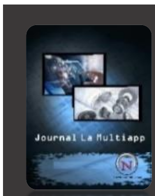

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Manufacture of Cellular Glass Using Oak Leaves as a Foaming Vegetable Agent

Lucian Paunescu[1], Sorin Mircea Axinte[1,2], Marius Florin Dragoescu[2], Felicia Cosmulescu[3]

[1]*Daily Sourcing & Research SRL Bucharest, Romania*
[2]*University "Politehnica" of Bucharest, Department of Applied Chemistry and Materials Science Bucharest, Romania*
[3]*Cosfel Actual SRL Bucharest, Romania*

**Corresponding Author: Lucian Paunescu*
Email: lucianpaunescu16@gmail.com

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Abstract

The manufacture experimentation of a cellular glass exclusively from mineral waste and natural residues using the unconventional technique of microwave irradiation was the objective of the research whose results are presented in the paper. The originality of the paper results from the use of oak leaves as a vegetable foaming agent as well as the use of microwave energy in heating processes of the raw material powder mixture for manufacturing thermal insulating materials for the building construction. Worldwide, these processes use only conventional heating techniques. The experimental results led to the conclusion that both the use of waste and residues, as well as the unconventional heating technique allow to obtain porous materials with structural homogeneity having apparent densities and thermal conductivities that can decrease up to 0.34 g/cm3, and 0.071 W/m•K respectively. The compressive strength corresponding to the materials with the lowest values of density and thermal conductivity has an acceptable value (1.2 MPa) for the field of application. The specific energy consumption is around 1 kWh/kg, being approximately at the same level with the values of industrial consumptions achieved by conventional techniques.

Introduction

The foaming (expanding) process of the glass waste involves the incorporation in the powder mixture of raw material of foaming agents, which at high temperature (700-1100 ºC) releases a gas in the softened mass of the glass. Being locked inside the material mass by an adequate control of its viscosity, the gas contributes to the formation of the porous structure specific to the cellular glass after cooling the expanded material. The nature of foaming agents used in industrial or experimental processes for manufacturing cellular glass is very diverse. Most often carbon or inorganic carbonaceous products are used (black carbon, coal dust, graphite, etc.), which by oxidation release carbon dioxide (CO_2). Also, calcium or sodium carbonates ($CaCO_3$ or Na_2CO_3), dolomite ($CaMg(CO_3)_2$) and gypsum ($CaSO_4$) are metal salts that provide foaming gases (CO_2 and SO_2, respectively) by decomposition. Silicon carbide (SiC) and silicon nitride (Si_3N_4) are considered very effective foaming agents leading to the production of cellular glasses with uniform microstructures and controlled pore sizes. Also, organic foaming agents containing carbon (sugar, starch, hydrocarbons, organic wastes) are used (Scarinci et al., 2005). Lately, several variants of new foaming agents have been experimentally tested on a small-scale. According to the literature, clam shells (Lunip et al., 2016), egg shells (Fernandes et al., 2013), ceramic lining of used casting molds (Vancea & Lazau, 2014), polishing glass powder organic residue (Attila et al., 2013), "water glass" without other foaming agent (Hesky et al., 2015; Owoeye et al., 2020), propyl gallate ($C10H12O5$) (Qu et al., 2015), liquid solution of polymethylmethacrylate (PMMA) dissolved in dichloromethane ($CH2Cl2$) (Scarinci et al., 2005). Foaming agents of vegetable origin (banana leaves (Arcaro et al., 2016), and yerba mate (da Silva et al., 2016), etc have been successfully tested in the manufacture of cellular glasses.

It should be noted that all industrial or experimental cellular glass manufacturing processes have exclusively used conventional heating techniques (electrical resistances or burning fossil fuels). Although it has been known for about 70 years, the unconventional method of fast and economical microwave heating of solid or liquid materials has been applied only in drying and low temperature heating processes, the best known application being in household in the food preparation. It has been known since 1990 that many types of solid materials (ceramics, organics, polymers, glass, metals, etc) could be efficiently microwave heated. Despite this, the progress in the world is very slow and the state of research is still experimental. A constant concern in the last four years for the use of microwaves in the field of cellular glass production has been manifested on an experimental scale in the Romanian company Daily Sourcing & Research, the results being presented in several international and national journals (Axinte et al., 2019; Paunescu et al., 2017a; Paunescu et al., 2020a).

