Porous thermal insulation building material made of recycled glass waste by microwave heating

Material de construcție poros termoizolant fabricat din deșeu de sticlă reciclată prin încălzire cu microunde

Lucian Păunescu[1], Sorin Mircea Axinte[2,3], Bogdan Valentin Păunescu[4], Felicia Cosmulescu[5]

[1]Cosfel Actual SRL
95-97 Calea Grivitei street, M4 room, sector 1, Bucharest 010705, Romania
E-mail: lucianpaunescu16@gmail.com

[2]Daily Sourcing & Research SRL
95-97 Calea Grivitei street, sector 1, Bucharest 010705, Romania
E-mail: sorinaxinte@yahoo.com

[3]Department of Applied Chemistry and Materials Science, University „Politehnica" of Bucharest
1-7 Gh. Polizu street, sector 1, Bucharest 011061, Romania
E-mail: sorinaxinte@yahoo.com

[4]Consitrans SA
56 Polona street, sector 1, Bucharest 010504, Romania
E-mail: pnscbogdan@yahoo.com

[5]Cosfel Actual SRL
95-97 Calea Grivitei street, M4 room, sector 1, Bucharest 010705, Romania
E-mail: feliss2014@gmail.com

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Rezumat. *Lucrarea prezintă rezultate experimentale obținute în procesul fabricării sticlei celulare utilizând încălzirea indirectă cu microunde. Intensificarea absorbției microundelor a fost realizată utilizând un creuzet din carbură de siliciu având peretele acoperit cu un film din oxid de ytriu. Încălzirea cu microunde și folosirea oxidului de ytriu reprezintă originalitatea lucrării. Rețeta conținând deșeu de sticlă, cenușă și carbură de siliciu a permis producerea sticlei celulare la 960-970 ºC cu consumuri economice de energie (sub 0,86 kWh/kg). Produsele au caracteristicile specifice sticlei celulare cu densitatea intre 0,27-0,33 g/cm^3, conductivitatea termică între 0,064-0,073 W/m·K și rezistența la compresiune între 1,35-1,45 MPa.*

Cuvinte cheie: sticlă celulară, încălzire cu microunde, deșeu de sticlă, cenușă de cărbune, carbură de siliciu, oxid de ytriu.

Lucian Paunescu, Sorin Mircea Axinte, Bogdan Valentin Paunescu, Felicia Cosmulescu

Abstract. *The paper presents experimental results obtained in manufacturing process of cellular glass using the indirect microwave heating. The microwave absorption intensification was achieved using a silicon carbide crucible having the wall coated with a yttrium oxide film. The microwave heating and the use of yttrium oxide are the paper originality. The recipe containing glass waste, ash and silicon carbide allowed cellular glass production at 960-970 ºC with economical energy consumption (below 0.86 kWh/kg). The products have specific characteristics of cellular glass with density between 0.27-0.33 g/cm^3, thermal conductivity between 0.064-0.073 W/m·K, and compressive strength between 1.35-1.45 MPa.*

Key words: cellular glass, microwave heating, glass waste, coal ash, silicon carbide, yttrium oxide.

1. Introduction

In the last 3-4 decades, most countries in the world have been concerned about recycling waste which is generated intensively and its storage in landfills is no longer an acceptable solution for the health of the environment and people. The glass industry, a major consumer of primary energy, has adopted the solution of reusing glass waste as a raw material in the production of new glass even before the initiation of the current global waste recovery measures. According to the literature [1], 1 kg of cullet replaces 1.2 kg of traditional raw materials (sand, sodium carbonate, and limestone) and the energy consumed in the glass manufacturing process decreases by about 3 % for every 10 % of cullet added to the starting material mixture. Also, the emission of greenhouse gases (mainly carbon dioxide) decreases by 5 % for every 10 % used cullet. However, the glass industry prefers its own glass waste resulting from the technological manufacturing process and avoids recycling the wastes predominantly accumulated from the post-consumer drinking bottle and the demolition and modernization of buildings due to the need of their selection by color (default, quality), which is an expensive operation.

If in several European countries, primarily Switzerland and Germany, there are very high recycling rates (90 %), in the United States recycling the residual glass is low (only 33 %), the raison being the diversity of glass quality requirement of the manufacturers as well as the long distances between the location of the collection and sorting centers and that of the industrial producer, which significantly increases the cost of the supply operation [1].

