

NONCONVENTIONAL TECHNIQUE FOR PRODUCING HIGH MECHANICAL STRENGTH GLASS FOAM FROM GLASS WASTE

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Abstract: In the work experimental results on the manufacture of glass foam with high mechanical strength from glass waste are presented. By replacing the usual conventional energy source with a nonconventional energy (electromagnetic waves) the heating efficiency allows a fast and economical manufacturing process. Calcium carbonate as a foaming agent and an addition of sodium silicate (aqueous solution) as a binder were used. By their physico-mechanical and morphological features (0.40-0.66 g/ cm³ the apparent density, 0.054-0.113 W/ m·K the thermal conductivity, 2.2-6.3 MPa the compressive strength, below 1.2 % the water absorption and under 2 mm the pore size), the foams are appropriate for their use as replacer of existing similar building materials on the market.

Keywords: glass foam, glass waste, sodium silicate, microwave, compressive strength, pore size

1. INTRODUCTION

Glass foam is a vitreous material with a porous macrostructure obtained by a thermal treatment at high temperatures (700 – 1200 °C, or even higher) of glass waste by addition of a foaming agent (silicon carbide, calcium carbonate, calcium sulphate, black carbon, etc.). The basic principle is the generation of a gas inside the powder mixture of raw material, which is released and blocked in the viscous mass of the heated material, producing by cooling a porous structure.

The properties of the glass foam are remarkable, combining the low density and thermal conductivity with resistance and stability to many mechanical, thermal, chemical agents or to the aggression of bacteria, insects, rodents, etc. For this reason, glass foams represent ideal solutions for their using as thermal or acoustic insulations or as suitable materials such as lightweight aggregates, pavements, outer panels, drainages, infrastructure foundation, sport grounds, road construction, etc. [1].

Currently, several glass foam types are industrially made, of which the most important are “Technopor” (made by Misapor Switzerland) and “Foamglas” (made by Pittsburgh Corning). The “Technopor” products have the compressive strength up to 6 MPa, being usable as building materials, which require high mechanical strength.

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The “Foamglas” products have lower compressive strength (up to 2.75 MPa) and are used as lightweight aggregates [2].

The literature presents also experimental results obtained in the world in the field of dense glass foam production. A manufacturing technique of this material with apparent density 0.46 g/cm^3 , high compressive strength (over 5 MPa) and low thermal conductivity ($0.36 \text{ W/m}\cdot\text{K}$) is described in [3]. Coal ash and waste glass are used as raw material in the weight ratio of 40/ 60. The foaming agent is calcium carbonate (0.5 wt. %) and borax is used as fluxing agent in the weight proportion of 30 %. An innovative manufacturing method of glass foam for panels of the building front with risk of terrorist attack is presented in an US Patent [4]. The material is made of silicate waste mixed with a foaming agent (silicon carbide, black carbon, carbonates or sulphates). The product, with the apparent density between $1\text{--}1.28 \text{ g/cm}^3$, can absorb much of the thermal shock of the blast. The paper [5] presents the influence of the use of soluble sodium silicate (up to 40 %) mixed with glass waste on the mechanical strength of glass foam. Calcium carbonate (1 %) was used as foaming agent. The process took place at $850 \text{ }^\circ\text{C}$. The apparent density of glass foam was 0.5 g/cm^3 and the bending strength reached 17.5 MPa. The ash of the bituminous shale and the cathode tube glass out of use were used with addition of calcium carbonate (5%) as foaming agent to produce glass-ceramic foam [1, 6].

The foaming took place at $900 \text{ }^\circ\text{C}$, followed by crystallisation treatment at $800 \text{ }^\circ\text{C}$. The porous product had the apparent density 0.35 g/cm^3 and the tensile strength 3 MPa. Another technique of obtaining a glass-ceramic foam with the apparent density 0.39 g/cm^3 and the compressive strength 2.4 MPa is described in [7]. The sintering/foaming temperature was $1140 \text{ }^\circ\text{C}$. The raw materials used were waste quartz sand (55%), coal gangue (40 %) and an unspecified additive with foaming role. A glass-ceramic foam from glass waste (50-65 %), copper-rich steelmaking slag (32-47 %) and silicon carbide (3%) as foaming agent was obtained by sintering at $800\text{--}1000 \text{ }^\circ\text{C}$. The compressive strength of the foamed material reached 9 MPa [8]. Another cellular product with high compressive strength (over 5 MPa) was obtained [9] from jarosit (40-70%), a hazardous iron-rich waste resulting from zinc hydrometallurgy, granite and mud residues (20-40 %) and glass waste (10-40 %). The sintering temperature was very high ($1400\text{--}1450 \text{ }^\circ\text{C}$).

