

HIGH POROSITY GLASS FOAM MADE WITH A LIQUID FOAMING AGENT BY MICROWAVE IRRADIATION

Marius Florin DRAGOESCU¹, Lucian PAUNESCU² and Sorin Mircea AXINTE³

¹University "Politehnica" of Bucharest, Department of Applied Chemistry and Materials Science, Research Center for Environmental Protection and Eco-Friendly Technologies, 1-7 Gh. Polizu street, District 1, Bucharest 011016, Romania, E-mail: mar_dmf@yahoo.com

²Daily Sourcing & Research SRL, 95-97 Calea Grivitei, District 1, Bucharest 010735, Romania, E-mail: lucianpaunescu16@gmail.com

³University "Politehnica" of Bucharest, Department of Applied Chemistry and Materials Science, 1-7 Gh. Polizu street, District 1, Bucharest 011016, Romania, E-mail: sorinaxinte@yahoo.com

ABSTRACT: The paper presents experimental results obtained in the manufacturing process of cellular glasses from commercial glass waste (soda-lime glass) as raw material, borax as a fluxing agent and aqueous NaOH solution as a foaming agent. Four variants were tested, in which the liquid foaming agent was increased from 3 to 7 %, while borax was reduced from 11 to 8 %. The sintering/foaming temperature increased from 710 °C (variant 1) to 780 °C (variant 4). Variant 3 was chosen as the optimal variant, sintered at 760 °C from 5.5 % NaOH, 9 % borax and 85.5 % glass waste. It had the following characteristics: apparent density of 0.26 g/cm³, porosity of 87.6 %, thermal conductivity of 0.069 W/m·K and compressive strength of 1.60 MPa. The specific energy consumption was low (0.89 kWh/kg) being at least at the level of consumption necessary for the manufacture of similar products by industrial conventional heating techniques.

KEYWORDS: aqueous solution, borax, glass waste, microwave heating, cellular glass.

1 INTRODUCTION

The Glass in the form of waste (especially from post-consumer drinking bottle and window glass waste from demolition and renovation of buildings) is a major source of risk to the health of planet by storing it on large grounds. In addition, the annual generation rate of this waste is constantly increasing. According to (Packaging waste, 2021), in 2019 the amount of glass waste generated in the EU reached 15.2 million tons. Also, the total amount of glass waste from demolition and renovation of buildings in the EU in 2013 was about 1.5 million tons (Hestin et al., 2016). Traditionally, part of the recycled glass waste, sorted by color, is re-used in the glass industry in the production of new glass products, for economic reasons. Processing operations for color selection of glass waste have quite high costs, being the main disadvantage of re-use of the waste.

In the last decades, a reorientation of the use of recycled glass waste to other application fields, especially to the manufacture of alternative building materials, has been felt in the world. Glass waste processing involves only the removal of contaminants (plastics, heavy metals, organic materials, etc.), washing and grinding to a high degree of fineness, without the need to selecting by color the glass. The manufacture of some materials

with thermal insulation properties (light weight, high porosity, low thermal conductivity) requires foaming the glass waste with a suitable expanding agent embedded into the waste mass, after a heat treatment at high temperatures between 750-1150 °C (Scarinci et al., 2005).

In the world, in recent decades, several European companies (from Belgium, Austria, Germany, the Czech Republic, Northern European countries) and the United States, to which China is also added by assimilating existing technologies, have conquered the world market of cellular glass made exclusively of glass waste. Several types of foaming agents are used in industrial manufacturing processes: carbonaceous products (coal, graphite, black carbon, glycerol), carbonates or sulfates (calcium carbonate, sodium carbonate, dolomite, gypsum), carbides and nitrides (silicon carbide, silicon nitride) (Scarinci et al., 2005).

Recent literature contains numerous papers on experiments by researcher teams of the world on the small-scale manufacture of cellular glass using a wide range of types of glass waste and other silicate waste [cathode ray tube (Lunip et al., 2016), borosilicate glass (Zhai et al., 2014), bituminous shale waste (Gorokhovskiy et al., 2002), metallurgical slag (Fidancevska et al., 2003), zinc hydrometallurgy waste (Pelino, 2000), quartz sand and coal gangue (Li et al., 2016), coal fly ash

(Chakartnarodom & Ineure, 2014), etc.] as well as foaming agents [agents of vegetable origin (Arcaro et al., 2016; da Silva et al., 2016), egg shell (Fernandes et al., 2013), organic agents (Attila et al., 2013), etc.], in order to improve the characteristics of the final products.