Under these conditions, a wide field of recycled glass application to other users has opened up. The use of glass as an alternative material for the construction sector is the main direction of valorization. Unlike other waste types, glass is completely recyclable and its properties remain unchanged even at the end of the product's life cycle [1].

According to [2], from a structural point of view, the glass can be classified into three categories: soda-lime glass (Na_2O-CaO-SiO_2) which is a combination of sodium silicate and calcium silicate, that dissolves at low temperature and can be efficiently

expanded or welded, potash-lime glass (K_2O-CaO-SiO_2) which is a mix of potassium silicate and calcium silicate, that dissolves at high temperature, being also called „hard glass", and potash-lead glass (K_2O-PbO-SiO_2) which is a mixture of potassium silicate and lead silicate, with excellent refractive power. Obviously, the soda-lime glass is the most widely used commercial glass found in the main glass products. This is the reason why this residual glass type is predominantly recycled and is preferred for various manufacturing techniques of expanded glass usable in construction.

The usual method of making the cellular glass is to incorporate into the finely ground glass-based raw material a solid or liquid expanding agent (carbonaceous products, carbides, or carbonates are preferred), which release a gas at high temperature as a result of a chemical reaction. The temperature range in which the reaction takes place must include the softening point of the glass, so that the medium in which the gas is distributed to have an adequate viscosity for its blockage and the formation of bubbles [3].

Since the 1980s, several industrial manufacturers have developed cellular glass manufacturing activities in Europe, the United States, and China using recycled glass waste, the products having as main characteristics light weight, high porosity, low thermal conductivity, and at least acceptable compressive strength (above 1 MPa), to which resistance to fire, water and steam, frost, corrosion, aggression of external agents, very high durability, chemical and physical stability, lack of toxicity, etc. are added [3, 4]. A diversification of features, especially mechanical and geometric exists, taking into account the requirements of the field of application of expanded products. Thus, manufacturers supply cellular glass in the form of blocks and boards for thermal insulation of building masonry (with apparent density of 0.165 g/cm^3, thermal conductivity of 0.05 W/m·K, and compressive strength of 1.6 MPa [5]), or as a filler for load bearing thermal insulation under foundation slabs, underground thermal insulation of energy fluid pipes and storage tanks, industrial floor tiles, road and railway construction components, bridge abutments, airport runway, drainage, sports fields, roof gardens, swimming pools, etc. (with bulk density between 0.11-0.17 g/cm^3, lump density between 0.22-0.38 g/cm^3, thermal conductivity between 0.05-0.08 W/m·K, and compressive strength up to 6 MPa [6-8]).

Industrially, the manufacture of cellular glass takes place in conveyor belt furnaces heated by conventional methods (electricity or thermal energy). In the case of thermal insulation blocks or boards production, raw material is loaded into metal molds, which are moved along the entire length of the furnace passing through the intense heating area, then through the tempering area and are discharged as cellular glass at the cold end. The porous gravel or aggregate manufacture involves placing the raw material on the conveyor belt in the form of a compact layer, moved on the all conveyor length and discharged at the cold end in the form of lumps with size up to 70-80 mm.

Testing the application of the unconventional technique of raw material microwave heating to obtain cellular glass adequate for their use as thermal insulation materials in construction were small-scale performed in the last years on adapted microwave oven in the Romanian company Daily Sourcing & Research. Except the

manufacturing recipes and the conventional heating techniques constantly used by the industrial producers, other attempts for technological improving were not reported in the literature. Changing the heating technique adopted by the Romanian company is singular in this area in the world and the result previously obtained showed an excellent energy efficiency compared to the conventional techniques. In terms of quality, the physical, thermal, mechanical, and microstructural features of expanded materials are comparable to those industrially achieved.

