

Manufacture of Glass Foam by Predominantly Direct Microwave Heating of Recycled Glass Waste

¹Sorin Mircea Axinte, ²Lucian Paunescu, ³Marius Florin Dragoescu, ⁴Ana Casandra Sebe

¹Department of Applied Chemistry and Materials Science, University "Politehnica" of Bucharest, Romania;

^{2,3}Daily Sourcing & Research SRL Bucharest, Romania;

⁴Cosfel Actual SRL Bucharest, Romania;

sorinaxinte@yahoo.com

ABSTRACT

The paper presents authors' contribution to the improvement of the manufacturing technique of foam glass using the microwave energy. Due to the physical and mechanical characteristics, this material, obtained by the sintering process of waste glass at high temperature, constitutes a viable replacer of existing similar materials, used especially in construction. Unlike the conventional heating methods used worldwide, the company Daily Sourcing & Research SRL Bucharest tested lately microwave heating techniques in the manufacturing process of foam glass. In the paper it is presented an original method based on the feature of the powder mixture composed by waste glass (over 97 wt.%) and the foaming agent (calcium carbonate) to absorb the microwave energy and convert it to heat since the ambient temperature, using a silicon carbide and silicon nitride (80/ 20 weight ratio) crucible with thin wall (2.5 mm), which allows both a preponderantly direct heating and partially an indirect heating of the material. The main parameters of the process (specific consumption of energy, heating speed, process temperature and duration) were significant improved compared to the previous experiments.

Keywords: Glass foam; Glass waste; Calcium carbonate; Microwave heating; Silicon carbide.

1 Introduction

Microwave heating is a fast, economical and "clean" process, known since the 1930s. However, until the end of the last century, the fields of applicability were very limited. The microwave applications in the household in the food preparation are well known. Industrially, the microwave energy was used only for vulcanization of rubber or for different drying processes. Only in the last decade, it has been experimentally found that many other types of materials can be efficiently heated with microwaves: organics, ceramics, polymers, metals, glass, etc., but the results remained at the level of experiment [1].

One such example is the field of industrial manufacture of glass foams from recycled glass waste. The manufacturing technique involves heating the raw material with a suitable foaming agent at high temperatures (between 700-1200 °C) to obtain its sintering and foaming. The main industrial

manufacturers (Misapor Switzerland, Pittsburgh Corning, etc.) of glass foam, usable as a substitute for existing building materials due to their remarkable physical and mechanical characteristics (mainly, low density, low thermal conductivity, acceptable mechanical resistance), still adopt conventional heating techniques (burning fossil fuels or electrical resistances)[2, 3]. It should be mentioned that even experimental works in this field, in fact many in recent years, are not interested in energy problems, but only technological. The explanation of the mistrust regarding the application of the microwave energy should be sought in the paper [4] published in 1997, which states that the commercial glass is very few microwave susceptible at ambient temperature, the heating becoming very efficient only after 500 °C. Other works also confirm that theoretically, due to the high content of microwave transparent materials (SiO_2 , Al_2O_3) in the glass composition, the heating process should take place with difficulty at ambient temperature due to the low electrical conductivity. Its value increases rapidly with increasing the temperature and simultaneously, the energy efficiency of the microwave heating significantly increases [5-7]. This statement was taken up by J. Hurley in 2003 in a UK market survey [2], which concludes that an industrial glass foam oven should have two distinct zones: a zone with temperatures below 500 °C heated by conventional methods, and one with temperatures above 500 °C microwave powered, which would be an unprofitable technology. In reality, however, due to the inherent presence in the glass composition of some contaminants (Fe_2O_3 , Cr_2O_3), even in small ratios, the microwave heating can occur with normal efficiency starting from the ambient temperature [8].

