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Unconventionally Made-Cellular Glass Aggregate

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Abstract

Improving the original manufacturing process in microwave field of a cellular glass aggregate using a recipe containing colored consumed drinking bottle, calcium carbonate ($CaCO_3$) as an expanding agent, sodium borate (borax) as a fluxing agent and sodium silicate (Na_2SiO_3) as a binder is shown in the work. The main adopted technological measures were the advanced mechanical processing of residual glass at a grain dimension below 100 μm and especially the use of a high electromagnetic wave susceptible ceramic tube with a wall thickness reduced from 3.5 to 2.5 mm for the protection of the pressed glass-based mixture against the aggressive effect of microwave field and, in the same time, to achieve a preponderantly direct heating with electromagnetic waves. Of the tested variants, a recipe with 1.6 % calcium carbonate, 6 % borax, 8 % sodium silicate and the rest residual glass was determined to be optimal. The cellular glass aggregate had the bulk density of 0.22 g/cm^3, heat conductivity of 0.079 W/m·K and compression strength of 5.9 MPa. The specific consumption of energy was very low (0.71 kWh/kg) below the range of reported values of the industrial processes consumption (between 0.74-1.15 kWh/kg).

Introduction

The cellular glass aggregate is one of the main assortments of cellular glass manufactured in the last 3-4 decades from recycled residual glass. This type of residue, predominantly from consumed drinking bottle and residual window glass resulting from demolition or modernization of existing buildings is found in the environment in very large amounts and its annual generation rate is excessively high. In general, two main types of cellular glass are commonly industrially made: blocks and boards for heat insulation of inner and outer walls of buildings and cellular glass aggregate for a wide variety of heat insulation of buildings from under floor insulation to filling the roof gardens and filling the foundation's infrastructure.

Combining very low density and low heat conductivity specific to cellular ceramics with compression strength, physical and chemical stability, non-flammability, water impermeability, resistance to insects, rodents, acids and bacteria, durability, etc. specific to glass, the cellular glass is a building material that successfully competes with similar existing materials (Hibbert, 2016).

The cellular glass aggregate is an excellent load-bearing for heat insulation and a lightweight aggregate as filler. The application field of cellular glass aggregate is very wide, also including roadway and railway construction, drainage, bridge abutments, airport runways, underground

heating pipes and storage containers, sports fields and others. (Cellular glass gravel, 2016; Geocell foam glass, 2017; MISAPOR, 2021; Ghafari et al., 2018).

Several European industrial manufacturers from Austria, Switzerland, Germany and the Nordic countries (Norway, Finland, Sweden) currently produce cellular glass aggregate. In the Nordic countries the production of cellular glass aggregate is focused on road construction materials, which can be explained by the special requirements they are subjected to due to extreme climatic conditions (mainly the freeze-thaw cycle) (Cosmulescu et al., 2020).

The basic method of manufacturing the cellular glass gravel is similar to that of producing any cellular glass by heat treatment at high temperature of a powder mixture composed of recycled glass waste, a foaming agent and possible other mineral additives to facilitate the foaming process. This process is generated by the thermal release of a gas produced by the foaming agent in the viscous glass mass, softened at a typical temperature. The gas forms bubbles that are trapped in this viscous medium and generate a cellular structure after the material cooling (Scarinci et al., 2005). At the manufacture of cellular glass gravel, the aim is to obtain denser porous products with significantly higher mechanical strength. Thus, certain additives that favor excessive foaming are avoided. On the other hand, the cooling of the foamed material is faster, intentionally allowing the cracking of the compact foam mass for easy detachment of irregular lumps with dimensions up to 60-70 mm which constitute the cellular glass aggregate.

All manufacturing recipes of industrial producers use recycled residual glass (mainly consumed drinking bottle and possibly residual window glass). The nature of the expanding agent greatly differs being gypsum ($CaSO_4$), limestone ($CaCO_3$) or silicon carbide (SiC) in the case of Misapor plants, glycerol ($C_3H_8O_3$) combined with sodium silicate (Na_2SiO_3) also called “water glass” in the case of Glapor plants. Geocell Company does not specify the nature of its expanding agent and Glamaco Company, which is an important designer and manufacturer of industrial equipment for manufacture of cellular glass aggregate, recommends SiC, manganese oxide (MnO), glycerol, Na_2SiO_3, coal powder, $CaCO_3$, $CaSO_4$ as expanding agents. One of Glamaco's recommended recipes contains the combined use of glycerol (liquid agent), $CaCO_3$ (solid agent) and Na_2SiO_3 (aqueous solution) (Cosmulescu et al., 2020).

