POROUS HIGH-STRENGTH BUILDING MATERIAL NONCONVENTIONALLY MADE FROM RESIDUAL GLASS

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ABSTRACT: A porous high-strength material having load-bearing thermal insulation properties suitable as light filling material for building foundation, drainage, road construction, underground insulation of heat pipes, etc. was experimentally made by sintering and foaming at 830-840 ºC applying the microwave irradiation to a pressed powder mixture composed of recycled post-consumer glass, glycerol, calcium carbonate, water glass and water. The manufacturing recipe adopted by authors is original by combining a liquid carbonaceous expanding agent (glycerol) with a solid agent (calcium carbonate) aiming at the increase of compressive strength at 6.5-7.6 MPa, the product bulk density being under 0.20 g cm^{-3}. The nonconventional technique of predominantly direct microwave heating is also original and more economical compared to conventional methods applied both in industrial processes and in small-scale experiments.
KEYWORDS: porous glass gravel, microwave heating, recycled post-consumer glass, glycerol, calcium carbonate, water glass.

1. INTRODUCTION

The oil crisis and the danger of global warming caused by greenhouse gas pollution (mainly carbon dioxide) initiated in the last decades of the 20th century have contributed to important changes in humanity's attitude towards irrational consumption of primary fuel (hydrocarbons and coal) as well as compared to the very large quantities of material waste deposited in landfills under the conditions of an excessive high annual rate of their generation. Thus, the need for efficient management of available resources resulting from industrial activities (as by-products) or those existing in nature and unexploited has been considered by researchers around the world. Consequently, in the world, technologies high consuming primary energy and generating air pollutants, producing materials required in industry, building, and other branches of the economy have been significantly reduced and products have been partially or completely replaced with similar more economical materials and using raw materials recovered or recycled from waste.

The glass is one of the most widespread materials globally, being used mainly as a container glass and as a flat window glass in building. Worldwide, around 130 million tons (Mt) are produced annually, of which 48 % are intended for container glass, 42 % for flat glass (construction and window glass, car glass, etc.), 5 % for tableware, and the rest for other types of glass products [1]. Due to its relatively short life cycle, the amount of residual glass (soda-lime glass type) in the form of post-consumer drinking bottle and flat glass waste resulting from building demolition and redevelopment is very high. The glass is a resource with unlimited recycling potential without loss its initial quality. The recycling rate of container glass is very high in the European Union countries, being on average 74 %, Sweden, Germany, and Italy having very high rates. However, the average recycling value of this type of glass in the world is much lower (below 40 %) [1]. The glass industry uses relatively low amount of waste as a raw material for the manufacture of new glass and prefers to recover its own residual glass resulting from the industrial manufacturing process, due to the high costs of waste processing for selection by color (so by quality) technologically required. However, other areas of application are suitable for the use of this waste as a very cheap raw material. The special properties of the parental glass (durability, chemical and physical stability, hardness, fire and humidity resistance, rodents, insects, and bacteria attack resistance, etc.) allow the manufacture (by heat expansion process) of some thermal insulation materials (named cellular glass or glass foam) attractive for the construction sector being able to compete with existing building materials in the market [2].

Several companies in the United States and Europe (Pittsburgh Corning, Misapor, Geocell, Glapor, etc.) began since 1980s industrial production of cellular glass for various applications in facilities in Austria, Germany, Switzerland, Belgium, Italy, Czech

Republic, Sweden, Norway, Finland, the United States, China, etc. Types of cellular glass can have characteristics suitable for their use since lightweight thermal insulation boards for interior and exterior walls of the building up to denser thermal insulation materials with high enough mechanical strength for lightweight foundations, roof gardens, pavements, drainage, road and railway infrastructure, sports fields, swimming pools, underground insulation of heat pipes and heat storage tanks, etc. [3].