Knox and Copley's theory was experimentally contradicted [9] by researchers from the Romanian company Daily Sourcing & Research Bucharest, who have recently conducted numerous experiments following the application of microwave heating in the field of glass foam production. The experimental results, published both in Romania and worldwide [9-14], confirmed the viability of the adopted solutions, the foamed products being qualitatively similar to those obtained by conventional methods.

An important aspect of the microwave heating of the powder mixture consisting of glass waste, foaming agent and any other mineral additions, is the difficulty of achieving the integral direct heating through the direct contact between the microwave field and the glass-based material. Several experiments have shown that the direct heating of the microwave glass waste is inadequate to obtain a homogeneous macrostructure in the mass of the foamed product [9]. It is known that the direct microwave heating of any solid material is initiated in its core, where the maximum temperature is reached, the heat being then transferred to the peripheral areas of the material [15]. The heating speed is very high (over 35-40 °C/min) and this fact is completely not indicated for the sintering / foaming process. The speeds recommended in the literature [3, 16] as well as those achieved in the own previous experimental processes are between 5-25 °C/min, varying depending on the type and quantity of the material, the type and weight ratio of the foaming agent, the manufacturing technique adopted, etc. Taking into account the above aspects, Daily Sourcing & Research has tested several types of microwave susceptible ceramic crucibles (silicon carbide, graphite, graphite + silicon carbide, silicon carbide + silicon nitride) as well as different thicknesses of their wall (between 2.5-20 mm). The use of such a crucible changes the type of microwave heating to indirect heating. This system slows down the process of making glass foam, because in the most cases the wall of the crucible absorbs all electromagnetic waves, heats up quickly, and transfers the heat to the material through the well-known conventional heating processes (mainly, thermal conductivity and radiation). Thus, the indirect heating takes place from the outside to the inside.

In the case of crucibles with wall thickness of 2.5-3.5 mm, it was found that the microwaves partially penetrate the ceramic wall, making both its heating and the direct contact with the material subjected to heating. In this way, the core of the material is heated, the heat transfer being carried out from the inside to the outside. On the other hand, simultaneously, the inner surface of the crucible transmits heat inward.

The experiments presented below show the results obtained in a predominantly direct heating case, using a crucible of silicon carbide and silicon nitride in the mass ratio 80/20, with the wall thickness of 2.5 mm.

2 Methods and materials

2.1 Methods

In principle, the production of glass foam from recycled glass waste is based on sintering at high temperature a powder mixture formed from glass waste and a suitable foaming agent. The foaming consists in the release of a gas in the softened mass of the mixture at a temperature close to its melting point, by thermal decomposition or oxidation of the foaming agent. Generally, the most commonly used foaming agents are carbonates (calcium carbonate, sodium carbonate), powder carbon, black carbon, silicon carbide, etc. The carbonates decompose, releasing carbon monoxide or carbon dioxide, while the other named agents oxidize, releasing the same type of gas. Due to the adequate viscosity of the material, the gas bubbles remain blocked in its mass, and by subsequent cooling they form a homogeneous porous structure. The experiment carried out in Daily Sourcing & Research took place in a 0.8 kW-microwave oven, of the type used in the household for food preparation, equipped with only one microwave generator, but adapted for operating conditions at temperatures up to 1200 °C (Figure 1a). The fine-grained powder material was loaded and pressed into a crucible of silicon carbide and silicon nitride in the weight ratio 80/ 20, having a wall thickness of 2.5 mm (Figure 1b). The crucible has the outer diameter of Ø 120 mm and the height of 100 mm and has a lid of a silicon carbide plate with a thickness of 10 mm. The total capacity of the crucible is 986 cm³, allowing a maximum load of the glass-based powder mixture of about 1.28 kg (average density of the pressed mixture being determined experimentally at about 1.3 g/ cm³). The wall of the crucible made of a material with high microwave susceptibility will partially absorb the radiation of the microwave field, while most of them will penetrate it and will come in direct contact with the material subjected to heating. This type of mixed heating (direct and indirect) has the role of increasing the efficiency of heating over the indirect one and, at the same time, of diminishing the undesirable effect of the destruction of the interior structure of the material, which characterizes the direct heating. Because the microwave heating process is inversely oriented compared to the conventional one, i.e. from the inside to the outside, the crucible containing the powder mixture is protected on all its outer surfaces with ceramic fiber mattresses to reduce heat loss outside the system.

