

# ALUMINO-SILICATE WASTES USED FOR MAKING CELLULAR GRAVEL BY NON-CONVENTIONAL DIRECT MICROWAVE HEATING METHOD

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**ABSTRACT:** Cellular gravel based on alumino-silicate wastes including residual clay brick recovered from construction demolition and fly ash resulted from coal burning in energy industry was experimentally made. The expanding process made with glycerin as a liquid organic material together water glass was obtained by direct electromagnetic wave warming without affecting the structural integrity of the product. The special effectiveness of the direct irradiation of the material led to extremely low specific electricity consumption (0.53-0.64 kWh/kg). The optimal version had the following characteristics: bulk denseness of 0.21 g·cm<sup>-3</sup>, heat conductivity of 0.063 W·m<sup>-1</sup>·K<sup>-1</sup>, compression resistance of 3.9 MPa, flexural strength of 2.4 MPa, and cell dimension within the limits of 0.2-0.5 mm. The use of alumino-silicate wastes in association with direct microwave warming keeping the physical, thermal, mechanical and microstructural properties unchanged (almost similar to industrial products) constitutes the paper originality.

**KEYWORDS:** cellular gravel, direct microwave warming, alumino-silicate waste, glycerin, water glass.

## 1. INTRODUCTION

At the end of the last century and the beginning of the new millennium, serious energy (oil crisis) and ecological problems (the risk of increasing the terrestrial atmosphere due to CO<sub>2</sub> release) have inaugurated a new stage in the approach to the relationship between production activities and environmental protection. Thus, the numerous wastes (plastics, metals, glasses, paper and cardboard, textiles, etc.) stored in landfills or thrown in a disorganized way in other terrestrial and aquatic areas that have alarmingly invaded the environment, have become interesting either for their reintroduction in the productive circuit, either for the manufacture of new materials under the conditions of low costs.

The construction sector was probably the most affected by the latest changes in the global policy approach due to the involvement of the most important material component (cement) in the manufacture of concrete both through the excessive consumption of fossil fuel and through the huge amounts of greenhouse gases emitted into the atmosphere. Recycled residual glass, but also other industrial by-products have become very important in this context, being intensively utilized for making the construction materials.

One of the new materials intended for the construction sector is cellular gravel with load-bearing, insulating, and mechanical strength properties. These foamed products made from

residual bottle with low additions of mineral materials with the role of expanding material have simultaneously several remarkable features: low denseness, reduced heat conductivity, low water-absorbent, fire-resistant, high-strength, resistant to rodents, insects, and bacteria. The field of application is wide. The cellular gravel is used as filler for foundation insulation, perimeter insulation of houses, pavement, road and railway structure, heat-insulating the underground pipes and cisterns, etc.

Concording with the paper [1, 2] and Quality Declarations of the main industrial manufacturers (Geocell-Austria, Misapor-Switzerland, and Glapor-Germany), the main features of porous gravel are: bulk denseness in the range of 0.12-0.25 g·cm<sup>-3</sup>, heat conductivity within the limits of 0.06-0.08 W·m<sup>-1</sup>·K<sup>-1</sup>, and compression strength between 5-6 MPa.

The main pore-forming agents industrially used are solid mineral materials in powder form (limestone, gypsum or silicon carbide). According to data from the literature [3], the German company Glapor adopted a liquid organic agent (glycerin) (1 wt. %) together with 12 wt. % water glass and very few kaolin obtaining foam bottle gravel with bulk denseness between 0.16-0.19 g·cm<sup>-3</sup>, and compression strength in the range of 4.9-6 MPa.

The method of liquid agent using has the advantage of favouring to obtain a finer porosity of the foamed material compared to the method of using solid

agents [4]. In an experiment performed in 2017 [5], 90 wt. % residual glass was mixed with 4 wt. % glycerin, 3 wt. % water glass, and 3 wt. % water addition for the purpose of manufacturing foam glass gravel by sintering-expanding in the temperature range of 800-850 °C. The product had fine porosity with the pore size under 0.3 mm. The heating process carried out in a usual electric oven was slow with an average heating rate of 8 °C/min and then continued with maintaining the foamed product at the expanding temperature during 30 min.

The optimal ratios of glycerin and water glass were experimentally determined [6] in a sintering-expanding process of residual bottle performed at 850 °C, the average warming rate being very high (50-80 °C/min). Under the conditions where the physical, heat, and mechanical characteristics of cellular bottle gravel specimens were approximately identical with industrial products, the weight ratios of glycerin and water glass were 1.5 wt. % and 5 wt. %, respectively.