Numerous experiments in the manufacture of cellular glass in microwave field have been performed using soda-lime glass waste as the main raw material and various expanding agent types. The heating technique adopted by authors has been the predominantly direct microwave heating by placing a SiC and Si_3N_4 ceramic tube or crucible with thickness wall between 2.5-3.5 mm between the material and the wave emission source. Using this technique and calcium carbonate as an expanding agent, very light weight cellular glass (apparent density between 0.16-0.19 g/cm^3), with good thermal insulation properties (thermal conductivity between 0.034-0.040 W/m·K and porosity between 91.4-92.7 %), the compressive strength having acceptable values (1.12-1.22 MPa) have been produced. The specific energy consumption has been values below 1 kWh/kg [9, 10]. Other experiments have led to obtaining (by heating at 900-905 ºC) dense cellular glasses with high compressive strength (14.1 MPa), apparent density between 0.77-0.82 g/cm^3 and thermal conductivity in the range 0.124-0.135 W/m·K [11], using colored glass waste, blast furnace slag (31-39.25 %), borax (7.8-8.1 %), titanium oxide (5 %), sodium phosphate (3 %) and sodium carbonate ($CaCO_3$) (5-6.5 %). Also, the specific energy consumption has been low (0.90-0.95 kWh/kg). Using a raw material mixture predominantly composed of clay (70.5-83.4 %) together with glass waste (2.6-15.5 %), coal fly ash (9 %), and silicon carbide (SiC) (5 %), the direct (100 %) microwave heating at 1055-1150 ºC without affecting the microstructural homogeneity has been possible [12]. The specific energy consumption has been very low, reaching the lowest value of 0.58 kWh/kg. The compressive strength has been between 7.8-8 MPa, apparent density between 0.60-0.69 g/cm^3, and thermal conductivity in the range 0.100-0.116 W/m·K. Manufacturing the cellular glass gravel by microwave irradiation technique has been also achieved, being used the main industrial manufacturing recipes successively including SiC, $CaCO_3$, glycerol as expanding agents, borax and sodium silicale as additives, the products being almost similar to those industrially manufactered [13].

Significant intensification of microwave absorption ability through the wall of an irradiated SiC crucible with a wave field can be obtained by coating the outer surface of crucible with thin layer of yttrium oxide (Y_2O_3) achieved by spraying. The dielectric properties of Y_2O_3 (tangent loss and dielectric constant) increase at high coating film temperature, enhancing the microwave absorption [14]. The method of Y_2O_3 nanoparticle using is known in the last years being applied in several domains, according to AzoNano, the leading online publication for the nanotechnology community [15], (television, microwave filters, inorganic synthesis of compounds, making fluorescent lamps, etc.). The method was not applied in manufacturing process

of cellular glass, its use in the present paper having original character. Previous tests of the authors in expansion process of glass waste using an existing Y_2O_3 coated crucible [16] showed enhancing the microwave absorbtion capacity and consequently, increasing the heat flux transferred to the material through indirect heating.

The work aims at manufacturing the cellular glass using the Y_2O_3 coated crucible containing a mixture of glass waste, coal fly ash, and SiC as an expanding agent, one of the common preparing recipes, to highlight the high energy efficiency of this process.

2. Method and materials

SiC is one of the most efficient expanding agents used in the manufacturing process of cellular glass from glass waste. In the industrial processes, its weight proportion is about 2 % [6, 13]. The soda-lime glas waste is the only raw material that makes up the finely ground starting mixture, coal fly ash being excluded because it raises the temperature limit required for sintering and expanding the material. According to [6], the Norwegian company Glasopor does not use commercial SiC, but waste SiC from the silicon industry for economic reasons. However, in other processes, coal fly ash is added together with glass and SiC due to its contribution to obtaining a homogeneous microstructure of the foamed product [17].

SiC using as an expanding agent involves the oxidizing atmosphere of the furnace, which must supply the oxygen necessary for the release of the foaming gas (CO_2 or CO) and a solid compound (SiO_2 or SiO) that enters into the molten glass composition.

The basic chemical reaction is (1), which is initiated at above 900 ºC.

$$SiC + 2O_2 = SiO_2 + CO_2 \quad (1)$$

If the oxygen concentration in the furnace atnosphere is insufficient for the reaction (1), the following reaction (2) takes place.

$$SiC + O_2 = SiO + CO \quad (2)$$

According to [3], the temperature range in which the two chemical reaction occur is 950-1150 ºC.

As stated above, the experiment described in the paper was based on the unconventional microwave heating of raw material placed into a SiC crucible whose outer side wall is coated with a yttrium oxide (Y_2O_3) film. The slightly truncate crucible with the outer diameter of the opening of 117 mm, the botton diameter of 95 mm, the height of 90 mm, and the wall thickness of 8 mm was purchased from China including also the Y_2O_3 film. The mixture was previously prepared by grinding and mixing the three compounds (glass waste, coal fly ash and SiC), water addition as a binder, loading into a metal mold, axially pressing and removing from the mold as a

compact cylindrical material (with density between 1.6-1.8 g/cm^3) having the diameter of about 7 cm and the height of about 6 cm.

