MICROWAVE HEATING PREPARATION OF A THERMAL INSULATION MATERIAL FOR BUILDING CONSTRUCTION USING GLASS WASTE AND COAL ASH

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ABSTRACT: The paper presents results of the manufacturing process of a porous high-strength glass-ceramic foam using colorless glass waste (between 35.5-50.0%) and coal fly ash (between 24.6-35.5%) as raw materials, sodium borate (between 25.0-28.0%) as a fluxing agent and calcium carbonate (between 0.4-1.0%) as a foaming agent. The heating technique was based on the conversion of microwave energy into heat, the sintering/foaming temperature varying between 802-815 ºC. The characteristics of the glass-ceramic foam samples were: apparent density between 0.38-0.45 g/cm^3, porosity between 78.6-81.9%, thermal conductivity in the range 0.049-0.064 W/m·K, compressive strength between 2.5-6.0 MPa and pore size below 0.60 mm. The specific energy consumption had very low values (0.72-0.82 kWh/kg) confirming the high energy efficiency of the predominantly direct microwave heating technique.
KEYWORDS: glass-ceramic foam, coal fly ash, glass waste, sodium borate, calcium carbonate, microwave heating.

1. INTRODUCTION

Several silicate by-products, such as coal ash, dust and fly ash trapped in the filters of waste incinerators, mixed with glass waste and heated at high temperature, in the presence of a foaming agent, lead to the formation of glass-ceramic foams. These are predominantly polycrystalline porous materials with a fine microstructure, obtained by controlled crystallization of glass. Some glass waste as windows glass are stable and difficult to crystallize. Commonly, a glass ceramic has not completely a crystalline microstructure (only 50 - 95%), while the rest is amorphous residual glass) [1]. Generally, the mechanical properties of glass ceramic foams are superior to those of precursor glass. In addition, they may exhibit other distinct properties that are beneficial to particular applications.

Coal ash, an industrial by-product generated from the combustion of coal in thermal power stations, consists of fly ash, bottom ash, boiler slag, and flue gas desulfurization material. Fly ash is the main component of coal ash being composed of spherical particles with sizes between 0.1-300 μm. This is predominately composed of silica, alumina, iron oxide and calcium oxide [2, 3]. More than 500 million tons of coal fly ash is generated annually worldwide [2].

According to [4], coal fly ash is currently used in many applications in pavement construction: asphalt concrete, Portland cement manufacture, embankment or fill, stabilized base, flowable fill, etc. Also, this industrial by-product is suitable for direct embedding in a ceramic powder without any other processing treatment. The coal ash partially replaces kaolin, feldspar and quartz [5]. It is frequently used in experimental manufacture of glass-ceramic foams as a raw material, generally, together with glass waste [2]. In recent decades, several studies on the use of fly ash for the manufacture of glass-ceramics have been reported in the literature.

A method of producing a light glass-ceramic foam from a powder mixture composed of 80 wt.% colorless post-consumer packaging glass and 20 wt.% coal fly ash is presented in the paper [6]. 2 wt.% silicon carbide (SiC) as a foaming agent was added to the powder mixture. The apparent density of the sintered product at 1000-1050 ºC was 0.2-0.4 g/cm^3 and the porosity had values between 70-90%. The most uniform pore distribution corresponded to a porosity of 75%, a compressive strength of 1.5 MPa and a relatively high thermal shock resistance. According to the XRD analysis, the main crystalline phase of this glass-ceramic foam was wollastonite and traces of SiC.

The paper [7] presents a similar experiment, the same material types being used, but the SiC weight ratio was reduced to 1 wt.%. The sintering temperature was 950 ºC, that led to the following glass-ceramic foam characteristics: apparent density

between 0.18-.35 g/cm^3, compressive strength in the range 0.9-1.8 MPa and a homogeneous microstructure with pore size between 1-3 mm.

