

# EXPERIMENTAL MANUFACTURE OF CELLULAR GLASS FROM GLASS WASTE IN THE MICROWAVE REACTOR

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**ABSTRACT:** Experimental results of the process of manufacturing cellular glass from glass waste and calcium carbonate (between 1.1-1.4 wt.%) in a microwave reactor are presented in the paper. Compared to all previous experiments, a significantly larger amount of raw material powder mixture was heated to 817-828 °C in a 3 kW-microwave reactor. The physical characteristics of the cellular glass samples were excellent: apparent density between 0.13-0.16 g/cm<sup>3</sup>, porosity between 92.7-94.1% and thermal conductivity in the range 0.041-0.046 W/m.K. The compressive strength had low values (1.09-1.18MPa), but in acceptable limits for using the cellular glass as an insulating material in construction. The specific energy consumption was higher (1.59-1.84 kWh/kg) due to the 15 mm-thickness of the wall of the silicon carbide crucible that fully absorbs the microwave radiation, the heating of the glass being indirect.

**KEYWORDS:** cellular glass, glass waste, calcium carbonate, microwave reactor, silicon carbide crucible, indirect heating.

## 1. INTRODUCTION

According to data provided by the United Nations, glass waste accounts for about 7% of all solid waste available in the world [1]. The annual generation rate of this waste is very high, especially of post-consumer container glass. The glass industry commonly recycles closed-circuit glass waste for the industrial production of the new glass, but the costs of the process of sorting colored glass are high. Much more cost effective is the use of glass waste as a basic raw material for the industrial manufacture of cellular glass as a replacement for building materials existing on the market. The cellular glass is a material with a homogeneous porous structure obtained by the heat treatment at high temperature (700-1100 °C) of a finely ground mixture of glass waste, a foaming agent (solid or liquid) and possibly various mineral additives with the role of improvement or facilitation of the foaming process. The mechanism of this process is the release of a gas following a chemical reaction (usually thermal decomposition or oxidation) of the foaming agent in the softened and viscous mass of the glass-based raw material. The temperature at which the gas is released must be correlated with reaching the softening point of the waste so that the gas meets a material with a suitable viscosity to be retained in the form of numerous bubbles. By cooling the bubbles blocked in the mass of material will form a homogeneous porous structure characteristic of cellular glass [2, 3]. In this way, the cellular material acquires unique physical and mechanical properties having simultaneously low density, low thermal conductivity and relatively high compressive

strength. In addition to these characteristics, the cellular glass is fireproof, waterproof, resistant to attack by rodents, insects, bacteria, non-toxic, non-deformable, chemically stable, etc. [2, 3]. Given all these properties, the cellular glass is suitable for a wide range of applications depending on the specific characteristics of the type of the manufactured glass foam. Thus, it can be used as an insulating material, floors, and wall tiles, architectural panels, filters, absorbers, gas sensor, in the case of very light materials or as lightweight aggregates, filling material in road and railway constructions, foundations, components of the construction of bridges, sports grounds, pavements, terraces, roof gardens, insulation of underground heating pipes and storage tanks, etc., in the case of porous materials with high mechanical strength [2-4].

According to [3], the first cellular glass were made during World War II in the United States being used as non flammable thermal insulation for the internal walls of ships and submarines. Glass waste began to be industrially used as a raw material only in the last decades of the 20<sup>th</sup> century, when the problem of recycling waste in general (plastic, metal, glass, paper) became an important concern especially in developed countries. The most well-known cellular glass companies were and still are Pittsburgh Corning in the United States and Misapor Switzerland in Switzerland, with several branches in Europe and China, which produce various types of cellular glass (thermal insulations as blocks, panels and plates, granules, lightweight aggregates, foam glass gravels or products of different shapes). Several companies, especially European, have the

production focused primarily on foam glass gravels used as filling materials with high mechanical strength in specific fields of application (Geocell Schaumglas, Glapor, Veriso, Technopor Handels, Hasopor, Glasopor, Foamit). The manufacturing recipes are very diverse, both in terms of the nature of the glass waste (container glass, flat glass, etc.), and especially the type of the used foaming agent (carbon black, coal powder, calcium carbonate, silicon carbide, glycerol, etc.) [3, 4]. According to [3], the largest expansions of glass waste (450%) were obtained using calcium carbonate (2 wt.%) as a foaming agent and colorless soda-lime glass as raw material. Industrially, glass foams produced in the form of blocks and various shapes had the apparent density between 0.10-0.17 g/cm<sup>3</sup>, porosity over 90% and thermal conductivity between 0.040-0.050 W/m·K [3]. Until now, the energy source used in the heating processes has been exclusively conventional (burning fossil fuels or electrical resistances). Also, numerous experimental results of small-scale tests performed by teams of researchers worldwide aiming to diversify foaming agents and appropriate mineral additives as well as the type of silicate waste are presented in the literature [4]. These experiments are also based on conventional heating techniques.

