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THE USE OF MICROWAVE FIELDS IN THE FOAMING PROCESS OF FLAT GLASS WASTE

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ABSTRACT
The paper presents experimental results obtained in the manufacture process of glass foam from clear flat glass waste and coal ash, using silicon carbide as foaming agent. The heating of the powder mixture was achieved in microwave field, unlike other experiments of this type presented in literature. The physical features of the glass foams obtained by unconventional heating are similar to those made by conventional methods, having the advantage of a superior macrostructural homogeneity, which ensures them a better mechanical strength.

INTRODUCTION
Worldwide the recycling of glass waste, existing in very large quantities, became a current concern in the last decades. Because it has not hazardous components, the bottle glass waste is used preponderantly as raw material in the foaming process for obtaining glass foam, a porous material with physical, mechanical and morphological features suitable for its use as insulating material in construction. The foaming method involves the use of one of foaming agents (calcium carbonate, calcium sulfate, sodium carbonate, silicon carbide, silicon nitride, black carbon, graphite, dolomite etc.) [1 – 5] embedded in low quantities in the powder mixture of raw material, which at high temperatures (700 – 1150 ºC) releases a gaseous component by decomposition or oxidation inside the viscous mass. It forms numerous blocked bubbles, which by cooling make up a homogeneous structure of pores.

According to the literature [6], there are several types of glass foam obtained from bottle glass waste industrially manufactured currently, the main being "Technopor", produced by Misapor Switzerland Company with branches in Germany, France and Austria and "Foamglas", manufactured by Pittsburgh Corning Company with branches in United States, Europe (Belgium, Czech Republic) and China. These products are achieved using conventional heating methods (fossil fuel combustion or electric resistance).

The flat glass waste includes clear cullet from standard flat glass and cullet mixture which can contain several glass types (sealed window units, laminated glass, mirrored glass, tinted glass, printed glass, old glass from wooden frames). Clear cullet is constituted from clear flat glass waste, or window manufacturing waste. According to [7], Great Britain manufactures 750,000 tonnes flat glass each year from which ¾ go into glazing products for buildings. In the industrial manufacturing process of flat glass, the recycled flat glass can be added in the raw material mixture and re-melted for producing new glass. Currently, the contain of flat glass recycled in Great Britain is 20 – 30%.

It is imperative that the contamination levels of this glass type be kept at minimum. The most common contaminants are metal drinks cans, floppy discs, floor sweepings, silicon carbide discs, spacer bars from sealed units (clear cullet only etc.

The flat glass waste mentioned above are not as interesting for producing glass foam. Previous tests experimentally conducted showed that it is possible a significant increasing of foaming process temperature and duration due to some components (e. g. Fe_2O_3) which influence this process. For diminishing these parameters, compounds (e. g. MnO_2) are used to enhance the oxidation of the powder mixture, which can cause the macrostructural inhomogeneity of glass foam [8].

The Romanian company Daily Sourcing & Research conducted in latest years tests on producing glass foams using microwave energy. The tests used as raw material bottle glass waste and the experimental results showed

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that the products have physical, mechanical and morphological features similar to glass foams produced by conventional methods [9 – 12].

The first tests conducted using clear flat glass waste and calcium carbonate, respectively silicon carbide and coal ash, indicated a slight increase of the process temperature and duration and a macrostructural inhomogeneity of products, in the first case, while, in the second case, the process temperature and duration increased significant, but the macrostructural homogeneity of the material was not affected compared to products obtained from bottle glass waste. According to [13], the high temperature of sintering/ foaming process corresponding to the high weight proportion of coal ash is due to the increase of weight concentration in the raw material mixture of Fe_2O_3 brought by ash in the powder mixture of glass waste, coal ash and silicon carbide on the temperature of glass foam manufacture is not decisively a feature of using microwave energy. There is a strong correlation between the electrical conductivity of glasses and the microwave absorption. The glass with high concentration of alkali (Na_2O and K_2O) can be heated effectively with microwave. The weight proportion of alkali in the coal ash composition is lower than in the glass waste, so that a mixture with more coal ash disadvantages the microwave heating process and requires higher temperatures.

Previous tests conducted in the Romanian company Daily Sourcing & Research highlighted that the most indicated solution for an efficient microwave heating way is their distribution in two types of simultaneous energy transfer: directly, through the penetration of the microwave susceptible material layer (made from silicon carbide) placed between the material subjected to the heating and the waves generating source, the heating being initiated inside the material and being propagated outside and indirectly, through the absorption of microwave in the silicon carbide layer, which is rapidly and intensely heated and transfers thermal energy by radiation. Thus, two opposable centers generating heat are formed. The optimal thickness of the silicon carbide layer experimentally determined with bottle glass waste was 3.5 mm.

Having into account the particularities of the foaming process of clear flat glass waste, mentioned above, the research conducted in the company Daily Sourcing & Research and presented in the paper was directed to determine the features of heat transfer, the functional parameters of the process and the physical, mechanical and morphological features of glass foam samples, in conditions of using silicon carbide and coal ash. The heating method was unconventionally, using the microwave energy.

