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MICROWAVE HEATING APPLICATION FOR MAKING CELLULAR GRAVEL USING RECYCLED RESIDUAL FLAT GLASS FROM CONSTRUCTION AS WELL AS ALUMINA-SILICATE INDUSTRIAL BY-PRODUCTS (FLY ASH AND SLAG)

BY

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Abstract. The production of cellular glass gravel based on glass waste and a suitable foaming agent has rapidly reached an industrial level in the last decades, satisfying the need for insulation-under slab and rooftops, light filling material for landscaping, roof gardens, green roofs, etc., having drainage and load bearing abilities. Not only glass waste was tested for the manufacture of cellular gravels, but also other material precursors, the most interesting being metallurgical slag and coal fly ash. They exhibited the capability to increase the mechanical strength of cellular products, but had a certain negative effect on their thermal insulation and physical properties. The current work aimed at finding an optimal correlation between the proportions of glass waste and those of slag and fly ash used in the material mixture. The own technical solution of applying predominantly direct microwave heating was maintained in this experiment.

Keywords: cellular gravel, microwave heating, drainage, load bearing, insulating.

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1. Introduction

A new recycled material (cellular glass gravel) that has caused a special interest in the field of construction works appeared in the last decades of the 20th century. The application fields of this gravel type have included road and railway construction, bridge abutments, insulation of thermal pipes and underground tanks, drainages, sports fields, building perimeter insulation, terrace insulation, insulation under ceilings, insulation of green flat roofs, load bearing heat insulation for under foundation slabs, etc. (Paunescu *et al*., 2022).

According to (Hibbert, 2016), European countries (mainly, Germany, Austria, Switzerland and Scandinavian area) contributed over 600 thousand m^3 per year of cellular glass gravel, the main raw material being recycled glass from post-consumer drinking bottles and flat glass waste from demolition, while the expanding agent was chosen from carbon black, graphite, coal tar, glycerol, calcium carbonate, sodium carbonate, silicon carbide, silicon nitride, etc. (Scarinci *et al*., 2005).

Special performances characterize the cellular glass gravel. The thermal insulation properties (low density, high porosity, and low heat conductivity) are complemented by resistance to fire, water, frost, corrosion, excellent durability, chemical and physical stability, lack of toxicity, resistance to rodent and insect aggression (Paunescu *et al*., 2022; Cellular glass, 2016).

Recently, cellular glass gravel is manufacturing in China at the performance level of European companies, the raw material being constituted entirely of recycled glass waste (Szeco, 2024). This material combines the physio-structural glass peculiarities with the insulation properties of a closed cell structure. Applications of this Chinese material are: insulation of basements under slabs as well as backfill, new floor in old buildings, light fill material for roof gardens, green roofs, and landscaping, insulation of rooftop, etc. Main heat-physical properties of the mentioned cellular gravel pieces are density of 0.12 $g \cdot cm^{-3}$, heat conductivity of 0.058 $W \cdot m^{-1} \cdot K^{-1}$, water uptake of 0.2 vol. %, and maximum sizes of 30 x 30 x 60 mm.

According to the comparative analysis study of the foam glass gravel types produced in the world (Cosmulescu *et al*., 2020), the most important companies in this manufacturing field are Geocell Schaumglas (Austria), Misapor Switzerland (Switzerland), and Glapor Werk Mitterteich (Germany). Considered one of the leading producers of cellular gravel, Geocell Schaumglas is based on coloured post-consumer drinking bottle (90%) and clear flat glass waste (10%) without the foaming agent being named. The thermal process temperature is almost 900°C and the final product has porous structure with closed cells, bulk density of 0.15 $g \cdot cm^{-3}$, heat conductivity of 0.08 $W \cdot m^{-1} \cdot K^{-1}$, and compression strength of 5.7 MPa. Currently, Misapor operates in two factories in Switzerland as well as one each in Italy and Germany. The company uses 98% recycled glass, the remaining two percent being represented by gypsum, limestone or SiC as expanding agents. The sintering process temperature is about 900°C for 30 min. The bulk density is between 0.16 - 0.19 $g \cdot cm^{-3}$ and the compact material density is between 0.21-0.25 $g \cdot cm^{-3}$. Heat conductivity of the compact material is 0.12 $W \cdot m^{-1} \cdot K^{-1}$ and water uptake can reach 6-10 vol. %. Compression strength is relatively high, being between 4.9-6.0 MPa. Unlike the first companies mentioned above, Glapor Werk Mitterteich uses in its production recipe 1% glycerol as a liquid foaming agent associated with 12% sodium silicate solution and very little kaolin. The basic raw material is also represented by flat glass waste or post-consumer drinking bottle in a proportion of 87%. Bulk density has values between 0.13-0.21 $g \cdot cm^{-3}$ and compression strength reaches 4.9-6.0 MPa. The porosity of cellular glass lumps is high, the pore size falling below 300 µm.