The originality of this paper consists both in the use of an unconventional technique for the production of cellular glass, and in the testing of a foaming agent also unconventional of vegetable origin (oak leaves).

The oak is a tree specific to geographic areas with temperate to tropical climate, being tall, with strong branches, wide and rich crown, with many relatively large leaves. It is found in Northern Hemisphere in Europe, Asia, America, and North Africa. The oak leaves are poisonous in large amounts to livestock including cattle, horses, sheep, and goats due to the toxin tannic acid, causing kidney damage, and gastroenteritis (Makkar & Singh, 1991).

Methods

Previous own experiments performed in the field of microwave manufacturing process of cellular glass from glass waste have shown that the direct contact of microwaves with glass-based raw material is inadequate due to the very high heating rate initially developed in the core of the material (over 40 ºC/min), which causes serious damage to the material (Paunescu et al., 2017a; Paunescu et al., 2017b). The optimal technical solution adopted by the authors (Figure 1) consists in tempering the effects of the direct microwave-material impact by placing between the wave generating source from the side wall of the oven and the mass of the heated material a ceramic barrier made of a susceptible high microwave material (Axinte et al., 2019; Paunescu et al., 2017a; Paunescu et al., 2017b). A SiC and $Si3N4$ ceramic tube (in the 80/20 mass ratio) with an outer diameter of 125 mm and a wall thickness of 5 mm (Figure 2) was adopted to partially absorb the microwave radiation in the tube wall intensely heating it, allowing its penetration in a lower proportion (Axinte et al., 2019). The optimal size of the tube wall thickness was determined exclusively based on previous experimental results. In this way, the microwave field facilitates the predominantly direct fast glass heating, without affecting its microstructure. A mixed microwave heating (direct and indirect) is performed, proving to be very energy efficient. The research presented in the paper did not use one of the usual types of foaming agents, being tested an agent from a vegetable material (oak leaves), dried, and very finely ground below 100 µm. The carbon existing in the composition of the oak leaves is a very active foaming agent favoring the expansion of the powder glass at temperatures between 750-900 ºC.

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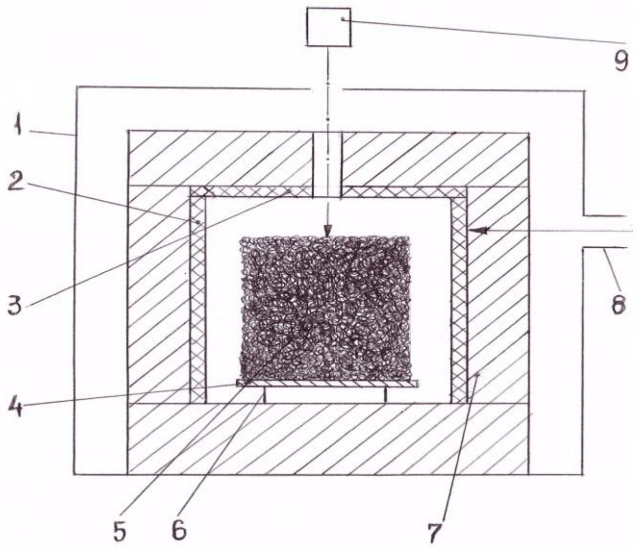

Figure 1. Constructive scheme of the experimental microwave equipment

1 – 0.8-kW-microwave oven; 2 –ceramic tube; 3 – ceramic lid; 4 – metal plate; 5 – pressed powder raw material; 6 – metal support; 7 – ceramic fiber mattress; 8 – waveguide; 9 – radiation pyrometer

Figure 2. Image of the SiC/Si_3N_4 ceramic tube

The materials used in the experiments were colorless, green and amber container glass waste (in equal weight proportions) ground in a ball mill, and sieved below 150 µm as well as oak leaves dried at 110 ºC for 2 hours in a muffle oven, ground in a laboratory electrical device, and sieved at a grain size below 100 µm. The container glass is the most common type of container, and it is a soda-lime glass.