Unlike the conventional methods applied in the world in the heating process for manufacturing glass foam, the results obtained in similar processes, described in the current paper, are based on the use of the microwave energy. The advantage of the application of this advanced technique is the achievement of the direct heating only on the material. The process is rapid, economical and environmentally friendly. Though the microwave heating system is currently applied in the domestic field, this is not yet industrially used than at a very low level [10].

The Romanian company Daily Sourcing & Research has already a good experience in the manufacturing area of glass foams in microwave field, experimental results being presented in literature [11, 12].

2. METHODS AND MATERIALS

2.1. Methods

The thermal process of decomposition of calcium carbonate takes place at high temperature between $730\text{--}900 \text{ }^\circ\text{C}$ [13]. The gas released after this process (the carbon dioxide) should meet a mass with appropriate viscosity of the powder mixture, the bubbles remaining stuck. By the gradual increasing of the material pressure, its expansion is forced and by cooling a porous structure is formed. The calcium oxide, released too by decomposition, enters into the softened glass mass and contributes to the formation of a melting with low viscosity favourable for the material foaming [1, 2].

Due to the importance of wetting the powder mixtures of raw material and foaming agent [1, 14] to obtain a structure with low pore size uniformly distributed, the solution of using the water glass or sodium silicate, as an aqueous solution, was adopted. This is a useful binder of solid materials such as vermiculite and perlite, containing 8.5 wt.% Na_2O , 27.8 wt.% SiO_2 and having the concentration of 36.8%. Water glass addition in the powder mixture of waste glass and calcium carbonate homogenizes its chemical composition and improves the most significant technological properties. Due to the chemical reaction between water glass and the surface of waste glass particles, sodium silicates containing chemical bound water are formed. This water is released at high temperatures ($600\text{--}620 \text{ }^\circ\text{C}$). Water glass addition facilitates the foaming of powder mixture, increases the quantity of the vitreous phase and decreases the propensity of glass for crystallization [15, 16].

The experimental making process of glass foam was carried out in the company Daily Sourcing & Research of Bucharest on a 3 kW-microwave reactor (Figure 1), previously designed and achieved for other experimental heating processes. The powder mixture has been loaded and pressed into a metal crucible, provided with a cover having a central hole for viewing the heated material. The crucible containing the mixture was introduced in the inner space of a silicon carbide cylindrical tube, whose wall has a thickness of 20 mm and is placed inside the cavity of the reactor. The silicon carbide belongs to the category of the microwave susceptible materials, being appropriate for the fast heating of the tube under the effect of the microwave irradiation. The use of the silicon carbide tube ensures the indirectly heating of the glass-based material, avoiding the direct contact between the microwave field and the glass. Previous experiments have shown the destructive influence of microwaves on the glass macrostructure [17]. The control of the heating process was performed with a Pyrovar type radiation pyrometer, mounted above the reactor cavity containing the metal crucible.

