NONCONVENTIONAL METHOD OF COLD PREPARING THE CELLULAR GLASS

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ABSTRACT: The preparation at room temperature of cellular glass from recycled residual glass and fly ash was made adopting a manufacturing method recently applied in the case of geopolymer foam. Sodium perborate in aqueous solution as a blowing agent and detergent as a surfactant have been experimentally tested in several versions. The optimal cellular glass product was obtained by mixing 2.6 % sodium perborate, 0.2 % surfactant, and 7.6 % water addition. The characteristics of this expanded product were: density of 0.65 $g\cdot cm^{-3}$, heat conductivity of 0.134 $W\cdot m^{-1}\cdot K^{-1}$, and compression resistance of 1.2 MPa, being satisfactory for its application as heat insulating product in construction. The main advantage of this method was the product manufacture in satisfactory quality conditions without supplementary energy consumption and greenhouse gas emissions (CO_2) into the atmosphere, like in the case of the usual manufacturing cellular products at high temperature.
KEYWORDS: cellular glass, room temperature, sodium perborate, surfactant, thermal insulation material.

1. INTRODUCTION

Recycling different types of waste (plastics, metals, glass, rubber, etc.) generated annually at a high rate has become an essential concern for humanity since the last decades of the previous century. On the one hand, the hydrocarbon crisis triggered in the mid-1970s and on the other hand, the excessive emissions of greenhouse gases causing the slow, but progressive destruction of the protective ozone layer, observed at the end of the millennium, were the basis of alert that constitutes the current period trend.

Residual waste comes first from consumed commercial drinking bottle and window glass from the demolition of buildings. Of the annual world glass production of about 130 million tons, 48 % are driking bottle and 42 % is window glass [1]. Glass recyclation in the EU represents only 21 % (27 million tons) of which 8.6 million tons constitutes container glass, according to The European Container Glass Federation-FEVE [2].

Usually, the recycled glass is mechanically processed (broken, finely ground and sieved), mixed with a blowing agent and possibly other mineral additives and subjected to the heat treatment of sintering/expanding in an industrial conventional heating furnace with belt conveyor. Cooling down of the foam product is done slowly into the oven, except for the manufacture of cellular glass gravel, which requires slightly forced cooling to create internal tensions in the material and facilitate the detachment of pieces with typical sizes. Cellular glasses thus produced are adequate as insulation materials in construction [3].

The current technique for the production of cellular glass using finely ground recycled glass waste as raw material is based on the diffusion of a gas in the soaked powder as a result of the decomposition or reaction of expanding agent. Pore-providing agents can be classified as neutralizers (such as calcium carbonate, sodium carbonate, dolomite, manganese oxide) and redox agents (such as carbonaceous products, silicon carbide, silicon nitride, aluminum nitride, titanium nitride). The thermal regime at which the expanding occurs is determined by the agent nature, being different from case to case (generally, within the limits of 700-1150 ºC) and depends on parameters of the reaction type [3, 4]. Industrially, carbonaceous products (coal, carbon black, glycerol), calcium carbonate and silicon carbide are most frequently used. The main disadvantage of the expansion process of raw material conditioned by reaching fairly high process temperatures is of course, the energy consumption. However, considering that by manufacturing the cellular glass, suitable materials for construction are made [5], which can replace traditional materials that require much higher energy consumption for manufacturing, resulting major greenhouse gas emissions, this method for making the foam glass is considered economic and ecological and with a sustainable future in the field of construction materials.

Possibilities of reducing the consumed energy were tested on a low experimental plant by Daily

Sourcing & Research SRL and Cosfel Actual SRL (Romania) by applying the unconventional method of microwave heating [6-8], but the excellent results remained in the experimental stage without industrial application.

The manufacture of aerated concrete [9] in the last decades of the 20th century offered an interesting solution [10] for the making the porous glass by the use of fine aluminum powder as a bubble-providing agent, lime-$Ca(OH)_2$ as an alkaline activator and sodium carboxymethyl cellulose (CMC) added to the composition of glass waste-based raw material with the role of foam stabilizer. The application of this cellular glass manufacturing technique had the advantage of being done at room temperature with minimal energy consumption. According to [4], a mixture consisting of 0.75 % aluminum powder, 1 % liquid solution of calcium hydroxide and up to 4 % sodium carboxymethyl cellulose (CMC) led to the formation of a slurry whose optimal viscosity was between 5-50 Pa·s and whose expansion reached 350-400 %.