Table 1. Chemical composition of glass waste

Chemical composition	**Container glass waste 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

Other oxides	0.05	0.01	0.05

The oak leaves (collected from freshly fallen leaves from the tree, completely avoiding the dead leaves) were chemically characterized by determining the carbon, hydrogen and nitrogen in their weight composition with an elemental analyzer Perkin-Elmer CHN 2400. Also, the weight contents of ash, volatile materials, total solids and fixed carbon were measured according to the Standard Test Methods ASTM E1755-01 (2020), ASTM E872-82 (2006), and ASTM E1756-08 (2008), respectively. The measurement results are shown in table 2.

Table 2. Dry weight composition of oak leaves

Volatile solids wt.%	**Fixed carbon wt.%**	**Ash wt.%**	**Carbon wt.%**	**Hydrogen wt.%**	**Nitrogen wt.%**
73.9	13.5	6.1	41.3	5.9	0.6

The high proportion of volatile solids (73.9 wt.%) indicates the presence of organic matter (lignocellulosic and carbon fractions, as components of biomass). By heating to 80-140 ºC, the volatiles are divided in gases such as light hydrocarbons (methane, ethane, propane and butane), carbon monoxide, carbon dioxide and tar, which up to almost 600 ºC decompose in proportion of over 90% into elementary components. The temperature and heating rate favor the efficiency of this process (Demibas, 2004). According to Magalhães et al. (2013), by thermal processing of oak leaves at temperatures between 80-140 ºC for 2-18 hours, it was experimentally found that higher temperatures, and longer durations favor higher purities of fractionated lignocellulosic materials.

The experiments were carried out in the company Daily Sourcing & Research Bucharest (Romania) on a domestic 0.8 kW-microwave oven adapted for operation at a much higher temperature than in the household (over 1000 ºC) used constantly in the latest applied research, and described in several scientific papers (Axinte et al., 2019; Paunescu et al., 2020a; Paunescu et al., 2020b). The temperature control of the heating and foaming process was performed with a radiation pyrometer axially placed above the oven at about 400 mm. The thermal protection of the powder material subjected to the heat treatment was obtained with ceramic fiber mattresses resistant to temperatures up to 1600 ºC.

The experimental method was based on the adoption of four experimental variants including different proportions of glass waste (between 45-75 wt.%), and oak leaves (25-55 wt.%) in the starting powder mixture. Using previous experimental data obtained in manufacturing processes that involved also a vegetable material (banana leaves) as a foaming agent (Arcaro et al., 2016), the values of the weight proportion of the oak leaves were adopted much higher than those of the mineral agents commonly used in the world. The dry powder mixture was wetted with 12 wt.% water to facilitate the cold pressing of the mixture. Table 3 presents the distribution of the components of the raw material mixture in the four adopted variahnts.

Table 3. Experimental variants for producing the cellular glass

Variant	**Glass waste wt.%**	**Oak leaves wt.%**	**Water addition wt.%**
1	75.0	25.0	12.0
2	65.0	35.0	12.0
3	55.0	45.0	12.0
4	45.0	55.0	12.0

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Result and Discussion

The amount of dry raw material used in each of the four variants was kept at 580 g, the glass waste amount varying between 261-435 g, and the amount of oak leaves having values between 145-319 g.

Unlike the conventional foaming processes in which the thermal regimes are pre-established, in the case of microwave heating at a constant wave emission power, the thermal process is considered completed when the foamed material has significantly increased its volume by climbing into the working enclosure comparative to the initial level. At this point, the temperature measured on the surface of the sample head has a short stabilization tendency followed by the beginning of a slow decreasing. These changes in the heating graph occur rapidly, and in order to obtain a cellular glass with optimal microstructure, the power supply of the source of electromagnetic waves must be stopped. It should be noted that the microwave heating system has no thermal inertia because the heating is selective targeting only the sample without the oven components (walls, vault, hearth, etc.) as in the case of conventional heating. Also, the direct microwave heating in which the field of electromagnetic waves comes in direct contact with the surface of the material is initiated in its core where the highest temperature develops, the heat being propagated through the mass of the material from the inside to the peripheral areas, radically opposite to the conventional heat transfer mechanisms. The above considerations were the basis for the development of the experimental foaming process of glass waste with oak leaves as a foaming agent in the four variants adopted. The main functional parameters including the foaming temperature, the process duration, the heating and cooling rates, the index of volume growth and the specific energy consumption are shown in table 4.