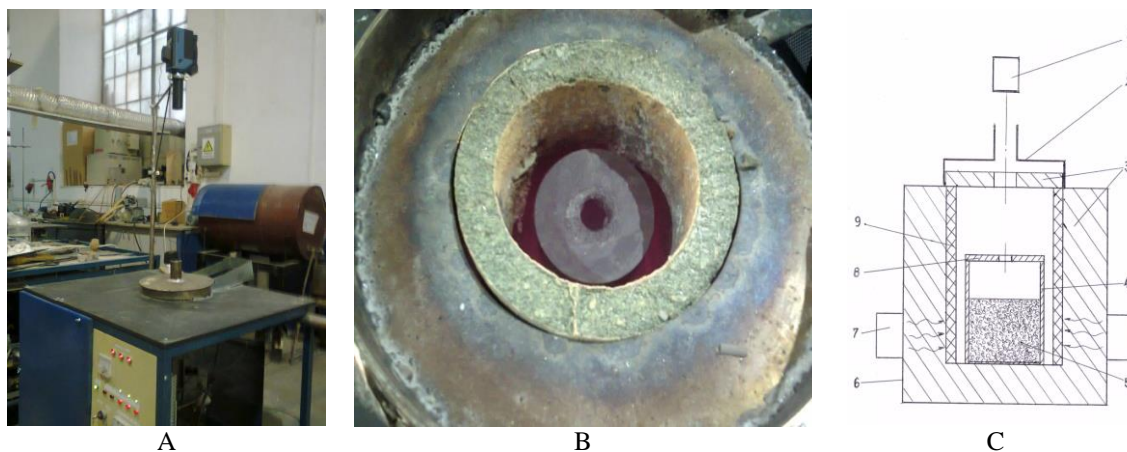


Fig. 1. The experimental microwave equipment: A – overall image of the microwave reactor; B – positioning of the metal crucible into the silicon carbide tube; C – the constructive and functional scheme of the equipment: 1 – radiation pyrometer; 2 – metal cover; 3 - ceramic fiber; 4 – metal crucible; 5 – pressed powder material; 6 – reactor cavity; 7 – microwave generator; 8 – metal crucible cover; 9 – silicon carbide tube.

2.2. Materials

The raw material used in experiments was a mixture of colorless (70 wt.%) and green (30 wt.%) bottle glass waste. The chemical composition of these wastes is indicated in Table 1.

Table 1. Chemical composition of the glass waste.

Glass waste	SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	MgO	Na ₂ O	K ₂ O	Cr ₂ O ₃	SO ₃
Colorless [18]	71.7	1.9	12.0	0.05	1.0	13.3	-	0.05	-
Green [19]	70.5	1.8	10.2	0.45	2.7	13.0	0.45	0.25	0.25

The recycled bottle glass waste was crushed and ground within a laboratory electrical device, its granulation size being under 150 μm. Calcium carbonate, having a very fine grain size under 40 μm, was used without further processing. After mixing and homogenization of the raw material within a small laboratory device, sodium silicate (also called water glass), an aqueous solution with the concentration of 36.8 %, was added over the powder mixture. Then, the wet mixture was blended. The load was pressed into the metal crucible at about 10 MPa.

2.3. Characterization of the glass foam specimens.

The glass foam specimens, made by the sintering and foaming experimental process, were tested in laboratory to identify the physico-mechanical and microstructural characteristics. The apparent density was measured by the gravimetric method [20]. The porosity was calculated by the comparison method of true and apparent densities of material, experimentally measured [21]. The water absorption of the specimen was determined by the method of its water immersion. The thermal conductivity was measured by the guarded-comparative-longitudinal heat flow technique. The mechanical strength was determined within an uniaxial press. The hydrolytic stability of the specimens was measured according to the standard procedure ISO 719:1985 [22, 23].

3. RESULTS AND DISCUSSION

3.1. Results

The main objective of experimentation was to determine the influence of the addition of sodium silicate (or water glass) on the characteristics of glass foams under the conditions of microwave irradiation. The raw material was soda-lime glass waste (between 99.0–99.5 wt. %) and calcium carbonate (between 0.5–1.0 wt. %) was adopted as foaming agent. Sodium silicate was successively added in mass ratios between 11–32 %. The composition of the powder mixture is indicated in Table 2.

Table 2. The powder mixture composition.

