Cheap building material prepared by unconventional heating of glass waste in alkaline solution

Material de construcție ieftin fabricat prin încălzirea neconvențională a deșeului de sticlă în soluție alcalină

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Rezumat. *Spumă de sticlă fabricată din deșeu de sticlă și soluție apoasă de silicat de sodiu (apă de sticlă) cu excelente proprietăți termoizolante (densitate între 0,25-0,33 g cm^{-3}, conductivitate termică între 0,059-0,069 W $(mK)^{-1}$, porozitate între 84,3-88,1 %) pentru aplicații în construcții a fost realizată cu succes folosind o tehnică originală de încălzire cu microunde predominant directă aplicată de autori, spre deosebire de metodele convenționale utilizate în mod obișnuit la fabricarea spumei de sticlă. Lucrarea a testat utilizarea apei de sticlă ca agent de spumare în condițiile particulare ale încălzirii cu microunde, deși capacitatea sa este cunoscută doar ca material de anvelopare în procesele de spumare cu agent carbonic lichid.*

Cuvinte cheie: spumă de sticlă, încălzire cu microunde, apă de sticlă, soluție apoasă alcalină.

Abstract. *Glass foam made of glass waste and aqueous sodium silicate solution (water glass) with excellent thermal insulation properties (density between 0.25-0.33 g cm^{-3}, heat conductivity between 0.059-0.069 W $(mK)^{-1}$, porosity between 84.3 -88.1 %) for building application was successfully performed using an original predominantly direct microwave heating technique applied by the authors, unlike the conventional methods commonly used in glass foam manufacturing. The paper tested the use of water glass as a foaming agent under the particular conditions of microwave heating, although its ability is known only as an enveloping material in foaming processes with liquid carbonaceous agent.*

Key words: glass foam, microwave heating, water glass, aqueous alkaline solution, thermal insulation properties.

1. Introduction

Glass foam remakably combines a multitude of properties (light weight, physical rigidity, resistant to fire, water, and steam, chemically inert, non-toxic, thermally insulator, frost resistant, resistant to rodent, insect, bacteria, resistant to compression, etc.) which make it attractive for several fields of application, primarily in construction. Initially (in the 1930s), the basic raw material was finely ground pure glass mixed with a gas-supplying material under high temperature conditions (most often carbon or carbonaceous materials). The heat treatment of mixture creates the conditions for the release and spread of gases in the thermally softened mass of the glass forming numerous bubbles, which by the final cooling turn into pores. Such a heat-insulating and non-flammable structure to glass foam was used by the United States during World War II for the inner lining of ships and submarines [1].

The onset of the world energy crisis and the perception of the danger of global overheating due to the uncontrolled emission of greenhouse gases in the last decades of the 20th century have significantly changed the attitude of humanity towards the huge reserve of waste accumulation worldwide.

One of the important quantitative wastes with very high annual generation rates is glass waste maintly from post-consumer drinking bottles as well as from the demolition or redevelopment of buildings in the form of flat glass (around 130 million tons, of which 48 % are container glass and 42 % flat glass) [2]. Recycling this waste is currently aimed at ensuring the basic raw material of industrial processes for the manufacture of glass foam. Numerous facilities have developed in recent decades in several European countries, the United States, and China, which industrially produce various types of glass foam using recycled glass waste as the main raw material. The most important companies that dominate the glass foam market are Misapor (Switzerland), Pittsburgh Corning (USA), Geocell (Austria), and Glapor (Germany) with branches in different countries [3]. The manufacturing recipes of each glass foam producer differ mainly by the nature of expanding agents, the most commonly used being black carbon, calcium carbonate, glycerine, and silicon carbide [1] in association with various mineral additives that favor the glass expansion process.

Small-scale laboratory science conducted in the last 20-25 years worldwide aims to further investigate the manufacturing feasibility of many other types of glass foam using various assortments of glass waste [4-7] and other silicate waste [8-11], a wide range of materials as expanding agents [5, 12-15] and additives [13, 16-18] as well as other ways of more efficient heat treatment of raw material mixture [19]. The purpose of this research is to obtain improved physical, thermal, and mechanical characteristics of glass foams, the use in the process of some natural products or industrial by-products considered wastes and increasing the energy efficiency of the manufacturing process by using unconventional techniques.