According to data from the literature (Hibbert, 2016; Cellular glass gravel, 2016; Geocell foam glass, 2017; MISAPOR, 2021; Ghafari et al., 2018; Cosmulescu et al., 2020), the features of cellular glass aggregates are: bulk density between 0.13-0.21 g/cm^3, density of compact material between 0.21-0.25 g/cm^3, porosity around 80 %, heat conductivity between 0.06-0.08 W/m·K (0.08-0.13 W/m·K for the cellular glass aggregate made for road construction-Foamit Company), compression strength between 4.9-6.0 MPa. The values of the characteristics corresponding to cellular glass aggregate manufactured for use especially in road construction are at the upper limits of the ranges mentioned above.

The industrial manufacturing processes of cellular glass aggregate take place in tunnel ovens with conveyor belt whose energy supply is based on conventional sources (consumption of electricity or heat energy by burning a gaseous fuel). The energy consumption of these processes is sporadically reported. According to data provided by Energocell (Energocell, 2016), one of the industrial producers of cellular glass aggregate, the average specific consumption is 140 kWh/m^3-cellular product (i.e. about 0.74-1.15 kWh/kg) being considered to be a very low energy consumption compared to that of the expansion process of polystyrene (an usual heat insulation material) of over 1500 kWh/m^3.

The energy aspects of the cellular glass manufacturing processes, including also the cellular glass aggregate, were a concern of the research team in Daily Sourcing & Research (Romanian company). In recent years, numerous tests have been carried out in the

experimental base, the manufacture of cellular glasses being performed using the unconventional electromagnetic wave heating technique. Approximately similar to the products conventionally made in industry, the experimentally manufactured materials in the Romanian company had very economical energy consumption. Numerous articles including these results have been published in Romanian and international literature.

The electromagnetic waves, which also include microwaves, have been discovered since the mid-20th century. Their ability to efficiently heat solids and liquids has been industrially applied mainly in drying or low temperature heating technologies. In the last 30 years it has been experimentally discovery that numerous materials (including also the glass) are adequate for efficient heating with electromagnetic waves (Kharissova et al., 2010). However, the industrial application of this advanced heating method of solids to higher temperatures is delayed and the research in the world is still in different testing stages. In these conditions, the Romanian company started a small-scale testing program of cellular glass manufacturing by applying own techniques of electromagnetic wave irradiation of recycled residual glass.