Specimen	Raw material composition, wt. %		
	Bottle glass waste	Calcium carbonate	Sodium silicate addition
1	99.0	1.0	11.0
2	99.0	1.0	25.3
3	99.1	0.9	25.3
4	99.2	0.8	26.2
5	99.3	0.7	28.0
6	99.5	0.5	32.0

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

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

Variant	Raw material amount, g	Glass foam amount, g	Process temperature, °C	Heating/cooling average rate, °C/ min	Index of volume swelling	Specific energy consumption, kWh/ kg
1	388.5	373.0	847	14.0/ 6.7	2.3	2.10
2	438.6	412.0	850	13.9/ 7.0	2.4	1.94
3	438.6	412.3	850	13.9/ 7.5	2.4	1.94
4	441.7	415.2	848	14.1/ 6.8	2.3	1.86
5	448.0	421.0	851	13.8/ 6.9	2.2	1.90
6	462.0	434.0	853	13.5/ 7.3	2.0	1.90

Corresponding to the data from Table 3, the raw material amount varied between 388.5–462.0 g due to the different additions of sodium silicate over the constant amount of glass waste of 350 g. The process temperature and the average heating rate had relative constant values of 847–853 °C and 13.5–14.1 °C/min, respectively. The specific energy consumption (between 1.86–2.10 kWh/kg) was significantly influenced by the glass foam quantity. By comparison with sintering and foaming processes of glass waste with similar ratio of calcium carbonate as foaming agent and without addition of sodium silicate, it was found a visible increase in the volume expansion of the material by the addition of this aqueous solution. In this case, the index of volume swelling had values between 2.0–2.4.

The physico-mechanical and structural characteristics of the glass foam are presented in Table 4.

Table 4. Characteristics of the glass foam specimens.

Specimen	Apparent density, g/cm ³	Porosity, %	Compressive strength, MPa	Thermal conductivity, W/ m·K	Water absorption, %	Pore size, mm
1	0.40	81.8	2.2	0.054	1.2	1.0–2.0
2	0.51	76.8	2.8	0.075	0.9	1.0–1.6
3	0.53	75.9	2.9	0.080	0.8	1.0–1.5
4	0.58	73.6	4.1	0.095	0.5	0.9–1.4
5	0.63	71.4	4.9	0.106	0.4	0.8–1.2
6	0.66	70.0	6.3	0.113	0.4	0.7–1.1

From the data contained in Table 4, the high increasing degree of the compressive strength values (from 2.2 to 6.3 MPa) with the increase of the sodium silicate ratio (from 11 to 32 wt. %) is remarkable. The apparent density and thermal conductivity increase significantly (between 0.40–0.66 g/cm³ and 0.054–0.113 W/m·K, respectively), but not as severe as the compressive strength. Implicitly, the porosity values decrease (from 81.8 to 70.0 %) with the increase of the apparent density. The water absorption is very low, under 1.2 %.

Pictures of longitudinal section of the glass foam specimens are shown in Figure 2.