The thermal control of the foaming process is based on the indications of a Pyrovar type radiation pyrometer mounted above the oven in its central axis (see Figure 1a). For viewing the upper surface of the material into crucible, holes of about 30 mm both in the upper metal wall of the oven and in the silicon carbide lid and the ceramic fiber mattresses layer that protects the ceramic lid were provided.

Because the foaming agent adopted during the experiment is calcium carbonate, the decomposition reaction that characterizes the foaming process is:



According to the literature [17], the reaction (1) can start at 740 °C and end at less than 900 °C. Generally, the foaming process of soda-lime glass (commercial glass bottle) takes place between 800-900 °C, varying depending on the quantity and quality of the glass waste, the weight ratio of foaming agent, fineness of granulation of materials, etc.

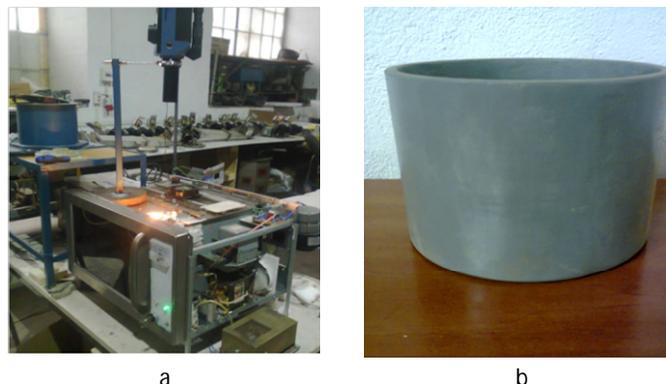


Figure 1. The experimental microwave equipment
a - 0.8 kW-microwave oven; b – ceramic crucible.

2.2 Materials

The raw material used in the experiments was that from the recycling of commercial glass containers. These are soda-lime glasses, with the largest spread between the glass waste. Only the colourless glass wastes were selected. The chemical composition of this type of glass is shown in Table 1.

Table 1. Chemical composition of the colourless soda-lime glass.

SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	MgO	Na ₂ O	Cr ₂ O ₃
71.7	1.9	12.0	0.05	1.0	13.3	0.05

The glass waste was crushed and ground into a ball mill. The grain size of the material was limited to maximum 130 µm following the sieving. Calcium carbonate as a foaming agent was used such as it was purchased from the market, having a very fine grain size below 40 µm. The powder mixture containing glass waste and calcium carbonate was homogenized together with the water addition quantity in a small laboratory device.

2.3 Characterization of the glass foam samples

The main physical, mechanical and morphological characteristics (apparent density, porosity, thermal conductivity, compressive strength, water absorption, hydrolytic stability and microstructural configuration) of the foam glass samples under the conditions described above were investigated in the laboratory in the companies Daily Sourcing & Research and Cosfel Actual as well as in the Department of Applied Chemistry and Materials Science of the University “Politehnica” of Bucharest, using common methods of analysis [18-21].

3 Results and discussion

3.1 Results

Four variants of the composition of the raw material and the foaming agent were tested in the 0.8 kW-microwave oven under the conditions of the predominantly direct heating operation described above. The tests included weight ratios of the glass waste between 97.8-99.4%, the rest being calcium carbonate (0.6-2.2%). Also, an addition of water (10%) was used, which reduces the viscosity of the foam mixture, contributes additionally to the foaming of the material and has the role of binder in pressing it before the thermal treatment begins [22]. The composition of each of the four tested variants is shown in Table 2.