A high-porosity glass-ceramic foam was prepared [8] using a soda-lime-silicate glass waste (84.5 wt.%) and coal fly ash (15 wt.%) as finely individually ground raw materials, additionally being added commercial silicon carbide (0.5 wt.%) as a foaming agent and water (6-8 wt.%). The fly ash had as main components SiO_2 (61.8 wt.%), Al_2O_3 (25.9 wt.%), Fe_2O_3 (6.1 wt.%), CaO (2.8 wt.%) and TiO_2 (2.1 wt.%). The mixture was mixed and compacted by pressing. The sintering temperature was varied between 925-975 ºC and the holding time between 10-60 min. The experimental results gradually led to increasing the pore size and to their transformation from closed, individualized and homogeneous to open, interconnected and inhomogeneous. The XRD analysis showed that the main crystalline phases formed at 925 ºC and kept up to 975 ºC were augite, diopside and wollastonite. By increasing the process temperature from 925 to 975 ºC the porosity increased from 39 to 67%, the water absorption increased from 39 to 181%, the bulk density decreased from 1.01 to 0.37 g/cm^3 and the compressive strength decreased from 9.3 to 0.95 MPa.

A glass-ceramic foam was prepared [9] from 84.75 wt.% glass waste, 14.75 wt.% coal fly ash as raw materials, 0.5 wt.% SiC and water. The wetted and pressed powder mixture was sintered at 950 ºC for 20 min. The volume expansion index of the foamed sample was 5.81 compared to the initial pressed volume of the mixture. The main characteristics of the final product were: apparent density of 0.267 g/cm^3, porosity of 81.55% and compressive strength of 0.98 MPa. The main crystalline phases observed by the XRD analysis were mullite and cristobalite.

Ultra-light ceramic foams were made from glass waste powder and coal fly ash as raw materials using the green sphere technique [10]. Sodium borate as a fluxing agent and SiC as a foaming agent were also added to the mixture. The optimal composition of the mixture contained 70 wt.% glass waste, 30 wt.% coal fly ash, 15 wt.% sodium borate and 0.5 wt.% SiC. The sintering temperature was between 680-780 ºC. The foamed product in this temperature range had bulk density between 0.14-0.41 g/cm^3, porosity between 82.9-94.1%, compressive strength in the range 0.91-6.37 MPa and thermal conductivity between 0.070-0.121 W/m·K. The manufacturing method was low-cost and environment friendly, being used the conventional heating technique with electric resistances.

Another manufacturing technique of glass-ceramic foam [11] was based on preparing a mixture containing glass waste (80 wt.%), fly ash (20 wt.%) and a sludge from dolomite ($CaMg(CO_3)_2$) and calcium carbonate ($CaCO_3$) as foaming agents (1-2 wt.%). The sintering/foaming temperature was 850 ºC. The foamed product had an apparent density between 0.36-0.41 g/cm^3 and a compressive strength between 2.4-2.8 MPa.

Using a powder mixture composed of a window glass waste (80 wt.%) and coal fly ash (20 wt.%) as raw materials, a sodium silicate solution (10 wt.%) as a binder and $CaCO_3$ (1 wt.%) as a foaming agent, pressed in molds, fired at 650 ºC for 30 min, then at 750 ºC for 1 hour, a glass-ceramic foam with apparent density of 0.421 g/cm^3, compressive strength of 5.87 MPa and water absorption of 2.1% was obtained. This product usable as a building material has higher mechanical strength, lower water absorption and a lower price compared to lightweight bricks existing on the market [12].

Glass-ceramic foams were experimentally made by the direct foaming method using coal fly ash and glass waste as raw materials, sodium borate as a fluxing agent and $CaCO_3$ as a foaming agent [13]. The optimal sintering temperature was 800 ºC for 45 min and the optimal manufacturing recipe included 40 wt.% coal fly ash, 60 wt.% glass waste, 30 wt.% sodium borate and 0.5 wt.% $CaCO_3$. The foamed product obtained by this recipe at the functional parameters mentioned above had the apparent density below 0.46 g/cm^3, thermal conductivity about 0.036 W/m·K and compressive strength over 5 MPa. The experimental results showed good thermal insulation properties of the glass-ceramic foam and simultaneously an excellent compressive strength.