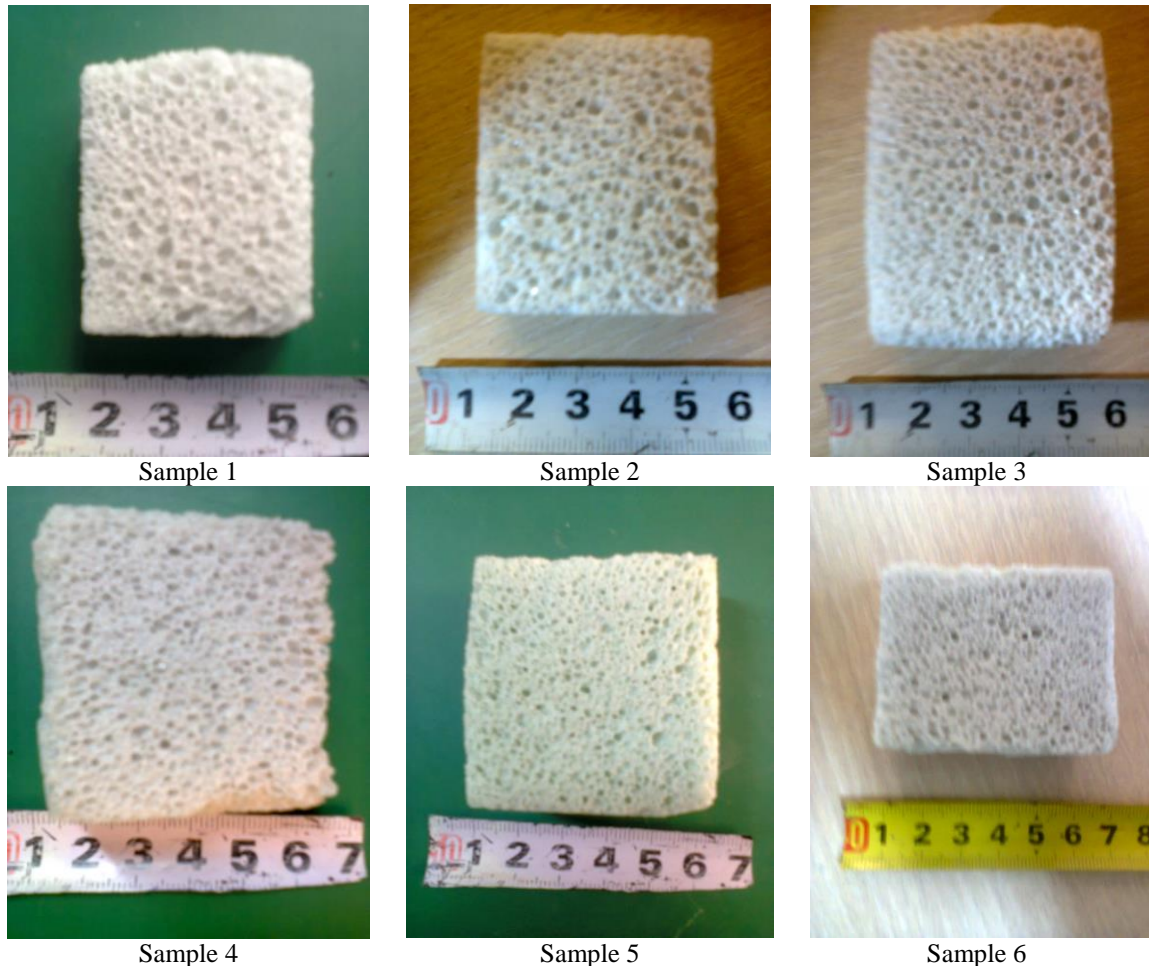


Fig. 2. Longitudinal section of the glass foam specimens.

According to these pictures, the specimens microstructure in longitudinal section has a homogeneous aspect, the pores being uniformly distributed. The pore size decrease from the specimen 1 (with 11 wt. % sodium silicate addition) to the specimen 6 (with 32 wt. % sodium silicate addition).

The microstructural analysis of the glass foam specimens was carried out with a Smartphone Digital Microscope. The pores sizes in the longitudinal section could be determined (Table 4) with this device. Microstructural images corresponding to specimens 5 and 6, characterized by the highest compressive strength values, are shown in Figure 3.

The hydrolytic stability of specimens was carried out according to the ISO 719 instructions. The procedure involved the use of a 0.01M HCl solution for the neutralization of the extracted Na₂O. The extracted Na₂O quantity was between 38-59 µg, the specimens being classified in the second hydrolytic class.

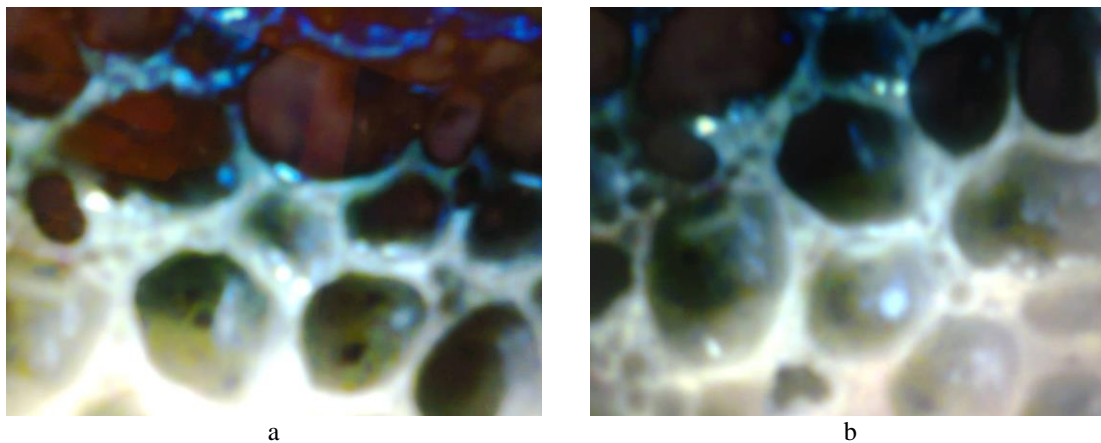


Fig. 3. Microstructural images of the glass foam specimens
a – specimen 5; b – specimen 6.

