Nonconventional Technologies Review
Romania, March, 2024

NON-CONVENTIONAL COLD PREPARATION OF CELLULAR GLASS UNDER ECONOMICAL AND ENVIRONMENTALLY FRIENDLY CONDITIONS

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ABSTRACT: Peculiarities of the modern cellular concrete manufacturing technique were borrowed in the expanding glass technique at ambient temperature presented in the work. Using aluminum dust in calcium hydroxide solution and adding carboxymethyl cellulose (CMC) as a froth fixer, cellular glass specimens with excellent insulating properties with minimal energy consumption could be experimentally obtained, as an alternative solution to conventional methods industrially used. The tests presented in this article intend to improve the mechanical resistance of the cellular product under the conditions of preserving existing insulating properties by adding to the starting mixture a very effective ultrafine powder (silica fume) contributing to the increase of strength and durability. Completing the manufacturing recipe with silica fume is the originality element compared to the previously applied solutions. The results confirmed the effectiveness of the new method, the compressive strength increasing up to 4.7 MPa under conditions of density below 0.35 $g \cdot cm^{-3}$ and heat conductivity below 0.079 $W \cdot m^{-1} \cdot K^{-1}$.

KEYWORDS: cellular glass, aluminum dust, $Ca(OH)_2$ solution, CMC, silica fume, compression strength.

1. INTRODUCTION

Ever since the last decades of the last century, several materials (plastic, glass, rubber, paper, metal, etc.) resulting from the continuously increasing human consumption have created overstocking in landfills. Solving these problems is possible by recycling the mentioned waste. The international community has already intervened by regulating this process at the zonal level, and each state has developed its own specific short-term and longer-term plans.

In principle, a sustainable recycling needs the recovered materials to have the ability to replace existing raw materials. On the other hand, these materials should be non-degradable, so that recycling not to be accompanied by value loss [1]. Glass has the advantage of preserving its molecular structure during remelting. Thus, glass foam produced from glass waste recycling constitutes an excellent opportunity.

Residual glass, primarily from post-consumer drinking bottles and from the demolition or modernization of old buildings, has a special peculiarity compared to other residual materials: it does not degrade over time, being suitable for reuse as a material with newly created value. Traditionally, the glass industry recycles residual glass for technological purposes, but only in proportion of 21 % (in 2018) [2]. The rest of the waste is available for its valorization in other domains (usually as construction materials obtained by foaming). Among European countries, Sweden has the highest glass waste recycling rate (over 95 %).

According to Scarinci et al. (2005) [3], the properties of cellular glass are attractive for users, being a light material, resistant to water and fire, anti-corrosive, resistant to acids, rodents, bacteria, and insects, non-deformable, non-toxic as well as having moderate, but acceptable mechanical strength. The material is suitable both for the heat and acoustic construction insulating of buildings, as well as for other applications under mechanical stress conditions such as road and railway structure, drainages, protection as a load-bearing material, thermal protection of underground heat pipes, sports fields, etc.

The current making technique of manufacturing cellular glass utilized in the world on an industrial scale is based on the heat treatment of sintering at high temperatures (800-1100 °C) of the mix containing residual glass and the expanding agent. The cellular structure is obtained through the release of gases in the pyro-plastic mass of thermally sintered glass. The gases are generated as the effect of some decomposition or oxidation reactions of the expanding agent. Agents consisting of carbonates and sulphates provide decomposition reactions and carbon products generate oxidation reactions [3-5].

According to the few data available in the literature regarding the specific consumption of conventional energy of industrially manufactured products, their values are around 1 $kWh \cdot kg^{-1}$. Numerous tests performed by authors of this paper having as a goal the manufacture of similar cellular glasses, under the conditions of using an 800 W-microwave oven, have identified decreasing the energy consumption (non-conventional) in general, under 1 $kWh \cdot kg^{-1}$, even reaching 0.6-0.7 $kWh \cdot kg^{-1}$ [6, 7].

Recently, the alkaline activation of ground glass waste has been designed and tested [8-13]. In principle, the direct contact between an alkaline solution with relatively low concentration (2.5 M-3 M NaOH or KOH) and commercial glass (silica-soda-lime glass type) generates gels of calcium silicate hydrate (C-S-H) on the surface of fine glass grains. By the mechanical stirring of the mixture and due to the chemical reactions that occur on the glass surface, the suspensions reach a higher viscosity, thus avoiding the loss of gas bubbles in captivity. The use of surfactants helps to stabilize the structure containing bubbles, which after drying forms the cellular structure.