The works presented in the literature mentioned above used conventional heat treatment techniques. The Romanian company Daily Sourcing & Research has initiated the application of efficient microwave heating methods, including also the use of coal fly ash in glass-ceramic foam manufacturing processes.

A solution adopted in 2016 [14] for preparing glass-ceramics in a 0.8 kW-microwave oven was the use of a metal crucible with a lid containing the pressed powder raw material introduced in a silicon carbide-based ceramic crucible with the outer diameter of 125 mm, the height of 100 mm and the wall thickness of 5 mm. Silicon carbide (SiC) was used as a foaming agent in a constant proportion of 5 wt.%, while the coal fly ash was varied between 0-19 wt.%, the rest being colorless glass waste (between 76-95 wt.%) recycled from post-consumer packaging bottle and mechanically pressed below

130 μm. The optimal sample was obtained from 80% glass waste, 15% fly ash and 5% SiC by sintering at 980 C. The specific energy consumption was 5.0 kWh/kg, the amount of foam material being below 120 g. Its main characteristics were: apparent density of 0.32 g/cm^3, porosity of 83.2%, thermal conductivity of 0.038 W/m·K and compressive strength of 1.5 MPa.

The paper [15] refers to foaming experiments of powder mixtures composed of colorless window glass waste (85.9-87.9 wt.%), coal fly ash (8.5-10.5 wt.%) as raw materials, SiC (3.5-3.6 wt.%) as a foaming agent and water addition (9.0-9.5 wt.%) using the microwave energy. The sintering temperature was between 980-995 ºC. The average heating rate varied between 11.2-12.0 ºC/min, the microwave heating method being predominantly direct and partially indirect due to the use of a SiC ceramic tube with the wall thickness of 3.5 mm placed between the emission source of electromagnetic waves and the heated pressed material. The foamed products characteristics were: apparent density between 0.32-0.42 g/cm^3, porosity between 80.9-85.4%, compressive strength between 1.4-2.1 MPa, thermal conductivity in the range 0.042-0.060 W/m·K and water absorption between 2.6-3.6%. The microstructural homogeneity was good in all tested variants, unlike the structural inhomogeneity of the samples made also from window glass waste by conventional heating techniques.

An almost similar experiment is described in [16], in which the glass waste was composed of a mixture of colored (colorless, green and amber) post-consumer packaging bottle. The fly ash had values between 9-11 wt.%, SiC between 2.8-3.6 wt.% and water addition was 14.5 wt.%. The sintering temperature varied between 958-966 ºC. The characteristics of glass-ceramic foams were: apparent density between 0.22-0.32 g/cm^3, porosity between 85.5-90.0%, compressive strength between 1.23-1.34 MPa and thermal conductivity in the range 0.043-0.060 W/m·K. The specific energy consumption was between 1.55-1.78 kWh/kg.

The aim of this paper is to manufacture a porous high-strength glass-ceramic foam from a powder mixture composed of glass waste and coal fly ash as raw materials, sodium borate (borax) as a fluxing agent and $CaCO_3$ as a foaming agent. The heating technique adopted was the predominantly direct and partially indirect microwave heating, which is the main original character of the work, unlike the conventional techniques currently used in the world in similar manufacturing processes.

2. METHODS AND MATERIALS

2.1 Methods

The most known and used method of producing glass foam is the incorporation of a foaming agent into the glass powder mixture and heating it to the sintering temperature [17]. There must be a correlation between the temperature at which the foaming agent releases a gas (or a gaseous compound) as a result of a decomposition or oxidation reaction and the softening temperature of the glass at which its viscosity becomes suitable for capturing and blocking the gas bubbles. By solidification, the bubbles form a specific porous structure.

