Alternative thermal insulation building material manufactured by expanding the glass waste with anthracite under the effect of microwave radiation

Alternativ material de construcție termoizolant fabricat prin spumarea deșeului de sticlă cu antracit sub efectul radiației microundelor

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Rezumat. *Sticlă celulară cu excelente proprietăți termoizolante (densitate de 0,27 g/cm^3 și conductivitate termică de 0,053 W/mK) și rezistență acceptabilă la compresiune (2,7 MPa) a fost produsă din deșeu de sticlă, fosfat disodic (5,7 %) ca agent de stabilizare a spumei și antracit (0,9 %) ca agent carbonic de spumare, prin sinterizare cu microunde la 793 ºC. Antracitul a fost folosit pentru prima oară în cuptoare cu microunde într-un proces original. Eficiența energetică a procesului (0,66 kWh/kg) a fost remarcabilă față de procesele convenționale datorită vitezei de încălzire semnificativ mai înaltă față de vitezele utilizate în procesele convenționale, fără afectarea microstructurii omogene a produsului. Acesta este adecvat utilizării ca material termoizolant in construcții.*

Cuvinte cheie: sticlă celulară, microunde, antracit, agent de spumare, fosfat disodic, material termoizolant.

Abstract. *Cellular glass with excellent thermal insulation properties (density of 0.27 g/cm^{3}and thermal conductivity of 0.053 W/mK) and acceptable compressive strength (2.7 MPa) was produced of glass waste, disodium phosphate, (5.7 %) as foam stabilizing agent and anthracite (0.9 %) as carbonaceous expanding agent by microwave sintering at 793 ºC. Anthracite was used for the first time in microwave ovens in an original process. The process energy efficiency (0.66 kWh/kg) was remarkable compared to the conventional processes due to the heating rate significantly higher than the rates used in conventional processes, without affecting the product homogeneous microstructure. This is suitable for using in building as a thermal insulation material.*

Keywords: cellular glass, microwave, anthracite, expanding agent, disodium phosphate, thermal insulation material.

1. Introduction

The last decades have been marked by a growing concern of the world community for the recycling of waste and industrial by-products. Annual waste generation rates from metal, plastic, glass, textiles, etc. or also coal fly ash, metallurgical slag, oil shale, different sludge and other by-product have excessive dynamics, so their storage in landfills has become insufficient to protect the environment. According to [1], continuing the current trend of waste recycling would be insufficient and the environment would be suffocated by them. Taking 2015 as a benchmark, the global amount of recycled waste should increase by 2030 from 363 Mt to 740 Mt.

Given that the glass is a material with relatively short life-time, large amounts of waste are annually generated (25.8 Mt in 2007 only in EU countries) [2]. In addition, the different types of glass as a chemical composition make it difficult and costly to sort waste for the reintroduction into the industrial production circuit of the new glass. In the last 3-4 decades, it has been found that recycled glass waste could be an excellent raw naterial for the manufacture of building materials similar to existing ones. The energy consumption for their production (about 500 kJ/kg) is significantly lower compared to the energy consumption required to make the glass (about 1500 kJ/kg) [3].

Several companies (Misapor Switzerland, Pittsburgh Corning, Glapor Werk Mitterteich, Geocell Schaumglas, etc.) focused their production activity in Europa, the United States, and China [4] on manufacturing cellular glasses with variable characteristics, but incorporating remarkable physical, thermal and mechanical properties (light weight, low density, low thermal conductivity, at least acceptable mechanical strength, resistance to fire, moisture, insects, rodents and bacteria aggression, resistance to corrosion and frost, high durability, chemical and physical stability, etc.) [5]. Due to this combination of properties, the cellular glass is especially attractive for the construction sector, being a thermal insulation material with load bearing capacity, usable both inside and outside the building for insulation and as a light filler for building foundations or for road and railways construction, drainages,

sports fields, swimming pools, insulation for underground heating pipelines and storage tanks, etc. [6, 7].

Except the industrial production, worldwide scientific research in the field of making various cellular glass types by improved techniques, use of various raw materials, expanding agents and additives is very active, numerous works being published in the literature.

The main method of foaming the powder mixture based on glass waste applied by all industrial manufacturers is the incorporation of a solid or liquid expanding agent and sintering the mixture at a temperature equivalent to its softening point. The expanding agent must be selected so that at the temperature mentioned above it releases a gas (or a gaseous compound) into the viscous mass of raw material. The gas generates bubbles, which are blocked inside the viscous material and by cooling the bubbles turn into pores (cells) forming the specific structure of the cell glass [5].

