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CELLULAR GLASS-CERAMIC FROM RECYCLED RESIDUAL GLASS, METAKAOLIN AND COAL FLY ASH

BY

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Abstract. In this paper, the care for environmental health was promoted, selecting raw materials that meet these requirements. Thus, recycled glass waste in predominant proportion, coal fly ash as a by-product of the energy industry, and a natural alumina-silicate material (metakaolin) were used. Silicon carbide (SiC) was chosen as an expanding agent. Although insignificantly used until now for high-temperature industrial processes, the microwave heating has been adopted to perform the sintering and expanding process. The results showed very good thermal insulation properties and high mechanical strength of the optimal expanded product.

Keywords: glass-ceramic, cellular, glass, metakaolin, coal fly ash.

1. Introduction

Considering the most modern applications of glass-ceramics, especially in the bio-medical field, this material is considered as an inorganic and non-metallic product containing at least one crystalline phase in an amorphous glassy

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matrix (Shearer *et al*., 2023). The remarkable combination of the glass-ceramic properties as well as the wide range of products made from them in the last 50-60 years create their peculiarity compared to traditional glass and ceramic products. The processing methods (firing glass and ceramics together, manufacturing based on additives as well as the use of laser) led to the completion of the glass-ceramic definition, its preparation being achievable through the controlled crystallization (devitrification) of glass (Deubener *et al*., 2018).

The current work was focused on techniques for the preparation of cellular glass-ceramic for thermal insulation applications. Several silicate-based wastes such as coal fly ash, metallurgical slag, filter dust from urban waste incinerators, red mud from hydrometallurgy as well as residual glass were in the attention of researchers and manufacturers of glass-ceramics. Rawlings *et al.*, 2006 show in a review of the development of waste-based glass-ceramic important accumulated knowledges regarding processes of turning silicate waste into valuable glass-ceramic products that can be used especially in construction as heat-insulating materials.

In another work (Taurino *et al*., 2014), the results of cellular glass-ceramic production by sintering residual borosilicate glass using an organic binder from dismantling the washing machines are reported. The sintering process took place at a relatively low temperature (up to 900°C). The softening initiation at 800°C was continued by crystallization at 845°C, being completed at 900°C with a volume growing of the expanded material of 40% within interval of about 50°C. The recycled glass was finely ground below 37 μm. The heating rate during sintering process was 10°C/min until the crystallization temperature was kept constant for 10 min. The apparent density decreased to 0.48 $g \cdot cm^{-3}$ and the total porosity had grown from 68 to 79%.

A method frequently used in the literature for making cellular glass-ceramics combining coal fly ash and residual glass for thermal insulating applications is presented in the paper (Zhu *et al*., 2016). The preparation of a glass foam using residual glass and fly ash is also mentioned in the work (Bai *et al*., 2014). The adequate temperature of the expanding process was around 950°C. Silicon carbide (SiC) was the blowing agent chosen in this work, which through oxidation has the ability to release CO_2. The results showed that foaming of the mix of glass and ash powders at 950°C for 20 min led to increasing by 5.8 times of the initial volume of the mix. The froth had a bulk density of 0.267 $g \cdot cm^{-3}$, compressive strength of 0.983 MPa and porosity of 81.5%.

According to Zhou *et al.*, 2022, a cellular glass-ceramic was made at 1000°C through sintering process utilizing recycled residual glass and titanium rich-blast furnace slag (Fan *et al.*, 2021). Borax as a flux agent was added, having effects on crystalline phase composition, physio-mechanical and heat properties as well as cellular glass-ceramic microstructure. According to the experimental outcomes, it has been found that borax contributes to lowering the softening temperature and also the viscosity, favouring expanding and crystallizing

processes. The crystalline phases of the foamed product remained unchanged, regardless of the added borax content. By increasing the borax content, the density, heat conductivity, and compression strength decreased, while porosity and water uptake increased. The optimal proportion of borax addition (6 wt. %) indicated the best performances of the cellular glass-ceramic obtained in this experiment.