The foaming agent adopted by authors was $CaCO_3$. The thermal decomposition of calcite (the most stable polymorph of $CaCO_3$) was analyzed by in-situ high-temperature X-ray powder diffraction [18]. The experimental results showed that the process of thermal conversion of $CaCO_3$ into calcium oxide (CaO) is initiated slowly and then occurs rapidly at temperatures above 750 ºC according to the reaction (1).

$$CaCO_3 = CaO + CO_2 \quad (1)$$

The experimental microwave equipment shown in Figure 1 (a) had as main component a 0.8 kW-microwave oven (b) of the type commonly used in the household, but constructively adapted for high temperature operation. The device for rotating the material subjected to heating was removed, several ceramic fiber mattresses being placed at the base of the oven. The pressed powder mixture (c) was freely deposited on a metal plate placed on the ceramic mattresses, being concentrically protected with a ceramic tube (d) with an outer diameter of 125 mm, a height of 100 mm and a wall thickness of 2.5 mm from a high microwave susceptible material (silicon carbide and nitride) provided with a lid of the same material. The thermal protection (e) of the pressed mixture, ceramic tube and lid with ceramic mattresses with high thermal resistance is very important to avoid the heat loss outside the system. The low thickness of the tube wall was adopted to obtain a predominantly direct and partially indirect microwave heating. The direct heating is achieved by the direct contact of the microwave flux with the pressed mixture. The microwave energy is converted to heat initially in the core of the irradiated material, which heats up intensely. The heat in the central area propagates volumetrically to the peripheral areas of the material [19]. Partially, the microwave energy is absorbed into the ceramic tube wall and also converted into heat. The wall is heated quickly and it

will transfer heat to the material by thermal radiation (indirect heating). This mode of mixed microwave heating proved to be very efficient in all previous experiments and was also adopted by the authors for the current experiment. Negative influences on the structure of the foam material by microwave irradiation were not found, given that commercial glass (soda-lime glass) is not suitable for direct microwave heating [20] at the maximum power provided by the oven without a microwave susceptible screen. The temperature control of the sintering/foaming process was performed with a radiation pyrometer mounted above the oven (a) visualizing the surface of the heated material through holes provided axially in the upper wall of the oven, the ceramic lid and the thermal insulation of the lid.

Figure 1. Experimental microwave equipment
a – overall experimental equipment; b – 0.8 kW-microwave oven; c – pressed powder mixture; d – SiC-based ceramic tube; e – ceramic fiber thermal insulation.

2.2 Materials

The materials used in the experiment described in the paper were: colorless glass waste and coal fly ash as raw materials, sodium borate (borax) as a fluxing agent and $CaCO_3$ as a foaming agent.

The glass waste was selected by color from a batch of recycled post-consumer packaging bottle. Then it was broken, ground in a ball mill and sieved at a grain size below 100 μm.

The chemical composition of this waste contained: 71.4% SiO_2, 1.9% Al_2O_3, 12.0% CaO, 0.05% Fe_2O_3, 1.0% MgO, 13.3% Na_2O, 0.1% K_2O, 0.05% Cr_2O_3 and 0.2% SO_3.

Coal fly ash purchased from Paroseni thermal power station (Romania) with the initial grain size below 200 μm was ground in a ball mill and sieved to dimensions below 63 μm. Its chemical composition included 46.5% SiO_2, 23.7% Al_2O_3, 7.9% CaO, 8.6% Fe_2O_3, 3.2% MgO, 6.0% Na_2O and 4.1% K_2O.

The borax was used as a fluxing agent due to the high Na_2O content known as the best fluxing material [21]. Purchased from the market at a grain size below 400 μm, the borax was ground in a laboratory electric device being used in the experiment at a grain size below 130 μm.

The calcium carbonate was used as a foaming agent without other mechanical processing such as it was purchased from the market due to the fine grain size below 40 μm.

3. RESULTS AND DISCUSSION

3.1 Results

Four variants of experimental manufacturing recipe were adopted also taking into account the results of quite similar experiments (using the same type of materials) described in the paper [13]. Table 1 presents the composition of the four variants.

Table 1. Experimental manufacturing recipes

Composition	Variant 1	Variant 2	Variant 3	Variant 4
Colorless glass waste (wt.%)	50.0	44.0	39.0	35.5
Coal fly ash (wt.%)	24.6	29.4	33.2	35.5
Sodium borate (wt.%)	25.0	26.0	27.0	28.0
Calcium carbonate (wt.%)	0.4	0.6	0.8	1.0
Water addition (wt.%)	12.0	12.0	12.0	12.0

As mentioned above, the testing of the experimental variants was performed on the 0.8 kW-microwave oven in the Romanian company Daily Sourcing & Research. The parameters of the foaming process of the mixtures based on glass waste and coal fly ash are shown in Table 2.