An experiment for the manufacture of cellular glass using glass waste (soda-lime glass) as raw material, borax as a fluxing agent and aqueous sodium hydroxide solution ($\text{NaOH}\cdot\text{H}_2\text{O}$) as a foaming agent was carried out recently, the results being published in literature (da Silva et al., 2019 a and b; Foreman, 2020). The combination of the fluxing agent and foaming agent proportions as well as the variation of the sintering temperature led to the variation of the porosity type (predominantly closed or predominantly open) being able to modify the product density in a wide range between 0.16-0.79 g/cm^3 , the maximum value of porosity reaching 92 %. The role of $\text{NaOH}\cdot\text{H}_2\text{O}$ was to reduce the value of the material density as well as to decrease the closed porosity ratio. The addition of borax in the presence of a small proportion of hydrated NaOH favored the densification and obtaining of a structure with closed pores. The thermally released gas that contributes to the glass waste foaming is steam (water vapor). At the transition temperature of the glass ($T_g = 587\text{ }^\circ\text{C}$) a crystalline phase of hydrated sodium and calcium silicate ($\text{Na}_2\text{CaSi}_2\text{O}_6\cdot 2\text{H}_2\text{O}$) is formed. Upon reaching the softening point of the glass-based mixture (above $700\text{ }^\circ\text{C}$) the release of water vapor from hydrated sodium and calcium silicate creates bubbles inside the mass with adequate viscosity of the glass, subsequently transformed by cooling into cells.

It should be noted that all industrial or experimental manufacturing processes noted above were performed by conventional heating techniques using electricity (electrical resistances) or thermal energy (fuel combustion). A faster and more economical technique according to the results obtained in numerous previous experiments (Paunescu et al., 2021 a and b; Dragoescu & Paunescu, 2020) was adopted and improved by the authors of the current paper within the Romanian company Daily Sourcing & Research. This is a predominantly direct microwave heating technique specially designed for the sintering/foaming processes of glass waste, in order to avoid the destruction of the internal structure of the glass-based mixture irradiated exclusively directly with microwave. The method by which the too intense flow of electromagnetic waves acts on the material subjected to heating in two ways (one predominantly direct and the other partially

indirect) allows to obtain cellular glasses with characteristics similar to those of products made by conventional techniques, under conditions in which the process occurs much more fast and the energy efficiency is higher.

The experimentation of manufacturing the cellular glass of glass waste, borax and an aqueous NaOH solution by applying the unconventional technique mentioned above is the objective of the current paper.

2 METHODS AND MATERIALS

2.1 Methods

Each In general, the use of a liquid foaming agent in the cellular glass manufacturing process promises a microstructure with fine and homogeneous porosity due to the easy penetration of the agent among the solid particles of the raw material. It is also the case of dissolving in water a powder material (NaOH) forming an aqueous solution. Sodium hydroxide can form several hydrate types ($\text{NaOH}\cdot n\text{H}_2\text{O}$). Among them the monohydrate ($\text{NaOH}\cdot\text{H}_2\text{O}$) with a concentration of its saturated water solution of 73.1 % corresponding to $62.6\text{ }^\circ\text{C}$ (Murch & Giauque, 1962), is the form under which NaOH is found on the market.

An important feature of sodium hydroxide dissolved in water is the complete decomposition into positive ions of sodium (Na^+) and negative ions of the OH^- group. At room temperature, the powder glass reacts slowly with the aqueous NaOH solution. Silica (SiO_2), existing in a high proportion in the composition of glass (around 70 % in the case of soda-lime glass), by reaction with hydrated NaOH forms sodium silicate (Na_2SiO_3) according to reaction (1):



As the temperature increases (to $700\text{-}800\text{ }^\circ\text{C}$ and even more), the intensity of this reaction intensifies (Kikuchi et al., 2016).

Sodium silicate (also known as "water glass") used alone as a liquid foaming agent in the process of making the cellular glass was investigated in (Heski et al, 2015). The result of these tests showed that the use of only water glass as a foaming agent leads to obtaining products with a very fine porous structure, with closed pores having dimensions between 4 nm and $800\text{ }\mu\text{m}$ and a compressive strength of 1.7 MPa. Much better known is the industrially applied method (Environmental, 2017) of combining the glycerol as a foaming agent with water glass and water as binders.