In the case of hazardous wastes whose vitrification is required, the use of slag has become an important component of reducing the cost of this operation. The recent research (Long *et al.*, 2024) proved the potential of preparing cellular glass-ceramics by sintering vitrification slag and biochar. The temperature required for the process was evaluated at 1000-1050°C. Turning the phase and structure of vitrification slag/biochar system during the sintering process showed that biochar has favoured the formation of aluminium oxide (Al_2O_3). However, the amorphous glassy phase was still predominant. The generation of the crystalline phase is essential in the formation of pores. In view of work's authors, an excellent glass froth with evenly distributed honeycomb structure could be created by sintering the vitrification slag with 2 wt. % C as well as 2 wt. % sodium pyrophosphate ($Na_3P_2O_7$) at 1050°C for 60 min.

A particular role in making processes of porous products through raw material expansion evidenced in the literature is played by clay powder. Several works (Ercenk, 2017; Susilawati *et al*., 2020; Iranfar *et al*., 2023) have used this addition material for the purpose of increasing the mechanical strength of froths resulting from applied foaming processes.

Some authors of the current paper had performed experimental research on producing cellular glass-ceramics since 2019 and results were published in the literature (Dragoescu *et al.*, 2019). Preparing cellular glass-ceramic was based on the solid mixture finely ground of residual glass, old clay brick waste recovered from building demolition, and coal fly ash, the chosen expanding agent being SiC. Sintering/expanding temperature of raw material was varied between 1000-1060°C, heating duration was between 53-62 min, and average heating rate had quite high values in the range of 16.8-18.5°C/min. Final products had apparent density between 0.53-0.78 $g \cdot cm^{-3}$, heat conductivity within the limits of 0.080-0.099 $W \cdot m^{-1} \cdot K^{-1}$, compressive strength between 1.62-3.35 MPa, and water uptake in the range of 3.1-4.0 vol. %. The special peculiarity of this experiment was the use of unconventional electromagnetic wave heating, a procedure that, despite its remarkable energy efficiency, is not applied in industrial processes of heating solids at high temperatures. There are known microwave utilizations on an industrial scale, but only in drying processes and in heating processes at low temperatures (Kharissova *et al*., 2010).

Few examples are known in the world, which confirm a real interest of researchers and manufacturers in hot sectors of industry to replace traditional conventional heating systems in industrial furnaces operating at high

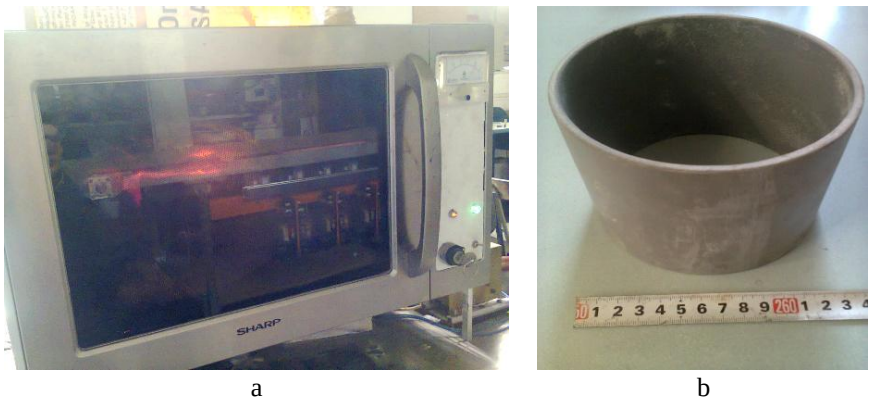
temperatures with the unconventional systems mentioned above (Hurley, 2003; Knox and Copley, 1997; Kitchen *et al.*, 2014; Jones *et al.*, 2002).