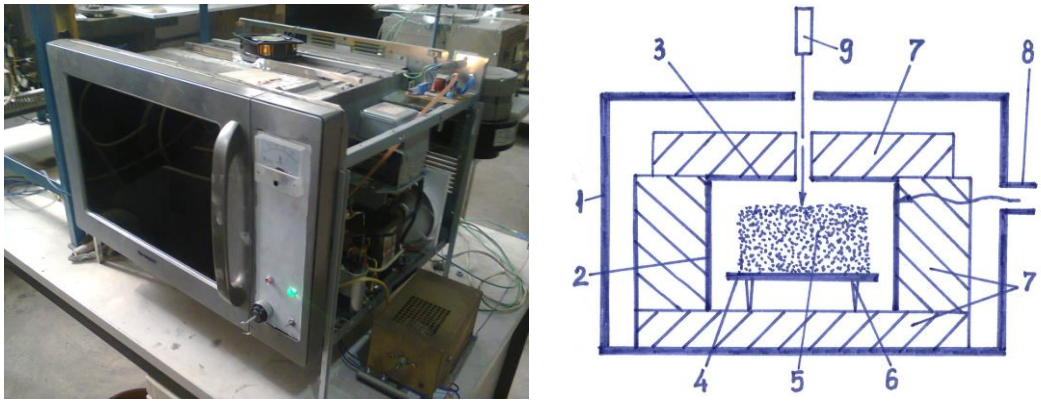
The paper (Cosmulescu et al., 2020) is an eloquent synthesis of the Romanian authors' results in the domain of unconventional manufacture of cellular glass aggregate. Manufacturing recipes composed of four material combinations in various weight proportions usually used in industrial production were tested under the conditions of application the own electromagnetic wave heating technique: colored consumed drinking bottle (green and amber) as the main material, $CaCO_3$ as an expanding agent, borax as a fluxing agent and an aqueous solution of Na_2SiO_3 as a binder (1), colorless residual window glass, glycerol as a liquid expanding agent associated with Na_2SiO_3 and water (2), container glass waste (colorless, green and amber), glycerol, Na_2SiO_3 and water (3) and consumed drinking bottle (colorless, green and amber), SiC as an expanding agent and added water as a binder (4). Analyzing the optimal experimental results achieved in the four recipe groups, it was concluded that the use of glycerol as a liquid carbonic expanding agent and consumed drinking bottle as raw material (3) leads to the best values of product characteristics: apparent specific gravity of 0.24 g/cm^3, porosity of 89.1%, heat conductivity of 0.063 W/m·K, compression strength of 5.9 MPa and pore dimension between 0.3-0.6 mm. It must be mentioned that due to the small number of cellular glass aggregate lumps manufactured, the bulk density could not be determined (as the density of this type of product is reported in the literature), but its value would be below 0.20 g/cm^3. All the features of cellular glass aggregate obtained by the experimental technique of electromagnetic wave heating correspond in terms of quality to the characteristics of industrially made materials. According to Cosmulescu et al., 2020, the use of residual window glass as main material (2) has little influence on the quality of the final product by slightly decreasing the compression strength to 5.3 MPa and increasing the pore dimension to 0.5-0.9 mm. The use of SiC as a solid expanding agent (4) increases the value of apparent specific gravity (0.35 g/cm^3), but also significantly increases the compression strength value (7.5 MPa). A less favorable situation was obtained in the manufacture of cellular glass aggregate by use colored consumed drinking bottle, $CaCO_3$, borax and Na_2SiO_3 (1). The apparent specific gravity of glass aggregate lumps had quite high values (0.62 g/cm^3), a heat conductivity approximately within acceptable limits (0.087 W/m·K), a high compression strength (7.4 MPa) and pore dimension between 1.0-1.6 mm. The main disadvantage of this product is the relatively high value of apparent specific gravity.

The improvement of the cellular glass aggregate manufacturing process using colored consumed drinking bottle, $CaCO_3$, borax and Na_2SiO_3 was researched later and the results are presented below.

Methods

The technological conditions of this making process of cellular glass aggregate have been presented in detail in the work (Paunescu et al., 2020a). The main cause of relatively high value of the apparent specific gravity of the expanded product was considered the insufficient mechanical processing degree of residual glass, which provided raw material with grain size below 250 μm. Currently, dimensions below 100 μm are used, the operations of breaking, grinding in the ball mill and sieving being carried out in the Bilmetal Industries Company Popesti Leordeni-Ilfov (Romania). A second technological novelty was the replacement of the ceramic tube made of SiC and Si_3N_4 (very high electromagnetic wave susceptible material) having a wall thickness of 3.5 mm with a similar ceramic tube with a wall thickness of only 2.5 mm. The ceramic tube was purchased from China. This constructive modification was experimentally verified, being adopted as the optimal solution for the ideal distribution of wave flows between the preponderant penetration of the tube wall with direct contact on the heated surface material (direct heating) and partial flow absorption into the mass wall of the tube, the conversion into heat and its transfer to the powder material mainly by the radiation process (indirect heating).

Excepting the change of the wall dimension of ceramic tube to protect the glass-based pressed mixture against too active irradiation process, the rest of the constructive components of the experimental equipment (Figure 1) used in all the tests performed lately (Paunescu et al., 2020b) have been kept unchanged.



A B

Figure 1. The experimental electromagnetic wave plant

A – 0.8 kW-electromagnetic wave oven; B – principle scheme of the experimental plant: 1- adapted oven; 2 – cylindrical ceramic tube; 3 – lid; 4 – sheet metal plate; 5 – pressed glass mixture; 6 – support; 7 – ceramic heat insulation; 8 – electromagnetic wave source; 9 – pyrometer.