According to the determinations performed in the paper (da Silva et al., 2019 b), at the transition

temperature of the glass (587 °C) the formation of a crystalline phase containing hydrated sodium and calcium silicate ($\text{Na}_2\text{CaSi}_2\text{O}_6 \cdot 2\text{H}_2\text{O}$) was observed. Its crystallization water is removed around 700 °C. Water vapor dissociates into hydrogen and oxygen and these are the only gases that contribute to the glass foaming spreading in the form of bubbles in the thermally softened mass of the mixture and remaining blocked until the porous material cooling.

Sodium and calcium silicate hydrolyzes to form free ions of Na^+ and Ca^{2+} as well as silicic acid ($\text{SiO}_2 \cdot 2\text{H}_2\text{O}$), which decomposes into hydrated silica gel.

The experimental microwave equipment adopted by the authors was identical to that used in previous experiments (Paunescu et al., 2021 a and b; Dragoescu & Paunescu, 2020).

Thus, the 800 W-oven with a single magnetron was the type commonly used in the household, but constructively adapted for operation at high temperature (above 1000 °C).

The rotation mechanism of the oven was eliminated due to the large volume of the assembly including the heating sample and the protection system against the excessive intensity of the microwave field as well as the thermal protection.

The same ceramic tube from a mixture of high microwave susceptible materials (SiC and Si_3N_4) with a diameter of 125 mm, a height of 100 mm and a wall thickness of 2.5 mm, provided with a ceramic lid was used as a screen to reduce the effect of electromagnetic radiation.

The material subjected to heating was previously pressed in the form of a cylindrical sample with a diameter of about 80 mm and a height of about 60 mm and deposited freely on a metal plate in the inner space of the tube.

An effective thermal protection from ceramic fiber mattresses was used around the surface of the tube and above the lid.

The temperature control inside the ceramic tube was performed with a radiation pyrometer axially mounted above the oven, which visualized the sample through 30 mm holes provided in the lid and in the upper wall of the oven.

An overview of the equipment has been previously presented in some papers of authors (Paunescu et al., 2019; Paunescu et al., 2021 a; Axinte et al., 2021) and its constructive and functional scheme is shown in Fig. 1.

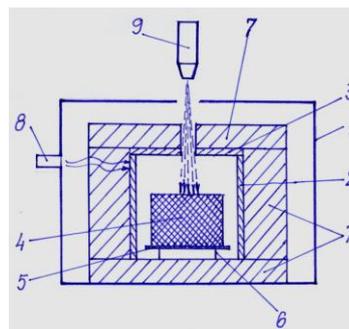


Fig. 1 The constructive and functional scheme of the equipment

1 – 800 W-microwave oven; 2- ceramic tube; 3 – ceramic lid; 4 – pressed sample; 5 – metal plate; 6 – support; 7 – thermal insulation; 8 – waveguide; 9 – pyrometer.

2.2 Materials

The materials used in this experiment were: post-consumer drinking bottle as raw material, sodium borate (borax) as a fluxing agent and 73 % aqueous NaOH solution as a foaming agent.

The post-consumer drinking bottle was constituted of colorless, green and amber recycled glass in approximately equal weight proportions. The glass waste was cleaned of possible contaminants (plastics, organics, metal), broken in a crusher, ground in a ball mill and sieved, the granulation of the powder being below 80 μm . According to some previous determinations, the oxide composition of the three types of commercial glass (soda-lime glass) used as raw material is shown in Table 1.

Table 1. Oxide composition (wt. %) of the glass waste types

Chemical composition	Colorless glass	Green glass	Amber glass
SiO_2	71.6	71.7	71.1
Al_2O_3	2.0	1.9	2.0
CaO	11.9	11.9	12.0
Fe_2O_3	0.1	-	0.2
MgO	1.0	1.1	1.0
Na_2O	13.2	13.2	13.1
K_2O	-	0.1	0.1
Cr_2O_3	0.05	0.05	-
SO_3	-	-	0.05

Borax was adopted as a fluxing agent due to its high content of Na_2O considered to be the most important chemical compound with flux properties, especially for the glass industry. Also, boron from its composition has a major role to increasing the

mechanical strength of the cellular glass made with borax addition.