Table 2. Parameters of the foaming process

Parameter	Variant 1	Variant 2	Variant 3	Variant 4
Dry	475/	475/	475/	475/

material/glass-ceramic foam quantity (g)	460	465	463	462
Process temperature (ºC)	802	805	809	815
Heating time (min)	32	33	34.5	36.25
Heating/cooling speed (ºC/min)	24.4/6.3	23.8/6.5	22.9/6.2	21.9/6.4
Volume expansion index	2.30	2.10	1.85	1.60
Specific consumption of energy (kWh/kg)	0.72	0.74	0.78	0.82

According to the data in Table 2, the sintering/foaming process took place at relatively low temperatures between 802-815 ºC, Na_2O from the composition of borax, but also from the composition of glass waste and coal fly ash, having the role of fluxing agent, influenced the decrease of sintering temperature. The microwave heating has shown high energy efficiency. The microwave absorption in the raw material mass is supplementary favored by the high proportion of alkali metal oxides (mainly Na_2O and K_2O). Consequently, the heating speed had high values (between 21.9-24.4 ºC/min) and the specific energy consumption was very economical (between 0.72-0.82 kWh/kg).

The determination of the physical, thermal, mechanical and morphological features of the four glass-ceramic foams was carried out using common analysis techniques. The apparent density was measured by the gravimetric method [22] and the porosity was calculated by the comparing method of the true and apparent density [23]. The thermal conductivity was investigated by the heat-flow meter method (ASTM E1225-04) and the compressive strength was determined using a TA.XTplus Texture Analyzer of Stable Micro Systems (ASTM C552-17). The water absorption was measured by the water immersion (for 24 hours) method (ASTM D570). The samples microstructure was analyzed with an ASONA 100X Zoom Smartphone Digital Microscope. The main physical, thermal, mechanical and morphological features of glass-ceramic foam samples are presented in Table 3.

Table 3. Main features of the glass-ceramic foam samples

Variant	Apparent density g/cm^3	Porosity %	Thermal conductivity W/m·K	Compressive strength MPa	Water absorption %	Pore size mm
1	0.38	81.9	0.049	2.5	2.0	0.40 – 0.60
2	0.40	81.0	0.052	3.6	2.3	0.20 – 0.50
3	0.42	80.0	0.055	4.6	2.5	0.15 – 0.40
4	0.45	78.6	0.064	6.0	2.5	0.05 – 0.30

Examining the data in Table 3, there is a strong tendency to increase the compressive strength of glass-ceramic foam samples from 2.5 to 6.0 MPa with the simultaneous increase of borax and $CaCO_3$. The main explanation is the presence of boron in the composition of borax. According to [24], the boron content in borax is 11.36% and the role of this chemical element in significantly increasing the mechanical strength has been highlighted in several papers. The combined consumption of $CaCO_3$ and borax to obtain a dense high-strength glass foam is known. In [25] the borax content was much lower (5 wt.%), but sufficient to produce a dense foamed material with compressive strength up to 6.2 MPa. Increasing the $CaCO_3$ content from 1.5 to 5% had a negative effect, with compressive strength decreasing to 2.5 MPa. The mentioned experiment did not use coal fly ash. The content of fly ash in the current work (24.6-35.5 wt.%) was higher than usual (up to 20 wt.%). The known role of fly ash is to facilitate the foaming process. The variation of its proportion did not show a clear improvement effect.

The apparent density of the samples had relatively low values (between 0.38-0.45 g/cm^3) acceptable for the use as thermal insulation materials. The thermal conductivity values were even low (0.049-0.064 W/m·K) compared to other products with similar apparent densities.

In order to have more complete data on the characteristics of glass-ceramic foams, their microstructural configuration was examined in Figure 2.