The industrial production of cellular bottle gravel based on recycled residual bottle in very high ratios (97-98 wt. %) and a pore-forming agent (1-2 wt. %) is performed by conventional warming of the mixture, regardless of whether the agent is solid or liquid.

Starting from 2020, the societies Daily Sourcing & Research SRL and Cosfel Actual SRL (Romania) have carried out research using their own equipment on the manufacture of foam bottle gravel through unconventional microwave warming. The basic material was recycled residual bottle, as in the case of industrial production. It was experimentally found that the bottle dust is not adequate for applying the direct electromagnetic wave warming method due to the damage to the structural integrity of the foamed material and it was necessary to place a ceramic screen made of highly susceptible microwave materials (silicon carbide and silicon nitride) between the emitting wave source and material. This heating technique diminished the effect of direct wave irradiation on the bottle waste-based mixture, eliminating the structural damage, but slightly reduced the energy effectiveness of microwave warming, the process being branched into a predominant direct heating and a partial indirect heating (through thermal radiation of the screen). Several tests were carried out in a 0.8 kW-microwave oven using as expanding agent silicon carbide [7], calcium carbonate together borax and water glass [8], glycerin together water glass [9]. The products have corresponded to requirements of construction applications and moreover, the specific

electricity consumption was very little (under 1 kWh/kg). The use of an improvised 10 kW-microwave oven under the conditions of sintering-expanding the residual bottle with SiC showed that this non-conventional warming method is adequate and it could be extended to higher production capacities [10].

Based on own experimental observations, authors of the current paper concluded that by substituting the residual bottle with recycled alumino-silicate waste from building demolition or alumina- and silica-rich industrial secondary products (such as fly ash, granulated blast furnace slag, etc.) the direct microwave warming process is possible without affecting the structural integrity of foamed material [11]. The features of cellular gravel specimens made with silicon carbide (SiC) as a pore-forming agent were corresponding to this type of construction material (bulk denseness in the range of 0.50-0.68 g·cm<sup>-3</sup>, heat conductivity between 0.078-0.095 W·m<sup>-1</sup>·K<sup>-1</sup>, and compression resistance up to 7.5 MPa. The specific electricity consumption was within the limits of 0.74-0.85 kWh/kg.

Starting from the latest own results regarding the possibility of extremely efficient microwave warming in terms of energy through direct irradiation of residual alumino-silicate, the current paper adopted the association of glycerin as a liquid expanding agent with water glass, which allows obtaining fine porousness, thus replacing the utilization of SiC from the previous experiment [11].

## 2. METHODS AND MATERIALS

### 2.1 Methods

In technical terms, the method chosen in this experiment is the direct microwave heating method through irradiation applied on the material surface. Although electromagnetic waves were discovered 80 years ago, the main applications on a large scale were communications and radar. In the energy field, applications were limited to drying processes and processes of solid heating at low temperature [12]. Also, on a large scale, the microwave application in the field of food preparation is well-known, but their industrial application for high temperature heating is still in various experimental stages.

Previous experiments carried out by the research team, which includes authors of the current paper, showed that the direct microwave warming of bottle dust causes destructive effects on its structural integrity, so it was necessary to diminish the microwave-glass impact by placing an absorbing screen of radiation between the emitting source and

material that reduced the intensity of the wave flux coming into direct contact with the heated sample [13].

Recent tests performed by the same Romanian research team have allowed to find that aluminosilicate waste with high content of alumina and silica can be directly microwave heated for foaming without observing negative effects on the structural composition of the cellular product. Thus, the warming rate reached extremely values up to 40 °C/min) and the electricity consumption was significantly diminished.

The warming process of a solid material through direct contact of waves with this is a completely different procedure by comparison with the conventional warming. Concurring with the literature [14, 15], the electromagnetic wave power is turned into warm, the area where the warming initiation occurs being concentrated into the middle of material. Thus, the highest temperature area is the central material zone of the irradiated material is the central area and the warm volumetrically propagates from the interior to the outside. For this reason, the peripheral area of the heated material should be thermally protected for avoid the loss of heat in the exterior zone. This method of warm propagation is completely different by comparison with the traditional heating, where the heat source is placed outside and the heating is generally initiated in the area of the refractory lining of the oven, which in turn transmits the heat to the material. Obviously, the heat effectiveness of direct microwave warming is significantly higher than that of traditional heating.