A new inorganic gel casting process for manufacturing the glass foam applied by Rinćon et al. in 2017 [14] is mentioned in the work [10]. The experiment described in the paper was based on container glass waste (below 75 µm) attacked with an aqueous alkaline solution of 2.5 M KOH. The mixture was magnetically stirred at 500 rpm lasting 3 hours at the ambient temperature. The gelation was obtained by warming the suspensions closed in plastic containers at 75 °C for 2 hours. Poly-oxyethylene octyl phenyl ether was added (4 % of the amount of glass) as a surfactant. Strong stirring at 2000 rpm for 10 min, drying at 75 °C for 24 hours, and firing in a muffle oven at 700 °C for 1 hour were the last operations, that led to obtaining the cellular glass product.

The manufacturing method presented in [11] used the expansion of waste glass in an aqueous alkaline medium through the reaction between metallic aluminum powder and calcium hydroxide ($Ca(OH)_2$). This procedure ensures the necessary conditions for the formation of pores at room temperature using the sludge rheology as the basic parameter. The preparation of suspensions was based on the fine mechanical processing of glass waste and mixing it with water (50-60 % of the solid quantity). Carboxymethyl cellulose (between 2-4 % of the suspension amount) was chosen as foam stabilizer and binder.

In another work [12], an expanded alkali-activated glass was produced at low temperature (about 80 °C). Aluminum dust was utilized as an expanding agent. The optimal mixture experimentally determined indicated the proportion value of aluminum dust at 10 %. The activator chosen in this test was composed of 10 M NaOH dissolved in water and sodium silicate (Na_2SiO_3) having SiO_2/Na_2O ratio of 3.23. The test results indicated that through this procedure the heat conductivity reached the value of 0.13 $W \cdot m^{-1} \cdot K^{-1}$, the froth density was 0.59 $g \cdot cm^{-3}$, and the compression strength had relatively high value for this type of product (5.52 MPa), i.e. 97 % more high compared to commercial cellular glass. The choice of aluminum dust as expanding agent was justified by the information that additionally it has a major role in the geopolymerization process of alumina-silicate wastes, increasing their mechanical strength.

The method of recycled glass expansion at the ambient temperature with aluminum dust in $Ca(OH)_2$ solution was tested by the authors of the work [13], including some authors of the current paper. The $Ca(OH)_2$ solution had the ability to remove the fine layer of Al_2O_3 existing on the surface of Al grains, allowing the aluminum hydration process to begin. CMC was also introduced into mix, acting as a froth stabilizer as well as a binder. The solids mix was charged and mixed into a metallic container. Deionized water was added over the solid mixture and mixing continued with an electrically driven propeller device until expansion was complete. The features of the best testing sample were denseness of 0.31 $g \cdot cm^{-3}$, heat conductivity of 0.07 $W \cdot m^{-1} \cdot K^{-1}$, and compression resistance of 1.32 MPa. The material quality was identical with conventionally made cellular products utilizable as insulating materials in construction.

The current work aimed at the manufacture of cellular glass from residual glass at room temperature by adopting the method of expanding the mixture with aluminum dust in $Ca(OH)_2$ solution. Unlike the previously applied techniques, the paper had intend to increase the mechanical strength maintaining the porous characteristic of the product structure by adding silica fume as an ultrafine powder to the mixture composition.

2. MATERIALS AND METHODS

2.1 Materials

The material listing utilized in this experiment includes post-consumer drinking bottle recycled from the commerce, aluminum dust, $Ca(OH)_2$ in powder state, CMC, and silica fume.

An approximately equal mixture of coloured glass (green and amber) of silica-soda-lime glass type was prepared by mechanical processing (crushing and grinding) being selected by sieving the particle dimension under 90 µm. The oxide composition of the two glass types was determined in the Metallurgical Research Institute of Bucharest (Romania) with the AXIOS type X-ray fluorescence spectrometer, the results being presented in Table 1.

Table 1. Oxide composition of glass type

Composition	Glass type (%)	
	Green	Amber
SiO_2	71.6	71.4
Al_2O_3	1.9	1.9
CaO	11.5	10.6
MgO	1.3	2.0
Na_2O	13.0	13.1
K_2O	0.1	0.6
Cr_2O_3	0.4	0.1
SO_3	0.2	0.3

The fine aluminum powder (under 1 mm) was produced on a testing plant in Daily Sourcing & Research Company, raw material being residual aluminum (recycled from commercial activities) melted by heating with electromagnetic waves. The metal melt was sprayed under nitrogen jets at the equipment base in the form of fine granules [15].