Generally, the gaseous products result through decomposition (e.g. calcium carbonate) or redox (e.g. oxidation of carbonaceous materials) reactions [5]. The main carbonaceous materials used as an expanding agent are: coke, graphite, anthracite, carbon black, glycerol, etc. In industrial processes the most commonly used are carbon black (Pittsburgh Corning, Misapor) and glycerol (Glapor).

The advantage of using carbon-based expanding agents is the lower cost and ability to oxidize at lower temperatures (750-850 ºC) compared to other expanding agents. From this point of view, it is considered that this agent type is suitable for the processes of foaming the glass, the obtained porous products having cells with small size. According to [5], the carbonaceous agent is used in weight proportions between 0.2-2 %.

The disadvantage of using carbonaceous materials in ovens that operate with an oxidizing atmosphere is the danger of premature oxidation of carbon particles. To avoid this disadvantage, the coating of fine carbon particles with a protective layer is necessary [8]. According to the literature [9], the optimal solution is an addition to the starting mixture of sodium phosphate (Na_3PO_4) or potassium phosphate (K_3PO_4) as a foam stabilizer. The temperature of the foaming process of the glass waste was reduced with the increase of the alkaline phosphate content.

The carbon black as a carbonaceous foaming agent (in proportion of 1 wt. %) was chosen by the authors of the paper [10] to produce a high mechanical strength cellular glass, using borosilicate glass waste, antimony oxide (Sb_2O_3) between 0-1.2 wt. % and disodium phosphate (Na_2HPO_4) as an additive (6 wt. %). The finely ground mixture was sintered at 750-800 ºC for 30 min in a graphite crucible placed in a conventional oven. Due to the presence of Sb_2O_3 in the glass mass, the viscosity and surface tension of the melt were diminished, also reducing the value of the sintering temperature. Also, the pore size was reduced and the density of the foamed material increased. Higher Sb_2O_3 ratios block the premature oxidation of carbon black during heating, but can lead to structural inhomogeneity. At the optimum sintering temperature of 775 ºC the compressive strength reaches 3.6-4.6 MPa, while at 800 ºC the mechanical strength decreases to 1.8-2 MPa. The samples sintered at 775 ºC had

the density of 0.408 g/cm^3, porosity of 84.6 %, compressive strength of 4.4 MPa and water absorption of 1.6 %.

According to [11], an analysis of the sintering/expanding process of borosilicate glass waste showed that by increasing the heating rate there is a tendency to reduce the pre-oxidation of carbon black, resulting an inhomogeneous distribution of pores in the foam structure. The optimum heating rate was determined experimentally at 8 ºC/min and the grain size of carbon black was considered optimal at 150 μm to obtain uniform pore size.

Charcoal (a lightweight carbon black residue) has been used as a carbonaceous expanding agent in the process of foaming the panel glass of an expired life-time cathode-ray-tube (CRT) [12]. Manganese oxide (MnO_2) between 5.4-7.2 % was added to the starting powder mixture as an oxygen-supplying agent for the release of carbon dioxide (CO_2). The grain size of charcoal and MnO_2 had values between 15-27 μm. The sintering temperature varied between 800-840 ºC. The optimum sample was obtained by sintering at 800 ºC using 1 % charcoal and 7 % MnO_2. The apparent density of the sample was very low (0.13 g/cm^3) as well as the thermal conductivity (0.042 W/mK).

It should be noted that the processes of expanding glass waste with carbonaceous agents both industrial and small-scale mentioned above have been carried out by conventional heating methods. Unlike these, the group of Romanian companies Daily Sourcing & Research and Cosfel Actual Bucharest (including the main authors of the current paper) have developed in the last 4-5 years an experimental program to test the production of cellular glasses exclusively using electromagnetic wave radiation.

According to [13], experiments aimed at foaming borosilicate glass waste included, among others, a carbonaceous expanding agent (activated carbon). The powder mixture included 92.8 % glass waste, 1 % activated carbon, 6.2 % Na_2HPO_4 and 10 % water addition. The sintering/expanding temperature was 820 ºC, the average heating rate being 15.3 ºC/min. The porous product had the following features: apparent density of 0.34 g/cm^3, porosity of 84.5 %, thermal conductivity of 0.055 W/mK, compressive strength of 2.5 MPa and pore size between 1-2.5 mm.