The process of heating the pressed glass powder took place in a typical domestic microwave oven for food preparation, adapted to temperatures around 1000 ºC (Figure 1A). The peculiarity of the direct irradiation process is the beginning of heating in the core of the irradiated material. The microwave power is converted into heat and the highest temperature is reached in the middle of material. The heat spreads in entire volume (volumetrically) of material from inside to outside (Kitchen et al., 2014). Also, the irradiation heating process is selective (Kitchen et al., 2014), being activated only the materials with microwave susceptibility. Thus, it is necessary a high heat protection of the cylindrical tube and the lid (using ceramic fibers with heat resistance up to 1200 ºC) so that the unprotected metal walls of the oven do not overheat above 60-70 ºC in case of a maximum thermal stress.

According to the principle scheme of the plant shown in Figure 1B, the material previously pressed into a mold is freely stored on a sheet metal plate (4) positioned about 20 mm above the bed of ceramic fiber mattresses (7) at the base of the oven (1) with a metal support (6). The material is protected with a high microwave susceptible cylindrical tube (2) provided with a ceramic lid (3) of the same material. Thick ceramic fiber mattresses (7) cover the tube and the lid. A radiation pyrometer (9) mounted above the oven at about 400 mm ensures the control of the temperature of the irradiated material (5), visualizing its upper surface through holes of 30 mm provided in the superior oven wall, lid and the heat insulation layer of it.

In principle, the expanding agent ($CaCO_3$) included in the powder mixture of raw material releases carbon dioxide (CO_2) by the decomposition reaction (1) that occurs at temperatures above 750 ºC (Karunadasa et al., 2019; Koizumi et al., 2011). In accordance with Scarinci et al., 2005, the temperature range at which the common commercial residual glass (soda-lime glass) is expanded using $CaCO_3$ is 800-900 ºC.

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

As in the experiment presented in the paper (Paunescu et al., 2020a), the residual glass was composed of consumed drinking bottle (green and amber). The oxide composition of the two types of residual glass is shown in Table 1.

Table 1. Oxide composition of the residual 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	Other oxides
Green	71.7	1.8	11.7	-	1.1	13.2	0.1	0.09	-	0.31
Amber	71.1	2.1	12.0	0.2	1.0	13.3	0.1	-	0.05	0.15

As stated above, the grain dimension of the residual glass used in this experiment was below 100 μm and the grain dimension of the expanding agent ($CaCO_3$) was less than 40 μm as commercially purchased.

The commercial borax (sodium borate) was used as a fluxing agent in the raw material mixture. The important content in Na_2O (30.8 %) (Borax, 2016) that is the most commonly used fluxes in ceramics, determined the borax role. Its initial granulation was reduced to values less than 130 μm by grinding in an electrical device and sieving. Due to the relatively high boron content of borax (13.8 %) (Miller, 1964), the mechanical strength of cellular glass aggregate is significantly increased.

The liquid solution (38 %) of sodium silicate (or "water glass") obtained from commercial source was used as a hardening material (binder), with major implication to increasing the mechanical strength of the expanded product (Ayadi et al., 2010).

Generally, the characterization of cellular glass aggregate used in the current paper was similar with those previously applied to characterize common cellular glasses. Thus, the gravimetric method (Manual, 1999) was applied to determine the apparent specific gravity. The bulk density was measured by the method of weighing a batch of undisturbed solid lumps completely introduced into a measured volume vessel (Scorgins, 2015; Bulk density, 2020). The porosity was measured by the comparison method of porous material density and the compact material "true" density (Anovitz & Cole, 2005) and the compression strength was measured with an analyzer TA.XTplus Texture type. The guarded-comparative-longitudinal heat flow method (ASTM E1225-04) was applied to determine the heat conductivity (Bianchi-Janetti et al., 2015). The water absorption of the cellular glass aggregate was measured by the method of its water immersion (ASTM D570) and the microstructural investigation of the porous products was carried out with a Smartphone Digital Microscope ASONA 100X Zoom type.

The experimentation of cellular glass aggregate manufacturing process using colored (green and amber in equal ratios) residual drinking bottle, $CaCO_3$, borax and Na_2SiO_3 was performed according to the data in Table 2.