The current work focused on the experimental manufacture of cellular glass with heat insulation properties (low density and low heat conductivity) and simultaneously with high compressive strength, i.e. the type of cellular glass gravel used as light filler in load-bearing thermal insulation applications.

The most well-known process for the manufacture of porous glass gravel used by industrial manufacturers is the preparation of a finely ground mixture of residual glass, an expanding agent, and sometimes other mineral agents, placed continuously in a layer of about 10 cm across the width of the conveyor belt of a tunnel oven heated by conventional methods (electrically or by burning a fossil fuel). The expanding agent decomposes or oxidizes depending on its nature within a known temperature range releasing gaseous products into the thermally softened mass of the glass-based mixture. The gases form numerous small bubbles, which by increasing the pressure due to the rising temperature expand generating a porous structure with polyhedral cells [2]. What is specific to the cellular glass gravel manufacturing process is the relatively high cooling rate of the expanded material, which on the one hand does not allow the process of collapsing the foam structure by coalescing pores and on the other hand creates internal stresses that crack the mass of the product facilitating the easy detachment of the gravel pieces at the cold end of the oven conveyor belt.

According to the few technical data provided by the manufacturers [3], the expanding agents used in industrial processes are: gypsum, limestone or silicon carbide (2 wt.%) (in Misapor company), glycerol (1 wt.%), water glass (12 wt.%) and kaolin (under 0.5 wt.%) (in Glapor). Glamaco, a very important supplier of industrial equipment for making the cellular glass gravel, recommends the use of silicon carbide, coal powder, calcium carbonate or gypsum as expanding agents, manganese oxide as an oxygen-supplying agent, and water glass as an enveloping material. The manufacturing recipe proposed by Glamaco includes (except the residual glass of 95 wt. %): glycerol, calcium carbonate as expanding agents and water glass (5 wt. %) as well as water addition and kaolin in very low ratio [4].

TECHNOpor products made by Misapor are representative for the type of cellular glass gravel with high compressive strength between 4.9-6.0 MPa, low heat conductivity between 0.075-0.095 W $(mK)^{-1}$, and low bulk density between 0.12-0.19 g cm^{-1}. They are fireproof, non-moisture absorbent (between 1-6 vol. %), non-deformable, resistant to frost, non-corrosive, resistant to various aggressions (insects, rodents, bacteria), etc. [5].

Unlike the conventional techniques of industrial manufacture of porous glass gravel, the team of authors of the present paper has experimentally made in the last years products with similar characteristics by applying the own original method of heat treatment using the conversion of electromagnetic power (microwave) into heat. The microwave warming of solids began to be industrially used at the end of the 20^{th} century, but only in drying and low-temperature warming processes, although the literature [6] acknowledges that this nonconventional process is quick, ecological, and cost-effective, suitable for efficient warming of various material types (organic, ceramic, polymeric, metallic, glass, etc.). The authors of the present paper have tested several making recipes almost similar to those used in industry by applying their own method of predominantly direct microwave heating. The paper [3] is a synthesis of these experiments in which colored (green and amber) container glass was used together with calcium carbonate ($CaCO_3$), borax, and sodium silicate (Na_2SiO_3) (i), clear flat glass together with glycerol, sodium silicate, and water (ii), container glass mixture (colorless, green, and amber) together with glycerol, sodium silicate, and water (iii) as well as the same mixture of recycled container glass together with silicon carbide (SiC) and water (iiii). The sintering/expanding temperature varied between 818-855 ºC in the case of the first three testing groups and was over 920 ºC in the case of experiments using SiC as an expanding agent. Heating rates slightly above 19 ºC/min favored obtaining specific energy consumptions around 1 kWh/kg, lower in the case of experiments using aqueous solution of glycerol and sodium silicate (0.86-0.88 kWh/kg). Tests for the manufacture of cellular glass gravel with $CaCO_3$, borax, and sodium silicate have shown that the products are dense, with relatively high apparent density (0.62 g cm^{-1}) and water absorption under 7 vol. %, but with the highest compressive strength (7.4 MPa). A similar