Table 2. Composition of the tested variants.

Variant	Colourless glass waste wt. %	Calcium carbonate wt. %	Water addition wt. %
1	97.8	2.2	10.0
2	98.5	1.5	10.0
3	99.0	1.0	10.0
4	99.4	0.6	10.0

The main functional parameters of the sintering and foaming process corresponding to the four compositional variants are presented in Table 3.

Table 3. Main parameters of the sintering/ foaming process.

Variant	Raw material quantity, g		Process temperature °C	Process duration min	Average heating speed °C/ min	Glass foam quantity g	Index of volume growth	Specific consumption of electricity kWh/ kg
	dry	wet						
1	490	539	832	35	23.2	475	2.40	0.98
2	490	539	830	34	23.8	476	2.30	0.95
3	490	539	829	33	24.5	476	2.25	0.92
4	490	539	828	34	23.8	474	1.80	0.96

According to the data in Table 3, the quantities of dry and wet powder mixture loaded in the crucible were kept at constant values (490 g and 539 g, respectively). In these conditions, the temperature and the duration of the process had slightly significant variations, between 828-832 °C and 33-35 min, respectively. The average heating speed reached high values between 23.2-24.5 °C/ min). The variation of the weight ratio of calcium carbonate (0.6-2.2 wt.%) clearly influenced the index of volume growth of the foamed material, which reached a maximum of 2.40, corresponding to the maximum value of the foaming agent ratio. The specific energy consumption had very low values (below 1 kWh/ kg) compared to other experimental microwave heating processes that use calcium carbonate as a foaming agent (over 2.5 kWh/ kg) made by Daily Sourcing & Research [9 , 10].

The physical, mechanical, and morphological characteristics of glass foams produced by predominantly direct microwave heating are shown in Table 4.

Table 4. Physical, mechanical, and morphological characteristics of glass foams.

Variant	Apparent density g/ cm ³	Porosity %	Compressive strength MPa	Thermal conductivity W/ m-K	Water absorption %	Pore size mm
1	0.18	91.8	1.18	0.040	1.1	1.5-3.0
2	0.20	90.9	1.20	0.042	0.8	1.0-1.6
3	0.19	91.4	1.21	0.043	0.8	0.8-1.5
4	0.20	91.0	1.21	0.044	0.6	0.7-0.9

Table 4 highlights that the glass foam samples produced by predominantly direct microwave heating have characteristics similar to those made by indirect microwave heating, and more so those manufactured by conventional methods. The apparent density and the thermal conductivity, which determine the quality of an insulating material usable in the building construction, have very low values (0.18-0.20 g/ cm³ and 0.040-0.044 W/ m· K, respectively). The compressive strength (1.18-1.21 MPa) is considered acceptable, the water absorption is very low, practically insignificant, and the pore distribution in the sample section is homogeneous.

Figure 2 shows images of the longitudinal sections of the four glass foam samples.

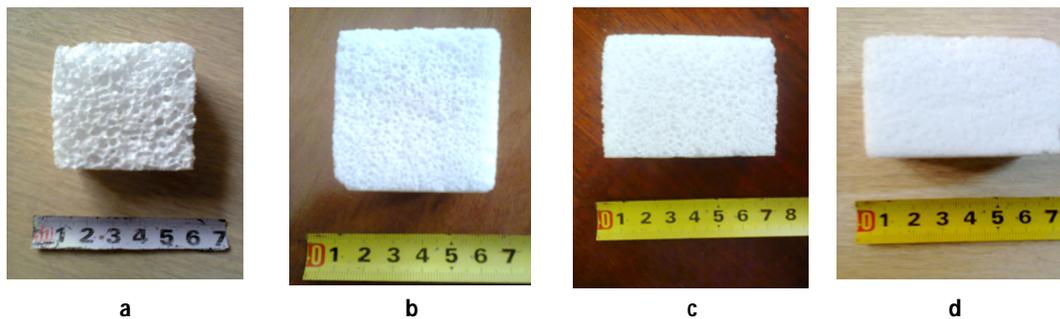


Figure 2. Images of the longitudinal sections of the glass foam samples
a – sample 1; b – sample 2; c – sample 3; d – sample 4.

