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PREPARING GLASS FOAM AT ROOM TEMPERATURE BY A NONCONVENTIONAL FOAMING TECHNIQUE

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ABSTRACT: The paper presents an innovative technique for manufacturing glass foams from recycled glass waste at room temperature by using aluminum powder and an aqueous solution of calcium hydroxide. The physical, thermal, mechanical and morphological characteristics of the foamed products (porosity between 82.9-86.2%, thermal conductivity in the range 0.061-0.081 W/m·K and compressive strength between 1.15-1.28 MPa) were similar to those of the foams manufactured by sintering at high temperatures. Glass foams produced by this new technique are suitable for use as thermal insulation materials for buildings.

KEYWORDS: glass foam, room temperature, glass waste, aluminum powder, foaming agent, high effectiveness

1. INTRODUCTION

The glass foams are porous, lightweight materials, with low thermal conductivity and high mechanical strength, non-deformable, non-toxic, resistant to fire and moisture, resistant to the freeze-thaw cycle, resistant to attack of rodents, insects, bacteria and acids [1]. Their manufacture on an industrial scale began in the last two decades of the 20th century being used as thermal insulation for interior and exterior walls of buildings, insulation in the perimeter of buildings, foundations, roof gardens, road and railway construction, insulation of underground pipe for thermal agent transport or underground tanks, sports fields, drainages, etc. [1, 2].

The conventional techniques for making glass foams from finely ground mixtures of glass waste incorporating small proportions of solid or liquid foaming agent take place at high temperatures (750-1150 °C) at which the foaming agent releases a gas or gaseous compound after a chemical reaction of decomposition or oxidation, which is blocked in the viscous mass, thermally softened of the glass waste [1]. The heating methods used worldwide are conventional (electrical resistances or burning fossil fuels) [3]. Experimentally, the nonconventional method of microwave heating has been tested on a small scale in the last four years in Romania [4].

It is known that polyurethane foams are manufactured at room temperature [5], but the strength of these products also used as thermal insulation is incomparably lower than that of glass-based foam products. However, a new experimental method of making porous concrete using a very fine aluminum powder in aqueous solution of calcium hydroxide [6] was adopted for experimentation the glass waste foaming practically at room temperature [7]. This innovative technique, applicable at room temperature is very attractive for the field of glass foam manufacturing.

The current work refers to experimental results obtained by the team of authors for the laboratory manufacture of glass foams applying the technique mentioned above.

2. METHODS AND MATERIALS

2.1 Methods

Unlike foaming agents commonly used in the manufacture of glass foams (black carbon, coal, glycerol, calcium carbonate, sodium carbonate, silicon carbide, silicon nitride, etc. [1]) which release gases at high temperatures, the aluminum powder in aqueous solution of calcium hydroxide ($Ca(OH)_2$) reacts with water releasing hydrogen at room temperature. This foaming agent recently tested in the manufacture of porous concrete has been adopted for various experimental tests in the manufacture of glass foams.

A fine aluminum powder (below 15 μm) with very high purity (over 99.9%), obtained by spraying the aluminum melt with converging nitrogen jets at high speeds, was incorporated (between 1.0-2.5 wt.%) into a finely ground powder (below 100 µm) of soda-lime silica glass waste (between 46.3-57.9 wt.%). The mixture of powder solid materials was completed with $Ca(OH)_2$ powder (between 0.5-0.9 wt.%) to remove the thin layer of Al_2O_3 from the surface of fine aluminum particles and initiate the hydration reaction of aluminum. Also, as a binder and foam stabilizer [8], carboxymethyl cellulose (between 2-4 wt.%) was added. Distilled water (between 38.6-46.3 wt.%) was added into the stainless steel cylindrical vessel (Figure 1) with the powder mixture to form a sludge.