Table 2. Composition of the experimental recipes

Composition	Recipe 1	Recipe 2	Recipe 3	Recipe 4
Residual glass, wt. %	85.4	84.4	83.4	82.4
$CaCO_3$, wt. %	1.6	1.6	1.6	1.6
Borax, wt. %	5.0	6.0	7.0	8.0
Na_2SiO_3, wt. %	8.0	8.0	8.0	8.0

Results and Discussion

The amount of raw materials prepared for this experiment was adopted at 570 g. With a density of about 1.75 g/cm^3, the dimensions of the cylindrical shape of hand-pressed raw material (less than 2.5 MPa) were: diameter of 8.5 cm and height of 5.7 cm.

The main functional data of the experimental making process in the 0.8 kW-irradiation heating oven are shown in Table 3.

Table 3. Functional data of the experimental process

Functional data	Recipe 1	Recipe 2	Recipe 3	Recipe 4
Starting material/ cellular glass aggregate amount, g	570/558	570/557	570/558	570/559
Sintering/expanding temperature, ºC	839	837	835	834
Process duration, min	39	38	37	35.5
Average heating speed, ºC/min	21.0	21.5	22.0	22.9
Average cooling speed, ºC/min	6.0	5.9	6.1	6.1
Index of volume increasing	1.60	1.48	1.42	1.35
Specific consumption of energy, kWh/kg	0.73	0.71	0.69	0.66

In accordance with the data in Table 3, the fluxing agent role of borax is highlighted by decreasing the temperature required for the expanding process of residual glass. In recipe 4 (with 8% borax) the process temperature reaches the minimum value of 834 ºC, while in recipe 1 (with only 5% borax) the process temperature reaches the highest value of 839 ºC. By default, the duration of the making process decreases from 39 to 35.5 min by increasing the weight ratio of borax, leading to improving the heat efficiency of the process, whose specific consumption reaches the minimum value of only 0.66 kWh/kg, lower than the minimum value (0.74 kWh/kg) of the industrial heat consumption reported by Energocell (Energocell, 2016), one of the leading European producers of cellular glass aggregate.

The main features of the cellular glass aggregate specimens achieved by microwave irradiation from residual glass, $CaCO_3$, borax and Na_2SiO_3 are presented in Table 4.

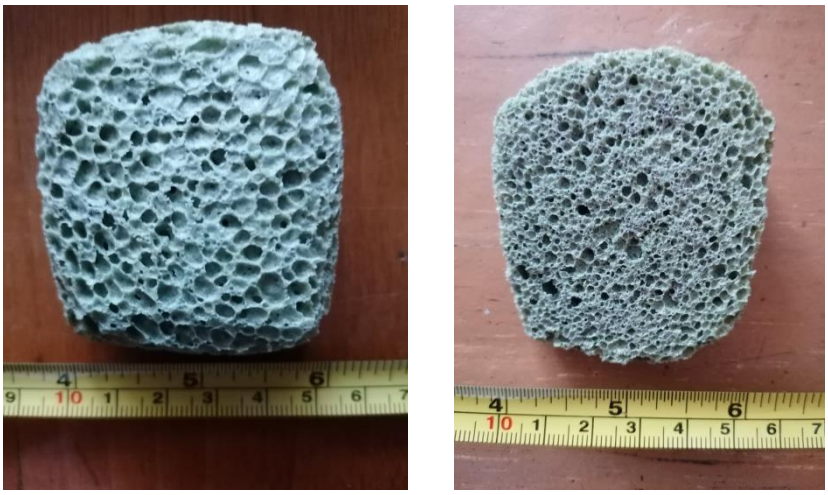
Table 4. Main features of the cellular glass aggregate specimens

Recipe	Bulk density/ apparent specific gravity g/cm^3	Porosity %	Heat conductivity W/m·K	Compression strength MPa	Water absorption vol. %	Cell size mm