The paper [14] reports the foaming of borosilicate glass waste in a 800 W-microwave oven using in the optimal variant carbon black (1 %) as a carbonaceous foaming agent, Na_2HPO_4 (5.9 %) as a foam stabilizing agent, Sb_2O_3 (0.8 %) as an oxygen supplying agent and water addition (10 %) as a binder. The optimal sintering temperature was 790 ºC and the heating rate was 24.8 ºC/min, unlike the conventional heating techniques with hearing rate around 10 ºC/min [15]. The main characteristics of the cellular product were: 0.34 g/cm^3 apparent density, 84.5 % porosity, 0.06 W/mK thermal conductivity, 2.2 MPa compressive strength and 0.4-0.7 mm pore size. The energy efficiency of the process (0.68 kWh/kg) was below the consumptions level reached in the industrial cellular glass conventionally manufacturing processes.

The current paper keeps the own original method of predominantly direct microwave heat treatment developed in the latest experiments of the Romanian group of companies and presented in the literature [16]. In addition, the originality of this

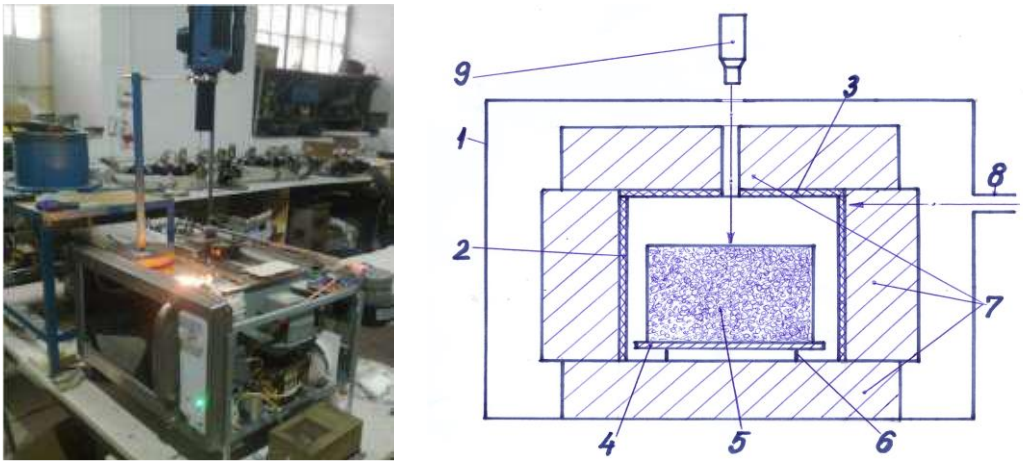
paper is the choice of the type of carbonaceous expanding agent (anthracite) which has not been previously tested in unconventional heating processes.

2. Methods and materials

The first experiments aimed at direct microwave heating of recycled soda-lime glass (commercial glass) powder, i.e. post-consumer drinking bottle and clear flat glass from demolition, showed that the core structure of the glass sample suffers serious damage due to the excessive contact intensity between the microwave field and the glass. The original solution adopted by the authors was to protect the material with a ceramic tube covered with a lid made of a mixture of SiC and Si_3N_4 (both components being excellent microwave susceptible materials) placed between the microwave source and the glass. The ceramic tube with an outer diameter of 125 mm, a height of 100 mm and a wall thickness of 2.5 mm purchased from China proved to be the optimal solution ensuring predominantly direct and partially indirect (by thermal radiation) microwave heating [16].

The experimental equipment shown in Fig. 1a is composed according to the scheme in Fig. 1 b of a microwave oven (1) equipped with a single 800 W-magnetron (8) of the kind used in household adapted for high temperature operation (up to 1200 ^oC), inside which is placed at the oven base a thermal insulation bed of ceramic fiber mattresses (7), on which a metal plate (4) is placed on a metal support (6), which will support the pressed powder mixture (5) prepared from the previously dosed materials. Above the pressed mixture is placed the ceramic tube (2) provided with a lid (3), supported on the thermal insulation bed. The outer surface of the tube and lid are protected with thick layers of ceramic fiber mattresses (7) to avoid the heat loss from the inside to the outside. The control of the heating process is performed with a radiation pyrometer (9) mounted above the oven on a support at about 400 mm, which visualizes the heated material through the holes provided in the upper wall of the oven and the ceramic lid.