mechanical strength (7.5 MPa) was reached in the case of using SiC, instead, the apparent density decreased to 0.35 g cm^{-1}. Experiments using the aqueous glycerol solution had low apparent density (0.24 g cm^{-1}), low heat conductivity [0.063 W $(mK)^{-1}$], but slightly lower compressive strengths (5.3 and 5.9 MPa, respectively). The conclusion of the paper mentioned above was that the optimal solution could be to use the recipe including 83 % mixture of glass container waste, 1 % glycerol, 8 % sodium silicate, and 8 % water, leading to products with a compressive strength of 5.9 MPa and low pore size between 0.3-0.6 mm.

The experiment presented in the paper combined for the first time two manufacturing processes of porous glass gravel used independently in industry. Recycled post-consumer drinking glass was the basic raw material. $CaCO_3$ as an expanding agent mixed with borax as a fluxing agent was one of the processes, while glycerol as an expanding agent forming the aqueous solution which also includes sodium silicate (water glass) and water was the second technological process. Each of these processes is commonly used as a technological method of manufacturing porous glass gravel. The aim of the experiment was to obtain a light porous product with higher compressive strength than those achieved by each of the mentioned technologies. This combination was possible due to the relatively similar range of temperatures at which the decomposition processes of expanding agents take place with the release of gaseous compounds that facilitate the foaming of glass.

2. MATERIALS AND METHODS

2.1 Materials

As mentioned above, the basic raw material of the experiment was recycled post-consumer drinking glass. Three types of glass (colorless, green, and amber) whose proportions were approximately equal had the oxide compositions [7] shown in Table 1.

Table 1. Oxide composition of glass

Oxide composition	**Glass type (wt. %)**		
	Colorless	Green	Amber
SiO_2	71.7	71.8	71.1
Al_2O_3	1.9	1.9	2.0
CaO	12.0	11.8	12.1
Fe_2O_3	-	-	0.2
MgO	1.0	1.2	1.1
Na_2O	13.3	13.1	13.3
K_2O	-	0.1	0.1
Cr_2O_3	0.05	0.09	-
SO_3	-	-	0.05

Processing the residual glass (selecting, washing, breaking, grinding, and sieving) was carried out in the Romanian company Bilmetal Industries SRL Popesti-Leordeni, Ilfov. The grain size of waste was below 80 μm.

$CaCO_3$ as an expanding agent was purchased from the market with a fine grain size below 40 μm. Its use in the experiment was performed without supplementary mechanical processing.

Borax has been added to the starting material mixture as a fluxing agent due to its high Na_2O content (30.8 %), known as an excellent fluxing material [8]. On the other hand, its boron content (about 11 %) favors the increase of the mechanical strength of cellular glass. The granulation of commercial borax below 400 μm required its grinding to reduce the grain size below 80 μm.

Glycerol ($C_3H_8O_3$) is a carbon-containing liquid expanding agent. Being in a liquid state, it has the ability to spread among the fine particles of the glass, contributing to a very fine porosity of cellular glass. In the oxidizing atmosphere of oven glycerol thermally decomposes into a wide range of components between carbon dioxide (CO_2) and pure carbon [9, 10].

Due to the high oxidizing capacity of carbon in the glycerol composition, there is a danger of premature oxidation of the glycerol before reaching the glass softening temperature. Therefore, the use of water glass (also in the liquid state) together with glycerol is technologically required to slow down the decomposition of glycerol and accelerate the sintering of glass.

The preparation of the starting mixture consisted of dosing and mixing the solid materials (residual glass, $CaCO_3$, and borax), dosing and mixing the liquid components (glycerol, water glass, and distilled water) in separate vessels, and then pouring the aqueous solution over the solid mixture. Further operations were mixing the wet mixture with a metal rod, loading it into a removable cylindrical metal mold and its axially pressing to about 6-8 MPa. Then, the pressed mixture was removed from the mold to be loaded freely into the microwave oven.