The aqueous NaOH solution was used in this experiment as a foaming agent. The formation at 587 °C of the crystalline phase containing hydrated sodium and calcium silicate and then the removal of the crystallization water facilitated the foaming of the glass-based mixture by means of hydrogen and oxygen resulting from the dissociation of water vapor. The preparation of the aqueous NaOH solution was carried out in a 1 liter half-filled graduated flask, to which 1 M NaOH (40 g) was added. Then the water amount in the vessel was completed to the upper graduated limit and the contents were mixed. This preparation method was adopted to avoid the violent reaction of water contact with solid NaOH.

To manufacture cellular glass by the technique described above, four variants of the recipe containing colored glass waste, borax and aqueous NaOH solution were adopted by the authors. The values corresponding to each variant are presented in Table 2.

Table 2. Variants of manufacturing recipe of cellular glass

Variant	Colored glass waste (%)	Borax (%)	Aqueous NaOH solution (%)
1	86.0	11.0	3.0
2	86.0	10.0	4.0
3	85.5	9.0	5.5
4	85.0	8.0	7.0

3 RESULTS AND DISCUSSION

3.1 Results

The three components of the starting mixture were mixed in a separate vessel. The wet mixture was then loaded into a cylindrical metal mold and pressed axially to about 5 MPa. After pressing, the material was removed and stored freely in the oven on the metal plate. The amount of wet material (470 g) was kept constant in all variants. The functional parameters of the heating process are presented in Table 3.

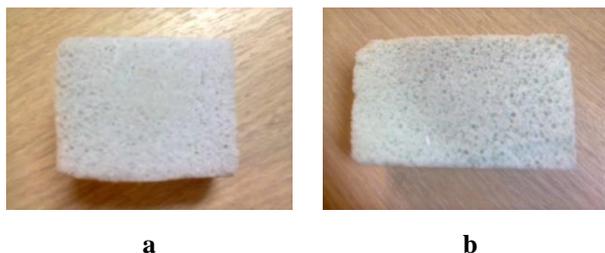
Table 3. Main functional parameters of the process

Parameter	Variant			
	1	2	3	4
Wet raw material/ cellular glass	470/	470/	470/	470/

amount (g)	328.7	329.0	328.9	328.7
Sintering temperature (°C)	710	730	760	780
Heating time (min)	25	26	28	30
Heating rate (°C/min)	27.6	27.3	26.4	25.3
Cooling rate (°C/min)	6.0	5.8	5.9	6.1
Index of volume increasing	1.40	1.50	1.80	2.10
Specific energy consumption (kWh/kg)	0.79	0.82	0.89	0.95

According to the data in Table 3, the sintering temperature of mixture increased from 710 °C to 780 °C corresponding to the proportion increase of aqueous NaOH solution from 3 % (variant 1) to 7 % (variant 4), although the borax proportion followed a decreasing slope between 11 % and 8 %. Due to the excellent energy efficiency of the predominantly direct microwave heating, the heating rate had much higher values (25.3-27.6 °C/min) compared to the heating rates applied in conventional heating (around 10 °C/min [3]). By default, the duration of the heating process was shorter (25-30 min) and the specific energy consumption had low values between 0.79-0.95 kWh/kg, at least at the level of the values registered in the conventional industrial processes (Energocell, 2014).

The section appearance of the four cellular glass samples obtained by the technique presented above is shown in Fig. 2. The influence of increasing the sintering temperature and the proportion of aqueous NaOH solution is obvious by increasing the size of cells that compose the sample porous structure. The high rate of predominantly direct microwave heating did not disturb the structural homogeneity of the products.



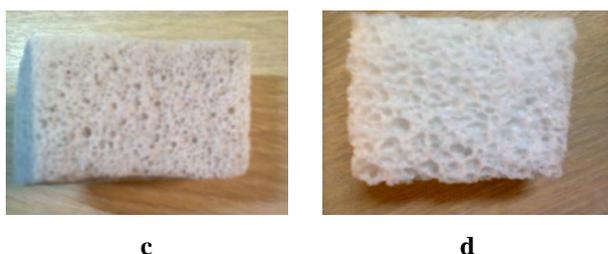


Fig. 2 Section appearance of the cellular glass products

a – variant 1 sintered at 710 °C; b – variant 2 sintered at 730 °C; c – variant 3 sintered at 760 °C; d – variant 4 sintered at 780 °C.