Foamed glass waste is not the only suitable precursor for making cellular products. According to (Scarinci *et al.*, 2005; Rawlings *et al.*, 2006), industrial wastes such as metallurgical slag, ash from coal burning in thermal power stations or from municipal waste incinerators, mud from zinc hydrometallurgy, etc., together with glass waste have the ability to produce different cellular glass types.

Material mixture consisting of TiO_2-rich blast furnace slag, glass waste, borax as a flux agent, and calcium carbonate ($CaCO_3$) as an expanding agent was sintered at 900°C for 30 min, producing a porous material with bulk density of 0.82 $g \cdot cm^{-3}$ and excellent compression strength (up to 25 MPa). By increasing the proportion of $CaCO_3$ to 5-7%, the expansion process was intensified. As a result, the compression strength decreased to 13.1-13.8 MPa and water absorbing had values below 3.7% (Sun and Wang, 2017; Wang *et al.*, 2018).

If the blast furnace slag is poor in TiO_2, it is necessary to add this oxide separately, zirconium oxide (ZrO_2) and calcium fluoride (CaF_2) as nucleating agents as well as glass waste (Ding *et al.*, 2015). Borax and sodium phosphate ($Na_3PO_4 \cdot 12H_2O$) as additives were also added as well as $CaCO_3$ (around 6%) as a blowing agent. Glass-ceramic foams obtained by introduction into mixture of 50% blast furnace slag led to excellent results (bulk density of 0.79 $g \cdot cm^{-3}$, compression strength of 14.3 MPa, and water uptake of 2.7%).

A mixture almost similar to that used in (Ding *et al.*, 2015) was tested in the experiment presented in (Grigoras *et al.*, 2020), which was mainly different due to the application of the unconventional microwave heating procedure. The temperature range at which the mixture was sintered was within the limits of 900-905°C. The blast furnace slag/glass ratio was applied in the range of 40/60 and 50/50. Due to the microwave heating, warming rate was extremely high (between 20.1-21.2$°C \cdot min^{-1}$). The optimal version resulted in this experiment including slag/glass ratio of 40/60, 7.8% borax, 5% TiO_2, 3% $Na_3PO_4 \cdot 12H_2O$, and 6.5% $CaCO_3$ had apparent density of 0.82 $g \cdot cm^{-3}$, heat conductivity of 0.135 $W \cdot m^{-1} \cdot K^{-1}$, and compression strength of 14.1 MPa.

By sintering at 1150°C, high proportions of coal fly ash (over 80%) in a mixture with 5% Na_2O as a fluxing agent and 3-9% $CaCO_3$ as an expanding agent allowed obtaining cellular glass-ceramics with bulk density between 1.55-1.59 g·cm^{-3}, porosity between 16.8-19.9%, and flexural strength in the range of 43.4-109.6 MPa. In terms of dimensional uniformity of cells and microstructural homogeneity, the version using 6% $CaCO_3$ was the most adequate (Ma *et al.*, 2018).

Some researchers from authors' team of the current paper aimed to find convenient technical solutions that would balance the very high level of mechanical resistance with the low thermal insulation properties of cellular products suitable for use as foam gravels in various construction applications (Paunescu *et al.*, 2022). The originality of this work was the complete non-utilized of glass waste, which is predominantly included in the composition of raw material used for the usual manufacture of foam glass gravels, the glass being replaced with alumina-silicate industrial waste (granulated blast furnace slag and fly ash). Avoiding glass waste allowed the direct microwave heating with extremely high warming rates (between 32.3 - 40.1°C·min^{-1}) without affecting the microstructural configuration of the foamed product. Bulk density was between 0.29 - 0.38 g·cm^{-3}, heat conductivity had values in the range of 0.092 - 0.105 W·m^{-1}·K^{-1}, and compression strength between 12.6-14.2 MPa.