2.2 Methods

In technological terms, the two glass expansion processes take place independently. According to [11], the decomposition of $CaCO_3$ takes place at over 750 ºC, CO_2 being released after the reaction (1).

$$CaCO_3 = CaO + CO_2 \quad (1)$$

The reaction is initiated at a slow rate and then develops rapidly. The work [2] considers that in case of soda-lime glass (commercial glass) the temperature range in which the foaming is optimal is 800-900 ºC.

The glycerol decomposition is initiated at low temperature (about 300 ºC) and the process increases in intensity at 750 ºC, according to [12]. The gaseous products released after the glycerol decomposition are: CO, H_2, CH_4, C_2H_4, CO_2, C_2H_6, C_3H_6, 1.3-butadiene, and isobutene.

According to the basic principle of the foaming process [2], the released gases remain trapped in the softened viscous mass of the glass, while the released solid products (calcium oxides) enter the composition of the molten glass.

Unlike conventional methods of heating the starting material mixture used in industrial processes, the predominantly direct microwave heating technique experimentally tested by the authors of the current paper is based on the conversion of microwave power into heat by contact of electromagnetic waves with the material subjected to heating. This nonconventional technique has fundamentally different peculiarities compared to the principles of the conventional heating. According to [13, 14], the heat is initially generated into the solid core, where the highest temperature is reached. The heat propagation throughout the material mass takes place volumetrically from the inside to the outside, i.e. completely the opposite of the heat transfer in conventional thermal processes. Also, another peculiarity of this technique is the selectivity, which means that only the targeted microwave-susceptible material is efficiently heated, while the other massive components of the oven (vault, walls, hearth, etc.) are heated to a very small extent. In this way, the energy efficiency of the heating process is obviously higher in the case of the nonconventional method highlighted in all the experiments previously performed by the authors.

The microwave susceptibility of materials is ensured by the existence of at least one component with this property. These materials are called dielectrics. In the case of glass, very low ratios of Fe_2O_3 or Cr_2O_3 in its chemical composition (as inherent contaminants) which are microwave-susceptible at room temperature, compensate for the microwave-transparent property of the glass due to the high content of SiO_2 and Al_2O_3. Also, carbon from the liquid expanding agent (glycerol) is an excellent microwave-susceptible material [15]. The increase of the oven temperature above 500 ºC leads to the increase of electrical conductivity of materials and at the same time the microwave susceptibility [16].

The experimental equipment for the production of porous glass gravel was the same used in previous tests, existing in the joint experimental base of the Romanian companies Daily Sourcing & Research SRL and Cosfel Actual SRL in Bucharest and presented in the literature [17-19].

The basic component of the equipment was an 800 W-domestic microwave oven equipped with a single magnetron, constructively adapted for operation at high temperature (over 1000 ºC). The pressed mixture was positioned freely on a thermal insulation bed of ceramic fiber mattresses placed on the bottom of the oven. The originality of the predominantly direct microwave heating system is the use of the ceramic tube provided with a lid made of high microwave-susceptible materials (SiC + Si_3N_4) with an optimal thickness of 2.5 mm, placed over the pressed mixture and supported on the thermal insulation bed. The propagation of heat from the inside of the material to the outside determined the protection of the outer surface of the tube and the lid with thick mattresses of ceramic fiber, in order to conserve the thermal energy from the inner space of the ceramic tube.

Figure 1 shows the constructive and operational scheme of the experimental equipment described above.