The current work is focused on glass foam manufacturing processes using a liquid expanding agent. The advantage of this type of agent compared to the preominantly used solid agents is the possibility of its easy spread through the free spaces between the fine particles of raw material mixture providing excellent conditions for homogeneous contact between the two phases. Consequently, a fine and evenly distributed microstructure of the foam is relatively easy to obtain folowing the sintering/foaming process.

The use in industrial production of glass foam gravel of glycerine as a liquid carbonaceous foaming agent is known from the literature. The peculiarity of this proces is the association of glycerine with sodium silicate (also called water glass) as an enveloping material for the fine carbon particles resulted by the thermal decomposition of glycerine preventing its premature oxidation [20, 21].

Industrially, the German company Glapor Werk Mitterteich uses a manufacturing recipe composed of recycled glass waste (87 %), glycerine (1 %), water glass (12 %), and minor quantities of kaolin (less than 0.5 %) [22]. According to the company's technical report [23], the main characteristics of the cellular product are: bulk density (compressed fill) between 0.125-0.155 g cm^{-3}, heat conductivity (compressed fill) of 0.078 W $(mK)^{-1}$, compressive strength (10 % compression) above 0.6 MPa. The advantages of the porous material manufactured by Glapor with liquid foaming agent are: light weight, the density of glass foam being only 20 % of the density of ordinary aggregates, excellent thermal insulating properties due to porosity with many cosed cells, resistance to acids, bases, bacteria, frost, moisture, fire, rodents and insects, water permeability. The material has applications in the following fields: load-bearing thermal insulation for foundation slabs with perimeter insulation, insulation under the ceiling, terraces, and balconies, flat roof insulation, road insulation, etc.

Recent research has aimed to test the use of water glass as the sole liquid foaming agent of glass waste without another common agent. The main reason was the high price of glycerine around 2800 USD/ton both in European markets and in the United States [24] compared to the price of a solid foaming agent widely used in glass foam manufacturing processes (calcium carbonate) which on European markets reaches 297 USD/ton.

The paper [25] presents experimental results obtained in the manufacture of glass foam from colored container glass waste with a grain size of 75, 150 and 250 μm as raw material and sodium silicate (water glass) as an expanding agent (in proportion of 15 %). The starting mixture was uniaxially pressed at 10 MPa and sintered successively at 800 and 850 ºC in a conventional oven. The results showed that the lowest grain size of the glass waste (75 μm) and the sintering temperature of 800 ºC were the optimal parameters for obtaining specimens with homogeneous microstructure characterized by small and closed pores. Glass grain size and higher temperatures led to coarse microstructures that negatively influenced the mechanical strength and heat conductivity of the specimens.

The thermal process of foaming glass waste using only water glass as an expanding agent was also experimented and presented in the paper [26]. Glass foam with very fine porosity and closed cells with a diameter of less than 0.8 mm was manufactured by the method of conventional heating of the pressed mixture of glass powder and aqueous solution of water glass. The optimum temperature of the heat treatment was between 800-850 ºC. The weight ratio of water glass tested in this experiment varied between 6-24 %, the thermal conductivity of the glass foam having decreasing values from 0.30 to 0.15 W $(mK)^{-1}$. The minimum value of thermal conductivity was achieved after the sintering process at 800 ºC. The compressive strength of 1.7 MPa could be achieved. The microscopic configuration of glass foam specimens showed the homogeneous distribution of closed cells that favors the thermal insulating properties of the material.