The new paper has proposed a mixture composed of residual glass (between 30-38%), granulated blast furnace slag (between 30-45%), and fly ash (in the range of 28-34%). The chosen expansion agent was silicon carbide (SiC) in proportions within the limits of 2-5%. Benefiting from the existence in the Romanian company Daily Sourcing & Research of adapted microwave heating equipment also used in own previous experiments, this efficient technique but generally not applied in the case of high temperature requirement was adopted as the technical solution for the sintering-expanding heat treatment of the above-mentioned mixture.

2. Methods and Materials

Due to the rather high weight proportion (over 30%) of the glass waste, the direct microwave heating had to be replaced with an own method of predominantly direct and partially indirect heating by positioning a ceramic tube made of SiC (80%) and Si_3N_4 (20%) with a wall thickness of 2.5 mm (experimentally determined). The vertical wall of the tube plays the role of a screen that limits the intensity of the microwave flux that comes into direct contact with the material subjected to heating through partial absorption of waves in the tube wall mass. Thus, the specimen heating process occurs from two opposing directions. It is already known that direct microwave heating is beginning in the central area of the material and volumetrically propagates from the inside to the outside, i.e. the opposite of conventional heating (Kitchen *et al.*, 2014; Jones *et al.*, 2002). On the other hand, the intense heat of the tube wall transmits thermal energy by radiation. Under the conditions of thermal protection with highly effective ceramic fibre mattresses, the heat is only diffused outwards to a small extent, but in the direction of the material subjected to heating.

The previously mechanically processed material mixture to values below 100 µm was pressed into a removable mould and then removed from the mould. The press-hardened sample was placed on a metal plate and metal supports put down on the thick thermal insulating layer of ceramic fibre mattresses at the base of the microwave oven. The microwave-susceptible ceramic tube was positioned over the sample to be heated, resting on the thermal insulation layer. The upper area of the tube was closed with a SiC-lid axially provided with a 30 mm-hole for viewing the heated material with a radiation pyrometer fixed on a metal rod about 400 mm above the oven. Using the radiation pyrometer thus positioned, the temperature on the surface of the heated material was monitored. The outer surface of the tube as well as the lid (except for the hole) were protected with ceramic fibre mattresses to greatly reduce heat losses outside the system.

The construction and operational scheme of the microwave heating equipment (a), the image of the microwave oven during operation (b), and the appearance of the ceramic tube acting as a protective screen against waves (c) are shown in Fig. 1.

a b c

Fig. 1 – Experimental microwave equipment
a – microwave equipment (1 – microwave oven; 2 – ceramic tube; 3 – ceramic lid; 4 – metal plate; 5 - pressed mixture; 6 – metal support; 7 – heat insulation; 8 – waveguide; 9 – pyrometer); b – image of the oven during the heat process; c – ceramic tube.

This experiment was based on the existence in the Romanian company Daily Sourcing & Research of stored reserves of industrial by-products (granulated blast furnace slag and coal fly ash) supplied by ArcelorMittal Galati and, respectively, Paroseni-Thermal power plant about 10 years ago. The granulated blast furnace slag had the grain size between 2-6 mm, requiring grinding in a ball mill and sieving to select sizes below 90 µm. Coal fly ash had particle sizes under 250 µm, requiring additional grinding to reduce the size below 60 µm. Clear flat glass waste recovered from building demolition was prepared by washing, grinding, and sieving, the selected grain sizes being below 70 µm. Chemical composition of the three raw materials used in this experiment is presented in Table 1.

Table 1

Chemical composition of raw materials (wt. %)

Material	SiO_2	Al_2O_3	CaO	MgO	Na_2O	K_2O	Fe_2O_3
Blast furnace slag	36.4	7.9	41.2	7.4	-	-	1.8
Coal fly ash	46.5	23.7	3.5	3.1	5.2	3.4	6.9
Clear flat glass	71.8	1.1	8.6	3.8	13.7	-	0.1

Commercially available silicon carbide (SiC) with a grain size under 10 µm was the foaming agent chosen in this experiment.