Under these conditions characterized by the lack of interest and confidence in the possibility of using microwaves in high thermal requirement processes, the team of researchers from Daily Sourcing & Research SRL (Romania) carried out several researches on foaming glass-based mixtures including suitable expanding agents and have applied electromagnetic waves successfully. Aiming at the manufacture of cellular glass-ceramic with improved performances compared to the previous experiment from 2019 (Dragoescu *et al*., 2019), the technique of using microwaves was kept as well as the experimental equipment intended for the realization of thermally efficient heating process.

On the other hand, the recovery of aged clay from the demolition of buildings was abandoned and the use of metakaolin available on the market was opted for. This alumina-silicate material is an anhydrous calcined state of kaolinite, a clay mineral. One of its major roles is to increase the resistance of the material in the composition of which it is added (often concrete, Basavaraj and Ravikumar, 2022), which in the case of a glass-ceramic froth this desideratum is of great interest.

2. Methods and Materials

As mentioned above the main peculiarity of the heating process used in this experiment was the application of unconventional technique of irradiation with microwave flux. A household microwave oven of 800 W, constructively and operationally adapted for operation at high temperatures of over 1100°C (Fig. 1a) was successfully used in several heating processes of this type in the Romanian company Daily Sourcing & Research.



a b

Fig. 1 – The experimental equipment
a – 800 W-adapted microwave oven; b – SiC and Si_3N_4-ceramic tube.

The oven is equipped with a single microwave generator placed on one of its side walls. To reduce the too strong effect of electromagnetic waves on the glass-based mixture, previously observed, between the heated sample and the emitting source, a ceramic tube made of SiC and Si_3N_4 in the 80/20 ratio procured from China (Fig 1b) was placed. The optimal thickness of the tube wall, experimentally determined, was 2.5 mm, which allows the predominant partial penetration of the microwave flow to come into contact with the sample. A lower proportion of waves was absorbed within the tube wall mass, heating it strongly. The direct specimen heating takes place by initiating this process in the volume core of the irradiated material, which then transmits the heat initiated in this central place to peripheral areas of the sample. The direct microwave heating is completely opposable than the traditional conventional heating processes. Volumetric propagation in the entire specimen mass ensures its rapid and effective heating (Kitchen *et al*., 2014; Jones *et al*., 2002). The combination of direct and indirect heating due to the radiation of the hot wall of the tube contributes to the remarkable energy efficiency of this thermal process (Axinte *et al*., 2019).

The previous predominantly direct heating processes of glass-based raw materials made by the Romanian team proved that, despite the much higher average heating rate (over 20℃/min) compared to the rates frequently used in conventional processes (below 10℃/min), the structural characteristics of the expanded specimens were not affected.

Materials used in the current experiment were: post-consumer green and colourless drinking glass, metakaolin, and coal fly ash as raw materials as well as SiC as an expanding agent.

Recycled glass was separated by colour, washed, broken, ground in a ball mill, and dimensionally selected by sieving. The grain size of waste was under 70 μm.

Metakaolin (or calcined clay) was purchased from the market, originating from China. The maximum size of its particles was 37 μm.

The coal fly ash from Paroseni Thermal Power Plant (Romania), stored as reserve material since 2016, had the grain size below 250 μm, additional grinding being necessary to lower the upper limit below 50 μm.

The chemical composition of residual glass, metakaolin, and coal fly ash is shown in Table 1.

Table 1

Chemical composition of raw materials (wt. %)

Raw material	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O
Geen glass	71.8	1.9	-	11.8	1.2	0.1	13.1
Colourless glass	71.7	1.9	-	12.0	1.0	-	13.3
Metakaolin	54.1	42.0	0.35	0.01	0.06	0.2	0.13
Coal fly ash	49.8	23.5	6.1	3.6	3.1	4.0	1.6

Silicon carbide (SiC) is considered by specialists as a very effective expanding agent having the ability to form a uniform and controlled cellular structure of glass froths. The main types of cellular glass (blocks and shapes) are generally produced using SiC. The temperatures at which SiC oxidation occurs with the release of CO_2 as an expanding gas are within the limits of 950-1150°C (Scarinci *et al*., 2005). The grain size of SiC utilized during the experiment was under 10 μm.