MATERIALS AND METHODS

Materials

As mentioned above, the composition of raw material and foaming agent contains clear flat glass waste, coal ash and silicon carbide. The clear flat glass waste was selected, cleaned, broken, finely ground in a ball mill and sieved between 63 – 130 µm. The fly ash, brought from Paroseni thermal power station at grain size between 63 – 80 µm, was ground in the ball mill and sieved below 63 µm. The chemical compositions of clear flat glass waste [7] and coal ash [11] are shown in Table 1.

Table 1. Chemical composition of raw material

RAW MATERIAL	Chemical composition, wt.%					
	SiO_2	Al_2O_3	MgO	CaO	$Na_2O + K_2O$	Fe_2O_3
Clear flat glass waste	71.1	1.3	3.9	9.3	14.2	0.2
Coal ash	46.5	23.7	3.2	7.9	10.1	8.6

The silicon carbide, used as foaming agent, purchased with the grain size between 63 – 80 µm, was ground in the ball mill and sieved below 32 µm.

Methods

The adapted method for producing glass foam into an enclosure, made from a microwave susceptible material (silicon carbide), simulates the working conditions of a tunnel furnace with the sidewalls coated with the silicon carbide layer. The powder mixture of raw material, including the foaming agent, pressed on the bottom of a large metal crucible with the opening down, has similar heating conditions as in the case of the depositing on the conveyor belt of the furnace. It is slowly heated, being continuously microwave irradiated as if moving along the tunnel furnace. The enclosure, thermal protected with thick layers of ceramic fiber, is introduced into an adapted domestic microwave oven powered with an only 0.8 kW microwave generator.

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The heating process in the microwave oven was monitored with a Pyrovar type pyrometer (measuring field: 700 – 2000 ºC) mounted above the oven in front of a viewing hole provided in the upper area of the heating equipment housing.

The microwave experimental equipment is shown in Fig. 1.

a *b* *c*

Fig 1 :The microwave experimental equipment

a – adapted 0.8 kW domestic microwave oven; b – the metal crucible and the silicon carbide tube; c – thermal protection with ceramic fiber of the silicon carbide tube placed in the oven.

As mentioned in the previous chapter, tests conducted in the Romanian company Daily Sourcing & Research on the adapted 0.8 kW microwave oven using clear flat glass waste, coal ash (9.5 – 10.5%) and silicon carbide (2.9 – 3.5%) led to the significant increase of temperature and duration required to reach the optimal conditions for foaming the raw material. Thus, the temperature values exceeded 1000 ºC, being reached 1005 – 1008 ºC and the process durations increased up to 102 – 107 min. Compared to the parameters of the similar microwave heating processes to obtain glass foams from bottle glass waste, coal ash and silicon carbide (960 – 985 ºC and 65 – 68 min), these parameters are very high. The heating speeds decreased below 10 º/ min (9.3 – 9.7 ºC/ min), compared to the usual heating process speeds in the case of bottle glass waste of 14 – 15 ºC/ min.

The intensify of the heating process is possible by increasing the share of direct heating (the microwaves come in direct contact with the material) compared to those of indirect heating (achieved by thermal radiation of the silicon carbide layer placed between the generating microwave source and the material. This desideratum was obtained experimentally by reducing the thickness of the silicon carbide layer from 3.5 mm to 3.0 mm.

The experiments were conducted for four heating variants, where the coal ash proportion varied between 9.5 – 10.5% and the silicon carbide had values between 2.5 – 3.5%. The method of wetting the powder mixture (about 9% water addition), currently used in the glass foam manufacturing process using bottle glass waste, was maintained. The weight proportions of the used materials are shown in Table 2.

Table 2. The weight proportions of the used materials

VARIANT	Component, wt.%			
	Clear flat glass waste	Coal ash	Silicon carbide	Water addition
1	88.0	9.5	2.5	9.0
2	87.4	9.8	2.8	9.0
3	86.6	10.2	3.2	9.0
4	86.0	10.5	3.5	9.0

The glass foam samples, resulted after the sintering/ foaming experimental process, were tested in laboratory to determine the physical, mechanical and morphological characteristics. Apparent density, porosity, thermal

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conductivity, compressive strength, hydrolytic stability, water absorption and crystallographic structure were determined by the current methods [14 – 17].

RESULTS AND DISCUSSION

Results

According to the adopted methodology for determining the functional parameters of the foaming process of the glass foam, four variants of producing this porous material from clear flat glass waste, coal ash and silicon carbide were tested on the adapted experimental microwave oven described above. The functional parameters of the sintering/ foaming process are shown in Table 3.