The microwave heating equipment was an oven with installed power of 800 W of the type commonly used in household, constructivelly adapted for operation at temperatures up to 1200 ºC (Fig. 1 a). The heating of microwave susceptible materials (the SiC crucible with Y_2O_3 film) is initiated in their core, unlike the conventional heating in which the walls, hearth and vault of the oven are initially heated. Therefore, very efficient thermal protection with ceramic fiber mattresses (Fig. 1 b) was made around the crucible (and above the corresponding lid), so that the unprotected metal walls of the microwave oven were kept below 65 ºC. The thermal process control was based on the correlation between the heating time and the heated material temperature, previously experimentally determined.

a b

Fig. 1. Images of the experimental equipment
a – adapted 800 W-microwave oven; b – ceramic fiber thermal insulation of the Y_2O_3 coated crucible.

The recycled glass waste from post-consumer colored drinking bottle was the main raw material of this experiment. Approximately equal weight proportions of drinking bottles differing by color (colorless, green and amber) were used. In terms of quality, the three glass waste types are soda-lime glasses, whose oxide composition varies within restricted limits according to Table 1. The analyses were performed by X-ray fluorescent spectrometry at the Metallurgical Research Institute of Bucharest.

Table 1

Oxide composition of glass waste types

Glass type	Oxide composition (wt. %)								
	SiO_2	Al_2O_3	CaO	Fe_2O_3	MgO	Na_2O	K_2O	Cr_2O_3	SO_3
Colorless	71.5	1.9	12.0	0.1	1.0	13.3	0.1	0.1	0.2
Green	71.2	1.8	10.2	0.4	2.2	13.0	0.5	0.2	0.3
Amber	71.4	1.9	10.3	0.3	2.3	13.2	0.6	0.1	0.3

The glass waste processing operations (washing, color selection, breaking, grinding and sieving) were performed in Bilmetal Industries SRL Popesti-Leordeni, Ilfov. The grain size of the waste after these operations was allowed below 100 μm.

Coal fly ash as an industrial by-product of the Romanian thermal power plant of Paroseni was purchased at a granulation below 250 μm, being reduced below 100 μm after grinding and sieving operations.

SiC as an expanding agent was purchased from the market at a fine grain size below 10 μm and was used in the experiment without further mechanical processing.

Four experimental variants based on the manufacturing recipe containing glass waste, coal fly ash, SiC, and water addition were adopted by the authors (Table 2). Given the value ranges of these components used in various previous experiments, coal fly ash was used between 9-11 % and SiC between 2.8-3.1 %. The recycled glass waste has resulted with values between 85.9-88.2 %, while the water addition was kept constant at 15 %.

Table 2

Adopted experimental variants

Variant	Recycled glass waste (wt. %)	Coal fly ash (wt. %)	SiC (wt. %)	Water addition (wt. %)
1	88.2	9.0	2.8	15.0
2	87.1	10.0	2.9	15.0
3	87.0	10.0	3.0	15.0
4	85.9	11.0	3.1	15.0

3. Results and discussion

The main parameters of the experimental manufacturing process of cellular glass by microwave irradiation of the crucible coated with Y_2O_3 film containing the material subjected to heating are shown in Table 3.

Table 3

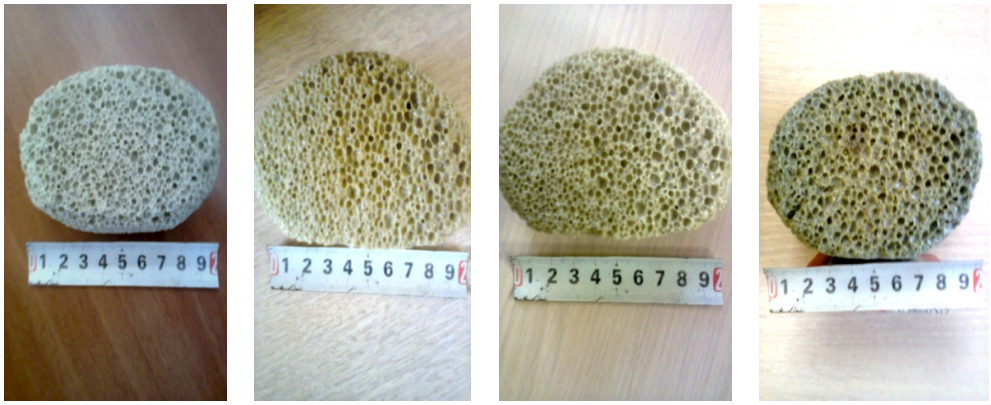
Parameters of the manufacturing process

Parameter	Variant			
	1	2	3	4
Dry raw material/cellular glass amount (g)	365/355	365/357	365/356	365/356
Sintering/foaming temperature (ºC)	960	965	967	970
Heating time (min)	32.5	33	33.5	34.25
Average heating rate (ºC/min)	29.5	28.6	28.3	27.7
Average cooling rate (ºC/min)	5.8	5.6	5.7	5.7

