezultat după descompunerea glicerolului. Utilizarea versiunii proprii a autorilor privind încălzirea cu microunde aplicată in ultimele experimente a reconfirmat viabilitatea soluției și remarcabila sa eficiență energetică. De asemenea, ca o măsură tehnică originală, proporția apei de sticlă in amestec a fost crescută la 8=12 % conducând la creșterea fineței porozității probelor.*

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Cuvinte cheie: porozitate înaltă, sticlă celulară, microunde, glicerol, apă de sticlă.

1. Introduction

A wide variety of cellular products using recycled residual glass was designed and made in the last few decades. Properties of these products could be modeled depending on several factors: foaming agent type and mineral addition materials, heating rate and holding time at the foaming temperature, final process temperature. Their appropriate choice allowed to obtain cellular materials with physical, thermal, mechanical, and microstructural characteristics quite different depending on the requirements of the application domain [1]. On industrial-scale, several cellular glass types are manufacturing mainly in the form of thermal insulation boards and blocks, the most known products in the world being „Technopor" under license of the Swiss company Misapor Switzerland and several branches in Europe and „Foamgla" under license of the North-American company Pittsburgh Corning with branches in the United States, Europe, and China. According to information in the literature, fine and ultra-fine porosity cellular glass are not industrially manufacturied, but only on an experimental small-scale and on the type of these products the current work is focused.

Recycling glass waste of which generation has been continuously increasing over the last 30-40 years, became a major problem, because storing this waste in landfills is now unacceptable for environmental reasons.

Recycling the glass waste and its reintroduction into the manufacturing process of new glass is practiced in the glass industry, but to a rather small extent due to the high costs of the quantitative sorting operations (by colour) of the waste. It has been experimentally found that glass recycling for the purpose of manufaccturing new material types, especially in construction, would be an appropriate procedure due to the attractive properties of this material after a relatively unexpensive processing (sintering, foaming). The so-called cellular glass simultaneously meets several properties such as: low weight, rigidity, non-degradability, non-toxicity, chemical resistance, compression resistance, resistance to humidity, steam, bacteria, insects, rodents, etc. [1].

Sodium silicate aqueous solution is considered a cheap and environmentally friendly category of alkali silicates. According to [2], the investigation of structural peculiarities of the sodium silicate aqueous solution subjected to heating showed that important structural changes occur through its reorganization due to the water elimination. The water amount remaining in the system complies with Arrhenius' theory [3], quantified by an energy activation of 30 $kJ \cdot mol^{-1}$. The measurement results carried out by the authors showed that the new structure becomes almost similar to the characteristic structure of hydrated silicate glasses.

In another work [4], the research of possibility to expand drinking bottle waste with water glass into air atmospheric-oven was performed. Experimental results indicated that by growing the water glass amount, heat conductivity and crystallinity of products decrease. The remaining crystal content affects however the forming

process of closed-porous froth. It has found that by the addition of only water glass, without another traditional expanding agent, the production mechanisms of cellular glass into air and respectively, into argon atmosphere are different. Under the conditions of carrying out the experiment without protection atmosphere, the lowest density of the expanded material was 0.123 $g \cdot cm^{-3}$ and the minimum value of heat conductivity was 0.053 $W \cdot m^{-1} \cdot K^{-1}$, the closed porosity being 50 %. The starting mixture was composed of glass waste mentioned above, water glass (12 wt. %) as an expanding agent as well as boron trioxide (2 wt. %), aluminum phosphate (2 wt. %), and tripotassium phosphate (2 wt. %) as additives.