Composition of experimental versions adopted for testing the production of cellular gravel under the conditions of moderate use of residual glass compared to known conventional industrial techniques as well as lower proportions of fly ash and slag compared to those tested in the microwave field by teams including authors of the current paper is presented in Table 2.

Table 2

Composition of experimental versions (wt. %)

Composition	Version 1	Version 2	Version 3	Version 4
Clear flat glass waste	38	35	32	30
Granulated blast furnace slag	30	35	40	45
Coal fly ash	28	30	32	34
Silicon carbide	2	3	4	5

The cellular gravel specimens experimentally produced by the sintering-expanding process of wastes were characterized by traditional analysis methods. The apparent density was measured using Archimedes' principle in accordance with the ASTM C373 standard (Manual, 1999). The bulk density was determined by reporting the weight of a batch of fully loaded lumps into a container of known volume. ISO 18754:2020 was applied to measure the porosity (Anovitz and Cole, 2015). The determination method of the heat conductivity (Janetti *et al.*, 2015) consisted of measuring the heat flow value that passes through a sample placed between two metal plates. One of the metal plate was heated and protected with insulating material and the other was cooled. A compression machine of 2000 kN capacity existing in the Metallurgical Research Institute with a loading rate of 0.2 MPa·s was utilized for determining the compression strength values. Water uptake of the porous specimen was measured by applying the method of its immersion under water (ASTM D570). The porous microstructure of the cellular gravel samples was identified with ASONA 100X Zoom Smartphone Digital Microscope.

3. Results and Discussion

Adopting the amount of dry raw material used in preparing the production recipes of 400 g, kept constant in all tried versions, operational parameters of the sintering process were those presented in Table 3.

Table 3

Operational parameters of the making process

Parameter	Version 1	Version 2	Version 3	Version 4
Dry raw material/cellular gravel (g)	400/ 388	400/ 389	400/ 387	400/ 389
Sintering temperature (°C)	900	902	905	908
Time process (min)	32	33	33.5	34
Heating rate ($°C \cdot min^{-1}$)	28.1	27.3	27.0	26.7
Cooling rate ($°C \cdot min^{-1}$)	5.1	5.3	5.2	5.2
Energy consumption ($kWh \cdot kg^{-1}$)	0.86	0.88	0.90	0.91

Due to the application of the own unconventional microwave predominantly direct heating method, the heating rate level was high (26.7-28.1$°C \cdot min^{-1}$), much higher compared to the heating rates used in conventional processes (around 10$°C \cdot min^{-1}$).

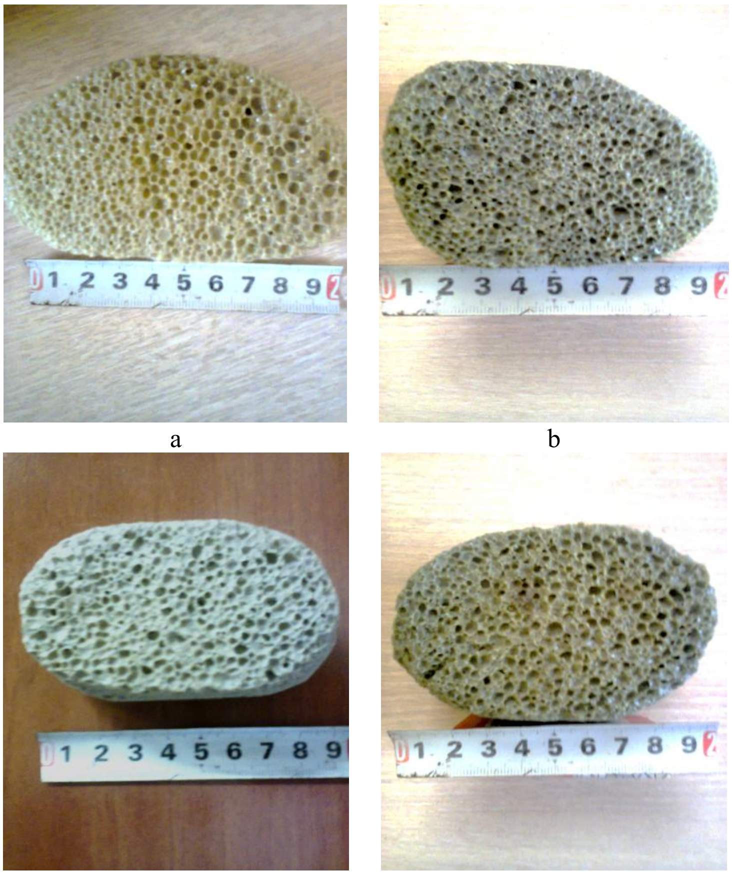
Images of the four cellular gravel samples produced within this experiment are shown in Fig. 2.