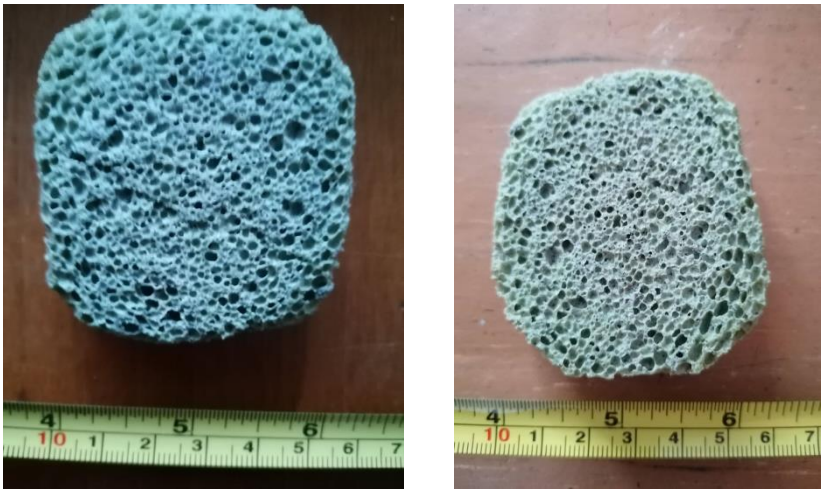
1	0.19/0.32	84.8	0.074	4.0	2.3	1.5-3.5
2	0.22/0.35	83.3	0.079	5.9	2.2	1.0-2.0
3	0.25/0.37	82.4	0.088	7.7	1.9	0.7-1.3
4	0.28/0.40	81.0	0.095	9.2	1.9	0.5-1.0

In order to determine the bulk density of a batch of cellular glass aggregate corresponding to each recipe tested, the experiments were multiplied at least three times to obtain a sufficient batch of specimens. Thus, this physical characteristic of glass aggregate lumps indicated in the literature in this form was determined. In accordance with the data in Table 4, the bulk density had values between 0.19-0.28 g/cm^3, being approximately similar to those of industrially made cellular glass aggregate. The specimens could be cut with the abrasive disc to sufficiently regular shapes, also allowing the calculation of their apparent specific gravity (between 0.32-0.40 g/cm^3). It was observed that variants with higher borax content had higher density values (determined by both methods). Higher values of compression strength (7.7 and 9.2 MPa, respectively), above the level of mechanical strength obtained in industrial making (maximum 6 MPa) correspond to the denser products (recipes 3 and 4). The porosity of the experimentally made materials had high values (81.0-84.8%), its values being more favored by the lower borax ratio in the starting mixture. The lower limits of the range of porosity values correspond to products with higher boron content. The cell size was generally uniform for each experimental recipe (from 1.5-3.5 mm in recipe 1 to 0.5-1.0 mm in recipe 4). The water absorption of the expanded products was low considering that the maximum allowed limit for this type of cellular glass is 10 vol. % (Cellular glass gravel, 2016).

Cross section of the cellular glass aggregate lumps obtained by making process using the electromagnetic wave irradiation are presented in Figure 2 and the microstructural configuration of the lumps are shown in Figure 3.



