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Glass-ceramic Foams Made of Very High Coal Fly Ash Weight Ratio by the Direct Microwave Heating Technique

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Abstract

A high mechanical strength glass-ceramic foam was produced by direct microwave heating at 853 ºC of a very high weight ratio of coal fly ash (82%), calcium carbonate (5%) as a foaming agent, sodium carbonate (13%) as a fluxing agent and water addition (10%). Due to the excellent energy efficiency of the direct microwave heating, the heating rate had a very high value (32 ºC/min), much higher than the heating rate of conventional processes and led to a very low value of the specific energy consumption (0.72 kWh/kg). The physical and mechanical characteristics of the optimal glass-ceramic foam sample were: apparent density of 1.44 g/cm3, porosity of 26.2%, thermal conductivity of 0.281 W/m•K, compressive strength of 41.3 MPa and water absorption of 0.5%. Given the features of the glass-ceramic foam (very high compressive strength, acceptable porosity and thermal conductivity, very low water permeability, fireproof, chemical stability, no-toxicity, etc., the application domain of this material type may include road and railway constructions, bridge abutments and retaining walls, foundations, drainages, sports grounds and other types of constructions that require high mechanical stress.

Introduction

Despite the global trend of protecting the environment by substantially reducing greenhouse gas emissions, large amounts of coal are burned in thermal power stations, the main by-products generated being fly ash (captured in electrofilters), bottom ash, boiler slag and waste gas desulphurization gypsum. The storage of these by-products could constitute major environmental problems. But currently, there are several high value-added application solutions for the by-products mentioned above. The fly ash is used in the manufacture of cement and concrete, soil fertilization, in the ceramic industry, as adsorbents for the removal of waste gases, organic compounds or heavy metals, for the synthesis of zeolite and geopolymers, for the recovery of precious metals, etc. The slag boiler is used for wastewater treatment, manufacture of concrete, cement, acoustic protection materials, pavements in road construction, etc. The application field of waste gas desulphurization gypsum includes using as a fertilizer and soil amendment agent, making the building plaster and calcium sulphate whiskers, making the fire-resistant panels, etc. (Li et al., 2017).

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Coal fly ash is a fine-grained powder and is made up of spherical glassy particles. The ash contains several valuable mineral resources such as SiO2, Al2O3, CaO, Fe2O3, etc. The chemical composition and fine granulation of the powder recommend this by-product for using as a raw material in the manufacture of glass-ceramics. Generally, the glass-ceramic properties are influenced by the main crystallization phases and their microstructures. It is difficult to manufacture glass-ceramic using only fly ash, without other additives because in most cases the ash does not contain adequate ratios of its components to make this ceramic material. The addition of network modifiers is necessary to achieve the full vitrification of fly ash. Na2O is the most efficient network modifier that can reduce the melting temperature and viscosity of the raw material and facilitates processing operations. In addition to the network modifiers, the main components of the raw material such as CaO and MgO and the nucleating agents TiO2 and ZrO2 are also required (Wang et al., 2015).

Coal fly ash is suitable for synthesizing CaO-Al2O3-SiO2 system glass ceramics. This system has several excellent properties such as high mechanical strength, dimensional stability, corrosion resistance. The main phases of this glass-ceramics system commonly contain anorthite, wollastonite, gehlenite and diopside (Wang et al., 2015). Numerous manufacturing process of glass-ceramic using fly ash as raw material together with glass waste and/or other silicate waste were experimentally tested. A light glass-ceramic was made from a silicate waste powder mixture composed of 20% coal fly ash and 80% glass waste. The method of sintering the pressed powder mixture at 1000-1050 ºC was used, in which 2% silicon carbide was added as a foaming agent. The apparent density of the expanded material was 0.2-0.4 g/cm3, porosity had values between 70-90% and compressive strength was around 1.5 MPa (Wu et al., 2006). A similar experiment using the same types of raw material and silicon carbide as a foaming agent in a weight ratio of only 1% was performed at a sintering temperature of 950 ºC with an average heating rate of 23.2 ºC/min. The process led to a glass-ceramic with an apparent density between 0.18-0.35 g/cm3, a compressive strength between 0.9-1.8 MPa and a homogeneous distribution of pores with dimensions between 1-3 mm (Fernandes et al.,2009).