Figure 1. The stainless steel cylindrical vessel

The mechanism of the hydrogen release process for scattering gas bubbles in the mass of glass-based sludge is as follows. Initially, there is a thin layer of Al_2O_3 on the surface of fine aluminum particles. The $Ca(OH)_2$ powder is relatively insoluble in water. Its acid dissociation constant is high enough that its aqueous solutions are basic, containing Ca^{2+} and OH^- ions. The $Ca(OH)_2$ aqueous solution has a safer pH even at the saturation level of 12.6. Solid by-products such as boehmite (AlOOH), bayerite ($Al(OH)_3$) and aluminum tetrahydroxide ($Al(OH)_4$) are successively formed and katoite ($Ca_3Al_2(OH)_{12}$) and gaseous hydrogen finally result. As in the case of foaming the glass waste with common foaming agents, the released hydrogen remains blocked in the viscous mass of the sludge in the form of gas bubbles. By solidifying the material, they turn into pores distributed throughout the volume of the product, constituting a specific porous structure. The passive layer of Al_2O_3 on the surface of the aluminum particles prevents the reaction between water and aluminum. The chemical reactions that take place at room temperature in this medium [6] are:

$$Al_2O_3 + H_2O = 2AlOOH \quad (1)$$

$$AlOOH + H_2O = Al(OH)_3 \quad (2)$$

$$Al(OH)_3 + OH^- = Al(OH)_4 \quad (3)$$

$$3Ca^{2+} + 2Al(OH)_4 + 3H_2O =$$

$$Ca_3Al_2(OH)_{12} + 6H_2 \quad (4)$$

To prevent the solidification of the glass-based sludge during the process and to favor the hydration reaction, the mixture is continuously stirred. When the material begins to expand, the stirring is stopped, its initial volume continuing to increase until the end. The product microstructure is typical for a glass foam.

2.2 Materials

The basic raw material was commercial post-consumer packaging bottle (soda-lime silica glass) consisting of a mixture of colorless (10 wt.%), green (45 wt.%) and amber (45 wt.%) glass with the chemical composition [9] indicated in Table 1.

Table 1. Chemical composition of the glass

Component	Glass type, wt.%		
	Colorless	Green	Amber
SiO_2	71.7	71.8	71.1
Al_2O_3	1.9	1.9	2.0
CaO	12.0	11.8	12.1
Fe_2O_3	-	-	0.2
MgO	1.0	1.2	1.1
Na_2O	13.3	13.1	13.3
K_2O	-	0.1	0.1
Cr_2O_3	0.05	0.09	-
SO_3	-	-	0.05
Other oxides	0.05	0.01	0.05

The glass was selected by color from a recycled batch of post-consumer bottle. After this operation it was broken, ground in a ball mill and sieved at a grain size below 100 µm in the Romanian company Bilmetal Industries SRL Popesti Leordeni-Ilfov.

The aluminum powder was obtained by a spraying method of molten aluminum with high speed nitrogen jets in an own conception installation of the Romanian company Daily Sourcing & Research. The powder aluminum had a very high purity (over 99.9%), the grain size being below 15 µm.

Commercially available fine $Ca(OH)_2$ powder (over 95% purity) was used in aqueous solution, where it dissociates into Ca^{2+} and OH^- contributing to hydrogen gas release reactions.

Carboxymethyl cellulose (known as CMC) in powder state was purchased on the market being used as a binder and foam stabilizer.

2.3 Characterization of the glass foam samples

The physical, thermal, mechanical and microstructural characterization of the glass foam samples obtained by foaming the glass waste at room temperature was performed by current analysis methods. The apparent density was measured by the gravimetric method [10] and the porosity was calculated by the comparison method between the porous sample density (apparent density) and the density of the same material type in compact state (true density) [11]. The compressive strength was determined using a TA.XTplus Texture Analyzer and the thermal conductivity was measured by the guarded-comparative-longitudinal heat flow (ASTM E1225-04 standard). The water absorption was determined by the water immersion method (ASTM D570 standard) and the samples microstructure was examined with an ASONA 100X Zoom Smartphone Digital Microscope.

3. RESULTS AND DISCUSSION

3.1 Results

The experimental process of producing glass foam at room temperature took place in a cylindrical stainless steel vessel loaded with solid materials in powder form over which the distilled water was slowly poured in the weight ratios indicated in Table 2. The amount of the dry solid material was 250 g in all experimental variants tested. A stainless steel metal wand was used to manually stirrer the sludge inside the vessel during the process.

Table 2. Experimental variants

Variant	**Glass waste wt.%**	**Aluminum powder wt.%**	**$Ca(OH)_2$ powder wt.%**	**Carboxymethyl cellulose wt.%**	**Distilled water wt.%**
1	46.3	2.5	0.9	4.0	46.3
2	49.8	2.0	0.75	3.3	44.15
3	53.0	1.5	0.6	2.6	42.3
4	57.9	1.0	0.5	2.0	38.6

According to the data in Table 2 regarding the proportions of raw materials used for the manufacture of glass foam at room temperature, weight ratios were used approximately within the limits applied to the experimental manufacture of porous concrete [6]. The ratio between the powder glass waste and the distilled water added to form a sludge varied between 1-1.5. The weight ratio between the powder aluminium and glass waste was in the range 0.0173-0.054 and the ratio between the powder $Ca(OH)_2$ and the powder aluminium was between 0.36-0.50.