In the glass expansion process, the direct contribution of water glass is well-known. However, this process mechanism has not yet been validated on an experimental basis. To prove this situation, Hriba et al. [5] made an experiment, in which the evolution of the mix containing catode ray tube waste and water glass was investigated during the foaming process. Structurally, water glass is perceived as a colloidal suspension of the Na_2SiO_3 aqueous solution, having as main components colloids and anions (or molecules) of silicate. Between these components there is the water as OH groups or water molecules. Drying the water glass solution leads to generation a xerogel with a glass-like structure, that by further heating softens and begins to expand due to the evaporation of the remaining water. As a result of these structural changes, connectivity of the pore network increases. The powder glass-water glass mix favours the sintering process and decreases the expanding temperature. Also, the presence of water glass allows to apply carbon-based foaming processes in atmospheric air without the danger of premature carbon burning. The current literature explains the expansion through releasing water structurally bound to water glass. However, the detection of water vapour at high temperatures during the water glass heating leads to the idea that water could be one of the main gases involved in expansion. Water glass as a soluble silicate reacts with CO_2 forming the akaline carbonate (Na_2CO_3) that constitutes a traditional expanding agent for the powder glass. So, the effect of the oxidizing atmosphere of the oven on the mixture containing water glass is a favourable one.

In another work [6], experimental results of using only water glass as an expanding agent were exposed. The conclusion was that this method is viable, cellular glasses with fine porosity and closed pores with diameters between 4 nm and 800 μm being obtained. The mechanical resistance of expanded products was 1.7 MPa. In conformity with the same paper, conventional cellular glasses (made by foaming with traditional agents) commercially available have significantly higher pore size reaching 3 mm, while their strength is lower.

Aspects related to the preparation of cellular glass of mixed colours and water glass (15 wt. %) as a foaming agent, the mixture being uniaxially pressed at 10 MPa were presented in [7]. The sintering process occurs at 800 and respectively, 850 °C. Results showed that by increasing the grain size of raw material and growing the temperature, the froth porosity increased, while the density decreased. The specimen microstructure indicated that the increase of pore homogeneity was favoured by the

fineness of glass grains and the higher sintering temperature led to larger pores, yet still remaining closed. Compression and flexural strength decreased with decreasing the grain size of raw material and increasing the sintering temperature. The heat conductivity did not exceed 0.25 $W \cdot m^{-1} \cdot K^{-1}$, this value being considered adequate for insulating material applications.

According to [8], making a cellular glass material with a high porous structure and density under 0.5 $g \cdot cm^{-3}$ requires the controled use of glycerol, a liquid carbonic agent ($C_3H_8O_3$) together with water glass, which have the capability to provide optimal properties.

The use of an industrial-scale making recipe of cellular glass gravel, a porous material with heat insulation and load-bearing properties, mainly including glass waste, glycerol, and water glass is that applied by the German company Glapor Werk Mitterteich [9]. According to technical data provided by the manufacturer, apparent density has values between 0.13-0.21 $g \cdot cm^{-3}$, heat conductivity around 0.078 $W \cdot m^{-1} \cdot K^{-1}$, compression strength in the range of 4.9-6.0 MPa, and the pore size under 300 μm [10].

The experimental analysis of influence of glycerol/water glass ratio used for making glass foam by the sintering/foaming process of glass waste on the morphological characteristics of specimens was made in the work [11]. Glycerol as an expanding agent (between 1-1.5 %) associated with water glass as an enveloping material for the carbon particles (between 2-5 %) were used in this experiment. The process temperature had values in the range of 800-850 °C, the heating rate having extremely high values between 26-166 $°C \cdot min^{-1}$. As a result, fine porosity materials with pore size within the limits of 0.25-2 mm were produced. The pore dimension varied depending on the heating rate value and the final temperature of the thermal process. The suitable proportions of glycerol and water glass were experimentally established being 1.5 % for glycerol and 5 % for water glass. The optimal temperature was 850 °C and the optimal heating rate was in the range of 50-80 $°C \cdot min^{-1}$. Heat conductivity had comparable values with that of similar industrial products (0.06-0.08 $W \cdot m^{-1} \cdot K^{-1}$).

Experimentally tested solution for manufacturing a lightweight glass froth is presented in [12]. The material mixture included container glass waste (92 %), water glass (3.5 %), and yellow glycerol (3.5 %) as a by-product of making biodiesel used as an expanding agent. Also, sodium carbonate (1 %) as a solid foaming agent was added. The final sintering/foaming temperature was 850 °C, the average heating rate being 10 $°C \cdot min^{-1}$. The sintered product was maintained at 850 °C for 30 min. The highest value of compression strength reached 16 MPa corresponding to the bulk density of 0.67 $g \cdot cm^{-3}$.