Table 4. Functional parameters of the foaming process.

Parameter	**Variant 1**	**Variant 2**	**Variant 3**	**Variant 4**
Dry/wet raw material amount, g	580/ 649.6	580/ 649.6	580/649.6	580/ 649.6
Foaming temperature, ºC	820	824	831	838
Heating duration, min	45	48	51	54
Average rate, ºC/min				
-heating	17.8	16.9	16.0	15.1
-cooling	5.6	5.9	5.5	5.7
Cellular glass amount, g	551	550	554	552
Index of volume growth	1.20	1.36	1.41	1.75
Specific consumption of electricity, kWh/kg	0.95	1.02	1.07	1.14

According to the data in table 4, the temperature of the foaming process, and implicitly, the duration of the process were influenced by the weight ratio of foaming agent / glass waste, which varied between 0.33 (variant 1) and 1.22 (variant 4). Therefore, higher proportions of oak leaves as a foaming agent required higher temperatures and durations for the expansion of the raw material to occur. The index of volume growth reached a maximum value of 1.75 at 838 ºC corresponding to variant 4, while in the variant 1 (with the lowest proportion of foaming agent) the value of the growing index was of only 1.20 at 820 ºC. Obviously, in terms of energy, the variants where the oak leaves ratio was lower had energy consumption lower reaching 0.95 kWh/kg (variant 1) and 1.02 kWh/kg (variant 2). However, the results of the physical, mechanical and microstructural characterization of the samples were very important for a complete evaluation of the experimental variants.

In order to determine the physical, mechanical and microstructural characteristics of the samples obtained by thermal foaming of glass waste with oak leaves in the microwave field,

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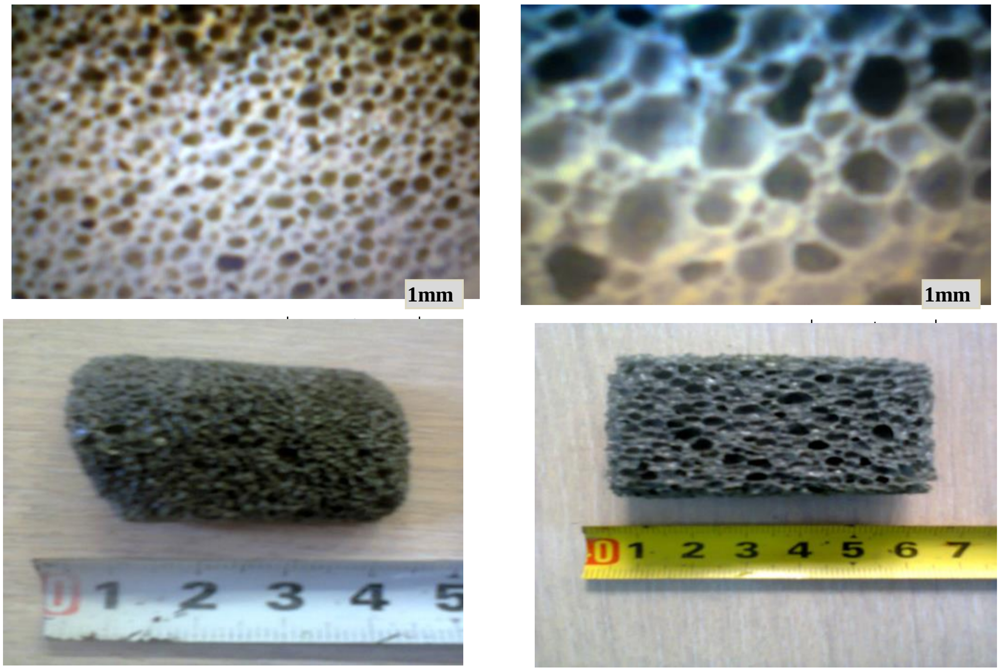
common analysis techniques were used. A Stable Micro Systems TA.XT Plus Texture Analyzer with a loading rate of 1 mm/min was used to determine the compressive strength of the cellular glass samples. The thermal conductivity was measured by means of the guarded-comparative-longitudinal heat flow, according to ASTM E1225-04 Standard Test Method for thermal conductivity of solids. The porous microstructure appearance of the cellular glass samples was identified with a Smartphone Digital Microscope. The apparent density was measured by the gravimetric method (Manual, 1999), and the porosity was determined by the method of comparing the density of the compact material (after melting and cooling) and the apparent density of the porous material (Anovitz & Cole, 2005). The water absorption was measured by the method of the sample immersion in water. The main characteristics of the cellular glass sample are shown in table 5.