According to the paper [5], a fast, economical and "clean" nonconventional energy source (microwave radiation) known since the second half of the 20<sup>th</sup> century was only used in drying or heating processes at moderate temperature. Only in the last 10-15 years it has been experimentally found that several types of material (organics, ceramics, metals, polymers, glass, etc.) are suitable for efficient microwave heating, but so far no real industrial applications are known.

In the glass industry this retention is explained by the fact that some components of glass (e.g. SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>) are not microwave susceptible materials and their heating from room temperature to about 500 °C would not be efficient, according to the paper [6] published in 1997. This information, correct only theoretically, was taken over later in 2003 in the paper [2], which concluded that at that time it would not be profitable to equip existing glass foam furnaces from the UK industry with microwave heating installations only in the temperatures area of 500-1000 °C being kept the conventional system in the area of 20-500 °C.

In the last three years, the Romanian company Daily Sourcing & Research Bucharest has performed experiments for the production of different types of cellular glass using the microwave energy. It has been experimentally proven [7] that the commercial

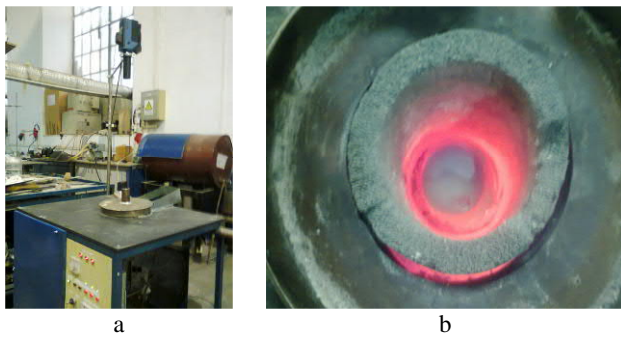
glass waste (soda-lime glass) can be efficiently microwave heated starting from room temperature due to the content, even very small, of inherent contaminants (Fe<sub>2</sub>O<sub>3</sub>, Cr<sub>2</sub>O<sub>3</sub>, etc.) that are susceptible microwave. Also, silicon carbide used either as a foaming agent in some cases or as a screen to protect the glass waste against the direct microwave irradiation is a highly microwave susceptible material. The works [8, 9] also theoretically claim that the glass can be efficiently microwave heated at room temperature.

Below, the paper presents experimental results of the manufacture of significantly higher quantities of cellular glass compared to the previous experiments from colorless post-consumer container glass waste and calcium carbonate as a foaming agent in various weight proportions, using a microwave reactor from the own experimental base.

## 2. METHODS AND MATERIALS

### 2.1 Methods

Previous experiments have been performed in a 0.8 kW-microwave oven, the largest amounts of cellular glass being less than 550 g. This time, a microwave reactor (Figure 1a) equipped with three microwave generators of 1 kW each placed equidistantly on the outer wall of the cylindrical cavity of the reactor was used. A silicon carbide crucible with a wall thickness of 15 mm was introduced in the reactor cavity, having a very efficient thermal protection of its external surface and the base with thick ceramic fiber mattresses. Inside the crucible was placed a detachable metal wall tube with a thickness of 2.5 mm, in which the powder mixture was pressed (Figure 1b). The upper opening of the ceramic crucible was protected with ceramic fiber and a metal lid provided with an Ø 30 mm hole through which the material temperature was measured during the process with a radiation pyrometer mounted above the reactor. The heating technique is based on the rapid absorption of the microwave field in the mass of the silicon carbide crucible which converts electromagnetic waves into thermal energy. Thus, the inner surface of the crucible continuously heats the wall of the metal tube by radiation and implicitly the glass-based powder mixture until the expansion of the material is observed. After stopping the power supply of the microwave generators, it is necessary to keep the foamed sample in the reactor for 40-60 minutes in order to avoid the cracking of the material caused by too fast cooling.



**Figure 1.** The experimental microwave reactor

a – overview of the 3 kW-microwave reactor; b – metal crucible positioning detail.