Some components of the authors' team of the current paper were previously interesed in producing fine porosity-cellular glass obtained by the use of the liquid carbonic expanding agent (glycerol) in association with water glass in order to avoid the premature burning of fine carbon particles resulted by decomposing the glycerol [13]. Colourless flat glass waste (83-83.7 wt. %) from building demolition constituted the basic raw material, while glycerol (1.0-1.8 wt. %), water glass (5.3-7.5 wt. %), and

water addition (7.7-10 wt. %) completed the starting mixture. Unlike all manufacturing processes mentioned above, in this paper the effect of high energy efficiency of electromagnetic waves was used as an unconventional heating procedure of solid materials. The process temperature varied within the limits of 810-824 °C, average heating rate being in the range of 19.1-20.3 °C·min^{-1}, leading to obtaining very low specific energy consumptions between 0.81-0.88 kWh·kg^{-1}. Results showed excellent heat insulation properties (apparent density between 0.20-0.26 g·cm^{-3}, porosity in the range of 85.5-88.2 %, and heat conductivity within the limits of 0.056-0.070 W·m^{-1}·K^{-1}, and in the same time high compression strength between 4.6-5.8 MPa.

In another work of Romanian authors [14], a different making method of a fine porosity-cellular glass using glass waste as raw material, borax as a fluxing agent (8-11 wt. %), and aqueous NaOH solution as a foaming agent (3-7 wt. %) was chosen. As in the paper [13], unconventional electromagnetic waves heating method in the own version was adopted. The process temperature had relatively low values (between 710-780 °C). Characteristics of the optimal making version were: apparent density of 0.26 g·cm^{-3}, porosity of 87.6 %, heat conductivity of 0.069 W·m^{-1}·K^{-1}, and compression strength of 1.60 MPa. The specific energy consumption value was low (0.89 kWh·kg^{-1}).

Starting from own results obtained in the manufacture of fine porosity-cellular glass by microwave-assisted in 2020 [13], the main objective of the current work was to improve the performance regarding the fineness of the cellular material porosity by modifying its making recipes. Specifically, the water glass content of the starting mixture was significantly increased from 5.3-7.5 wt. % to 8.0-12.0 wt. % with growing the addition water content (between 10-13 wt. %).

2. Methods and materials

In general, the carbonic expanding agents can generate glass froth either by reaction with oxygen existing in the interstitials of the softened glass particles in the oxidizing atmosphere of the furnace or by carbon reduction of some glass components [1, 15]. For example, Na_2SO_4 from the glass can be reduced to Na_2S and dissolved into the glass molten. Thus, the reaction of SO_4^{2-} (from the glass) with the carbon forms S^{2-} (in glass) as well as CO and CO_2, that are pore-forming gases.

Carbon-containing liquid expanding agents (such as glycerol) develop the possibilities of penetration between the fine particles of powder glass. Glycerol from the glass mixture decomposes in the oxidizing conditions of the oven, generating a large range of chemical components from CO_2 and CO to pure carbon and hydroxyl compounds [15].

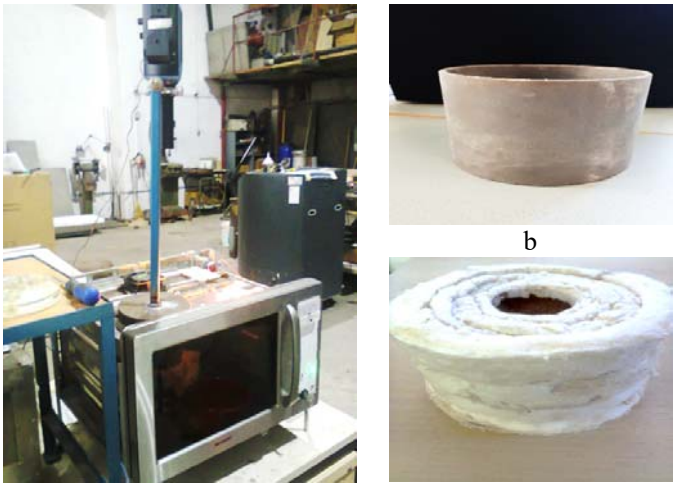
As stated above in this paper, in the case of using the carbon-expanding agent (e.g. glycerol), the role of water glass is essential in the foaming process to avoid premature burning the carbon resulting from the decomposition of this organic agent in the oxidizing environment of the furnace. Thus, water glass creates a protective layer

to envelop fine carbon particles, delaying the burning process and therefore the formation of CO and CO_2 as pore-forming gases.