Table 5. Physical, mechanical, and microstructural characteristics of the cellular glass samples

Var.	**Apparent density g/cm^3**	**Porosity %**	**Thermal conductivity W/m·K**	**Compressive strength MPa**	**Water absorption %**	**Pore size mm**
1	0.84	58.0	0.178	3.4	0.9	0.1-0.25
2	0.64	68.1	0.123	2.9	1.4	0.2-0.6
3	0.49	75.5	0.095	2.0	1.3	0.3-0.65
4	0.34	83.0	0.071	1.2	1.6	0.4-0.75

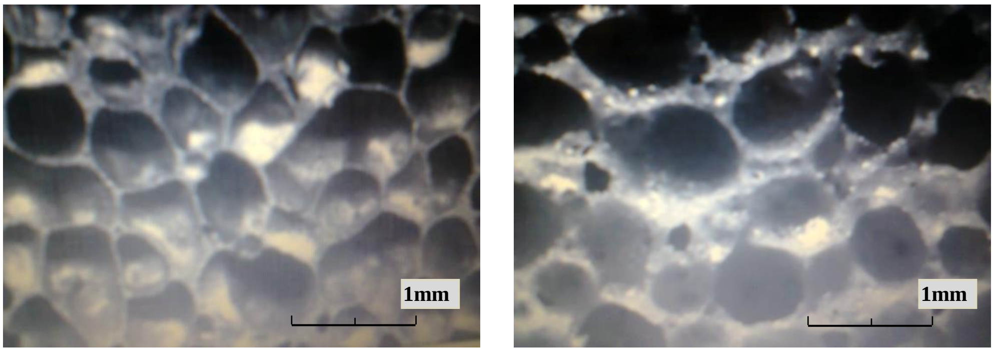
Pictures of the section of cellular glass samples manufactured in the four experimental variants are shown in Figure 3. Microstructural images of these samples to identify the distribution and the size of the pores are presented in Figure 4.



C D

Figure 3. Pictures of sections of the cellular glass samples

a – sample 1, heated at 820 ºC; b – sample 2, heated at 824 ºC;
c – sample 3, heated at 831 ºC; d – sample 4, heated at 838 ºC.



A B

Figure 4. Microstructural images of the cellular glass samples

a – sample 1; b – sample 2; c – sample 3; d – sample 4.

Analyzing the data in Table 5, significant differences are observed between the main physical and mechanical characteristics of the cellular glass samples manufactured in the four variants. Thus, sample 1 obtained with 25 wt.% oak leaves is a dense porous material (with high apparent density of 0.84 g/cm^3, and low porosity of 58%) which gives it a high thermal conductivity of 0.178 W/m·K and a relatively high compressive strength of 3.4 MPa. Increasing the proportion of vegetable foaming agent up to 55 wt.% radically changes the appearance and characteristics of the samples. Sample 4 has a low apparent density for this type of cellular glass (0.34 g/cm^3) its porosity reaching a high value of 83%. The thermal conductivity of the material is low (0.071 W/m·K), suitable for use as thermal insulation material in building construction. Also, the compressive strength decreased up to 1.2 MPa (lowl but acceptable) is typical for thermal insulation materials. The water absorption is within very low limits (between 0.9-1.6%) proving that the material is practically impermeable to water and moisture.

The appearance in section of the four samples (Figure 3) indicates homogeneous microstructures with uniform distribution of the pores. Their dimensions according to Figure 4 and Table 5 have low values from 0.1-0.25 mm (variant 1) to 0.4-0.75 mm (variant 4).