Ultra-light glass-ceramics were manufactured using 30% fly ash, 70% glass waste, 15% sodium borate (borax) as a fluxing agent and 0.5% silicon carbide. The material sintered at 680 ºC had the apparent density of 0.14 g/cm3, porosity of 94.1%, thermal conductivity of 0.07 W/m•K and compressive strength of 0.91 MPa (Mi et al., 2017). Higher weight ratios of fly ash (50-70%) were used in the glass-ceramic manufacturing process presented in (Chen et al., 2011) together with sodium borate as a fluxing agent and sodium silicate as a foaming agent. The optimal sample was obtained after sintering at 800 ºC using 70% fly ash. The sample had excellent comprehensive properties, the value of the compressive strength reaching 3.44 MPa. All glass-ceramic foams manufactured by the techniques presented above were achieved by conventional heating methods. Glass-ceramic foams were manufactured (Dragoescu et al., 2019) using as raw material green container glass (52.8-66.0%), old clay brick waste (22.0-35.2%) and coal fly ash (9%) as well as silicon carbide (3%) as a foaming agent. The adopted manufacturing technique was a mixed microwave heating (partly direct, partly indirect). The experiments were performed in a 0.8 kW-microwave oven, the sintering temperature being between 1000-1060 ºC and the average heating rate being 16.8-18.5 ºC/min. The specific energy consumption had values between 2.91-3.46 kWh/kg. The products had the apparent density between 0.53-0.78 g/cm3, porosity between 68.8-78.3% and compressive strength in the range 1.62-3.35 MPa, the highest value corresponding to the sample with the maximum clay ratio (35.2%). Another method of manufacturing glass-ceramic foams using clay brick waste (75-83%), coal ash (15-23%), silicon carbide (2%) and water addition (25%) is presented in the paper (Paunescu et al., 2020a). The ceramic foam was produced in the 0.8 kW-microwave oven by the technique of the direct microwave heating. The sintering temperature had values in the range 1115-1145 ºC and the average

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heating rate was between 31.3-35.3 ºC/min. The specific energy consumption had very low values between 0.74-0.85 kWh/kg. The foamed products had the apparent density between 0.50-0.68 g/cm3, porosity between 59.5-68.6% and compressive strength in the range 3.8-7.5 MPa.

The paper (Axinte et al., 2020) presents results obtained in the experimental manufacture of a silicon carbide ceramic foam from silicon carbide (40-75%), red clay waste (15-40%), coal fly ash (10-20%) and water addition (17%). The tests occurred in a 0.8 kW-microwave oven by direct microwave heating at the sintering temperature between 1560-1610 ºC with the average heating rate between 30.6-32.8 ºC/min. The specific energy consumption had values between 1.29-1.42 kWh/kg. The ceramic foam had the apparent density between 1.05-1.39 g/cm3, porosity between 36.8-51.4% and compressive strength in the range 28.3-58.0 MPa. In the paper (Ma et al., 2018), a conventional manufacturing technique of a high strength glass-ceramic foam is presented using extremely high coal fly ash content (between 83-95%), calcium carbonate (between 3-9%) as a foaming agent and Na2O (5%) as a fluxing agent. The sintering temperature was 1150 ºC. The samples characterization led to the following results: bulk density between 1.55-1.59 g/cm3, apparent porosity in the range 16.8-19.9%, bending strength between 43.4-109.6 MPa, thermal expansion coefficient between 4.1-5.6•10-6 K-1. The sample made with 6% CaCO3 shows the best microstructure homogeneity with pore size between 0.1-0.4 mm.

Previous tests performed in the Romanian company Daily Sourcing & Research have shown that the microwave heating for sintering/foaming powder mixtures of any aluminosilicate material can be done directly with maximum efficiency without affecting the core structure of the material. These structural defects have been identified in all cases where the raw material is glass powder or it is in a relatively high proportion in a mixture based on aluminosilicate materials (Paunescu et al., 2017). According to the literature (Kitchen et al., 2014), the direct microwave heating of a solid material is initiated in its core, where a high temperature is reached in a short time. The heat transfer through the mass of the material is done from the inside to its peripheral areas. Another important feature of the direct microwave heating is its selective character (Jones et al., 2002), being heated only the material subjected to this process, not the other massive components of the oven (walls, vault, hearth). These completely different characteristics compared to the conventional heating methods (electrical resistances or burning fossil fuels) lead to a reduction of the heating process time and to a very low specific energy consumption. In the case of the experiment described in this paper, the raw material is coal fly ash, whose chemical composition shown in Table 1 indicates that it is an aluminosilicate material.