$Ca(OH)_2$ (traditionally called slaked lime), commercially available at over 95 % purity, was chosen in this experiment due to its contribution in aqueous solution (1:6 concentration) to the hydrogen release after the dissociation process into Ca^{2+} and OH^- ions and participation to reactions that occur on the surface of fine aluminum particles [16].

Carboxymethyl cellulose (CMC) is an anionic cellulose as a fine powder, soluble in cold water. It is used as a known froth stabilizer. CMC has the ability to reduce density and improve water-absorbing and compression resistance in porous concrete [17].

Silica fume is an amorphous polymorph state of SiO_2, being a by-product of silicon metallurgy. It is an extremely fine dust (with particle dimensions under 1 µm). Used especially in manufacturing process of porous concrete, it contributes to the significant increase of strength and durability [18].

2.2 Methods

Unlike the conventional method of foaming residual glass, in which the expansion agent provides a gas following a chemical reaction at high temperature, higher than that causes the significant decrease of the silicate material viscosity, the expanding technique at the ambient temperature using aluminum powder mixed in the glass powder mass as well as an aqueous medium of $Ca(OH)_2$ totally differs. The $Ca(OH)_2$ solution has the ability to remove the Al_2O_3 layer formed on the surface of Al granules, facilitating starting and developing the aluminum hydration process.

According to Kaneshira et al. [16], in the area that separates the Al granules and $Ca(OH)_2$ solution, several staged transformation reactions of Al_2O_3 into boehmite, bayerite, and aluminum tetrahydroxide ($Al(OH)_4$) occur. The last reaction (1) between Ca^{2+} ions and $Al(OH)_4$ in water leads to the formation of katoite ($Ca_3Al_2(OH)_{12}$), that is a crystalline substance, and hydrogen gas.

$$3Ca^{2+} + 2Al(OH)_4 = Ca_3Al_2(OH)_{12} + 6H_2 \qquad (1)$$

Hydrogen is the gas suitable for creating bubbles into the silicate mass and thus the generation of the cellular structure of the material. Facilitating the formation of bubbles as well as the development of the hydration reaction are conditioned by the continuous agitation of the glass-based sludge until the expansion of the material begins. Growing its volume continues for some time even after stopping the stirring.

2.3 Investigation methods for measuring features of specimens

Common methods were utilized in the characterization procedure of cellular specimens. Density was determined by the gravimetric methodology [19] and porosity was measured by comparing the true and apparent density of the cellular glass specimen [20]. Using TA.XTplus Texture Analyzer the measure of compression resistance was carried out and applying the heat-flow procedure (ASTM E1225-04) the heat conductivity was determined. Water immersion technique (ASTM D570) was applied for identified the specimen water-absorbing. Utilizing ASONA 100X Zoom Smartphone Digital Microscope, the cellular product microstructural appearance was analyzed.

3. RESULTS AND DISCUSSION

3.1 Results

Designing the testing variants of material mixtures tested in this experiment was based, in principle, on results presented in the literature as well as on own results obtained during previous similar tests. Thus, experimentally verified basic parameters were adopted such as: $Ca(OH)_2$/aluminum report in the range of 0.36-0.55, aluminum/residual glass ratio between 0.025-0.080, CMC/residual glass report within the limits of 0.045-0.125, and deionized water/residual glass report in the range of 0.66-1.00.

Six testing versions were chosen for testing the cellular glass manufacture at room temperature, according to Table 2. The main operating parameters of this process are shown in Table 3.

Table 2. Composition of testing versions (wt. %)

Composition	Version					
	1	2	3	4	5	6
Cellular glass	60.5	56.8	53.2	49.7	46.2	42.7
Aluminum powder	1.3	1.7	2.2	2.7	3.1	3.6
$Ca(OH)_2$ powder	0.6	0.8	0.9	1.1	1.3	1.5
CMC	2.6	3.1	3.8	4.4	5.1	5.8
Silica fume	0.4	0.6	0.7	0.9	1.0	1.1
Deionized water	38.0	39.5	41.1	42.7	44.3	46.0

Table 3. Main operating parameters of the making process

Parameter	Version					
	1	2	3	4	5	6
Dry/wet raw material quantity (g)	250/ 345	250/ 349	250/ 353	250/ 357	250/ 361	250/ 365
Process temperature (°C)	23	23	23	23	23	23
Process time (min)	21	20	19	18	17	16
Index of volume increase	2.2	2.3	2.6	2.7	3.0	3.2
Cellular glass quantity (g)	258	258	257	256	255	254

In accordance with the data in Table 3, the making technique of cellular glass was made at ambient temperature (23 °C). The operation duration was much reduced compared to the conventional processes of hot foaming residual glass, being in the range of 16-21 min. With the increase in the expanding agent ratio (aluminum dust) from 1.3 to 3.6 wt. %, the increase in volume of the specimen due to the cold expansion was significant, starting from 2.2 (in variant 1) and reaching 3.2 (in variant 6).