The experiment presented in this work was conducted at the experimental base of the Romanian company Daily Sourcing & Research on an 800 W microwave-powered testing equipment. The equipment shown in Fig. 1 consists of a microwave oven commonly used in households for food preparation, adapted through constructive and operational modifications to allow much higher temperatures (up to 1200 °C) (a). The oven is equipped with a cylindrical tube made of SiC and Si_3N_4 (b) highly sensitive to microwaves, with the diameter of 1250 mm, height of 100 mm, and wall thickness of 2.5 mm, placed in the central area of the inner space of the oven on a 3 mm-metal plate. The location of the tube (made in China) has the role of tempering the intensely destructive effect of electromagnetic waves on the glass-based raw material upon their direct contact. The optimal size of the tube wall thickness was previously experimentally determined by the research team of Daily Sourcing & Research [16]. In this way, a lower, but predominant proportion (70-80 %) of emitted waves through the single waveguide placed in one of the oven walls completely penetrates the oven wall and comes into direct contact with the material subjected to heating. The remaining proportion of waves are absorbed in the wall mass, which is quickly and intensely heated, so that its hot inner surface at over 1000 °C transmits heat through thermal radiation to the same material. Direct microwave heating has some peculiarities that give it a higher energy efficiency than conventional heating systems. Thus, the heating process is started inside the material in its central area. There, the microwave power is converted into energy and the core of the material becomes the point with the highest temperature. Thus, the heat propagates volumetrically from the inside to the outside. The propagation mode is completely opposite to that which characterizes the heat transfer in conventional heating [17, 18]. Conventional methods require first heating the entire set of massive materials that make up the traditional oven (vault, walls, hearth, etc.) and only then the material effectively takes up the heat emitted by the conventional heating source. On the contrary, unconventional microwave heating has the ability of selectivity [18], i.e. the heating is focused only on the material susceptible to microwaves, the other materials not having the capacity to absorb the waves are insignificantly heated. Refractory materials that are traditional components of the oven masonry are practically not susceptible to microwaves, having in their composition high contents of silica and alumina.

Although electromagnetic waves have been known since the mid-20th century and their properties have been identified since the early years, their application has only materialized in the field of communications and radars and very few in the area of industrial heating processes, and those only at low temperatures in the range of 100-500 °C [19].

The list of materials chosen for this experiment includes: green and amber post-consumer drinking bottle as raw material, glycerol as a liquid carbonic expanding agent, aqueous solution of water glass, mainly used to avoid the premature burning of carbon resulted from the glycerol decomposition as well as distilled water addition for supplementary dilution of aqueos solution.



a b c

Fig. 1. Images of the experimental equipment
a – microwave oven including the radiation pyrometer for measuring the glass surface temperature, b – SiC and Si_3N_4 ceramic tube; c – thermal protection of the tube.

The post-consumer drinking bottle was selected from green and amber drinking bottles in relatively equal weight proportions. After washing, the waste was broken, ground in a ball mill, and sieved for elimination glass particles over 80 μm. The oxide composition of the two glass waste previously measured with the AXIOS type fluorescence spectrometer from the Romanian Metallurgical Research Institute showed the following composition values [20] (Table 1).

Table 1

Oxide composition of glass types

Glass type	Oxide composition (wt. %)							
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	Cr_2O_3
Green	71.8	1.9	=	11.8	1.2	13.1	0.1	0.1
Amber	71.1	2.0	0.2	12.1	1.1	13.3	0.1	-

In terms of chemistry, glycerol or glycerine ($C_3H_8O_3$) represents the simplest trihydoxy alcohol, being a slightly viscous liquid (1.28 $g \cdot cm^{-3}$), water and alcohol-soluble. Glycerol is found in all natural oils and fats in the form of long chain fatty acid esters (known also as glycerides). Also, it is naturally found in combined form in all vegetable and animal fats and oils, commonly as a triglyceride with fatty acids. Different industrial processes are used for obtaining glycerol, the most known being the propylene synthesis through the oil hydrolysis. If the glycerol proportion in vegetable oils is within the limits of 8-14 %, the yield of glycerol through industrial processes is about 10 % [21]. Glycerol grades for non-food aplications can reach the maximum purity of 95 wt. %. The glycerol used in this experiment commercially purchased (originated from the Netherlands) had 89 wt. % of purity.