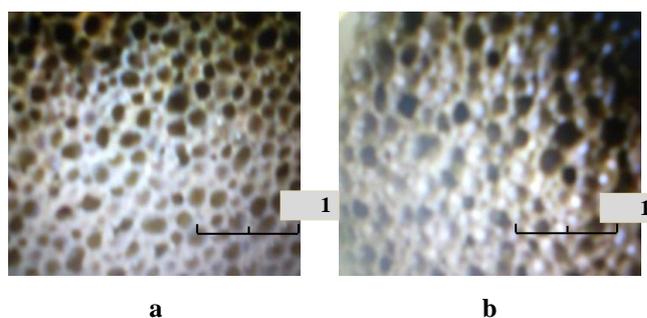
The cellular glass samples were subjected to the common methods of determining the main characteristics. The apparent density was measured by the gravimetric method (Manual, 1999). The porosity was calculated by the method of comparing the true and apparent density (Anovitz & Cole, 2005). The thermal conductivity was measured by the 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 by the water immersion (for 24 hours) method (ASTM D570) and the microstructural configuration of the cellular glasses was investigated with an ASONA 100X Zoom Smartphone Digital Microscope.

The results of physical, thermal, mechanical and morphological features measurements and determinations of the investigated cellular glass products are shown in Table 4.

Table 4. Main physical, thermal, mechanical and morphological characteristics of the cellular glass products

Characteristic	Variant			
	1	2	3	4
Apparent density (g/cm ³)	0.45	0.39	0.26	0.17
Porosity (%)	78.6	81.4	87.6	91.9
Thermal conductivity (W/m·K)	0.091	0.085	0.069	0.048
Compressive strength (MPa)	2.14	1.95	1.60	1.58
Water absorption (vol. %)	2.25	2.10	2.20	2.00
Cell size (mm)	0.1-0.3	0.1-0.4	0.4-0.6	0.6-1.0

Analyzing the data in Table 4, it results that the use of a liquid foaming agent, such as aqueous NaOH solution, favors obtaining a homogeneous microstructure with uniform cells distribution. The experiment included the combination use of this type of foaming agent with a fluxing agent (borax). A higher proportion of borax (10-11 %) in association with a reduced proportion of NaOH (3-4 %) as in the case of variants 1 and 2 favors the densification of the glass foam and obtaining a closed porosity. Consequently, the apparent density of cellular glass has slightly high values (0.45 g/cm³ and 0.39 g/cm³, respectively), an acceptable porosity (78.6-81.4 %) and a thermal conductivity slightly above the performance limit of 0.080 W/m·K, which together indicate the thermal insulation properties of the material above the acceptable average level in the field of building materials. Significantly higher proportions of NaOH of 5.5-7 % and slightly lower of borax (8-9 %) as in the case of variants 3 and 4 as well as a significant increase of the sintering/foaming temperature (from 710-730 °C corresponding to variants 1-2 at 760-780 °C corresponding to variants 3-4) significantly modified the thermal insulation properties of the foamed products. The apparent density was reduced to low values (0.17-0.26 g/cm³), the porosity reached 91.9 % in the case of variant 4 and the thermal conductivity greatly decreased to values between 0.048-0.069 W/m·K. The compressive strength followed a slightly decreasing slope from manufacturing variant 1 to variant 4. The mechanical strength values are located at a relatively high level compared to other similar porous materials, reaching 2.14 MPa in the case of variant 1 and a minimum of 1.58 MPa, more than acceptable for a thermal insulation material for buildings, in the case of variant 4. The water absorption had normal values for a cellular glass (below 2.25 vol. %). The analysis of microstructural peculiarities of the four variants of foamed products was performed by investigating the pictures presented in Fig. 3.



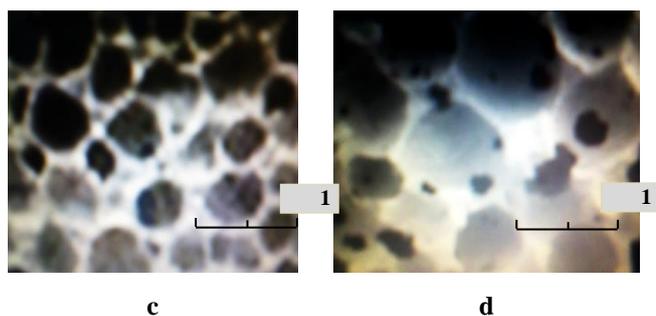


Fig. 3 Microstructural configuration of the cellular glass products

a – variant 1 sintered at 710 °C; b – variant 2 sintered at 730 °C; c – variant 3 sintered at 760 °C; d – variant 4 sintered at 780 °C.