The direct microwave heating (predominant in this case) is initiated in the core of the irradiated material, the microwave power being converted into heat. The heat thus generated volumetrically propagates throughout the mass of the material from the inside to the outside. Another characteristic of direct microwave heating is selectivity, which means that only the microwave-sensitive material is heated, not other massive components of the oven. Thus heating is done quickly and efficiently in terms of energy, being completely different compared to the conventional heating [17, 18].



a b

Fig. 1. Experimental microwave equipment
a – overal image of the equipment; b – constructive scheme of the equipment:
1 – microwave oven; 2 – ceramic tube; 3 – ceramic lid; 4 – metal plate;
5 – pressed powder mixture; 6 – metal support; 7 – thermal insulation;
8 – waveguide; 9 – pyrometer.

The foaming process of glass waste using carbonaceous materials as an expanding agent is based on the oxidation reactions of carbon with formation of CO_2 or CO according to the following ecuations.

$$C + O_2 = CO_2 \qquad (1)$$

$$C + 0.5O_2 = CO \qquad (2)$$

The gas bubbles containing CO_2 and/or CO are spread into the viscous mass of glass forming by cooling a fine porosity structure [5]. Only an excessive increasing of the process temperature would create a structure with semi-open pores due to the increase of gas presure inside the bubbles and increasing the glass volume.

According to [5], the glass foaming with carbonaceous materials occurs in the range 800-900 ºC and the gaseous products are strongly influenced by the type of carbonaceous materials and by the glass composition (mainly, the sulfate content). Except the reaction with atmospheric oxygen existing in the oven and in the spaces between the glass particles, carbon can react with some constituents of the glass (alkalis, sulfates and water).

As noted above, the carbonaceous expanding agent adopted in the experiment presented in this paper was anthracite, a hard sort of coal having the highest carbon content (between 86-97 %) [19]. Chemically, anthracite may be considered as a transition stage between common bituminous coal and graphite. Its standard features (maximum limits) are: 15 % moisture, 20 % ash, 10 % volatiles, 73 % fixed carbon and 1 % sulfur. This carbonaceous expanding agent was ground in a ball mill and sived at the grain size of 15 μm.

The basic raw material used in the experiment was recycled post-consumer drinking bottle (green, colorless, and amber in approximately equal ratios). The oxide composition of the three soda-lime glass types [20] is presented in Table 1.

Table 1

Oxide composition of the glass 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
Green	71.8	1.9	11.8	-	1.2	13.1	0.1	0.09	-
Colorless	71.7	1.9	12.0	-	1.0	13.3	-	0.05	-
Amber	71.1	2.0	12.1	0.2	1.1	13.3	0.1	-	0.05

Processing the glass waste used in esperiments consisted in washing, color selection, breaking, grinding and sieving. These operations were performed in the Romanian company Bilmetal Industries SRL Popesti-Leordeni, Ilfov and led to the final granulation below 80 μm.

The composition of the starting material mixture was supplemented with the addition of Na_2HPO_4 as a foam stabilizing agent and distilled water as a binder. As mentioned above, the role of Na_2HPO_4 is to prevent the premature oxidation of anthracite before reaching the foaming temperature by enveloping its fine particles with a protective layer, under the conditions that the process takes place in an oxidizing environment. Na_2HPO_4 with a purity of 95 % was purchased from the market as a Chinese crystalline product and was ground in an electrically operated laboratory device at a grain size below 30 μm.

Three experimental variants of the starting mixture components were adopted including recycled glass waste (between 92.7-93.3 wt. %), Na_2HPO_4 (between 5.7-6.3), anthracite (between 0.9-1.1 wt.%) and water addition (12 wt. %) having the weight proportions shown in Table 2.

Table 2

Composition of the starting mixture

Variant	Recycled glass waste (wt. %)	Na_2HPO_4 (wt. %)	Anthracite (wt. %)	Water addition (wt. %)
1	93.3	5.7	0.9	12.0
2	93.0	6.0	1.0	
3	92.7	6.3	1.1	

Similar methods to characterize the foamed products used in all previous experiments performed in Daily Sourcing & Research and Cosfel Actual companies were also applied in the current work to determine the apparent density by gravimetric method [21], porosity by comparison of apparent density and „true" density [22], compressive strength with a TA.Xtplus Texture Analyzer, thermal conductivity by the guarded-comparative-longitudinal heat flow (ASTM E1225-04), water absorption by

water immersion method (ASTM D570) and microstructural investigation of the cellular glass samples with an ASONA 100X Zoom Smartphone Digital Microscope.