Method

Previous tests performed in the Romanian company Daily Sourcing & Research have shown that the microwave heating for sintering/foaming powder mixtures of any aluminosilicate material can be done directly with maximum efficiency without affecting the core structure of the material. These structural defects have been identified in all cases where the raw material is glass powder or it is in a relatively high proportion in a mixture based on aluminosilicate materials (Paunescu et al., 2017). According to the literature (Kitchen et al., 2014), the direct microwave heating of a solid material is initiated in its core, where a high temperature is reached in a short time. The heat transfer through the mass of the material is done from the inside to its peripheral areas. Another important feature of the direct microwave heating is its selective character (Jones et al., 2002), being heated only the material subjected to this process, not the other massive components of the oven (walls, vault, hearth). These completely different characteristics compared to the conventional heating methods (electrical resistances or burning fossil fuels) lead to a reduction of the heating process time and to a very low specific energy consumption. In the case of the experiment described in this paper,

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the raw material is coal fly ash, whose chemical composition shown in Table 1 indicates that it is an aluminosilicate material.

Table 1. Chemical composition of coal fly ash

Component	SiO_2	Al_2O_3	CaO	MgO	Na_2O	K_2O	Fe_2O_3
Weight ratio, wt.%	46.5	23.7	7.9	3.2	6.0	4.1	8.6

Considering that during the direct microwave heating the heat transfer is done from the inside of the material to the outside, a very efficient thermal protection of the material is required. The solution adopted by the authors was the use of several layers of ceramic fiber mattresses that wrap the pressed powder material (Figure 1a). This is placed at the base of the oven on a bed of ceramic fiber mattresses. Also, the same type of thermal protection is provided to cover the upper area of the material. The heating oven was a 0.8 kW-microwave oven of the type commonly used in the household for food preparing adapted for operation at high temperature (Figure 1b). The oven rotation mechanism has been deactivated. The control of the heating process of the material was performed with a radiation pyrometer (with the measuring domain between 600-2000 ºC) mounted above the oven at about 400 mm (Figure

1b). To visualize the upper surface of the heated material, the upper metal wall of the oven was provided with a hole with a diameter of 30 mm and also a similar hole was made in the ceramic protection layer of the upper area of the material.

A **B**

Figure 1. Pictures of the experimental microwave equipment

a - ceramic fiber protection of the pressed powder material; b - overall image of the 0.8 kW-microwave oven including the radiation pyrometer positioning

The main chemical reactions of the foaming process are: decomposition at over 400 ºC (Toxic, 1988) of sodium carbonate:

$$Na_2CO_3 = Na_2O + CO_2 \quad (1)$$

$$Na_2O + SiO_2 = Na_2SiO_3 \quad (2)$$

decomposition in the temperature range 750-900 ºC (Ducman, 2004; Karundasa et al., 2019) of calcium carbonate:

$$CaCO_3 = CaO + CO_2 \quad (3)$$

Na_2O resulting from the decomposition of Na_2CO_3 acts as a fluidifier reducing the fluxing temperature. By its reaction with SiO_2 (reaction 2) from the silicate composition

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results Na_2SiO_3 (sodium silicate or "water glass") which is an usual binder of solids contributing to the increase of the mechanical strength of the foamed product.

The materials used in the process of manufacturing glass-ceramic foams were coal fly ash, calcium carbonate as a foaming agent, sodium carbonate as a fluxing agent (being a supplier of Na_2O) and water addition. Coal fly ash was purchased from Paroseni (Romania) thermal power station having the chemical composition shown in Table 1 and the initial grain size below 250 μm, which was reduced to below 80 μm by grinding in a ball mill and sieving. Calcium carbonate used in experiments had the grain size below 40 μm. Sodium carbonate with an initial grain size below 350 μm was ground in a laboratory electrical device and sieved below 80 μm.

The water addition was necessary because the silicate materials foaming with calcium carbonate is possible only with wet material particles to avoid the particle adhesion due to the partial leaching of alkali metal oxides (Na_2O, K_2O) at temperatures below the foaming temperature with calcium carbonate (Scarinci et al., 2005).

Results and Discussion

Based on some data provided by the literature and the own experience, four experimental variants were adopted to be tested in conditions of the direct microwave heating (Table 2).

Table 2. Experimental variants of glass-ceramic foams

Component	Variant 1	Variant 2	Variant 3	Variant 4
Coal fly ash, wt.%	80	82	84	86
Calcium carbonate, wt.%	7	5	3	1
Sodium carbonate, wt,%	13	13	13	13
Water addition, wt.%	10	10	10	10

The main functional parameters of the manufacturing process of glass-ceramic foam performed on the 0.8 kW-microwave oven are presented in Table 3.