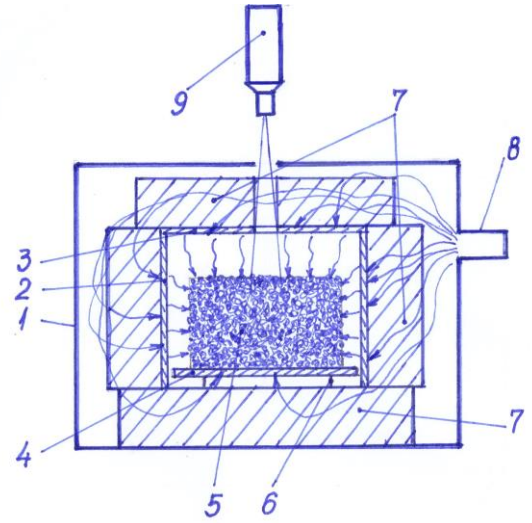


Figure 1. Constructive and operational scheme of the experimental equipment
1 – microwave oven; 2 – ceramic tube; 3 – ceramic lid; 4 – stainless steel plate; 5 – pressed mixture; 6 – metal support; 7 – thermal insulation; 8 – magnetron waveguide; 9 – radiation pyrometer.

2.3 Characterization methods of the products

Porous glass gravel samples, resulted after the sintering/expanding experimental process, were

tested in laboratory to determine the physical, thermal, mechanical, and morphological features. Apparent/bulk density, porosity, heat conductivity, compressive strength, absorption of water, and microstructural organization were determined by common methods of analysis. Thus, the apparent density was determined by the gravimetric method [20], while the bulk density was measured by reporting the mass of a batch of fully loaded pieces in a container of known volume. The porosity was calculated by the comparison method of the true and apparent density of the material, experimentally measured [21]. The compressive strength was determined with TA.XTplus Texture Analyzer and the heat conductivity was measured by the guarded-comparative-longitudinal heat flow technique, according to ASTM E1225-04. The absorption of water characteristic of the sample was measured by the method of its immersion in water (ASTM D570) and their microstructural organization was identified with an ASONA 100X Zoom Smartphone Digital Microscope.

3. RESULTS AND DISCUSSION

3.1 Results

Three experimental variants (Table 2) were adopted by the authors to test the manufacture of porous glass gravel using simultaneously the combination of two recipes of expanding agents ($CaCO_3$ together with borax as well as glycerol in aqueous solution including water glass and distilled glass). The heating technique was nonconventional by predominantly direct microwave heating.

Table 2. Composition of experimental variants

Composition	**Experimental variant (wt. %)**		
	No. 1	No. 2	No. 3
Residual glass	93.3	90.2	86.9
$CaCO_3$	0.5	0.8	1.1
Borax	1.2	2.0	3.0
Glycerol	1.0	1.0	1.0
Water glass	4.0	6.0	8.0
Distilled water	9.0	9.0	9.0

According to the data in Table 2, $CaCO_3$ was used in a usual range between 0.5-1.1 wt. %, while glycerol was kept constant at 1.0 wt. %. Borax as a fluxing agent had values between 1.2-3.0 wt.% corresponding to borax/$CaCO_3$ ratios between 2.4-2.7. The weight proportion of glycerol (1 wt. %) is usually used at the industrial making of cellular glass gravel exclusively with glycerol and water glass and the water glass/glycerol ratio (between 4-8) is also commonly in this case.

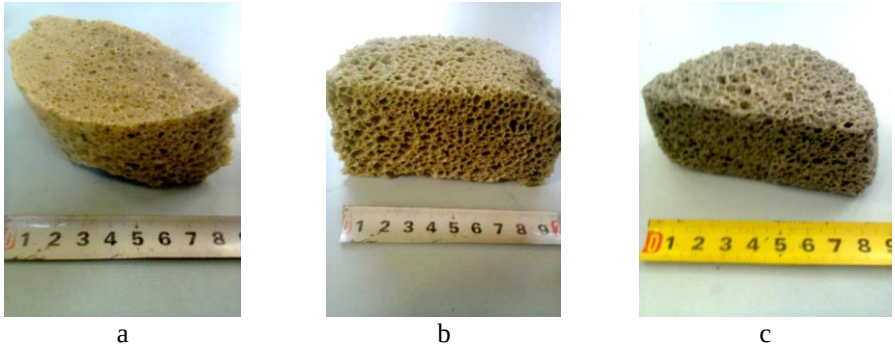
The functional parameters of the experimental process are presented in Table 3.