Index of volume growth	1.80	1.90	1.95	2.10
Specific energy consumption (kWh/kg)	0.81	0.82	0.84	0.86

The dry raw material amount was adopted and kept in all experimental variants at the value of 365 g. As mentioned above, the experimental methodology was used to determine the temperature of the materials subjected to heating based on the correlation between heating time and temperature, experimentally identified by previous measurements, the thickness and quality of the thermal insulation layer being kept constant. Thus, the variation of the heating duration between 32.5-34.25 min led to increasing the final temperature of the expanded material from 960 to 970 ºC. The average heating rate had very high values (27.7-29.5 ºC/min), significantly above the usual level of this functional parameter, indicating the remarkable effect of microwave absorption in the wall of the crucible coated with Y_2O_3 film. The increase in volume of the expanded material was determined to be a doubling of the initial volume, i.e. a common increase in the use of SiC (around 3 %) and coal fly ash (around 10 %). The specific energy consumption recorded very low values (less than 0.86 kWh/kg), confirming the energy efficiency of the innovative tested heating process.

Significant images of the cross section with homogeneous porosity of the cellular glass samples manufactured in the four variants are shown in Fig. 2.



a b c d

Fig. 2. Appearance of the cellular glass products
a – variant 1; b – variant 2; c – variant 3; d – variant 4

Common methods of analysis were used to characterize cellular glass samples from a physical, mechanical, thermal, and morphological point of view. The apparent density was measured by the gravimetric method [19]. The porosity was calculated by the comparison method of the “true” and apparent density of the material, experimentally measured [20]. The compressive strength was determined with a TA.XTplus Texture analyzer. Using the guarded-comparative-longitudinal heat flow method (ASTM E1225-04) the thermal conductivity was measured. The water absorption for 24 hours was determined by the water immersion method (ASTM

D570). The microstructural appearance of the four products was investigated with ASONA 100X Zoom Smartphone Digital Microscope. The results of the features determination of cellular glass samples are presented in Table 4.

Table 4

Features of cellular glass samples

Variant	Apparent density (g/cm^3)	Porosity (%)	Thermal conductivity (W/m·K)	Compressive strength (MPa)	Water absorption (vol. %)	Pore size (mm)
1	0.33	84.3	0.073	1.45	1.5	0.1-0.4
2	0.31	85.2	0.070	1.42	1.8	0.2-0.7
3	0.30	85.7	0.066	1.41	1.6	0.2-0.8
4	0.28	86.7	0.064	1.36	1.8	0.3-0.9

The thermal regimes adopted for the treatment of the four mixtures being relatively close in value led to quite low variations of the expanded samples features. Thus, the apparent density had values between 0.28-0.33 g/cm^3, considered small enough for use in construction as thermal insulation material. Also, the high porosity (84.3-86.7 %) and the low thermal conductivity (0.064-0.073 W/m·K) ensure adequate thermal insulation properties of the products. The level of compressive strength was more than acceptable (1.36-1.45 MPa) for this type of cellular glass and water absorption was determined at low values, below 1.8 vol. %.

The macrostructural aspect of the four foamed products in Fig. 2 showed a relatively fine and homogeneously organized porosity. The microstructural investigation of these products presented in Fig. 3 confirmed this appearance.