Images of the samples microstructure identified with a Smartphone Digital Microscope are shown in Figure 3.

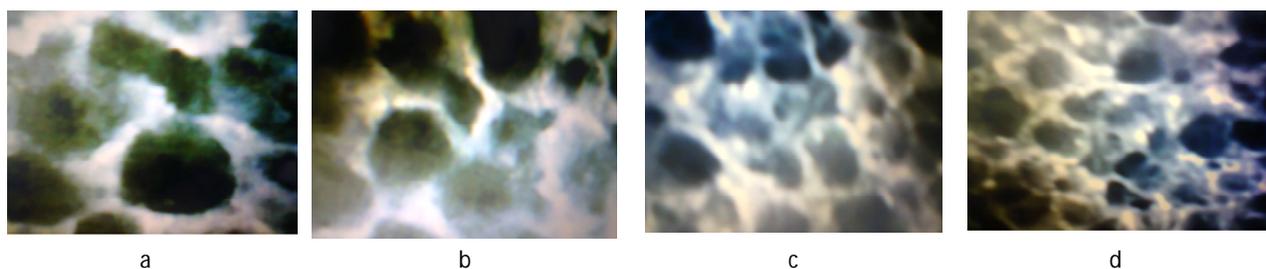


Figure 3. Images of the samples microstructure
a – sample 1; b – sample 2; c – sample 3; d – sample 4.

The tests for determining the hydrolytic stability of samples, using 0.15 ml of 0.01M HCl solution to neutralize the extracted Na₂O, showed that the stability joins in the hydrolytic class 2.

3.2 Discussion

The main objective of the research presented in the paper is to reduce the specific energy consumption in the process of manufacturing glass foam by predominantly direct heating without changing the product characteristics. It should be noted that the literature does not provide information on energy consumption. The only information comes from the market survey [2] referring to an average consumption of 100 kWh/ m³-glass foam (i.e. about 0.40-0.67 kWh/ kg-glass foam, corresponding to the foam density between 0.15-0.25 g/ cm³). The specific energy consumption obtained in experiments, under conditions of a discontinuous operation of the oven, was between 0.92-0.98 kWh/ kg. According to the paper [1], an industrial microwave equipment should allow a high-energy power sources, the use of an unique internal protection feature, an uniform exposure to microwave, contributing to a significant reduction of the specific energy consumption with over 25% comparing to a small experimental equipment.

4 Conclusion

Glass cullet and especially post-consumer glass containers are a waste with a very high annual generation rate worldwide. The recycling of this waste has become a requirement both ecologically and materially due to the efficiency of its reuse in the production of glass foams as replacers of existing building materials on the market or in the manufacture of new glass as a raw material.

The use of the microwave energy is a fast, economical and "clean" technique applied far less in industry. In recent years, the Romanian company Daily Sourcing & Research has made efforts to implement this unconventional technique in the field of glass foam manufacturing.

An unconventional heating technique with high energy efficiency is presented in the paper, being tested under experimental conditions in an adapted 0.8 kW-microwave oven of the type of those very widespread in the household for preparing the food.

Using a low-cost foaming agent in the category of carbonates (calcium carbonate) and, implicitly, a thermal process at relatively moderate temperatures of 800-850 °C as well as a predominantly direct microwave heating technique, low specific energy consumption values (0.92-0.98 kWh/ kg) have been obtained.

Theoretically, by applying this technique to an industrial scale, the energy efficiency could be considerably higher by at least 25%.

The physical, mechanical and morphological characteristics of the glass foams produced under these conditions are almost similar to those made by conventional techniques.

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