The microwave equipment used in this experiment (Figure 1) was a 0.8 kW-electromagnetic wave oven usually used in domestic operations, but constructively modified for applications at much higher thermal levels (at least 1100 °C). The thermal protection of the oven base was provided by ceramic fiber bed resistant up to 1200 °C. A 3 mm-metallic plate was placed on the ceramic bed and the material in a pressed state was freely deposited above it. The material was also protected with ceramic fiber mattresses both on the side wall and above it. The thermal monitoring was done with a radiation pyrometer fixed above the furnace at about 400 mm. To visualize the sample surface, the upper wall of the plant was provided with 30 mm-hole.

In terms of chemistry, the making method of cellular gravel has adopted the foaming technique with a liquid organic expanding agent (glycerin-C<sub>3</sub>H<sub>8</sub>O<sub>3</sub>) commonly used together with water glass [4]. This organic material decomposes by heating around 850

°C releasing a variety of chemical compounds such as CO<sub>2</sub>, CO, pure C, hydroxyl compounds, etc. The role of water glass mixed with glycerin is to protect the carbon particles formed after decomposition against their premature oxidation. The gases released in the thermally softened basic material remain trapped as bubbles. By increasing the pressure inside the bubble as a result of the temperature increasing, the gas bubbles increase their volume and thus the material expands.



Figure 1. Microwave equipment used in experiment

## 2.2 Materials

The used solid materials were residual clay brick recovered from building demolition and coal fly ash previously supplied by the Romanian Thermal power plant from Paroseni. Preparing the residual clay brick was manually made by breaking the brick and selecting the clay component, the particles with sizes under 110 µm being chosen after sieving. Coal fly ash that had initial sizes under 230 µm, was ground and selected the particle size being under 90 µm.

Oxide composition of the two residual aluminosilicate is presented in Table 1.

Table 1. Oxide composition of aluminosilicate wastes

Composition	Residual clay brick (wt. %)	Fly ash (wt. %)
SiO <sub>2</sub>	56.4	46.5
Al <sub>2</sub> O <sub>3</sub>	27.4	23.7
CaO	1.2	5.2
Fe <sub>2</sub> O <sub>3</sub>	7.2	8.6
MgO	1.4	3.2
Na <sub>2</sub> O	1.0	6.0
K <sub>2</sub> O	4.4	4.1

**Table 3.** Operational parameters of the procedure

Operational parameter	Experimental version			
	1	2	3	4
Wet basic material/cellular gravel quantity (g)	527/390	527/393	527/391	527/392
Sintering-expanding temperature (°C)	831	838	847	865
Time process (min)	20	21	22	24
Average rate (°C/min)				
- warming	40.6	39.0	37.6	35.2
- cooling	8.4	8.4	8.5	8.3
Specific electricity consumption (kWh/kg)	0.53	0.56	0.58	0.64

The liquid parts of the mixture for preparing the cellular gravel were glycerin as a carbonic expanding agent, water glass ( $\text{Na}_2\text{SiO}_3$ ) whose role was mentioned above, and water as a binder. They were mixed together for 4 min before mixing with the solid components. Glycerin and water glass were commercially purchased being available.

Four testing versions were chosen in this experiment characterized by variation of weight proportions ratio of residual clay brick and fly ash and keeping constant liquid component proportions (1 % glycerin, 10 % water glass, and 6 % water). The weight ratio of residual clay had increasing values from 72 to 84 %, while the fly ash ratio decreased from 28 to 16 %. Table 2 presents the composition of each experimental version.

**Table 2.** Composition of experimental versions

Composition	Version (wt. %)			
	1	2	3	4
Clay waste	72	76	80	84
Fly ash	28	24	20	16
Glycerin	1	1	1	1
Water glass	10	10	10	10
Water	6	6	6	6

### 2.3 Investigation methods of specimens

Usual methods were applied for investigating the cellular gravel specimen characteristics. Bulk denseness and porousness were measured by applying the Archimedes' method (water intrusion method) according to ASTM D792-20 standard. The heat-flow method was utilized for determining the heat conductivity (ASTM E1225-04) [16]. The compressive resistance was measured with TA.XTplus Texture analyzer and the flexural strength was identified by conducting the three-point bend test on the specimen (ASTM D790) [17]. The method of immersing the specimen under water (ASTM D570) was applied to measure the water-absorbing capacity. Microstructural peculiarities of specimens were examined with ASONA 100X Zoom Smartphone Microscope.

## 3. RESULTS AND DISCUSSION

### 3.1 Results

Utilizing a constant quantity of wet basic material (527 g) in all experimental versions, operational parameters of the sintering-expanding procedure had values presented in Table 3.