This innovative cold manufacturing technique of foamed glass was also tested by the authors of this work [11]. The initiation of the aluminum hydration reaction occurred due to the calcium hydroxide solution, which contributed to the removal of the fine film of aluminium oxide from the aluminum particle surface. Mixing the glass powder with the metal powder, alkaline activator solution, and distilled water was continued at room temperature until the expansion of material was completed. The procedure time was within the limits of 15-20 min and the volume growth was identified in the range of 230-370 %, the growth corresponding to the increase in the content of aluminum (from 1.3 to 3.5 %), $Ca(OH)_2$ (from 0.7 to 1.3 %), and CMC (from 2.6 to 5.6 %). The characteristics of specimens were: density in the range of 0.25-0.34 $g \cdot cm^{-3}$, heat conductivity between 0.059-0.079 $W \cdot m^{-1} \cdot K^{-1}$, and compression resistance within the limits of 1.20-1.37 MPa.

The current work also constitutes the taking over of cold expanding method applied in the case of geopolymer foam [12] by the use of sodium perborate as a blowing material and detergent as a surfactant. The method is based on the alkaline activating features of the washing liquid containing alkyl sulfates and alkyl ethoxylate sulfates, both anionic. The length of chain specific for the hydrophobic zone of surfactant has a major influence on the kinetic migration as well as on the surface activity, a fact proven by the stability of the foam [13].

2. METHODS AND MATERIALS

2.1 Methods

The manufacturing method was based on a technique applied in the case of geopolymer foam production. The main component of mixture was recycled residual glass and the expanding material was sodium perborate monohydrate. The activation of the fine glass powder was achieved by using the detergent as a surfactant. Molecular adsorption of the surfactant on the gas-liquid separation surface was the mechanism of cold foaming the material. In the kinetic terms, the migration at the interfaces as well as the surface activity were determined by the length of chain of the hydrophobic zone of surfactant. As mentioned above, the surfactant contains alkyl sulfates and alkyl ethoxylate sulfates, both anionic, having alkaline activation properties. The starting material mixture had also in its composition a secondary product of energy making industry (fly ash), also used in the case of geopolymer foam manufacturing.

The preparation of the mixture components was carried out separately for the solid materials (glass waste and coal fly ash) and, respectively, for the liquids (sodium perborate powder in aqueous solution). Then, the slurry was slowly cast over the mixture in solid state in a glass vessel and the two mixture types were stirred (together with the addition of surfactant) with about 800 rpm for 15-20 min until the expansion of the material has been completed.

2.2 Materials

The list of components for this test included: consumed drinking bottle (60 % colourless, 25 % green, and 15 % amber glass), fly ash, detergent as a surfactant, sodium perborate monohydrate as a blowing material as well as water addition.

The recycled glass was mechanically modified by breaking, grinding in a ball mill, and sieved. The dimension of grains chosen for the experiment was under 80 μm.

Coal fly ash as a by-product was supplied by Paroseni-Thermal power plant at the maximum grain size of 200 μm, requiring a supplementary mechanical processing to reduce the granulation of fly ash under 100 μm.

The oxide components of glass types and fly ash is indicated in Table 1.

Table 1. Oxide components of raw materials

Composition	Raw material (wt. %)			
	Colourless glass	Green glass	Amber glass	Fly ash
SiO_2	71.7	71.8	71.1	52.5
Al_2O_3	1.9	1.9	2.0	23.3
CaO	12.0	11.8	12.1	6.1
Fe_2O_3	-	-	0.2	7.5
MgO	1.0	1.2	1.1	2.5
Na_2O	13.3	13.1	13.3	0.8
K_2O	-	0.1	0.1	2.2
Cr_2O_3	0.05	0.09	-	-
SO_3	-	-	0.05	0.7
Other oxides	0.05	0.01	0.05	-

Sodium perborate monohydrate (BH_2NaO_4) as a blowing agent with very fine grain size was purchased from China.

2.3 Methods of investigation of cellular glass characteristics

Using the gravimetric method [14], it was determined the density. The procedure of determining by comparation of "true" density and porous material density [15] was applied to identify the specimen porosity. The measurement of heat conductivity was carried out using HFM 446 Lambda device by the heat-flow method (SR EN 1946-3:2004) and TA.XTplus Texture Analyser was utilized to determine the compression resistance. Measuring the volumetric content of water absorbed by the material (ASTM C1585-20) was made applying the method of submersion of the sample under water. The microstructural features of foamed glass samples were investigated with the Smartphone Digital Microscope-ASONA 100X Zoom type.