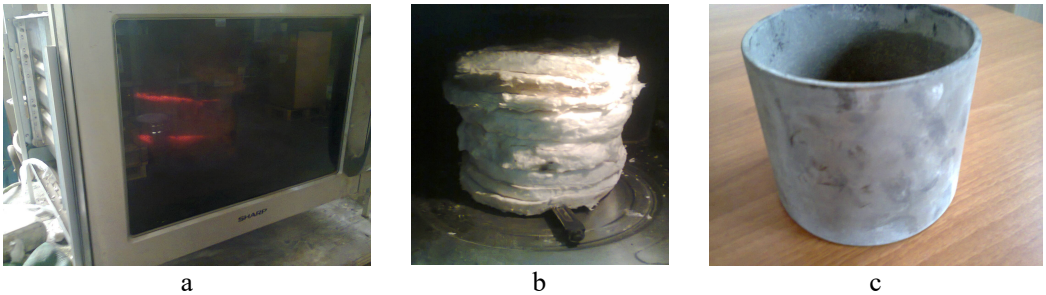
Another paper [27] includes in its content the possibility of using the aqueous solution of sodium silicate as the only foaming agent in the process of making glass foam. Analyzing the role of the addition of water glass in the mixture of cathode ray tube (CRT) panel glass, Mn_3O_4 and carbon as a foaming agent, the paper also investigates the peculiarities of using only sodium silicate mixed with CRT panel glass waste. The aqueous solution of water glass releases water during the heating process of the mixture, observed at temperatures above 400 ºC. Practically, by heating the water glass begins to foam by itself. However, the stability of the glass foam thus obtained is poor. Experimentally, it has been shown that water glass can be used as the sole foaming agent for the production of glass foam without the contribution of another ordinary agent. However, the heat conductivity of the foam has quite high values. In the experiment presented in [27], the weight proportion of water glass was between 0-24 %. It is assumed that the contribution of water glass as a foaming agent is based on the release of more strongly bound water. Often before the heating process begins for sintering, the solid material mixture is dried. Thus, the water content decreases with the release of water with less strong bonds. The glass foam produced from dry mixture has lower density and smaller pore size compared to the foam obtained from a wet mixture when heated to the same temperature. According to the results, the specimens of glass foam prepared at a lower foaming temperature from mixtures containing 24 % water glass had a density similar to that of specimens from mixtures with 12 % water glass at higher foaming temperature. In both cases the increase of the foaming temperature over 800 ºC led to the increase of the proportion of open porosity simultaneously with the increase of the pore size. The heat conductivity of glass foam containing 12 % water glass varied between 0.040-0.063 W $(mK)^{-1}$ and that of the foam containing 24 % water glass had values between 0.043-0.050 W $(mK)^{-1}$.

The research that was the basis of the current paper aimed to test the manufacture of glass foam from colorless post-consumer drinking bottle using only aqueous solution of sodium silicate as a foaming agent. The originality of the experiment was the application of the unconventional microwave heating technique, unlike the conventional methods used in the experiments presented above.

2. Methods and materials

Aqueous solutions of soluble sodium silicates in which the SiO_2/Na_2O ratio is higher than 1.5 are known as water glass. The sodium silicate water glass has an adhesive nature and this property is more accentuated with the reduction of the water content. These materials dehydrate easily. Sodium silicate solutions, especially those with a higher SiO_2/Na_2O ratio, are used as an adhesive. Research on the $Na_2SiO_3 \cdot xH_2O$ system has shown the existence of five crystalline sodium metasilicates that are hydrates containing 5-9 molecules of water in addition to the anhydrous material [28]. According to [29], the contact between a soda-lime glass and a neutral or slightly alkaline aqueous solution causes an exchange of hydrogen-alkali ions by a controlled diffusion. The silica network remains stable and is not affected. The method of foaming glass waste using water glass is based on breaking water crystallization bonds and releasing it in the form of vapor. The released water vapors are blocked in the thermally softened mass of the glass forming bubbles, which by cooling turn into a network of pores specific of glass foam.

Unlike the experiments described in [25-27], performed by conventional heating methods, the tests on foaming glass waste using aqueous solution of water glass presented in this paper were performed by applying the own predominantly direct microwave heating technique [19]. 800 W-microwave oven of the type usually used in the household but constructively adapted for high temperatures (above 1000 ºC) was the experimental equipment. The raw material mixed with the aqueous solution of water glass and pressed was freely deposited on a metal support placed on the ceramic fiber thermal insulation at the base of the oven. A cylindrical ceramic tube of SiC and Si_3N_4 with the diameter of 125 mm, height of 100 mm and wall thickness of 2.5 mm was placed between the pressed material and the emission source of the microwave field. The tube had a lid made of the same material. Because the peculiarity of the direct microwave heating consists in initiating the process in the core of material [30, 31] inverse to the conventional heating, efficient thermal protection with ceramic fiber mattresses of the outer surface of the ceramic tube and lid was required. A radiation pyrometer axially mounted above the oven visualizing the heated material through 30 mm-holes provided in the upper wall of the oven and the ceramic lid allowed the control of its temperature evolution. The experimental microwave equipment is shown in Fig. 1.



a b c

Fig. 1. Experimental microwave equipment
a – 800 W-microwave oven; b – thermal protection of ceramic tube and lid; c – ceramic tube.