Investigating the physio-mechanical, heat, and morphological characteristics of specimens led to the results presented in Table 4.

Table 4

Main characteristics of cellular gravel specimens

Characteristic	Version 1	Version 2	Version 3	Version 4
Apparent density ($g \cdot cm^{-3}$)	0.39	0.43	0.44	0.56
Bulk density ($g \cdot cm^{-3}$)	0.27	0.29	0.29	0.42
Porosity (%)	79.8	78.1	78.0	75.3
Heat conductivity ($W \cdot m^{-1} \cdot K^{-1}$)	0.095	0.106	0.105	0.114
Compression strength (MPa)	6.3	10.3	12.4	12.7
Water uptake (vol. %)	3.5	3.7	3.8	4.0
Pore size (mm)	0.2-0.8	0.2-1.0	0.3-0.9	0.5-1.1



a b

c d

Fig. 2 − Physical appearance of cellular gravel specimens
a – version 1; b – version 2; c – version 3; d – version 4.

The pore size was identified from images representing microstructural peculiarities of cellular gravel specimens (Fig. 3). According to these images, the samples had a generally uniform distribution of closed cells. The pore size was the smallest in version 1 (between 0.2-0.8 mm), in which blast furnace slag, fly ash, and foaming agent (SiC) had the lowest proportions compared to the other experimental versions, except for clear flat glass which had the highest value. The last version made under the conditions of using the highest values of slag, fly ash, and SiC and the lowest value of glass waste, the pore size was the largest (between 0.5-1.1 mm).

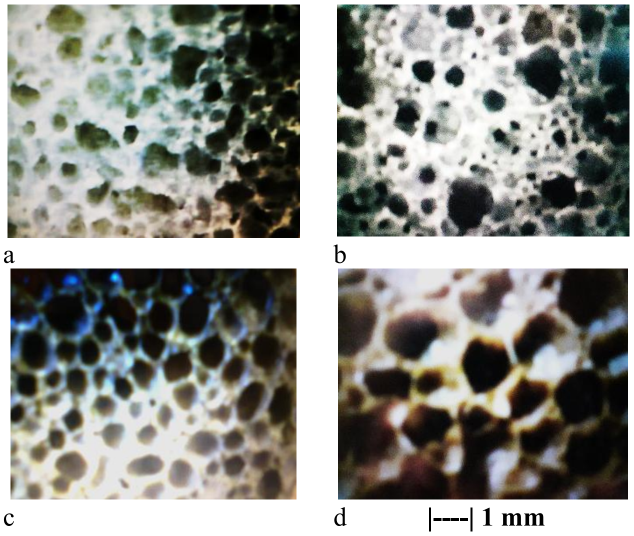


Fig. 3 – Microstructural peculiarities of cellular gravel specimens
a – version 1; b – version 2; c – version 3; d – version 4.

Examining the data in Table 4 allowed the identification of results corresponding to experimental versions 2 and 3 that are almost similar in terms of density, heat conductivity, and porosity, but obviously different in terms of the value of compression strength, which in the case of version 3 is higher than version 2 by about 23.3%. This observation demonstrates that the use of higher proportions of slag (from 35 to 40%), coal ash (from 30 to 32%), and SiC (from 3 to 4%) as well as of lower proportion of clear flat glass (from 35 to 32%) did not lead to a deterioration in the thermal insulation properties of the cellular gravel specimen, but instead caused an increase in its compressive strength. Therefore, experimental version 3 can be considered to be the optimal version, through which the best correlation between thermal insulation properties and mechanical strength was obtained.