3. Results and discussion

The parameters of the testing process of manufacturing the cellular glass by the unconventional technique presented above are shown in Table 3.

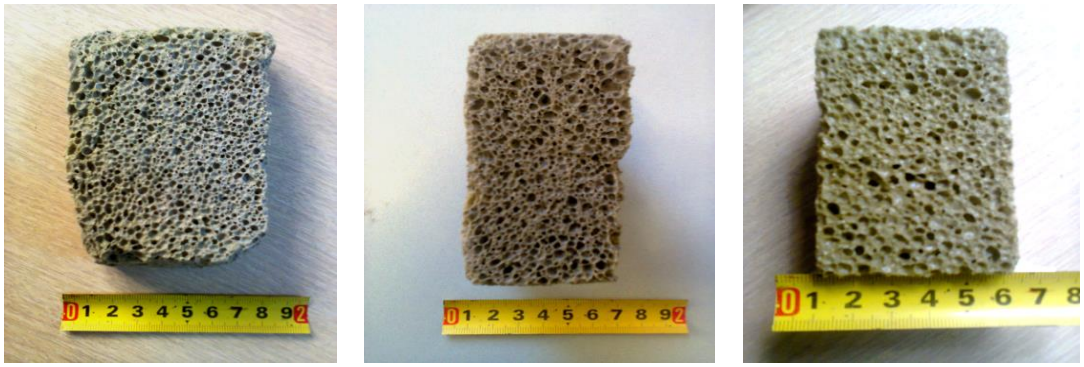
Table 3

Parameters of the manufacturing process of cellular glass

Variant	Dry raw material amount (g)	Process temperature (ºC)	Process time (min)	Average rate (ºC/min)		Cellular glass amount (g)	Specific energy consumption (kWh/kg)
				Heating	Cooling		
1	470	793	31	24.9	5.2	486	0.66
2	470	801	33	23.7	5.3	485	0.69
3	470	810	35	22.6	5.3	487	0.75

The dry raw material amount was kept constant at 470 g for the three experimental variants. As a characteristic of the foaming processes of glass waste which use carbonaceous expanding agents, the sintering/foaming process temperature had low values (between 793-810 ºC). The microwave heating rate (between 22.6-24.9 ºC/min) significantly exceeded the usual values of this parameter in conventional processes (around 10 ºC/min) and contributed to achieving very economical specific energy consumption (0.66-0.75 kWh/kg) without influencing the cellular glass quality.

The cross section of the cellular glass samples obtained after the heat treatment is presented in Fig. 2.



a b c

Fig. 2. Cross section of the cellular glass samples
a – variant 1; b – variant 2; c – variant 3.

According to the images in Fig. 2, the fineness of the porosity of samples decreases from variant 1 made at 793 ºC with 5.7 % Na_2HPO_4 and 0.9 % anthracite to variant 3 made at 810 ºC with 6.3 % Na_2HPO_4 and 1.1 % anthracite. Therefore, a

slightly higher proportion of anthracite as an expanding agent increases the temperature of the foaming process and leads to more coarse macrostructures.

Table 4 presents the main physical, thermal, mechanical and microstructural characteristics of the cellular glass samples determined accordind to the methods of analysis mentioned above.

Table 4

Characteristics of cellular glass samples

Variant	Apparent density (g/cm^3)	Porosity (%)	Thermal conductivity (W/mK)	Compressive strength (MPa)	Water absorption (vol. %)	Pore size (mm)
1	0.27	87.1	0.053	2.7	1.6	0.1-0.5
2	0.25	88.1	0.049	2.2	1.4	0.2-0.8
3	0.22	89.5	0.046	1.8	1.4	0.3-1.0

The data in Table 4 show that the unconventional method of predominantly direct microwave heating using anthracite as an expanding agent favors obtaining cellular glasses with good thermal insulation properties (apparent density between 0.22-0.27 g/cm^3, thermal conductivity between 0.046-0.053 W/mK and porosity between 87.1-89.5 %) and at the same time with sufficiently high compressive strength (1.8-2.7 MPa). The water absorption is low with values below 1.6 vol. %. These characteristics indicate that the cellular glass manufactured under the conditions presented in the paper is suitable to be used as thermal insulation material in building, being able to replace the existing materials.

Examination of the microstructural characteristics of the cellular glass samples (Fig. 3) indicates that the best dimensional uniformity of the pores is achieved in the case of variant 1 (pore size between 0.1-0.5 mm), the microstructural homogeneity decreasing in the case of variants 2 and 3. The sample corresponding to variant 3 tends to have a more coarse distribution of pores, which are in the range of 0.3-1.0 mm.