Water glass (sodium silicate-Na_2SiO_3), whose role has already specified above in this work, was procured from the market in form of an aqueous solution with 38 % concentration.

The investigation techniques of physical, heat, mechanical, and microstructural characteristics of cellular glass products were in general those usually applied for this cellular material type. Apparent density was measured using Archimedes' principle in accordance with the ASTM C373 standard and ISO 18754:2020 was applied for measuring the porosity. The compression strength of fine porosity-cellular glass samples was determined with a hydraulically operated compression testing machine with the pressing capacity of 107 MPa, according to the ASTM C133-97 (2015) standard. The water-absorption of specimens was identified in accordance with ASTM C373-18 standard by their immersion under water. Microstructural appearance of specimens was examined with Biological Microscope MT5000 model (1000 x magnification).

3. Results and discussion

Composition of the mixture versions adopted for experimentally making the fine porosity-cellular glass is presented in Table 2.

Table 2

Composition of the mixture versions for producing cellular glass

Composition	Version 1 (wt. %)	Version 2 (wt. %)	Version 3 (wt. %)	Version 4 (wt. %)
Green and amber post-consumer drinking bottle	81.00	78.75	76.50	73.25
Glycerol	1.00	1.25	1.50	1.75
Water glass	8.0	9.0	10.0	12.0
Water addition	10.0	11.0	12.0	13.0
Water glass/glycerol ratio	8.00	7.20	6.67	6.86

According to the data in Table 2, the water glass/glycerol weight ratio had values between 8 and 6.67, slightly decreasing from the first to the latest versions. Glycerol and water glass as well as water addition had growing values within the limits of 1-1.75 %, 8-12 %, respectively 10-13 %.

Operational parameters of the expanding process of fine porosity-cellular glass are shown in Table 3.

Table 3

Operational parameters of the expanding process of cellular glass

Version	Raw material/ cell glass amount (g)	Process temperature (°C)	Heating time (min)	Average rate (°C·min^{-1})		Specific energy consumption (kWh·kg^{-1})
				Heating	Cooling	
1	550/525	815	35	22.71	5.2	0.69
2	550/527	817	36	22.14	5.3	0.71
3	550/526	820	38	21.05	5.2	0.75
4	550/527	825	41	19.63	5.3	0.81

Cellular glass specimens corresponding to the four material mixtures mentioned above (Fig. 2) were obtained in the temperature range of 815-825 °C. According to the operational parameters shown in Table 3, by using the technique of microwave heating predominantly direct and partially indirect, reaching very high heating rates in all the four experimental versions (between 19.63-22.71 °C·min^{-1}) has been possible and also relatively short heating times until the appearance and development of foaming into the material mass (between 35-41 min).

The energy efficiency of the microwave-assisted procedure was excellent also in this case, the specific energy consumption being in the range of 0.69-0.81 kWh·kg^{-1}, lower orat most equal to the consumption industrially registered using conventional heating methods.

a b c d

Fig. 2. Section images of the fine porosity-cellular glass
a – version 1; b – version 2; c – version 3; d – version 4.

The physical, heat, mechanical, and microstructural features of the cellular glass specimens are presented in Table 4.

Table 4

Physical, heat, mechanical, and microstructural features

Version	Aparent density (g·cm^{-3})	Porosity (%)	Heat conductivity (W·m^{-1}·K^{-1})	Compression strength (MPa)	Water absorption (vol. %)	Pore size (μm)
1	0.20	88.3	0.055	2.1	2.3	40-120
2	0.21	87.5	0.058	2.5	2.3	60-230
3	0.23	86.7	0.061	2.7	2.5	70-240
4	0.15	90.8	0.049	1.8	2.1	20-90

In accordance with the results in Table 4, the work objective was reached. Fine porosity of cellular glass specimens was obtained in all tested experimental versions, the pore size being under 240 μm. The lowest values of cell diameter, fully closed, were reached in the case of version 4 characterized by the combined use of water glass and glycerol in the weight ratio of 12.0/1.75, i.e. 6.86, the temperature at the

moment of stopping the heating proces being the highest of the four version (825 °C). The identified range of pore size was 20-90 µm.