3.2. Discussion

Previous own tests have clearly highlighted that the appropriate heating rate for obtaining a homogeneous structure of glass foam should be in the range 10–15 °C/min. Under condition of using the microwave energy, this desideratum is only possible if the material is indirectly heated. This involves placing a compact wall of a microwave susceptible material (e.g.: silicon carbide) between the microwave emission source and the heated material. In this way, the wall is heated quickly and efficiently by absorbing the electromagnetic waves, then transferring the heat to the material placed or not in a metal crucible by radiation and eventually thermal conductivity.

The comparison of the mechanical and physical features of glass foams produced in the microwave field from glass waste with calcium carbonate as a foaming agent and the addition of sodium silicate (aqueous solution) and those of glass foams previously manufactured by the same nonconventional heating method, with calcium carbonate and water addition as a binder, showed major differences. Thus, the compressive strength of the specimens with sodium silicate, up to 6.3 MPa, was much higher than that of the reference specimens (1.12-1.22 MPa). Instead, the physical features were slightly affected. The apparent density was significantly higher (up to 0.66 g/cm³) than that of specimens without the addition of sodium silicate (0.15-0.19 g/cm³). Also, the thermal conductivity had much higher values (0.054-0.113 W/m·K) than the values of the same characteristic of the reference specimens (0.034-0.040 W/m·K) [22]. Porous products with high mechanical characteristics are suitable for use as building materials requiring resistance at high mechanical stress such as outer panels, pavements, drainages, foundations, sport grounds, road construction, lightweight aggregates, etc. [1].

By comparing the main physico-mechanical features of glass foam obtained by the addition of sodium silicate and using the nonconventional heating method with those of glass foams (or glass-ceramic foams) made by conventional heating methods, there is a good similarity between these products. Differences between the compressive strength values are due to the nature of the silicate wastes used in the process and not the heating technique.

Although the specific energy consumption of the two types of heating processes cannot be compared due to lack of information in literature, theoretically, the advantage of the nonconventional method is obvious. For the microwave oven used for tests, the hourly energy consumption is 0.8 kWh. Replacing the microwave generator with a natural gas burner to achieve the same thermal effect would be equivalent to a fuel consumption of only 0.08 Nm³/h, meaning a negligible consumption that would not allow the material heating up to over 800 °C in an hour.

4. CONCLUSIONS

The paper aimed to develop a manufacturing technique of building materials with insulating properties and, in the same time, with high mechanical strength, from recycled glass waste, calcium carbonate as a foaming agent and addition of aqueous solution of sodium silicate, using the microwave energy.

Six compositional variants, where the bottle glass waste ratio varied between 99.0–99.5 wt. %, the calcium carbonate ratio was reduced from 1.0 to 0.5 wt. % and the sodium silicate addition ratio was increased from 11.0 to 32.0 wt. %, were tested.

The experiments were carried out on an existing experimental microwave equipment in the Romanian company Daily Sourcing & Research.

The effect of the sodium silicate addition on the glass foam characteristics was identified comparing similar products made in microwave field with and without sodium silicate. Thus, the compressive strength reached maximum 6.3 MPa than 1.12-1.22 MPa, in the case of foams produced without sodium silicate. Instead, the apparent density and the thermal conductivity were slightly affected, their values being higher compared to the reference products.

The high values of the mechanical strength, together with the relative low values of apparent density and thermal conductivity recommend the glass foams obtained by additions of sodium silicate up to 32 wt. % as insulating materials for construction that require resistance at high mechanical stress such as outer panels, pavements, drainages, foundation, sport grounds, road construction, lightweight aggregates, etc.

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