## 2.2 Materials

The basic raw material was a colorless post-consumer container glass having the following chemical composition (wt.%): 71.7% SiO<sub>2</sub>; 1.9% Al<sub>2</sub>O<sub>3</sub>; 12.0% CaO; 0.05% Fe<sub>2</sub>O<sub>3</sub>; 1.0% MgO; 13.3% Na<sub>2</sub>O; 0.05% Cr<sub>2</sub>O<sub>3</sub>. The glass waste was thermally washed in an own conception microwave oven at 250 °C to remove the organic contaminants. Then, the glass was ground in a ball mill and sieved at a grain size below 32 μm. The calcium carbonate as a foaming agent was used at the grain size below 5 μm as it was purchased from the market.

By this very advanced processing of glass waste, the selection of a type of waste suitable for a high foaming (soda-lime glass) and a foaming agent with extremely small grain size was aimed at the experimental manufacture of a cellular glass with extremely low apparent density, high porosity and very low thermal conductivity, like to similar products industrially made by conventional heating methods.

## 2.3 Characterization of the cellular glass samples

The cellular glass samples experimentally manufactured were characterized by traditional analysis methods. The main physical, mechanical and morphological characteristics were: apparent density, porosity, thermal conductivity, compressive strength, water absorption and microstructural configuration of the samples. The apparent density was measured by the gravimetric method [10]. The porosity was calculated by the comparison method [11] between the porous sample density and the density of the same material type in compact state obtained by melting followed by cooling to the room temperature. The determination method of the thermal conductivity [12] consisted of measuring the thermal flow value that passes through a sample of standard dimensions (50 mm-thickness) placed between two metal plates. One of the plate was heated and protected with insulating material and the other was cooled. An own conception device was

used to determine the compressive strength by developing an axial pressing force generated with a hydraulically operated piston. The last pressing force axially applied to the sample before to crack was considered the compressive strength value. The tested sample had a cylindrical shape with the diameter of 80 mm and the height of 70 mm. The water absorption of the porous sample was measured by the traditional method of its water immersion (ASTM D 570). The porous microstructure of the cellular glass samples was identified with a Smartphone Digital Microscope.

## 3. RESULTS AND DISCUSSION

### 3.1 Results

To manufacture the cellular glass samples from glass waste with calcium carbonate as a foaming agent, four experimental variants were adopted in which the weight proportion of foaming agent had values between 1.1-1.4% and the proportion of glass waste between 98.6-98.9%. The water addition as a binder was kept constant at 8%. Generally, these proportions correspond to those provided by the literature for cellular glass manufacturing processes using conventional heating methods.

**Table 1.** Experimental variants to made cellular glass

Variant	Colorless glass waste wt. %	Calcium carbonate wt. %	Water addition wt. %
1	98.9	1.1	8.0
2	98.8	1.2	8.0
3	98.7	1.3	8.0
4	98.6	1.4	8.0

The main functional parameters of the manufacturing process of cellular glass: amounts of the dry raw material and cellular glass, process temperature, heating time, heating and cooling rate, expansion of the glass volume and specific energy consumption are shown in Table 2.

The main physical, mechanical and morphological characteristics of the cellular glass samples: apparent density, porosity, thermal conductivity, compressive strength, water absorption and pore size are presented in Table 3.

**Table 2.** The main functional parameters of the manufacturing process of cellular glass

Variant	Dry raw material/ cellular glass amount  g	Sintering- foaming temperature  °C	Heating time  min	Average rate, °C/min		Expansion of glass volume  %	Specific consumption of energy  kWh/kg
				Heating	Cooling		
1	980/ 951	817	38	21.0	5.4	340	1.59
2	980/ 953	820	40	20.0	5.4	350	1.67
3	980/ 950	824	42	19.1	5.7	370	1.76
4	980/ 952	828	44	18.4	5.3	390	1.84