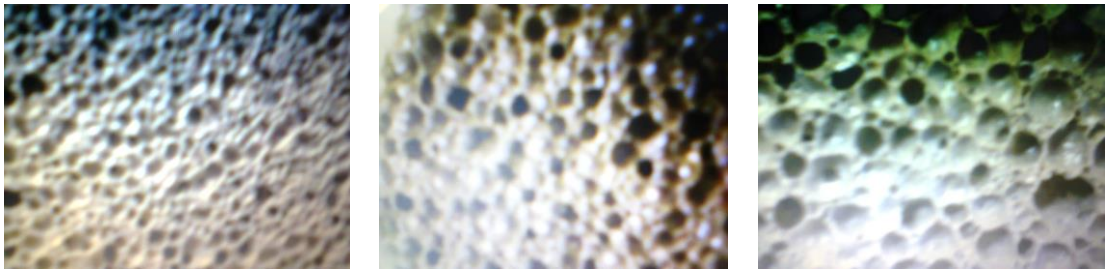


Fig. 3. Microstructural configuration of the cellular glass samples
a – variant 1; b – variant 2; c – variant 3.

Centralized analysis of information on microwave field cell glass manufacturing with the use of anthracite as a carbonaceous expanding agent including physical, thermal, mechanical, microstructural characteristics and functional process

parameters (temperature, heating rate and specific energy consumption) led to establishing variant 1 as optimal. The cellular glass corresponding to this variant sintered at 793 ºC with heating rate of 24.9 ºC/min reached a minimum value of energy consumption (0.66 kWh/kg) being a very economical process compared to the other two experimental variants, but also to the consumption achieved in conventional industrial processes (0.75-1.15 kWh/kg [23]). As a peculiarity of the unconventional foaming process, it was observed that, despite the significantly higher heating rate compared to conventional processes, the microstructural characteristics of the porous product are not affected.

The comparison of the characteristics of the foamed products obtained in this experiment with those previously made with different carbonaceous expanding agents (carbon black, charcoal, activated carbon) showed generally a good similarity, especially in the case of applying the unconventional method [13, 14]. By using the anthracite, adopted in the experiment presented in the current paper, a cellular glass with apparent density (0.27 g/cm^3) and thermal conductivity (0.053 W/mK) was produced, even slightly lower than the results of applying the unconventional technique mentioned above, thus increasing the thermal insulation properties of the final product. In addition, the specific energy consumption was kept at a very low level.

4. Conclusions

The aim of the research that formed the basis of this paper was to test anthracite as a carbonaceous expanding agent in a process of manufacturing cellular glass from recycled glass waste (post-consumer drinking bottle) by microwave heating using the own original heating method predominantly direct. This method has already been successfully applied by authors in several previous experiments and presented in the literature. The main originality of the paper is the testing for the first time in the world of foaming glass waste with anthracite as an expanding agent embedded in the starting powder mixture, which also contains Na_2HPO_4 as a foam stabilizing agent, under the conditions of applying the microwave irradiation technique. Three experimental variants were tested including glass waste (92.7-93.3 %), Na_2HPO_4 (5.7-6.3 %), anthracite (0.9-1.1 %) and water addition (12 %) as a binder. The powder mixture was microwave sintered at 793-810 ºC using heating rates between 22.6-24.9 ºC/min, significantly higher than the rates used in conventional processes. The very low level of specific energy consumption (0.66-0.75 kWh/kg) is remarkable. Heating rates did not affect the physical, thermal, mechanical and microstructural characteristics of the products. The values of these features were: apparent density between 0.22-0.27 g/cm^3, thermal conductivity between 0.046-0.053 W/mK, porosity between 87.1-89.5 %), compressive strength between 1.8-2.7 MPa, water absorption below 1.6 vol. % and pore size below 1 mm. All variants had properties suitable for use as alternative thermal insulation building material, but variant 1 which used 0.9 % anthracite, 5.7 % Na_2HPO_4 and sintering temperature 793 ºC was chosen as the optimal variant. It had

the highest apparent density of 0.27 g/cm^3, but the other characteristics were excellent (thermal conductivity of 0.053 W/mK and compressive strength of 2.7 MPa). Very fine porosity with pore size between 0.1-0.5 mm represented the appearance of the cross section of this foam sample.

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