According to Fig. 3, the microstructural appearance of the samples made in variants 1 and 2 sintered at 710-730 °C is characterized by homogeneity, cells with small dimensions, between 0.1-0.3 mm (sample 1) and between 0.1-0.4 mm (sample 2). The samples obtained by sintering at higher temperatures (760 °C and 780 °C, respectively) have a tendency (mild in sample 3 and more pronounced in sample 4) to form open cells by communication between neighboring cells. The apparent density of these samples decreases, but the compressive strength tends to remain relatively constant or to decrease very easily, because intercellular communication channels are formed especially in denser areas of the cellular structure known as "struts", which ensures a good mechanical strength of the porous material matrix (Tulyaganov et al., 2006). The cells dimension is increasing compared to those of samples 1 and 2, having the following ranges of values: 0.4-0.6 mm (variant 3) and 0.6-1.0 mm (variant 4).

Analyzing the parameters of the manufacturing process and the characteristics of the four cellular products obtained by the unconventional technique of microwave heating, the optimal variant was chosen by the authors. This is variant 1 made by sintering at 760 °C a mixture composed of 85.5 % glass waste, 9.0 % borax and 5.5 % aqueous NaOH solution. Using a high heating rate of 26.4 °C/min, the specific energy consumption was only 0.89 kWh/kg. The main features of the final product were: apparent density of 0.26 g/cm³, porosity of 87.6 %, thermal conductivity of 0.069 W/m·K and compressive strength of 1.60 MPa. The cells size was between 0.4-0.6 mm, the microstructure appearance being homogeneous. If the characteristics of the product are similar to those of glass foams produced by conventional techniques with moderate heating rates, the energy efficiency of the process described in the current paper is higher.

3.2 Discussion

The application of electromagnetic wave energy (microwaves) in cellular glass manufacturing processes is generally a very fast and economical method (Kharissova et al., 2010), but which has proven to be inadequate by direct irradiation of silicate materials (including the glass) according to tests performed by authors in 2017 (Paunescu et al., 2017). For this reason, given the use of an oven with a useful volume of 20 L powered by microwaves with an installed power of 800 W, the direct heating of a powder silicate material (a commercial soda-lime glass) of about 400-500 g could be too intense, requiring protection from a highly microwave-absorbing material and, at the same time, with a thickness low enough (2.5 mm) to also allow its penetration for a less intense contact with the silicate material. This original technical solution was the basis of all subsequent experiments, including the current one.

The significantly higher heating rate (above 25 °C/min) compared to the usual rates recommended in conventional processes (around 10 °C/min) did not negatively influence the properties of the cellular glass.

4 CONCLUDING REMARKS

The paper aimed to manufacture a cellular glass with thermal insulation properties (low bulk density, high porosity and low thermal conductivity) using an unconventional technique of heat treatment of glass-based raw material. This technique is fundamentally different from conventional techniques commonly applied in industrial processes, using electromagnetic waves (microwaves). The work originality consists in adopting the predominantly direct and partially indirect microwave heating solution by placing between the radiation emitting source and the material a ceramic tube made of SiC and Si₃N₄, strongly microwave absorbing, but with a very thin wall thickness (2.5 mm), which also allows its penetration by the microwave flux. Mixed heating diminishes the effect of integral direct heating, which previously proved to be too intense causing major damage to the internal structure of the glass subjected to the foaming process.

In this experiment, performed on a 800 W-microwave oven adapted for high temperature operation in the Romanian company Daily Sourcing & Research, four cellular glass manufacturing recipes were tested using glass waste (between 85-86 %) as raw material, borax (between 8-11 %) as a fluxing agent and aqueous NaOH solution (between 3-7 %) as a liquid foaming agent. The sintering/foaming temperature had an increasing

slope from 710 °C to 780 °C as the proportion of foaming agent increased and the proportion of borax decreased. The optimal variant was considered by authors the variant achieved by sintering at 760 °C of the mixture composed of 85.5 % glass waste, 9 % borax and 5.5 % aqueous NaOH solution. The porous product characteristics were: apparent density of 0.26 g/cm³, porosity of 87.6 %, thermal conductivity of 0.069 W/m·K and compressive strength of 1.60 MPa, values almost similar to those of products of the same type manufactured by conventional techniques. The specific energy consumption was economical being determined at 0.89 kWh/kg, at least at the level of the values registered in the conventional industrial processes.

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