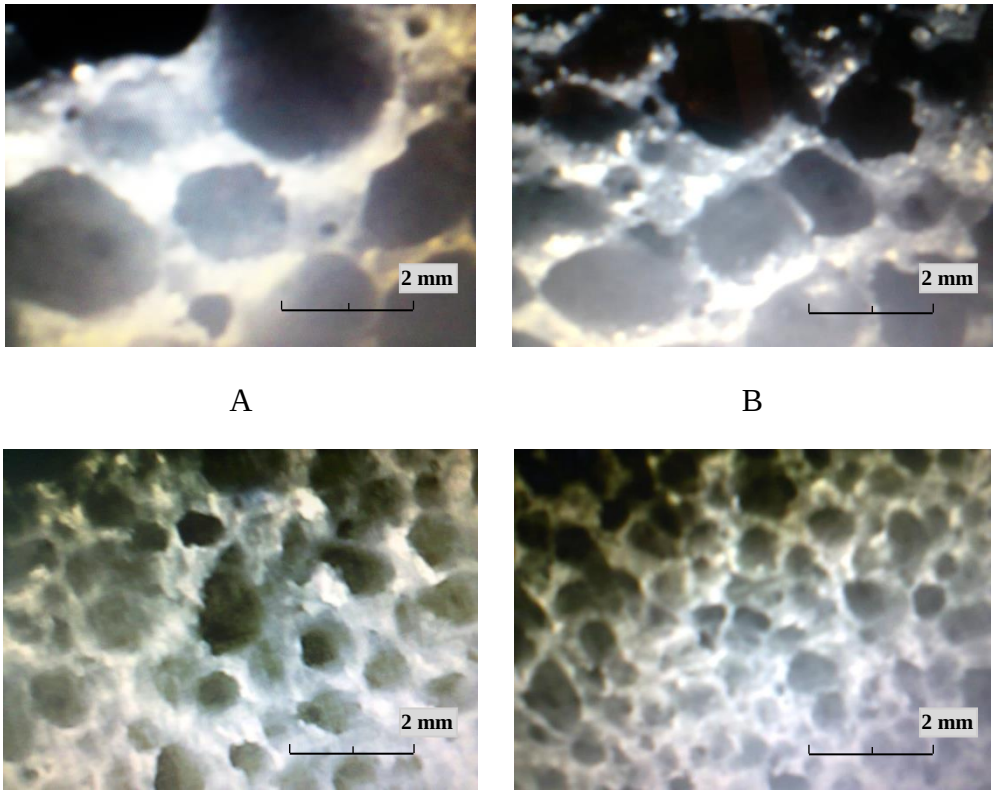
A B



C D

Figure 2. Cross section of the cellular glass aggregate lumps

A – recipe 1; B – recipe 2; C – recipe 3; D – recipe 4.



A B

C D

Figure 3. Microstructural configuration of the lumps

A – recipe 1; B – recipe 2; C – recipe 3; D – recipe 4.

The images in Figures 2 and 3 confirm the observations regarding the appearance of the cellular glass aggregate specimens and their microstructural peculiarities noted above.

The objective of the work on improving cellular glass aggregate made with $CaCO_3$, borax and Na_2SiO_3 in terms of quality was achieved through the applied technological changes that led to a significant reduction of the density of expanded products.

The comparative analysis of the features of cellular glass aggregate specimens allowed the selection of the optimal recipe, whose quality performances are to a large extent similar to those of industrially made cellular glass aggregate. This optimal recipe was designated recipe 2 made of 84.4 % colored residual drinking bottle, 1.6 % $CaCO_3$, 6 % borax and 8 % Na_2SiO_3 by microwave heating at 837 ºC with the average heating speed of 21.5 ºC. The product has the bulk density of 0.22 g/cm^3 and the heat conductivity of 0.079 W/m·K, i.e. the physical properties that give it a light weight and a good heat insulation. The cell size is between 1.0-2.0 mm, optimal for ensuring the homogeneity of the insulation. Simultaneously, the compression strength of 5.9 MPa is high enough, suitable for typical mechanical requirement for quality cellular glass aggregate. Higher compression strengths (7.7 and 9.2 MPa corresponding to recipes 3 and 4) are not required, especially as the bulk density increases to values of 0.25 and 0.28 g/cm^3, which slightly exceed the recommended limits. Recipe 1 has a low bulk density (0.19 g/cm^3), but the compression strength decreases up to 4.0 MPa and the microstructural configuration is quite large and slightly inhomogeneous.

Conclusion

The paper aimed to improve the process of experimental wave irradiation manufacturing the cellular glass aggregate using a recipe composed of colored (green and amber) residual drinking bottle, $CaCO_3$ as an expanding agent, borax as a fluxing agent and Na_2SiO_3 solution as a hardening material. Reducing the granulation of residual glass below 100 μm and especially, decreasing the thickness of the high microwave-susceptible ceramic tube from 3.5 to 2.5 mm, used as a screen to protect the pressed glass-based mixture against the aggressive effect of the electromagnetic wave irradiation, were the main technological measures adopted by the authors. As a result, the quality of cellular glass aggregate was improved by significantly reducing the bulk density in the range 0.19-0.28 g/cm^3 and the apparent specific gravity, respectively, between 0.32-0.40 g/cm^3, the reduction of heat conductivity in the range 0.074-0.095 W/m·K and keeping a high level of compression strength between 4.0-9.2 MPa. The qualitative analysis of the four experimentally made cellular glass aggregate specimens led to the identification of recipe 2 as the optimal recipe with very close characteristics compared to industrially made products. Thus, the bulk density was 0.22 g/cm^3, heat conductivity was 0.079 W/m·K, compression strength was 5.9 MPa, cell size between 1-2 mm and water absorption 2.2 vol. %. The specific energy consumption of the cellular glass aggregate making process was very low (between 0.66-0.73 kWh/kg) below the reported values of consumption in industrial processes (0.74-1.15 kWh/kg). The specific consumption of energy of the optimal recipe was 0.71 kWh/kg.

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