Investigation of physico-mechanical, heat, and microstructural features of cellular glass-ceramic specimens made in this experiment was achieved using common analysis procedures. The apparent density was performed by the gravimetric method (Manual, 1999) and the porosity was identified by comparing method of the true and apparent density according to (Anovitz and Cole, 2015). The apparent density of the froth was determined by measuring the mass and dimensions of specimens, while the true density of the powdered froth was determined with a picnometer. The heat conductivity was measured by the heat-flow meter method (ASTM E1225-04). The compressive strength was determined with TA.XTplusC Texture Analyzer from Stable Micro Systems (ASTM C552-17). Water uptake was measured by the water-immersion method (for 24 hours) according to ASTM D570 and the microstructure appearance of specimens was analyzed using ASONA 100X Zoom Smartphone Digital Microscope. The oxide composition of raw materials was identified with X-ray fluorescence spectrometer AXIOS type.

3. Results and Discussion

Four mixture combinations have been tried within this experiment. The composition and dosage of each component material, including green and colourless residual glass in approximately equal proportions, metakaolin, coal fly ash, SiC, and distilled water addition are indicated in Table 2.

Table 2
Composition of mixture versions

Composition	Version 1 (wt. %)	Version 2 (wt. %)	Version 3 (wt. %)	Version 4 (wt. %)
Residual glass	70.1	69.0	67.6	65.7
Metakaolin	16.0	16.0	16.0	16.0
Coal fly ash	12.5	13.3	14.4	16.0
SiC	1.4	1.7	2.0	2.3
Distilled water addition	20.0	20.0	20.0	20.0

The data in Table 2 shows that the weight proportion of expanding agent (SiC) was significantly grown from 1.4 to 2.3 wt. %. The same increasing trend

had also coal fly ash in the range of 12.5-16.0 wt. %, creating conditions for facilitating the development of cellular structures (Zhang *et al*., 2007). Metakaolin, dosed at the constant value of 16 wt. % for all four experimental versions, was introduced into the starting mixture due to its ability to increase the strength of the ceramic froth. The residual glass amount had high values within the limits of 65.7-70.1 wt. %. Additionally, 20 wt. % of distilled water was required.

Basic operational parameters specific to the cellular glass-ceramic making process by unconventional microwave heating are indicated in Table 3.

Table 3
Operational parameters

Parameter	Version 1	Version 2	Version 3	Version 4
Dry/wet raw material amount (g)	350/ 421	350/ 422	350/ 420	350/ 421
Process temperature (°C)	965	970	980	995
Heating time (min)	36	38	41.5	45
Average rate (°C/min) - heating - cooling	26.25 5.0	25.00 4.9	23.13 5.1	21.67 5.0
Cellular glass-ceramic amount (g)	332.5	334.2	332.7	331.9
Specific energy consumption ($kWh \cdot kg^{-1}$)	1.13	1.18	1.30	1.41

The total dry quantity of solids, as components of each mixture used in versions specified in Table 2, was kept constant at 350 g. The sintering/expansion process took place at temperatures between 965 C (version 1) and 995 C (version 4), while the process duration has grown from 36 min (version 1) to 45 min (version 4). Correspondingly, the average heating rate reached the highest value (26.25°C/min) in the case of version 1, while the lowest value (21.67°C/min) corresponded to version 4. Considering the quantities of the final foamed product exposed in Table 4 (between 331.9-334.2 g), the specific energy consumption corresponding to each experimental version was within the limits of 1.13-1.41 $kWh \cdot kg^{-1}$, the minimum value being attributed to version 1.

As mentioned in the work (Scarinci *et al*., 2005), SiC utilization as an expanding agent imposed the temperature range at which CO_2 can be released as a result of SiC-oxidation reaction, i.e. above 950°C. Limiting to the minimal thermal level the sintering process, including also the mix expanding, led to the creation of a fine cellular structure, which did not benefit from sufficient time and temperature to grow in size. Increasing the temperature to higher levels facilitated the expansion of the cells containing CO_2 released, due to increasing the internal

pressure of cells. In experimental version 4, characterized by conducting the heating process up to the highest temperature of 995°C, the degree of expansion of the gas cells reached a level at which the volume increase of the porous structure was the highest compared to the other versions. The size of cells has considerably increased. In many cases of similar experiments, the intercommunication of neighbouring cells was reached.