The raw material used in experiment was recycled glass waste coming from colorless post-consumer drinking bottle. The oxide composition of this glass type contains: 71.7 % SiO_2, 1.9 % Al_2O_3, 12.0 % CaO, 1.0 % MgO, 13.3 % Na_2O, and 0.1 % other oxides. Waste processing included the following operations: selection by color, washing, drying, breaking, grinding in a ball mill, and sieving. The grain size of glass after these operations was below 60 μm. The water glass was purchased from the market as a 30 % aqueous solution.

The characterization of the glass foam specimens was performed by the following methods. The gravimetric method [32] was used to determine the apparent density and the method of comparing the "true" density with the apparent density [33] was used to calculate the porosity of the specimens. Determining the heat conductivity was performed by the heat-flow method (ISO 9869-1: 2014, reviewed and confirmed in 2019) and the TA.XTplus Texture analyzer was used to identify the compressive strength (EN 826-2013). To measure the volumetric proportion of absorbed water in the material it was applied the method of specimen immersion in water (for 24 hours) (ASTM D570). The microstructural peculiarities of cellular glass-ceramic specimens were examined with ASONA 100X Zoom Smartphone Digital Microscope.

3. Results and discussion

Three experimental variants were adopted for the mixture including glass waste, water glass, and distilled water as an additional binder. The weight proportions of these components are presented in Table 1. The ranges of component dosage values were chosen based on the results of previous preliminary tests.

Table 1

Composition of the experimental variants

Variant	Glass waste (wt. %)	Water glass (wt. %)	Distilled water (wt. %)
1	83.0	12.0	5.0
2	81.5	12.5	6.0
3	80.0	13.0	7.0

Mixing the fine glass waste powder with water glass and distilled water in the proportions indicated in Table 1 led to obtaining a sludge, which was loaded into a cylindrical metal mold with a detachable wall in which it was axially pressed at approx. 2-3 MPa. The compacted wet mass was then removed from the mold and freely placed in the microwave oven. In all experimental variants the amount of wet material was kept constant at 480 g.

The heating process for the manufacture of glass foam carried out in the microwave oven had the functional parameters shown in Table 2.

Table 2

Functional parameters of the process

Variant	Wet raw material/glass foam amount (g)	Sintering/ expanding temperature (ºC)	Heating time (min)	Average rate (ºC/min)		Index of volume increasing	Specific energy consumption (kWh/kg)
				Heating	Cooling		
1	480/385.5	788	28	27.4	6.0	1.35	0.76
2	480/385.1	793	28.5	27.1	5.8	1.45	0.77
3	480/384.6	808	30	26.3	5.8	1.90	0.81

According to the data in Table 2, the measured temperature of the end of sintering and foaming process of raw material was in the range 788-808 ºC, the highest value corresponding to the maximum addition of water glass and distilled water (variant 3). Due to the remarkable energy efficiency of the predominantly direct microwave heating, the average heating rate reached very high values (26.3-27.4 ºC/min) and the process time was short (between 28-30 min). Implicitly, the specific energy consumption was in a very small range of values (0.76-0.81 kWh/kg).

The appearance of the glass foam specimens in cross section obtained following the experimental manufacturing process is shown in Fig. 2. The images indicate the fineness of the microstructure of specimens 1 and 2 sintered at temperatures below 800 ºC and having in the starting mixture lower proportions of water glass and distilled water. The specimen 3 sintered at 808 ºC and having the highest proportions of water glass and water is characterized by a less fine, but homogeneous microstructure with closed pores whose size does not exceed 1 mm.

a b c

Fig. 1. Appearance of glass foam specimens
a – specimen 1 heated at 788 ºC; b – specimen 2 heated at 793 ºC;
c – specimen 3 heated at 808 ºC.

The investigation of the physical, thermal, mechanical, and microstructural characteristics of the glass foam specimens performed with the methods indicated above allowed identifying the peculiarities of each specimen. Table 3 shows the results of these investigations.