Table 3. Functional parameters of the process

Parameter	**Variant**		
	No. 1	No. 2	No. 3
Wet raw material amount (g)	450	450	450
Sintering/expanding temperature (ºC)	830	835	840
Heating time (min)	37	39	45
Average rate (ºC/min) - heating - cooling	 21.9 7.5	 20.9 7.5	 18.2 7.5
Index of volume increasing	1.35	1.60	1.78
Porous product amount (g)	391	393	390
Specific energy consumption (kWh/kg)	0.99	1.03	1.20

According to the data in Table 3, the sintering/expanding temperature of the manufacturing process was in the range 830-840 ºC. The heating rate had higher values (18.2-21.9 ºC/min) compared to those used in the industrial conventional processes without affecting the structural homogeneity of products. Due to the high energy efficiency of the unconventional process the specific energy consumption had low values between 0.99-1.20 kWh/kg.

The appearance of porous products manufactured by the unconventional heating technique is shown in Figure 2.



a b c

Figure 2. Appearance of porous glass gravel made by the unconventional heating technique
a – variant 1; b – variant 2; c – variant 3.

According to the images in Figure 2, the products made by the nonconventional technique using the combination of the two expanding agents and the additives borax and water glass have the adequate appearance in physical terms, with fine porosity and good macrostructural homogeneity. The product obtained according to variant 1 (with the $CaCO_3$/glycerol ratio = 0.5 and the water glass/glycerol ratio = 4) has the finest porosity. Increasing the two ratios to 0.8 and 6 respectively, contributes to obtaining a product with increasing porosity, but with similar structural homogeneity. The product according to variant 3 (with the highest values of the ratios mentioned above of 1.1 and 8 respectively) has the appearance of a sufficiently fine porosity, but the structural homogeneity is slightly affected.

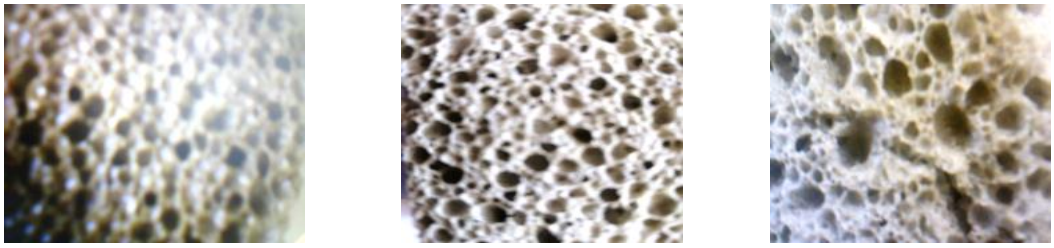
The determination of the main characteristics of porous glass gravel samples allowed the identification of the quality of these products (Table 4).

Table 4. Characteristics of the porous glass gravel samples

Characteristic	**Variant**		
	No. 1	No. 2	No. 3
Apparent/bulk density (g cm^{-1})	0.30/0.15	0.32/0.16	0.41/0.19
Porosity (%)	85.7	84.8	80.5
Thermal conductivity [W $(mK)^{-1}$]	0.074	0.073	0.090
Compressive strength (MPa)	6.5	6.9	7.6
Water absorption (vol. %)	1.2	0.8	0.9
Pore size (mm)	0.2-0.5	0.2-0.6	0.2-1.0