Surface images of each specimen, identifying the peculiarities of cell distribution, are presented in Fig. 2.

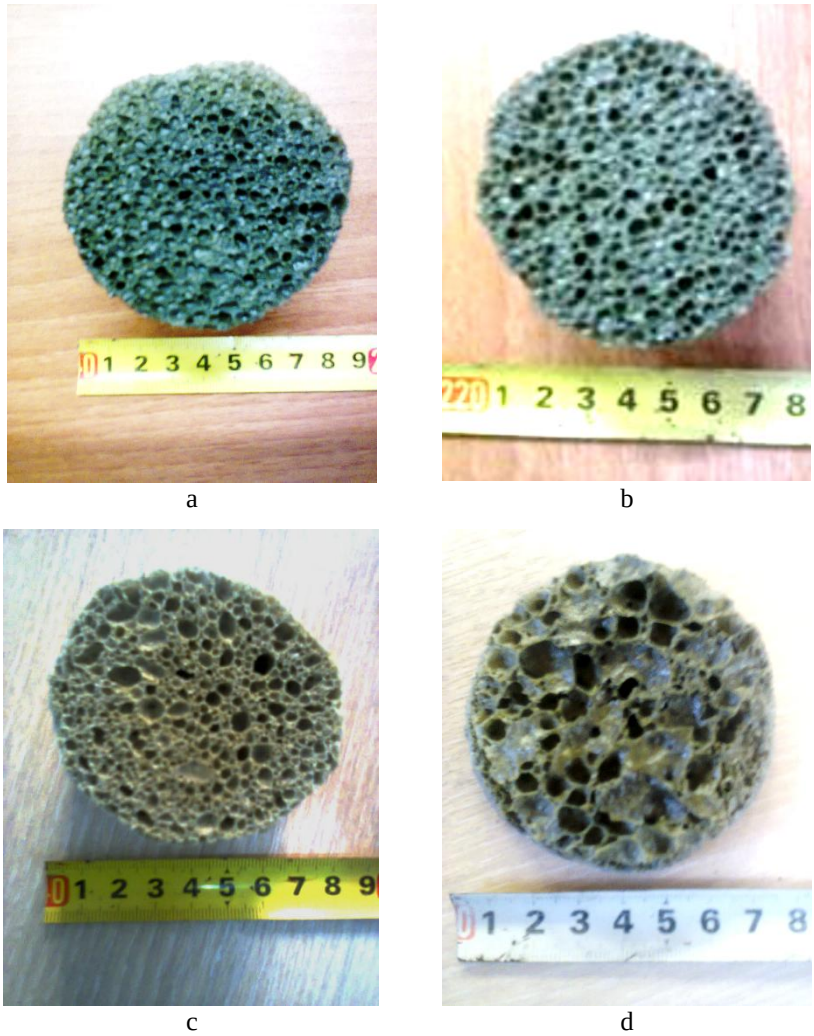


Fig. 2 – Surface images of cellular glass-ceramic specimens
a – version 1; b – version 2; c – version 3; d – version 4.

Using the investigation methods mentioned above, the physico-mechanical, heat, and microstructural characteristics were determined. Results are presented in Table 4.