The appearance of cellular glass specimens in the six testing versions is presented in Figure 1.

Differences between the specimen surfaces of cellular glass subjected to various expansion regimes are obvious in images exhibited in Figure 1.

The microstructural aspect of cellular specimens prepared at ambient temperature is shown in Figure 2.

a b

c d

e f

Figure 1. Appearance of cold preparing cellular glass specimens
a – specimen 1; b – specimen 2; c – specimen 3; d – specimen 4;
e – specimen 5; f – specimen 6.

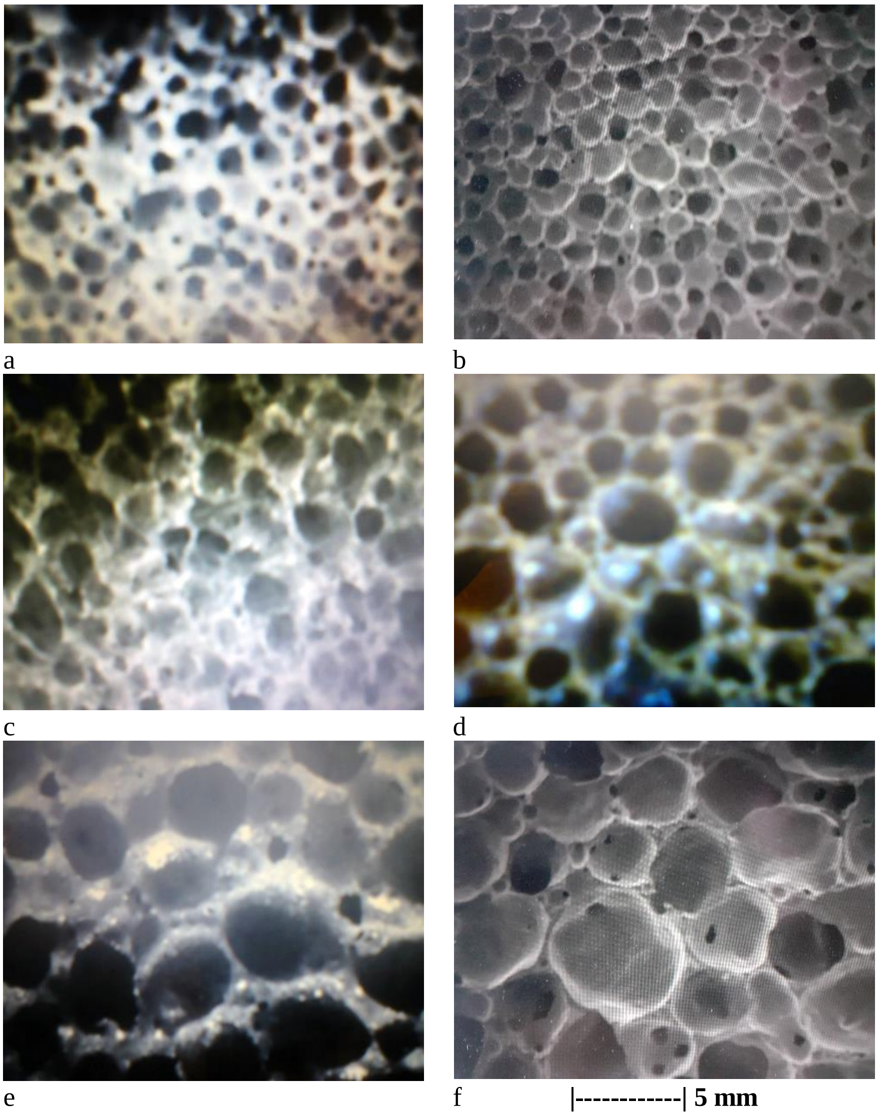

Figure 2. Microstructural appearance of cold preparing cellular glass specimens
a – specimen 1; b – specimen 2; c – specimen 3; d – specimen 4;
e – specimen 5; f – specimen 6.

The microstructure of analyzed samples is according to Figure 2 generally homogeneous, the pore size being increasing depending mainly on the weight proportions of aluminum dust and $Ca(OH)_2$ solution, increasing from sample 1 to sample 6.