Tests carried out in 2019 (Axinte *et al*., 2019) involving authors of the current paper showed that the completely direct microwave heating of glass waste in the 800 W-oven in foaming processes causes destruction of the internal structure of the expanded product, the solution adopted by the Romanian research team being the placement of a microwave-susceptible screen between the wave emitting source and the material subjected to heating. Compared to other works in which microwave heating is applied, in the current study the solution of tempering the intensity of the microwave field on the material by placing the protective screen in the form of a ceramic tube absorbing electromagnetic waves between the emission source and the material was adopted. Experimentally, it has been found that the optimal thickness of the screen should be 2.5 mm and further, cylindrical tubes made of SiC (80%) and Si_3N_4 (20%) made in China were used. Subsequent experiments have demonstrated that silicate materials other than glass (slag, ash, and other similar wastes) support direct microwave irradiation emitted in the 800 W-microwave oven and consequently, rapid and efficient heating in order to achieve foaming. These considerations were the basis of the experiments carried out previously and currently. In principle, the rather massive presence of slag and ash in the mixture prepared in this experiment together with the glass waste implied the application of the mentioned method of predominantly direct microwave heating.

4. Conclusions

The work aimed at the manufacture of a cellular gravel intended for various special insulations in buildings, roof gardens, landscaping around the building, drainages and load bearing constructions, the preparing mixture including flat glass waste, slag, fly ash, and SiC as an expanding agent. The peculiarity of the adopted procedure was applying the own unconventional method of predominantly direct microwave heating, under the conditions in which industrial techniques used in the world are exclusively conventional. The conducted experiment aimed to find an optimal correlation between the thermal insulation properties of cellular gravel and its mechanical strength as a result of utilizing different proportions of the raw materials mentioned above, including the expanding agent. The results allowed to identify the optimal solution by investigating the physio-mechanical, heat, and morphological characteristics of the obtained specimens. It was found that the optimal experimental version was the one that used 32% clear flat glass waste, 40% blast furnace slag, 32% fly ash, and 4% SiC, the mixture being sintered at 905°C with a heating rate of $27°C\cdot min^{-1}$. Bulk density was $0.29\ g\cdot cm^{-3}$, heat conductivity was $0.105\ W\cdot m^{-1}\cdot K^{-1}$, and porosity was 78%, while the compression strength reached a good value of 12.4 MPa. The experiment presented in this paper confirmed that the version of cellular gravel preparation using also other industrial by-products together with glass waste leads to almost similar qualitative results.

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APLICAREA ÎNCĂLZIRII CU MICROUNDE PENTRU FABRICAREA PIETRIȘULUI CELULAR UTILIZÂND STICLĂ PLATĂ REZIDUALĂ RECICLATĂ DIN CONSTRUCȚII, PRECUM ȘI PRODUSE SECUNDARE INDUSTRIALE ALUMINOSILICATICE (CENUȘĂ ZBURĂTOARE ȘI ZGURĂ)

(Rezumat)

Producerea pietrișului din sticlă celulară pe bază de deșeu de sticlă și a unui agent adecvat de spumare a atins rapid un nivel industrial în ultimele decenii, satisfăcând nevoia pentru izolația sub plăci și acoperișuri, material ușor de umplutură pentru amenajări peisagistice, grădini de acoperiș, acoperișuri verzi, etc., având capacități de drenare și încărcare portantă. Nu numai deșeul de sticlă a fost testat pentru fabricarea pietrișului celular, ci și alți precursori, cei mai interesanți fiind zgura metalurgică și cenușa de cărbune zburătoare. Acestea au manifestat capacitatea de a crește rezistența mecanică a produselor celulare, dar au avut un anumit efect negativ asupra proprietăților lor termoizolante și fizice. Lucrarea actuală a vizat găsirea unei corelații optime între proporțiile deșeului de sticlă și acelea ale zgurii și cenușii zburătoare utilizate în amestecul material. Soluția tehnică proprie a aplicării predominant directe a încălzirii cu microunde a fost menționată în acest experiment.