Concluding the results of the physical, mechanical and microstructural characteristics of the four experimental variants, samples 3 and 4 with weight proportions of oak leaves as foaming agent of 45 and 55%, respectively, were adopted as the best for this type of cellular glass. Their characteristics are close to those of the cellular glass made of glass waste, and common foaming agents (such as carbon, calcium carbonate, silicon carbide, etc.) demonstrating the good foaming ability of vegetable agents. Also, the comparison with data from the literature (Arcaro et al., 2016) regarding the use of another type of vegetable foaming agent (banana leaves) shows the similarity between the characteristics of the two types of cellular glass of vegetable origin (porosity of 87.5%, thermal conductivity of 0.06 W/m·K, and compressive strength of 1.17 MPa for an optimal proportion of the foaming agent of 50 wt.%, and the foaming temperature of 850 ºC).

The process of manufacturing the cellular glass from container glass waste, specific to human civilization, using as a foaming agent a natural residue (oak leaves), and a fast and economical unconventional energy source (microwave radiation) is the ideal combination of the need for elimination of a waste with a high increasing generation rate, the manufacture of a material with insulating properties, mainly for the building construction, and the use of a type of unconventional economical and non-polluting energy.

According to the literature (Kharissova et al. 2010), the advantages of using microwaves as a heat generator for glass-based raw materials are: higher heating rate, selective heating, better control on the heating process, low size of the equipment (Rahaman, 2007; Kolberg & Roemer, 2001).

Although silica (SiO_2) and alumina (Al_2O_3), present in high proportions in the glass composition, are not microwave susceptible materials at low temperatures (below 500 ºC), other components of it (Na_2O, K_2O) significantly favor the microwave absorption (Kolberg & Roemer, 2001) and Cr_2O_3, Fe_2O_3, although in small amounts, substantially benefits the microwave susceptibility of glass at room temperature (Jones et al., 2002).

Thus, the soda-lime glass waste existing in large quantities in the world, and used intensively as a raw material in conventional cellular glass manufacturing processes (Paunescu et al., 2017b) was adopted in the experiments described above using the unconventional microwave heating technique.

Typically, information in the literature on the specific energy consumption of cellular glass manufacturing processes is omitted. According to Paunescu et al. (2020b), the company Energocell presented in 2014 its own average industrial consumption of 140 kWh/m^3, i.e. between 0.80-0.93 kWh/kg, for conventional heat treatment processes. The specific consumption of the experimental manufacturing process of cellular glass described in the current paper is slightly higher between 0.95-1.14 kWh/kg), but it should be considered that a low power experimental equipment (0.8 kW) with a discontinuous operation was used, and the foaming agent was less effective compared to the agents commonly used in the world. Also, according to Kharissova et al. (2010) an industrial-scale microwave equipment would have a significantly higher energy efficiency by up to 25% compared to the 0.8 kW-microwave oven. This would mean that the specific consumption achieved in the experimental process could decrease up to 0.71-0.86 kWh/kg in industrial microwave heating condition.

Conclusion

The testing of a vegetable foaming agent (oak leaves) in the cellular glass manufacturing process by microwave irradiation of the powder glass-based raw material was the objective of the research presented in the paper. The microwave equipment on which the tests were performed was a 0.8 kW-oven commonly used in the household for food preparation, adapted to operate at high temperatures. Unlike the conventional technique of using common foaming agents, the testing of a vegetable agent imposed the need to significantly increase its weight proportion in the mixture with glass waste, starting from 25 wt.%, and reaching up to 55 wt.% in four experimental variants. Operating at a heating rate between 15.1-17.8 ºC/min for durations between 45-54 min, the conditions for the release of carbon dioxide in the viscous mass of the glass, and its foaming at temperatures between 820-838 ºC were achieved. The physical, mechanical, and microstructural characteristics of the cellular glass samples confirmed the possibility of obtaining porous materials with a homogeneous microstructure fulfilling the requirements imposed on thermal insulating products applicable in the building construction. The best samples were considered to be those made of 45 wt.% glass waste and 55 wt.% oak leaves (variant 4) as well as 55 wt.% glass waste and 45 wt.% oak leaves (variant 3). Foamed at 838ºC and, 831 ºC respectively, the samples had apparent density of 0.34-0.49 g/cm^3, thermal conductivity of 0.071-0.095 W/m·K, and compressive strength of 1.2-2.0 MPa. The use of microwaves has once again proved to be a very energy efficient solution for solids heating processes, the specific energy consumption being around 1 kWh/kg, approximately at the level of the specific consumption of conventional industrial processes, with the possibility of reducing it by up to 25 % by using microwave equipment on an industrial-scale.

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