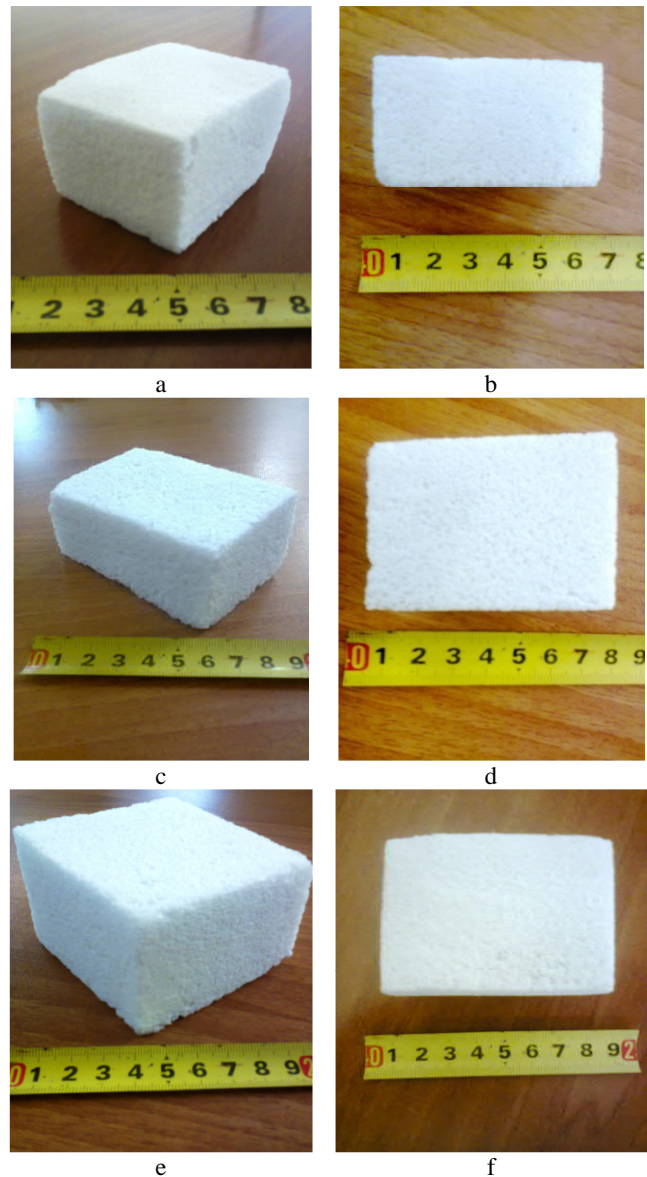
**Table 3.** The main physical, mechanical and morphological characteristics of the cellular glass samples

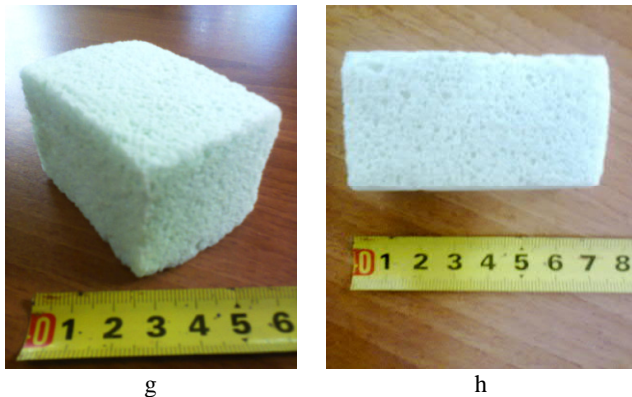
Variant	Apparent density g/cm <sup>3</sup>	Porosity  %	Thermal conductivity W/m·K	Compressive strength MPa	Water absorption %	Pore size  mm
1	0.15	93.2	0.046	1.18	0.2	0.5 – 0.8
2	0.13	94.1	0.041	1.16	0.3	0.6 – 0.9
3	0.16	92.7	0.046	1.14	0.5	0.6 – 1.0
4	0.14	93.6	0.044	1.09	0.6	0.7 – 1.4

According to the data in Table 2, the amount of the mixture of glass-based raw material powder and foaming agent was significantly higher compared to all previous experiments, in which this amount did not exceed 550 g. The average heating rate of the mixture had high values between 18.4-21.0 °C/min, allowing finishing the sintering-foaming process of glass waste in 38-44 min. The initial volume of the mixture increased by expansion on over 3.5 times. The specific energy consumption of the process, given that the microwave heating was exclusively indirect through the silicon carbide crucible with a wall thickness of 15 mm, had values between 1.59-1.84 kWh/kg, higher than 2 times compared to previous experimental results obtained in the heat treatment of much lighter samples (less than 550 g).

Analyzing the data in Table 3, the physical characteristics of the cellular glass samples were remarkable. The values of the apparent density were extremely low (between 0.13-0.16 g/cm<sup>3</sup>), the porosity had values of over 92.7% and the thermal conductivity was very low (below 0.046 W/m·K). Also, the impermeability of the samples was almost maximum. The compressive strength of cellular glass had low values (1.09-1.18 MPa), however acceptable for building applications as insulating materials.

Pictures of the cellular glass samples (overview and section) are presented in Figure 2.