3. RESULTS AND DISCUSSION

3.1 Results

Four testing versions were adopted for experimentation the chosen technical solutions. Table 2 presents these versions.

The composition of versions tested in this experiment included solid mixtures of recycled glass waste and fly ash in weight ratios kept practically constant between 2.072-2.076 % and liquid mixtures represented by sodium perborate in aqueous solution. The sodium perborate content was reduced from a maximum value of 3 % (version 1) to 1.3 % (version 4). The surfactant was added at the end in the final mixture, its content increasing uniformly from 0.1 % (version 1) to 0.4 % (version 4).

Cellular glass specimens produced by the method described above were investigated for determining their characteristics. The results are indicated in Table 3.

Table 2. Components of tested versions

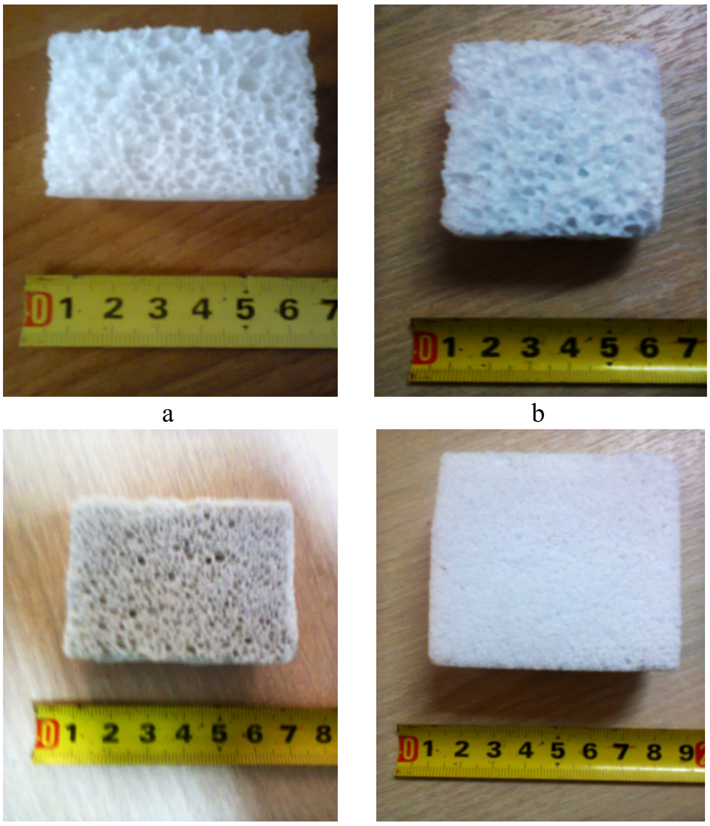
Version	Residual glass (wt. %)	Fly ash (wt. %)	Surfactant (wt. %)	Sodium perborate (wt. %)	Addition of water (wt. %)
1	65.4	31.5	0.1	3.0	7.8
2	65.6	31.6	0.2	2.6	7.6
3	65.8	31.7	0.3	2.1	7.4
4	66.3	32.0	0.4	1.3	7.2

Table 3. Characteristics of porous glass specimens

Version	Density ($g \cdot cm^{-3}$)	Porosity (%)	Heat conductivity ($W \cdot m^{-1} \cdot K^{-1}$)	Compression resistance (MPa)	Absorption of water (vol. %)	Pore size (mm)
1	0.58	72.4	0.123	1.0	11.4	0.7-1.9
2	0.65	69.0	0.134	1.2	13.6	0.6-1.1
3	0.74	64.8	0.161	1.6	16.0	0.4-0.8
4	0.82	61.0	0.183	2.1	16.2	0.3-0.7