The measuring results that characterize the experimentally manufactured cellular glass samples are presented in Table 4.

Table 4. Characteristics of cellular glass samples

Characteristic	**Variant**					
	1	2	3	4	5	6
Density ($g\cdot cm^{-3}$)	0.35	0.32	0.30	0.28	0.25	0,23
Porosity (%)	83.3	84.8	85.7	86.7	88.1	89.0
Heat conductivity (10^{-3} $W\cdot m^{-1}\cdot K^{-1}$)	79	76	71	64	60	53
Compression resistance (MPa)	4.7	4.5	4.0	3.7	3.4	3.2
Water-absorbing (vol. %)	2.3	2.2	2.0	2.1	2.2	2.0
Pore size (mm)	0.5-1.0	0.6-1.7	0.8-2.0	1.0-2.4	1.6-4.1	2.0-4.9

Results presented in Table 4 show good interconnection between the mechanical and physical peculiarities of specimens, given that the objective of the work was to achieve an adequate balance between the thermal insulation properties and those of mechanical strength of the cellular products. The density had values included between 0.23-0.35 $g\cdot cm^{-3}$, their level being reduced. Correspondingly, the porosity of samples was very high starting from 83.3 % in the case of the sample made in variant 1 and reaching 89.0 % corresponding to the most porous specimen (version 6). The heat-insulating character was completed by the low values of thermal conductivity between 0.053-0.079 $W\cdot m^{-1}\cdot K^{-1}$. The mechanical strength of the cellular material reached acceptably high values of over 4 MPa in the case of the first three variants and slightly lower, but satisfactory, in the case of the other experimental variants. Water-absorbing was in normal limits for this type of cellular product, being within the limits of 2.0-2.3 vol. %. The pore size in the case of specimens with reduced consumption of expanding agent was relatively small, but it reached quite high values in the case of samples corresponding to variants 5 and 6.

3.2 Discussion

The cold preparation of cellular glass materials is a much more economical alternative to the current industrial production of cellular glass, because the process practically does not require energy consumption. The use of aluminum powder as an expanding agent (through the release of gaseous hydrogen) is an innovative solution tested in other fields such as: hydrogen production and cellular concrete manufacturing. The adaptation of these technical solutions to making the cellular glass was and is one of the concerns of authors of the current work.

Given that hydrogen is an ideal energy carrier, because through oxidation it only generates water, its production is an intense concern of researchers. One of the attractive solutions is that of using aluminum to generate hydrogen. Corrosion of aluminum in water occurs in both acidic and basic environments, the second option being preferable because the alkali is regenerated. The general (cumulative) reaction is the following:

$$2Al + 6H_2O + 2NaOH = Al(OH)_3 + 1.5H_2 \qquad (2)$$

The advantages offered by the generation of hydrogen through the corrosion of aluminum are: wide availability of aluminum, high energy density, low price, no energy consumption is necessary [21].

Cellular concrete was designed and industrially making since the beginning of the 20th century using aluminum powder with a role in the expanding technique of the material mix. Following the reaction of aluminum with fine sand or fly ash, a porous structure is formed based on hydrogen bubbles, constituting the so-called aerated concrete. $Ca(OH)_2$ was used in the mixture and its reaction with silica fume with high pozzolanic properties leads to the paste hardening and the increase of mechanical strength of cellular concrete [22].

According to [23], aluminum is widely used as an expanding agent in the manufacture of lightweight geopolymer concretes. In the first stages of the geopolymerization process, the aluminum reaction favours the creation of aluminum hydroxide gel, which is deposited on the fine particles of fly ash and thus the fly ash dissolution rate is reduced, delaying the development of the material strength. Through the reaction of metallic aluminum, the release of alumina in the aqueous solution increases, leading to improving the bonds between the unreacted particles as well as the microstructure. Partially consuming the sodium hydroxide (NaOH) used as a catalyst, the aluminum dust protects the alkali content of the geopolymer matrix formed by carbonation.

Among the tested experimental variants, versions 1 and 2 were considered optimal.

4. CONCLUSION

The proposed objective of the work to obtain a suitable interconnection between the mechanical and physical peculiarities of cellular specimens has been achieved. Using aluminum dust in aqueous $Ca(OH)_2$ solution and adding carboxymethyl cellulose (CMC) as a froth stabilizer and silica fume for increasing the strength and durability, the cellular product reached relatively high compression resistanceincreasingincreasing up to 4.7 MPa, keeping its excellent heat-insulating properties.

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