Table 4

Physico-mechanical, heat, and microstructural features of the cellular products

Feature	Version 1	Version 2	Version 3	Version 4
Apparent density ($g \cdot cm^{-3}$)	0.73	0.60	0.52	0.42
Porosity (%)	71.4	75.0	81.2	84.9
Heat conductivity ($W \cdot m^{-1} \cdot K^{-1}$)	0.092	0.080	0.071	0.062
Compressive strength (MPa)	5.62	4.84	2.96	1.02
Water uptake (vol. %)	4.1	3.8	3.4	3.1
Pore size (mm)	0.6-1.2	1.2-2.3	1.3-2.7	2.3-4.6

Analyzing the main experimental results showed in Table 4, the excellent values of the compressive strength can be observed, especially of specimens 1 (5.62 MPa) and 2 (4.84 MPa), but even of specimen 3 (2.96 MPa). Also, heat conductivity has values at the level of requirements for applications in the field of thermal insulation materials in the case of specimens made by experimental versions 2-4 (between 0.080 and 0.062 $W \cdot m^{-1} \cdot K^{-1}$). The most appreciable values of the apparent density were also obtained in the case of versions 2-4 (between 0.60-0.43 $g \cdot cm^{-3}$).

The compressive strength offers a wide range of values in the four experimental versions reaching very good values for a cellular material, especially the specimen corresponding to version 1 (5.62 MPa), but the thermal insulation properties (density, heat conductivity) are the least adequate of all the versions tried in this experiment.

On the other hand, the sample made by version 4 has a rather low value of the compressive strength (only 1.02 MPa), obviously the lowest compared to those of versions 1-3, despite the fact that version 4 benefits from the more advantageous values of density (0.43 $g \cdot cm^{-3}$), heat conductivity (0.062 $W \cdot m^{-1} \cdot K^{-1}$), and porosity (84.9%).

Comparing the four experimental versions, it is obvious that the choice of the optimal version means a decision in which the accepted ratio of physico-thermal properties in comparison with the mechanical ones is established. Based on these considerations, it seems that the specimen made by version 2 could be the optimal solution. Having a fairly high compressive strength (4.84 MPa), apparent density of 0.60 $g \cdot cm^{-3}$ and heat conductivity of 0.080 $W \cdot m^{-1} \cdot K^{-1}$, this

sample seems to ideally combine mechanical resistance and thermal insulation properties.

Peculiarities of microstructural appearance of the four cellular products are shown in Fig. 3.

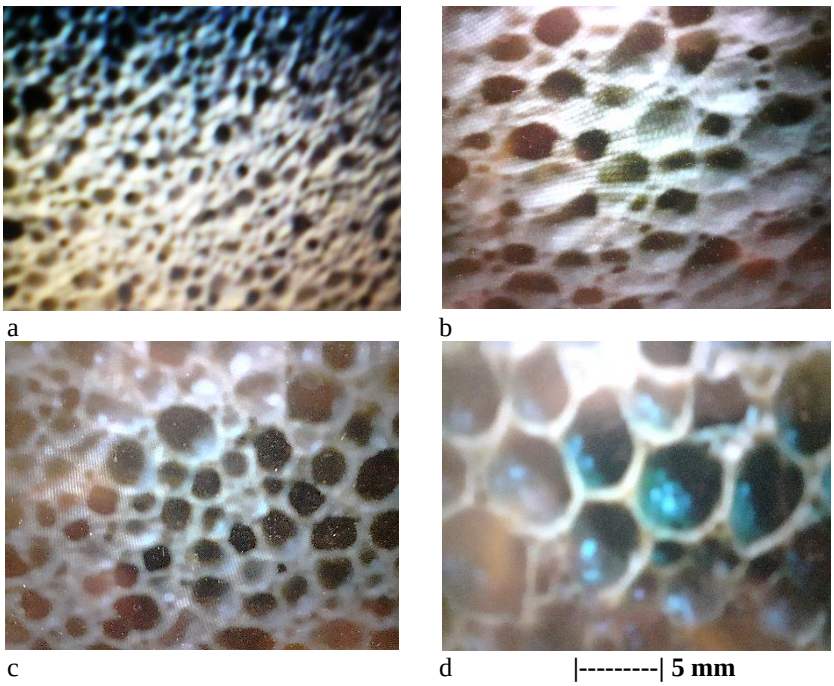


Fig. 3 – Peculiarities of microstructural appearance of the cellular products
a – version 1; b – version 2; c – version 3; d – version 4.