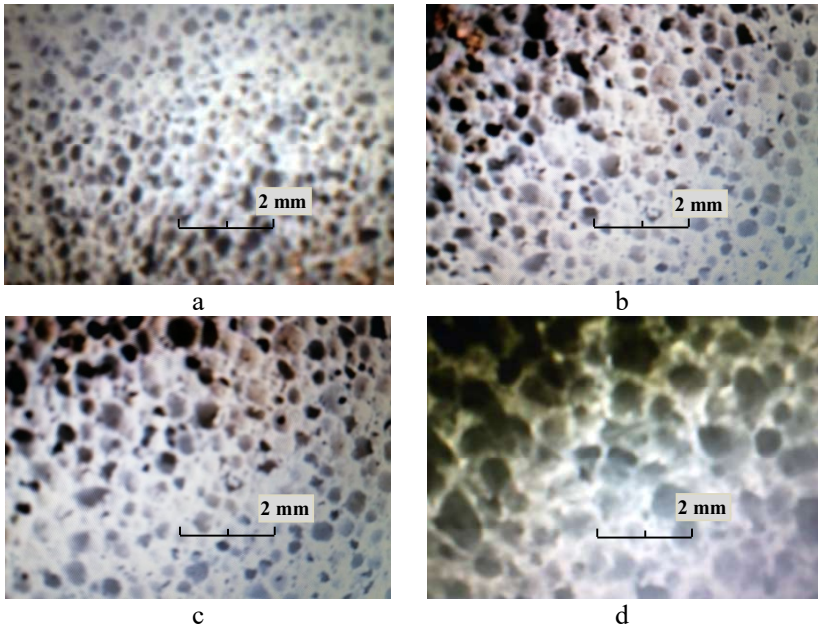


Fig. 3. Microstructural pictures of the cellular glass samples
a – variant 1; b – variant 2; c – variant 3; d – variant 4

Closed round pores formed the microstructure of the cellular glass samples. Their distribution was even according to Fig. 3. The size of cells was less than 1 mm, the dimensional ranges being shown in Table 4. The lowest dimensions characterized the sample obtained in variant 1, with the lowest proportions of SiC (2.8 %) and coal fly ash (9 %) and the highest dimensions belonged to the sample made with 3.1 % SiC and 11 % coal fly ash, corresponding to variant 4.

Comparing the features of products manufactured by microwave irradiation through SiC crucible coated with Y_2O_3 with that industrially made by conventional heating methods (without coal fly ash) [5] and especially with that experimentally obtained also by conventional methods using the same raw material types (glass waste and coal fly ash) and the same expanding agent type (SiC) (apparent density between 0.2-0.4 g/cm^3, optimal porosity of 75 %, and corresponded compressive strength of 1.5 MPa) [17] it can be observed that the differences in terms of qualyty are low. The main advantage of the method presented in the paper is the higher energy efficiency compared to conventional methods, the specific energy consumption being between 0.81-0.86 kWh/kg. In the industrial production of cellular glass and also in the numerous small-scale experiments presented in the literature, the energy consumption is not an important parameter, the predominant being the interest for the quality of the product. The literature provides few data on this. However, the few information shows that the average specific energy consumption of industrial processes is around 140 kWh/m^3, i.e. approximatelly between 0.80-1.16 kWh/kg [21]. It should be noted that the use of industrial-scale microwave equipment could increase the energy efficiency of the heating process by up to 25 % compared to a low power oven of the type used in this experiment [22].

4. Conclusions

The objective of the paper was to manufacture cellular glass with specific properties for use as thermal insulation material for buildings, from recycled post-consumer drinking bottle, coal fly ash and silicon carbide as an expanding agent, under the conditions of applying the unconventional method of microwave heating of the SiC crucible coated with Y_2O_3 film containing the pressed powder raw material. Knowing the property of Y_2O_3 as a remarkable microwave absorber as well as silicon carbide as a high microwave susceptible material, the original solution of using a SiC crucible coated with Y_2O_3 film was adopted for the process of indirect heating of raw material at temperatures of 960-970 ºC at which the sintering and expansion of the material takes place due to the formation of the porous structure. Also, the use of microwave heating in the cellular glass manufacturing process, unlike the conventional methods applied on an industrial scale, is one of the main originality elements of the work. As a consequence of the simultaneous use of microwave heating and the intensification of the process with Y_2O_3 film, the heating rate reached very high values (up to 29.5 ºC/min) and the specific energy consumption was reduced to 0.81-0.86 kWh/kg, i.e. at

the minimum limit of consumption in industrial processes. In terms of quality, the four variants of cellular glass had good thermal insulation properties (low apparent density, high porosity, and low thermal conductivity) required for use in building, adequate compressive strength and very homogeneous pore distribution in the material structure. The industrial application of the small-scale tested solution is feasible on a conveyor belt tunnel furnace by replacing the refractory vault with SiC plates coated with Y_2O_3 on the outer surface and mounting the microwave generators above the plates.

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