Apparent density corresponding to the specimen made by version 4 was extremely low (0.15 $g \cdot cm^{-3}$), it also influencing the very small value of heat conductivity (0.049 $W \cdot m^{-1} \cdot K^{-1}$) and the high value of porosity (90.8 %). Despite extremely low physico-thermal features of this specimen, compression strength of the mentioned product was more than satisfactory (1.8 MPa).

Although, the other tried versions allowed increasing the apparent density to 0.20-0.23 $g \cdot cm^{-3}$ as well as the heat conductivity to 0.055-0.061 $W \cdot m^{-3} \cdot K^{-3}$, the mechanical strength did not significantly increase, reaching a maximum of 2.7 MPa in the case of version 3.

The evolution of microstructural appearance of the fine porosity-cellular glass specimens under the influence of different proportions of glycerol and water glass introduced in the starting mixture of the four experimental versions is shown in Fig. 3.

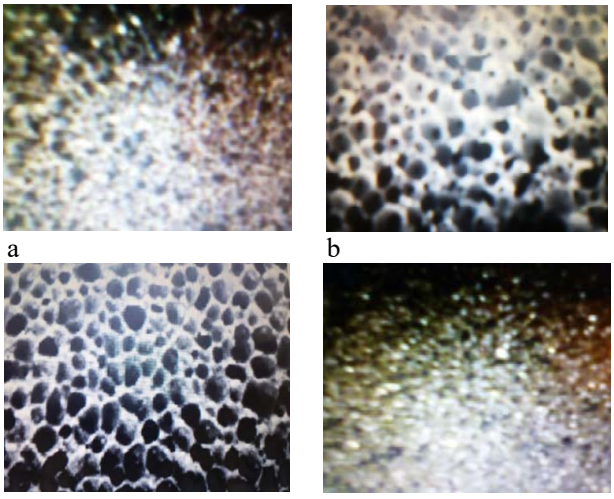


Fig. 3. Microstructural appearance of the fine porosity cellular glass specimens
a – version 1; b – version 2; c – version 3; d – version 4.

The simultaneous growing of glycerol and water glass in the proportions mentioned in Table 2 showed that firstly, the cellular glass registers increasing the pore size and then, the tendency to form a fine porosity structure is exhibited. The explanation could be that by tha use of glycerol in a lower amount the foaming process is normally favoured and in addition, the water glass contribution, whose role is to protect the carbon particle surfaces created through the glycerol decomposition, is beneficial for delaying this process. By simultaneous growing the glycerol and water glass content, increasing the specimens porosity is evident, their size reaching over 200 µm. Under the water glass/glycerol ratio value of about 7, the contribution of water glass to form fine porosity becomes more important than the contribution of

further increase of the glycerol content on the foaming and thus pore dimension enters in the reduction trend, reaching below 90 μm.

6. Conclusions

Having in view the work objective regarding making a fine porosity-cellular glass under conditions of higher energy efficiency compared to production of other similar products, the experimental results can be considered as a success in technological terms. In the experimental version (4) based on the combined use of water glass and glycerol in the ratio of 6.86 (water glass representing 12.0 wt. % and glycerol 1.75 wt. %), the apparent density reached extremely low value of 0.15 $g \cdot cm^{-3}$ as well as heat conductivity registered 0.049 $W \cdot m^{-1} \cdot K^{-1}$. Despite these low values of physical and thermal properties, the cellular material maintained a more than satisfactory level of compressive strength of 1.8 MPa. Obtaining these performances was made through the original growing the proportion of water glass up to 12 wt. % and choosing suitable ratio between water glass and glycerol. Also, adopting the unconventional microwave heating technique, unused in the world in heating processes of solids at temperatures above 500 °C, constituted an important originality character of the paper.

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