According to the microscopic investigation, it seems that in the case of version 4 the degree of expansion of the gas cells reached a level at which the volume increase of the porous structure was the highest compared to the other versions. The size of cells has considerably increased, however the cells keeping the features of some closed structures, without the interconnection of neighbouring cells. The distribution uniformity of cells was remarkable in all the microstructures corresponding to the four analyzed specimens.

The current work presents an economical and environmentally friendly technical solution using mixtures of raw materials that include large proportions of recycled residual glass (over 65 wt. %), coal fly ash (12.5-16 wt. %), and metakaolin (16 wt. %). All these raw materials are alumina-silicate materials, of which fly ash is a by-product of the energy generation industry, residual glass is an inexhaustible source of silicate waste from the beverage consumption of modern human civilization, and metakaolin comes from processing clay, an important source of natural alumina-silicate material.

Silicon carbide (SiC) was chosen from among several effective expanding agents. Its oxidation reaction carried out at temperatures above 950°C is the source of emission of a gaseous compound such as CO_2, which being blocked into thermally softened glass-based raw material mass creates numerous gas bubbles that then by the expanding material cooling turn into pores.

The appropriate combination of raw materials as well as the optimal choice of the expanding agent led to preparing mixtures that favoured obtaining cellular glass-ceramics with excellent physico-thermal and at the same time, mechanical properties.

Operational parameters that characterized the four sintering and expanding processes of glass-based raw materials showed the optimal temperature range between 965-995°C. Use of the own method for predominant direct microwave heating allowed to reach heating rates within the limits of 21.67-26.25°C/min, much higher than usual values in conventional processes (under 10°C/min). Specific energy consumption had low values decreasing up to 1.13 $kWh \cdot kg^{-1}$, while in the case of the optimal version it was 1.18 $kWh \cdot kg^{-1}$. In most cases, the literature does not provide data on this energy efficiency indicator, because ensuring the quality of the foamed product is considered the main priority.

Physico-mechanical, heat, and microstructural features of the optimal cellular glass-ceramic product were at the level of the best products of this type reported in the literature. Apparent density was of 0.60 $g \cdot cm^{-3}$, porosity of 75%, and heat conductivity of 0.080 $W \cdot m^{-1} \cdot K^{-1}$. Compressive strength had a high value (4.84 MPa), while pore size was within the limits of 1.2-2.3 mm. Water uptake had a value considered normal for this material type (3.8 vol. %).

4. Conclusions

The work aimed to promote techniques with favourable impact on environmental quality both in terms of the raw material nature and the energy source for the sintering and expansion process without emissions of polluting gases into the atmosphere. Materials were silicate waste (recycled residual glass), an alumina-silicate by-product of the energy industry (coal fly ash) as well as a natural alumina-silicate material (metakaolin). Electromagnetic waves (microwaves) were chosen as energy carriers, although they are not yet used on an industrial scale for high-temperature processes, but they represent an excellent alternative to burning fossil fuels. In terms of quality, experimentally made cellular glass-ceramic products showed excellent thermal insulation properties as well as a sufficiently high level of compressive strength.

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VITROCERAMICĂ CELULARĂ DIN STICLĂ REZIDUALĂ RECICLATĂ, METACAOLIN ȘI CENUȘĂ DE CĂRBUNE ZBURĂTOARE

(Rezumat)

În această lucrare, a fost promovată grija față de sănătatea mediului, selecționând materii prime care îndeplinesc aceste cerințe. Astfel, au fost utilizate deșeu de sticlă reciclat în proporție predominantă, cenușă de cărbune zburătoare ca un produs secundar al industriei energetice și un material silicoaluminos natural (metacaolinul). Carbura de siliciu (SiC) a fost aleasă ca agent de expandare. Deși utilizată nesemnificativ până acum în procese industriale la temperatură înaltă, încălzirea cu microunde a fost adoptată pentru realizarea procesului de sinterizare și expandare. Rezultatele au arătat foarte bune proprietăți termoizolante și o înaltă rezistență mecanică a produsului expandat optim.