Increasing the residual clay/fly ash ratio (2.57-5.25) influenced the temperature values of the sintering-expanding process, which increased from 831 to 865 °C. The very high energy effectiveness of direct microwave warming led to the process completion in very short times between 20-24 min due to extremely high warming rates (in the range of 35.2-40.6 °C/min). Concomitant with investigating the microstructural peculiarities of specimens, the very high warming rates did not cause damage to the structural homogeneity and integrity. The cooling rates (8.3-8.5 °C/min) used at the finish of the expanding process were higher than those practiced for the manufacture of cellular glass aimed at creating internal tensions in the mass of specimens for easy breaking of lumps at the usual sizes (60-70 mm). As a consequence of the excellent energy effectiveness of the non-conventional warming process, the specific electricity consumption was very low (0.53-0.64 kWh/kg), lower than the consumption of industrial cellular gravel producing processes (below 1 kWh/kg).

The main features of cellular gravel specimens investigated concurring with the methods noted above are shown in Table 4.

**Table 4.** Features of cellular gravel specimens

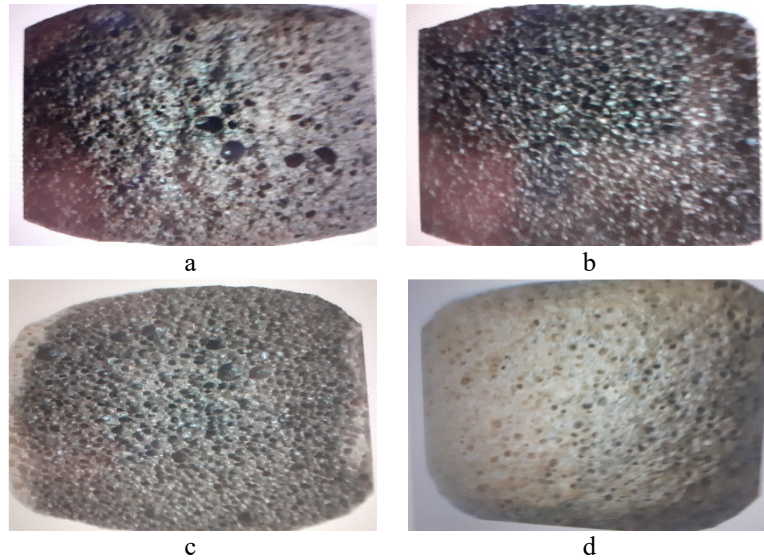
Characteristic	Version			
	1	2	3	4
Bulk denseness ( $\text{g}\cdot\text{cm}^{-3}$ )	0.17	0.21	0.24	0.27
Porousness (%)	84.0	82.3	80.1	78.9
Heat conductivity ( $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ )	0.059	0.063	0.068	0.074
Compression resistance (MPa)	3.5	3.9	4.6	5.4
Flexural strength	1.9	2.4	2.9	3.4

(MPa)				
Water-absorbing (vol. %)	4.1	4.6	6.5	7.8
Pore size (mm)	0.3-0.9	0.2-0.5	0.1-0.3	0.05-0.2

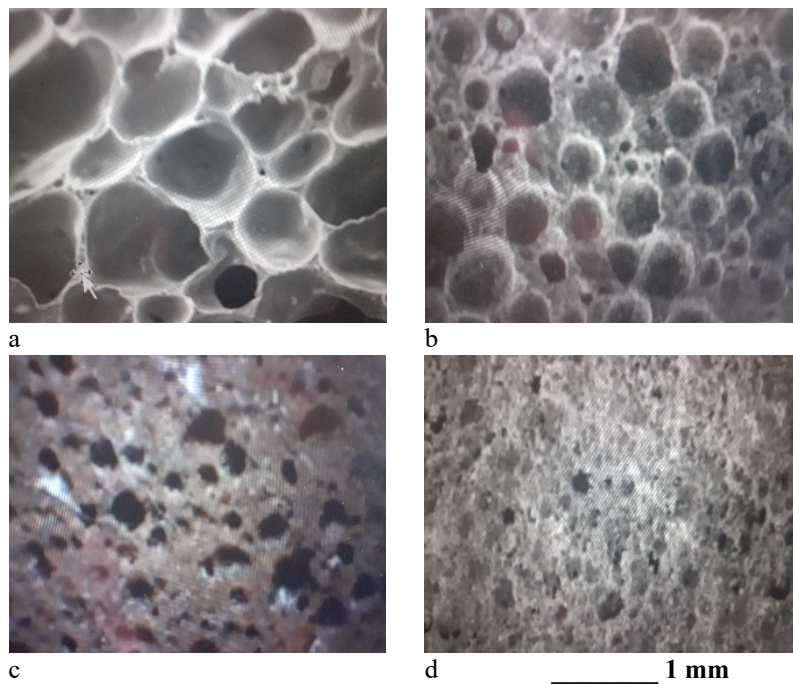
The compression strength falls between 3.5-5.4 MPa and flexural strength between 1.9-3.4 MPa. Water-absorbing has normal values for the first two versions (4.1-4.6 vol. %) where the clay content is lower, but there is the increasing trend up to 7.8 vol. % reaching this value for the clay content of 84 wt. %. The pore sizes are very low, with the largest range (0.3-0.9 mm) in the case of version 1 and the smallest (0.05-0.2 mm) in the case of version 4.