Table 3

Physical, thermal, mechanical, and microstructural features of specimens

Variant	Apparent density (g cm^{-3})	Porosity (%)	Heat conductivity [W $(mK)^{-1}$]	Compressive strength (MPa)	Water absorption (vol. %)	Pore size (mm)
1	0.33	84.3	0.069	1.85	1.5	0.10 – 0.30
2	0.30	85.7	0.065	1.73	1.1	0.20 – 0.50
3	0.25	88.1	0.059	1.40	0.9	0.40 – 0.90

Analyzing the data from Table 3, the thermal insulating properties of the experimentally obtained foams can be noticed being determined by the low values of the apparent density (0.25-0.33 g cm^{-3}) and of the heat conductivity [0.059-0.069 W $(mK)^{-1}$] as well as the high values of porosity (84.3-88.1 %). The compressive strength is within the acceptable limits of glass foams usable as thermal insulation material (1.40-1.85 MPa). Also, the porous material is not water absorbent, the volumetric proportion experimentally determined being only between 0.9-1.5 vol. %.

Examining the microstructural configuration of the glass foam specimens (Fig. 3) confirms the fineness of their microstructure and homogeneity of the pore distribution in the material section. The pore size of maximum 0.90 mm is indicated in Table 3.

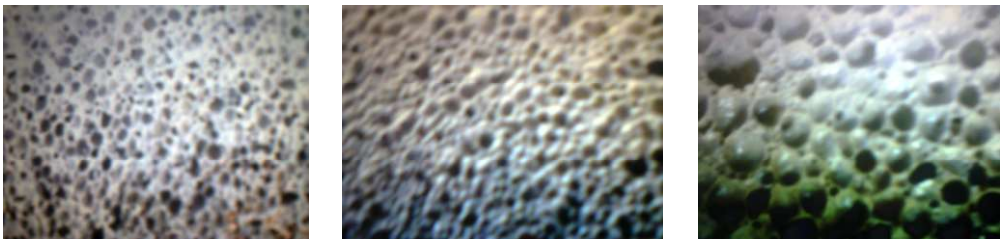


Fig. 3. Microstructural configuration of glass foam specimens
a – specimen 1; b – specimen 2; c – specimen 3.

The experiment described in the paper confirmed that the use of aqueous solution of sodium silicate as the only glass expanding agent is feasible and does not require a commonly more expensive agent for similar processes of manufacturing a glass foam with thermal insulation properties required in the building. The main difference between adopting the unconventional technique of predominantly direct microwave heating compared to the conventional methods applied in the experiments [25-27] mentioned above is the very high heating rate (26.3-27.4 ºC/min) compared to much lower values (under 15 ºC/min) recommended in the literature [1] without affecting the homogeneous organization of the microstructure of final products. The consequence of this advantage is the high level of energy efficiency of the unconventional technique, the specific energy consumption being diminished up to 0.76 kWh/kg. Glass foam manufacturers do not provide clear information on the energy efficiency of their conventional manufacturing processes. However, it can be deduced that even in continuous industrial conditions a value below this limit cannot

be obtained. Moreover, according to [34] the transition from a small-scale experiment to an unconventional industrial-scale process could increase its energy efficiency by up to 25 %.

4. Conclusions

The paper aimed to test the use of aqueous solution of sodium silicate as the only foaming agent in the experimental manufacturing process of glass foam from recycled glass waste without the contribution of another commonly used agent. The originality of the process was the application of the own technique of predominantly direct microwave heating unlike conventional methods of heat treatment of the powder mixture composed of glass and foaming agent. The predominantly direct microwave heating controlled by the wall thickness (2.5 mm) of a ceramic screen made of strongly microwave susceptible materials placed between the source emitting electromagnetic waves and the irradiated material allowed to reach very high heating rates (between 26.3-27.4 ºC/min) compared to their recommended range (5-15 ºC/min) used in industrial processes. Glass foam produced by sintering at 788-808 ºC had thermal insulation properties suitable for building materials (density between 0.25-0.33 g cm^{-3}, heat conductivity between 0.059-0.069 W $(mK)^{-1}$, porosity between 84.3-88.1 %) as well as compressive strength between 1.40-1.85 MPa. The excellent energy efficiency of the heating technique led to very economical specific energy consumptions (between 0.76-0.81 kWh/kg).

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