The main functional parameters of the process are presented in Table 3.

Table 3. Main functional parameters of the experimental process

Parameter	**Variant**			
	1	2	3	4
Dry/wet raw material (g)	250/ 365.8	250/ 360.4	250/ 355.8	250/ 346.5
Process temperature (ºC)	25	25	25	25
Process time (min)	17	17	16.5	16.5
Index of volume growth	3.0	2.8	2.6	2.4
Glass foam amount (g)	245.0	247.2	248.3	249.8

The experimental process according to Table 3 had a required duration of 16.5-17 min, during which the initial volume of glass-based raw material grew 2.4-3 times by expansion. The process temperature was 25 ºC, confirming that the manufacture of glass foam occurs at room temperature.

The main physical, thermal, mechanical and morphological characteristics of glass foam samples are shown in Table 4.

Table 4. Main physical, thermal, mechanical and morphological characteristics of glass foam samples

Characteristic	**Variant**			
	1	2	3	4
Apparent density (g/cm^3)	0.26	0.29	0.31	0.33
Porosity (%)	86.2	84.8	83.8	82.9
Thermal conductivity (W/m·K)	0.061	0.069	0.075	0.081
Compressive strength (MPa)	1.15	1.21	1.25	1.28
Water absorption (vol.%)	2.3	2.5	2.4	2.7
Pore size (mm)	2.2-4.2	1.5-2.5	1.2-2.3	0.8-1.2

According to the data in Table 4, the glass foam samples characteristics are similar with those of glass foam made by conventional foaming techniques.

The apparent density and thermal conductivity that determine the glass foam thermal insulation property have low values (0.26-0.33 g/cm^3 and 0.061-0.081 W/m·K, respectively). Also, the samples porosity was high (of over 82.9%). The compressive strength had relatively low but acceptable values (between 1.15-1.28 MPa) typical for light foam products used as thermal insulation in building construction. The water absorption in the mass of glass foam was within the usual limits of conventionally manufactured products (2.3-2.7 vol.%).

Longitudinal section appearance of the glass foam samples experimentally made at room temperature is presented in Figure 2. A large table example is presented in Table 2.

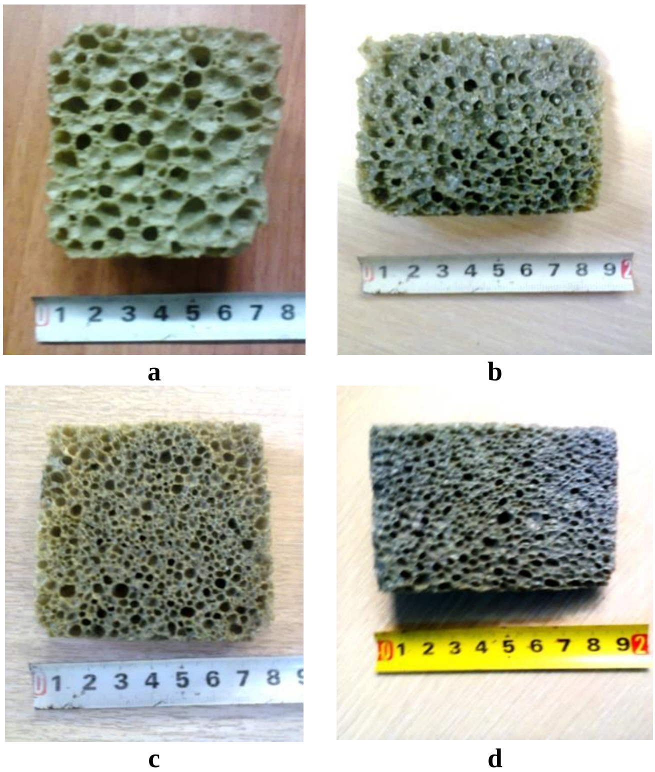

Figure 2. Longitudinal section of the glass foam samples
a – sample 1; b – sample 2; c – sample 3; d – sample 4.

Pictures of the microstructural configuration of the four glass foam samples are shown in Figure 3.