Images of cellular gravel samples are presented in Figure 2 and the microstructural peculiarities of these specimens are shown in Figure 3.

Analyzing the data in Table 4, excellent thermal insulation properties result (bulk denseness in the range of 0.17-0.27 g·cm<sup>-3</sup>, heat conductivity within the limits of 0.059-0.074 W·m<sup>-1</sup>·K<sup>-1</sup>, and porousness between 78.9-84.0 %). The mechanical strength has acceptable values within the requirements for the type of cellular gravel.



**Figure 2.** Images of cellular gravel samples  
a – version 1; b – version 2; c – version 3; d – version 4.



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

### 3.2 Discussion

The cellular gravel manufacturing industry developed in the last decades has focused its attention on recycled residual bottle used as the basic material. The manufacturing technique is based on expanding the mixture, also including a mineral expanding agent in very low proportions through the conventional heating of material at high temperatures (800-1100 °C).

Authors of the current paper have tried to make the manufacturing process more effective by applying the non-conventional method of microwave warming, faster and more economical, but have concluded that this warming type is inappropriate in the case of bottle as a silicate material due to the serious damage at the structural level of the foamed material.

It was experimentally found that residual alumino-silicate materials can be directly irradiated, obtaining their fast and economic heating without affecting the structural characteristics. Thus, excluding the use of alumino-silicate glass waste, various industrial by-products and other types of waste (fly ash, metallurgical slag, construction material waste such as bricks, concrete, etc. coming from demolitions, red mud, ash from waste incinerators, etc.) can be used for the manufacture of new products by direct microwave heating.

The association of the use of electromagnetic waves for efficient heating at high temperatures as well as waste or industrial by-products as raw material constituted the basis of research and originality of making process of cellular gravel.

The sintering-expanding process of alumino-silicate waste composed of residual clay brick and coal fly ash was performed using the non-conventional method of direct microwave heating. Cellular gravel specimens were made with specific characteristics of industrially manufactured materials and in addition, the specific energy consumption had lower values compared to industrial products.

Among the tested versions, version 2 made from 76 % residual clay, 24 % fly ash as solid components as well as 1 % glycerin, 10 % water glass, and 6 % water as liquid components was considered the best for applications in construction. Its bulk denseness was  $0.21 \text{ g}\cdot\text{cm}^{-3}$ , thermal conductivity was  $0.063 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ , compressive resistance reached 3.9 MPa, and flexural strength was 2.4 MPa. The cell dimension was within the limits of 0.2-0.5 mm, the product having fine porousness. The specific

electricity consumption value was very low (0.56 kWh/kg).

### 4. CONCLUSION

The work concerned the experimental production of cellular gravel for applications in construction from waste of building demolition (residual clay brick) and a by-product of the energy industry (coal fly ash) by sintering-expanding the material mixture using the association of glycerin as a liquid organic expanding material, water glass, and water. The energy source was the transformation of electromagnetic wave power into heat through the direct meet with the irradiated material. The goal was to test a fast and economic manufacturing method of cellular gravel compared to conventional heating techniques industrially used.

The originality of making process of cellular gravel was the association of the use of electromagnetic waves as the energy source and alumino-silicate waste (residual clay brick) or industrial by-products (coal fly ash) as basic materials.

The optimal product made with 76 % residual clay, 24 % coal fly ash, 1 % glycerin, 10 % water glass, and 6 % water had bulk denseness of  $0.21 \text{ g}\cdot\text{cm}^{-3}$ , thermal conductivity of  $0.063 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ , compressive resistance of 3.9 MPa, and flexural strength of 2.4 MPa. The product had fine porousness, its cell size being within the limits of 0.2-0.5 mm.

Although the discovery of electromagnetic waves was made 80 years ago, their use in industry for high temperature warming procedures of solids is still at a low level in the world. The present paper could contribute to the increase of manufacturers' interest for applications in the near future, considering the excellent energy effectiveness of this method (0.53-0.64 kWh/kg).

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