Table 3. Main functional parameters of the foaming process

Parameter	Variant 1	Variant 2	Variant 3	Variant 4
Dry/wet raw material amount, g	500/550	500/550	500/550	500/550
Process temperature, ºC	850	853	855	856
Heating time, min	25	26	27	28
Average rate, ºC/min heating - cooling	 33.2 5.3	 32.0 5.1	 30.9 5.4	 29.9 5.3
Index of volume growth	1.60	1.40	1.30	1.25
Glass-ceramic foam amount, g	480	482	481	480
Specific energy consumption, kWh/kg	0.70	0.72	0.74	0.78

According to the data in Table 3, the measured temperature of the experimental foaming process of coal fly ash varied between 850-856 ºC, its value increasing slightly with the increase of the weight proportion of coal ash. Due to the excellent energy efficiency of the direct microwave heating, the heating rate had very high values (between 29.9-33.2 ºC/min) compared to the heating rate of the conventional process (between 10-20 ºC/min)

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and the process time was very short (25-28 min). Consequently, the value of the specific energy consumption had very low values (0.70-0.78 kWh/kg). The physical, mechanical and microstructural characteristics of the glass-ceramic foam samples were determined by common methods. The apparent density was measured by the gravimetric method (Manual, 1999) and the porosity was calculated by the method of comparing the true and apparent density (Anovitz & Cole, 2005). The thermal conductivity was determined by the guarded-comparative-longitudinal heat flow (ASTM E1225-04 standard) and the compressive strength was measured using a Stable Micro Systems TA XT Plus Texture Analyzer. The water absorption was determined by the water immersion method (ASTM D570 standard). The samples microstructure was examined with a Smartphone Digital Microscope. To investigate the crystallographic structure of the glass-ceramic, X-ray diffraction (XRD) was used according to the standard EN 13925 – 2: 2003 using a X-ray diffractometer Bruker-AXS D8 Advance with CuKα radiation. The main physical, mechanical and microstructural characteristics of the glass-ceramic foam samples are presented in table 4.

Table 4. Physical, mechanical and microstructural characteristics of samples

Variant	Apparent density g/cm^3	Porosity	Thermal conductivity W/m·K	Compressive strength MPa	Water absorption %	Pore size
1	1.41	27.7	0.275	38.4	0.8	0.3 – 0.8
2	1.44	26.2	0.281	41.3	0.5	0.2 – 0.5
3	1.48	24.1	0.284	47.2	0.6	0.2 – 0.5
4	1.50	23.1	0.289	52.8	0.3	0.1 – 0.3

According to the data in Table 4, the glass-ceramic foam samples made of coal fly ash were dense, with an apparent density between 1.41-1.50 g/cm^3 and thermal conductivity between

0.275-0.289 W/m·K. The samples porosity was low (between 23.1-27.7%). The main physical-mechanical characteristic aimed by adopting the type and weight ratio of the starting materials was the compressive strength that had high values between 38.4-52.8 MPa, comparable with the silicon carbide ceramic foam (28.3-47.6 MPa (Axinte et al., 2020) and 44.8-56.5 MPa (Paunescu et al., 2020b), respectively), a porous material sintered at high temperature recognized for its high mechanical strength. Also, the glass-ceramic foam samples were almost waterproof (below 0.8% the water absorption).

Figure 2 shows pictures of the four glass-ceramic foam samples having the appearance of dense materials (especially samples 2-4) and Figure 3 shows microstructural images of the samples.

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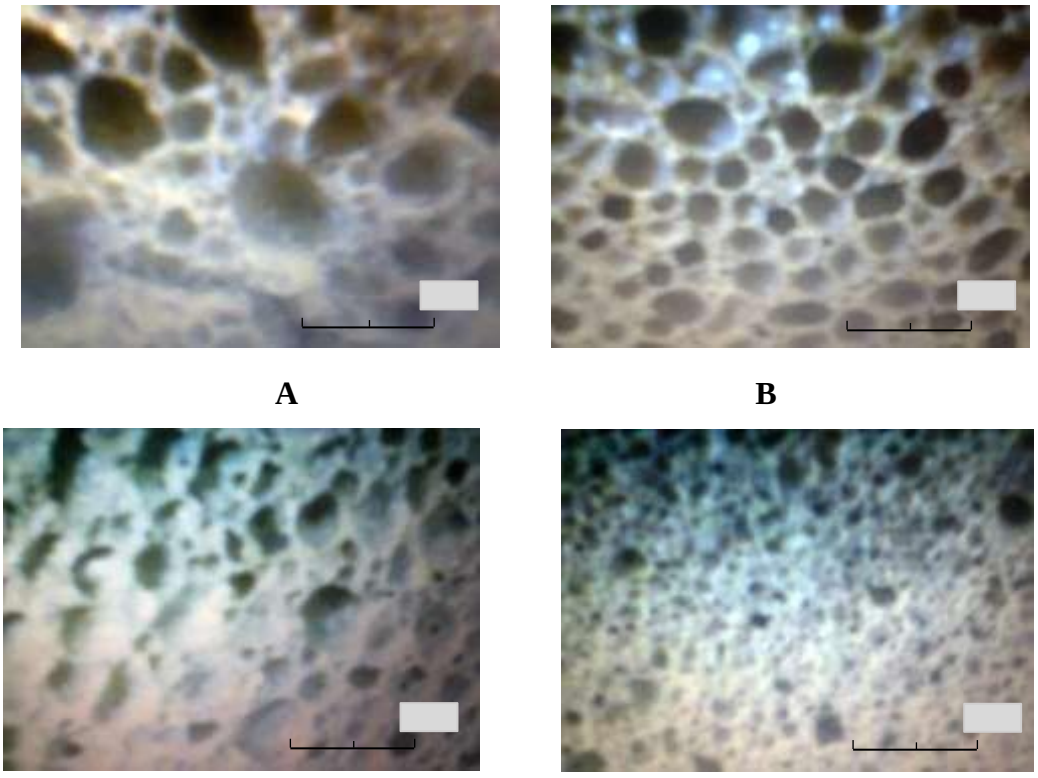
A **B**