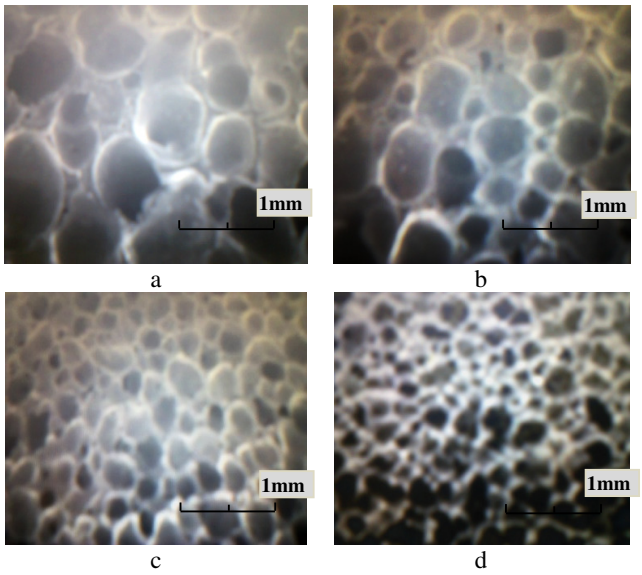

Figure 2. Microstructural configuration of the glass-ceramic foam samples
a – sample 1 heated at 802 ºC; b – sample 2 heated at 805 ºC;
c – sample 3 heated at 809 ºC; d – sample 4 heated at 815 ºC.

Pictures of the microstructural configuration of the glass-ceramic foam samples show their homogeneity. Closed pores with very small dimensions have a uniform distribution. The pore size varies from 0.05-0.30 mm (variant 4) to 0.40-0.60 mm (variant 1).

3.2 Discussion

In terms of quality, the glass-ceramic foams obtained by the nonconventional technique presented in the paper were compared with foamed products made by a conventional technique using the same type of starting materials [13]. Comparing the functional parameters of the current experiment with those shown in the paper [13] it is found that the range of heating speeds (21.9-24.4 ºC/min) is almost similar to the optimal value of about 22 ºC/min, which allows to obtain the highest compressive strength (5.2 MPa) as well as the optimal combination between apparent density (0.46 g/cm^3) and porosity (78.5%). Also, the optimal value of the process temperature indicated in [13] is very close to the range of experimentally determined temperature values (802-815 ºC) according to Table 2.

If in terms of quality it can be considered that the products manufactured by unconventional technique are almost similar to those obtained by conventional techniques, the comparison of the energy efficiency of the two thermal processes is impossible, because this information is avoided in the literature. However, the fact that the microwave heating of solid materials is faster and more economical compared to conventional heating techniques is recognized in the literature [26].

4. CONCLUSION

Experimental results obtained in the manufacturing process of a high-strength glass-ceramic foam are presented in the paper.

The used heating technique was nonconventional by converting the microwave energy into heat unlike the conventional techniques commonly applied in the world in glass-ceramic foam manufacturing processes and this is the originality of the work.

Colorless glass waste from recycled post-consumer packaging bottle and coal fly ash in proportions of 35.5-50% and 24.6-35.5% respectively, as raw materials, borax between 25-28% as a fluxing agent and $CaCO_3$ between 0.4-1% as a foaming agent were used to make a finely ground mixture wetted with 12% water addition.

The measured sintering/foaming temperature was in the range 802-815 ºC, the heating speed varying between 21.9-24.4 ºC/min.

The determination of the physical, thermal, mechanical and morphological characteristics of the glass-ceramic foam samples led to the following results: apparent density between 0.38-0.45 g/cm^3, porosity between 78.6-81.9%, compressive strength between 2.5-6.0 MPa and thermal conductivity in the range 0.049-0.064 W/m·K. The water absorption was measured between 2.0-2.5%. The pore size varied from 0.05-0.30 mm (variant 4) to 0.40-0.60 mm (variant 1).

The specific energy consumption was very economical (between 0.72-0.82 kWh/kg) due to the excellent energy efficiency of the microwave heating technique.

The glass-ceramic foams made by the method described in the paper are usable as thermal insulation materials in construction works that require mechanical stress.

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