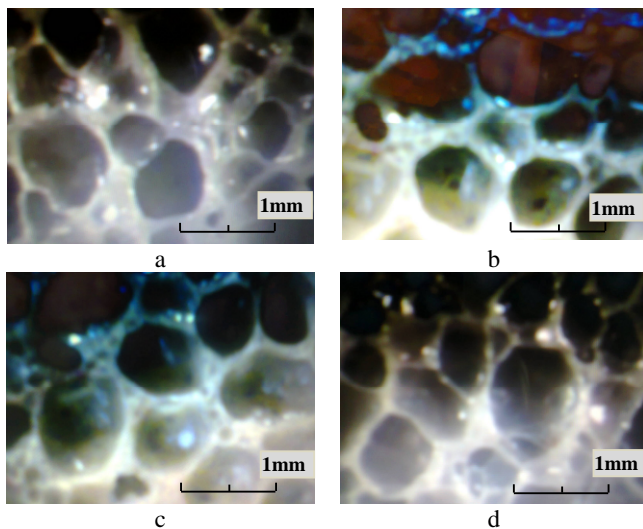




**Figure 2.** Pictures of the cellular glass samples  
 a, b – sample 1; c, d – sample 2;  
 e, f – sample 3; g, h – sample 4.

The main characteristic of the appearance of the four samples is the fine microstructure and the homogeneity of the pore distribution. The difference between the cellular glass sample made with 1.1% calcium carbonate (variant 1) and that with 1.4% (variant 4) is the range of pore size, which in variant 1 is between 0.5-0.8 mm and in variant 4 is between 0.7-1.4 mm. Given the very low values of the apparent density of the samples, the homogeneous distribution of the pores is what contributes to the acceptable level of their compressive strength.

Pictures of the microstructure of the cellular glass samples are shown in Figure 3.



**Figure 3.** Microstructure of the cellular glass samples  
 a – sample 1; b – sample 2; c – sample 3; d – sample 4.

The interpretation of the images in Figure 3 should be done together with the pore size data in Table 3.

### 3.2 Discussion

As noted above, the current work aimed at the experimental manufacture of cellular glass from glass waste by microwave irradiation under the

conditions of significant increase of the quantity of raw material from 200-550 g to 980 g and of the maximum power of the microwave equipment from 0.8 kW to 3 kW. So, instead of the oven of the type currently used in the household, a microwave reactor of own conception was used. Due to the much increased capacity, the protective screen made of microwave susceptible material used in previous experiments was replaced with a larger silicon carbide crucible, including the wall thickness (15 mm). The change in the wall thickness implicitly led to the change of the mixed microwave heating system (partially direct, partially indirectly) and the switch to exclusively indirect heating mode. Thus, the high microwave absorbing silicon carbide crucible has become the only massive component of the equipment that at the end of the process contains a significant amount of accumulated heat (about 77% of the total heat consumed). This explains the higher level of the specific energy consumption (1.59-1.84 kWh/kg) compared to the values obtained (less than 1 kWh/kg) in the most recent experiments [13]. In the perspective of the industrial application of the expected technical solution, this disadvantage will no longer exist because the industrial process will not be a discontinuous one.

### 4. CONCLUSION

A lightweight cellular glass (below  $0.16 \text{ g/cm}^3$ ) with very high porosity (over 92.7%) and very low thermal conductivity (below  $0.046 \text{ W/m}\cdot\text{K}$ ) was manufactured in a microwave reactor.

The physical characteristics of the experimental samples were similar to those industrially made by conventional heat treatment techniques.

The paper aimed for the increase of the amount of glass-based raw material up to 980 g using a suitable for this purpose microwave equipment, i.e. a 3 kW-microwave reactor.

Using the average heating rate up to  $21 \text{ }^\circ\text{C}/\text{min}$ , the foaming process of glass waste at maximum  $828 \text{ }^\circ\text{C}$  could be completed in less than 44 minutes.

A silicon carbide crucible with a wall thickness of 15 mm was used as a protection screen against the destructive effect of the direct microwave heating on its internal structure.

The porous structure of the foamed material was very fine, less than 1 mm in the case of variants with calcium carbonate between 1.1-1.3% and only partially over 1 mm in the case of the variant with 1.4% calcium carbonate, in all cases the pore distribution being very homogeneous.

The obtained cellular glass had an extremely low permeability to water, practically zero.

The compressive strength of the cellular glass had relatively low values (1.09-1.18 MPa), but in acceptable limits for using as an insulating material in construction.

Due to the thickness wall of the crucible and the total absorption capacity of the microwaves, the material heating mode was indirect. As a consequence, the specific energy consumption of the process increased by about 2 times the consumption corresponding to the mixed microwave heating applied in previous experiments.

In the perspective of the industrial application of a thick layer of silicon carbide on the side walls and the vault of the furnace, the disadvantage of the relatively high level of energy consumption will no longer exist because the industrial process will not be a discontinuous one.

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