C **D**

Figure 2. Pictures of the glass-ceramic foam samples

a - sample 1, heated at 850 ºC; b - sample 2, heated at 853 ºC;

c - sample 3, heated at 855 ºC; d - sample 4, heated at 856 ºC.



A **B**

C **D**

Figure 3. Microstructural images of the glass-ceramic foam samples

a - sample 1; b - sample 2; c - sample 3; d - sample 4

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According to Figure 3, the four samples have microstructures with variable pore size from the dimensions range 0.3-0.8 mm (sample 1) up to the range 0.1-0.3 mm (sample 4). In terms of microstructural homogeneity, sample 2 is obviously the best, the pore size ranging between 0.2-0.5 mm. The criterion of the uniformity of pore distribution in the ceramic material section was the one predominant in choosing sample 2 as the optimal sample of the experiments presented in the paper. Sample 2 was made of 82% coal fly ash, 5% calcium carbonate as a foaming agent, 13% sodium carbonate as a fluxing agent and 10% water addition. The temperature of the sintering/foaming process was 853 ºC, the average heating rate was 32 ºC/min and the specific energy consumption was 0.72 kWh/kg. The physical and mechanical characteristic of the sample were: apparent density of 1.44 g/cm^3, porosity of 26.2%, thermal conductivity of 0.281 W/m·K, compressive strength of 41.3 MPa and water absorption of 0.5%. The XRD analysis of sample 2 identified the presence of the following crystalline phases in the glass-ceramic foam thermal treated at 853 ºC: albite, anorthite, gehlenite, wollastonite and diopside. Given the characteristics of the optimal glass-ceramic foam sample (very high compressive strength, acceptable porosity and thermal conductivity, very low water permeability, fireproof, chemical stability, no-toxicity, etc., the application domain of this material type may include road and railway construction, bridge abutments and retaining walls, foundations, drainages, sports grounds and other types of constructions that require high mechanical stress.

Due to the type of the used raw material, the glass-ceramic foam made of coal fly ash is a low-cost building material being a viable option for replacing similar materials on the market. It should also be mentioned that the used technique of direct microwave heating of the powder mixture based on coal fly ash, suitable for aluminosilicate materials, allows to obtain very low specific energy consumptions due to the peculiarities of the microwave absorption and heat transfer mode inside the material mass, completely different from the characteristics of the conventional heating.

Conclusion

The paper aimed to find a technique for manufacturing a high mechanical strength glass-ceramic foam using as raw material a very high weight proportion (82%) of coal fly ash, calcium carbonate (5%) as a foaming agent, sodium carbonate (13%) as a fluxing agent and water addition (10%). The direct microwave heating technique was used as the energy source to obtain a very low specific energy consumption of 0.72 kWh/kg. From the four performed experimental tests, the optimal sample was chosen based on microstructural features (uniformity of pore distribution). The physical and mechanical characteristics of the optimal sample were: apparent density of 1.44 g/cm3, porosity of 26.2%, thermal conductivity of 0.281 W/m•K, compressive strength of 41.3 MPa and water absorption of 0.5%. The sintering/foaming process occurred at 853 ºC. Given the characteristics of the optimal glass-ceramic foam sample (very high compressive strength, acceptable porosity and thermal conductivity, very low water permeability, fireproof, chemical stability, no-toxicity, etc., the application domain of this material type may include road and railway constructions, bridgen abutments and retaining walls, foundations, drainages, sports grounds and other types of constructions that require high mechanical stress.

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