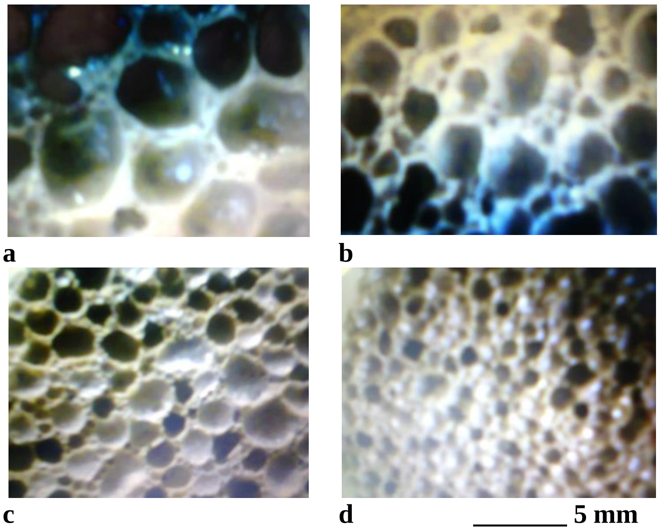

Figure 3. Microstructural configuration of the glass foam samples
a – sample 1; b – sample 2; c – sample 3; d – sample 4.

Generally, the microstructures are homogeneous with a uniform pore distribution. The pore size varies from 0.8-1.2 mm (variant 4) to 2.2-4.2 mm (variant 1) being influenced by the weight proportion of the foaming agent (aluminum powder), which decreases from the maximum value of 2.5 wt.% (variant 1) up to 1.0 wt.% (variant 4). From a microstructural point of view, the glass foam samples obtained by the manufacturing method at room temperature also correspond to the requirements of this type of porous material.

3.2 Discussion

According to the literature, the use of powder metallic aluminum as a foaming agent in the manufacture of porous concrete is an experienced method that provides good results. Given that the mechanism of the hydrogen release process (with a major role in foaming) under the conditions described above does not require heat, the reactions taking place at room temperature, this method, applied to concrete, has already been tested on a small scale [7] in case of foaming the glass waste.

The current paper mentions that the fine aluminum powder used in experiments was produced by the team of authors by an original technique made in the company Daily Sourcing & Research.

The experimental results presented in the paper showed that all the tested variants led to obtaining appropriate glass foams in terms of quality, similar to those manufactured by sintering at high temperature. Obviously, this manufacturing technique is cheaper and high effectiveness due to energy saving, although the aluminum powder is a foaming agent with higher costs compared to foaming agents commonly used in foaming glass.

4. CONCLUSION

An innovative technique for making glass foam by foaming glass waste at room temperature using metallic aluminum powder in an aqueous solution of $Ca(OH)_2$ has been experimented on a small scale, by extending the method applied to porous concrete.

The aluminum powder was obtained by an original method in an own conception installation of the Romanian company Daily Sourcing & Research. The powder aluminum had a very high purity (over 99.9%), the grain size being below 15 µm.

The mechanism of the process of releasing gaseous hydrogen following chemical reactions in aqueous medium does not require heat, being a cheap process, interesting for the field of glass foam manufacturing.

The raw material loaded into a cylindrical stainless steel vessel was composed of finely ground glass waste, aluminum powder, $Ca(OH)_2$ powder in an aqueous solution, carboxymethyl cellulose as a binder and foam stabilizer and distilled water.

The distilled water was added to the mixture of solids forming a glass-based sludge, which was continuously stirred with a metal rod for 16.5-17 min, until the material expanded completely in the vessel 2.4-3 times.

The glass foams produced by the foaming technique at room temperature (25 ºC) had the apparent density of 0.26-0.33 g/cm^3, porosity of 82.9-86.2%, thermal conductivity between 0.061-0.081 W/m·K, compressive strength of 1.15-1.28 MPa and water absorption of 2.3-2.7 vol.%.

From a microstructural point of view, the glass foam samples obtained at room temperature corresponded to the requirements of this type of porous material. The microstructures were homogeneous with a uniform pore distribution. The pore size varied from 0.8-1.2 mm to 2.2-4.2 mm being influenced by the weight proportion of the foaming agent (aluminum powder).

The characteristics of glass foams produced by the foaming method at room temperature are similar to those conventionally manufactured. Foam products are suitable for use as thermal insulation materials for buildings.

5. REFERENCES

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