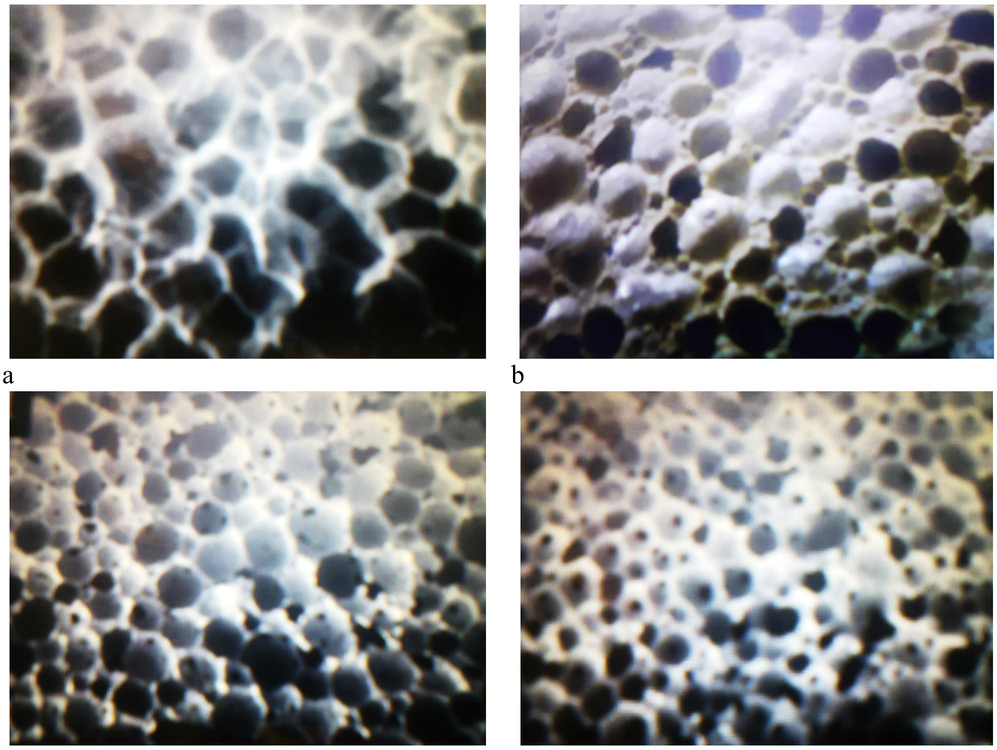
Based on Table 3 data, the heat-insulating properties of the specimens are sufficiently low, particularly in versions 1 and 2 (density within the limits of 0.58-0.65 $g \cdot cm^{-3}$, and heat conductivity between 0.123-0.134 $W \cdot m^{-1} \cdot K^{-1}$), compression resistance having satisfactory values of 1.0-1.2 MPa. The compression resistance is increasing (1.6-2.1 MPa) in versions 3 and 4, but the heat-insulating properties are slightly increased (density in the range of 0.74-0.82 $g \cdot cm^{-3}$, and heat conductivity between 0.161-0.183 $W \cdot m^{-1} \cdot K^{-1}$). Under these conditions, version 2 could be chosen as the optimal version of the experiment.

The images of the four foamed glass specimens is presented in Figure 1 and microstructural aspects of porous glass samples were examined in Figure 2.



a b c d

Figure 1. Images of foamed glass specimens
a – variant 1; b – variant 2; c – variant 3; d – variant 4.



a b c d **2 mm**

Figure 2. Microstructural features of porous glass specimens
a – variant 1; b – variant 2; c – variant 3; d – variant 4.

The microstructural homogeneity of the four images corresponding to the tested versions is obvious. The pore size varies decreasingly from variant 1 (0.7-1.9 mm) to variant 4 (0.3-0.7 mm). The pore size values for all variants are shown in Table 3.

3.2 Discussion

The experiment described above demonstrated that a cold manufacturing procedure of geopolymer foam applied in the case of cellular glass manufacturing is viable for obtaining satisfactory results. The manufacture of these cellular products at room temperature makes a major contribution to the effectiveness of energy process.

Physico-mechanical, thermal, and morphological performances of the foamed glass optimal specimen include density of 0.65 $g \cdot cm^{-3}$, porosity of 69 %, heat conductivity of 0.134 $W \cdot m^{-1} \cdot K^{-1}$, compression resistance of 1.2 MPa, absorption of water of 13.6 vol. %, and pore dimension within the limits of 0.6-1.1 mm. These characteristics are suitable for using the product as a thermal insulation material in construction, supplementary having the advantage of energy efficiency.

4. CONCLUSION

The application for the first time of a recent technique for manufacturing geopolymer foam at room temperature in cold preparation of foam glass constituted the originality of method presented in the current work. Eliminating the heat treatment of manufacturing process allowed avoiding the energy consumption characteristic of the usual techniques industrially applied. Even if unconventional economic and ecological methods based on the use of electromagnetic waves were experimentally tested in recent years, they only slightly reduced the energy consumption. The method of preparing foamed glass at room temperature, taking over the principle recently applied to the manufacture of geopolymers, proved to be adequate, the foamed product obtaining physico-mechanical and thermal characteristics recommended for the use in construction: density of 0.65 $g \cdot cm^{-3}$, heat conductivity of 0.134 $W \cdot m^{-1} \cdot K^{-1}$, and compression resistance of 1.2 MPa.

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