The examination of the data in Table 4 shows that the products made by manufacturing variants 1 and 2 have characteristics suitable for the purpose of the research, i.e. excellent thermal insulation properties (bulk density of 0.15-0.16 g cm^{-1}, thermal conductivity of 0.073-0.074 W $(mK)^{-1}$ and porosity of 84.8-85.7 %) and simultaneously high values of compressive strength of 6.5-6.9 MPa. In addition, the water absorption is very low of 0.8-1.2 vol. %. The sample corresponding to variant 3 has also thermal insulation material properties, but with slightly higher bulk density and thermal conductivity values [0.19 g cm^{-1} and 0.090 W$(mK)^{-1}$ respectively]. The water absorption has also very low value (0.9 vol. %). Instead, the compressive strength reaches a high value of 7.6 MPa.

The microstructural configuration of porous glass gravel samples is shown in Figure 3.



a b c

Figure 3. Microstructural configuration of porous glass gravel samples
a – variant 1; b – variant 2; c – variant 3.

As can be seen from the images, the microstructural configuration of the samples corresponding to variants 1 and 2 indicates the excellent homogeneity of the pore size and their distribution (between 0.2-0.5 mm and between 0.2-0.6 mm, respectively). Instead, the microstructural homogeneity of the sample made in variant 3 is unsatisfactory (pore size between 0.2-1.0 mm and uneven pore distribution). This analysis shows that an addition to the starting mixture of more than 1 % CaCO3 and more than 2 % borax together with the aqueous solution of glycerol and water glass is not indicated, although this combination contributes to increasing the compressive strength up to 7.6 MPa.

3.2 Discussion

The experimental results obtained by the team of authors confirm the achievement of the paper objective, i.e. the manufacture by an original method of unconventional heat treatment of porous glass gravel with high mechanical characteristics above

the level of similar industrial products (4.9-6.0 MPa, specific to TECHNOpor products of Misapor).

The original manufacturing recipe characterized by the simultaneous use of two usual methods included $CaCO_3$ (0.5-1.1 %) together with borax (1.2-3 %) and aqueous solution of glycerol (1 %) together with water glass (4-8 %). Of the three tested variants, variant 2 (0.8 % $CaCO_3$, 2 % borax, 1 % glycerol, 6 % water glass, and 9 % distilled water addition as a binder) was adopted as optimal with the following characteristics: 0.16 g cm^{-1} bulk density, 0.073 W $(mK)^{-1}$ thermal conductivity, 6.9 MPa compressive strength, 0.8 vol. % water absorption, and 0.2-0.6 mm pore size.

The specific energy consumption of the process was low (1.03 kWh/kg). According to [6], the application of the nonconventional method on an industrial scale would allow the reduction of the consumption obtained on a small scale by up to 25% due to the improvement of the energy efficiency of the heating process.

4. CONCLUSION

The aim of the paper was the experimental manufacture of porous glass gravel from recycled residual glass by an original heat treatment method for sintering/expanding at 830-840 ºC having high mechanical features. Similar material industrially made in the world by conventional techniques has load-bearing thermal insulation properties being suitable as light filling material for building foundation, drainage, road construction, underground insulation of heat pipes, etc. Its compressive strength value is between 4.9-6.0 MPa. The original manufacturing recipe was characterized by the simultaneous use of two usual methods including $CaCO_3$ (0.5-1.1 %) together with borax (1.2-3 %) and aqueous solution of glycerol (1 %) together with water glass (4-8 %), and distilled water (9 %). The optimal variant based on 0.8 % $CaCO_3$, 2 % borax, 1 % glycerol, 6 % water glass, and 9 % distilled water was sintered at 835 ºC. The main characteristics of the optimal porous product were: 0.16 g cm^{-1} bulk density, 0.073 W $(mK)^{-1}$ thermal conductivity, 6.9 MPa compressive strength, 0.8 vol. % water absorption, and 0.2-0.6 mm pore size, being suitable for the mentioned area of application. In addition, the value of the compressive strength is higher compared to those industrially obtained through the usual technologies.

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