Table 3. Parameters of the sintering/ foaming process

VAR.	Sintering/ foaming temperature ºC	Average speed		Process duration min	Index of volume growth	Specific consumption of electricity kWh/ kg
		Heating ºC/ min	Cooling ºC/ min			
1	978	13.6	8.2	71	2.65	1.55
2	983	13.3	8.5	73	2.80	1.59
3	988	13.2	8.0	74	2.85	1.68
4	992	13.1	7.8	75	2.45	1.78

Unlike the parameters of the heating process achieved previously with the same proportions of raw material and foaming agent (temperatures: 1005 – 1008 ºC, process durations: 102 – 107 min and heating speeds: 9.3 – 9.7 ºC/ min) mentioned in the subchapter “Methods”, by diminishing the wall thickness of silicon carbide tube from 3.5 to 3.0 mm and increasing the share of direct microwave heating, the temperature range was reduced at 978 – 992 ºC, the process durations were significant diminished at 71 – 75 min and the heating speeds have reached 13.1 – 13.6 ºC/ min. The index of increasing the foamed material volumeis comparable with those obtained in the process of glass foam manufacture from bottle glass waste: 2.45 – 2.85. The specific consumption of energy (between 1.55 – 1.78 kWh/ kg) is not representative for the process, because the discontinuous operation regime of the experimental equipment leads to high heat losses compared to an industrial continuous furnace. Theoretically, the processes which use of the microwave field are rapidly, “clean” and economical [18].
The physical, mechanical and morphological features of the samples are shown in Table 4.

Table 4. Physical, mechanical and morphological features

VARIANT	Apparent density g/ cm^3	Porosity %	Compressive strength MPa	Thermal conductivity W/ m ·K	Water absorption %	Pore size mm
1	0.31	85.9	1.35	0.041	0.9	0.5 – 1.5
2	0.29	86.8	1.31	0.039	1.2	0.8 – 1.7
3	0.28	87.3	1.30	0.040	1.0	0.8 – 1.9
4	0.25	88.6	1.27	0.038	1.2	1.0 – 2.5

Except for sample 4, whose porosity is very high, the apparent density values are in the range 0.28 – 0.31 g/ cm^3, being common to the glass foams obtained from clear flat glass waste. Thermal conductivities have low values, suitable for the use of these products as insulating materials in construction. Like the most glass foams, the water absorption is very low, practically non-existent. The compression strength is relatively high, with values of over 1.3 MPa in the case of the three samples with porosities between 85.9 – 87.3%.
Images of the longitudinal section through the samples are shown in Fig.2.

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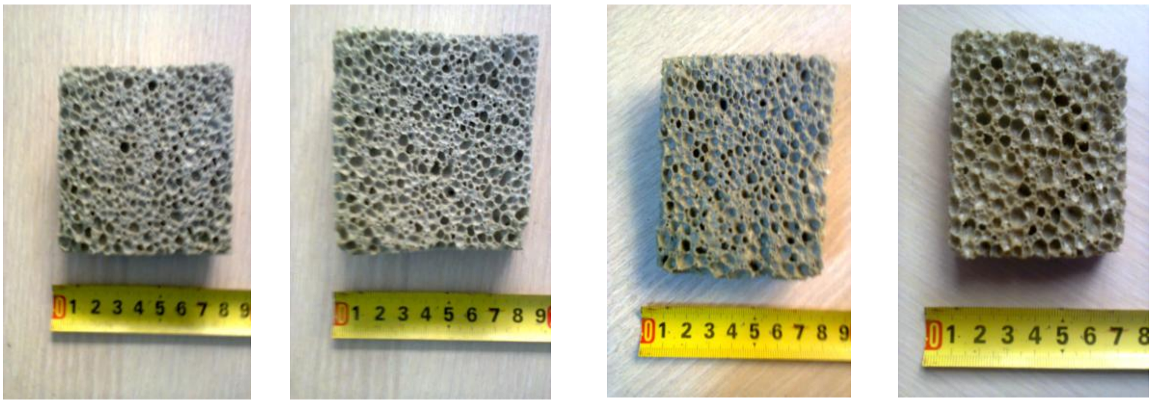

Sample 1 *Sample 2* *Sample 3* *Sample 4*

Fig.2 Images of the longitudinal section through the samples

According to the images from Fig. 2, the structural homogeneity of all samples is remarkable. The pores distribution in the longitudinal section of the samples is uniformly. The pore size of sample 1 is between 0.5 – 1.5 mm. Sample 2 has pores in the range 0.8 – 1.7 mm and the pore size of sample 3 varies between 0.8 – 1.9 mm. Sample 4 has larger pores with dimensions between 1.0 – 2.5 mm.

All samples were subjected to the XRD analysis. In all cases, the main crystalline phase identified after the thermal treatment is wollastonite-2M ($CaSiO_3$) and traces of silicon carbide (SiC). Though silica (SiO_2) there is in high proportions in raw material, cristobalite is not detected in the foamed samples.

The tests for determining the hydrolytic stability of samples, using 0.15 ml of 0.01M HCl solution to neutralize the extracted Na_2O, showed that the stability joins in the hydrolytic class 2, the extracted Na_2O equivalent being in the range 34 - 53 μg.

DISCUSSION

The experimental results were obviouslyinfluenced by the microwave field and the share of the two heating types (direct and indirect) applied to the powder mixture. Thus, the process temperature and duration and implicitly the heating speed were controlled. Previous tests conducted both by conventional methods and unconventional showed that the foaming process of the clear flat glass waste is difficult compared to that of the bottle glass waste. The main problem is the difficulty to obtain the macrostructural homogeneity of products, that influences their physical and mechanical features values. By the using of microwave field this disadvantage was practically eliminated.

CONCLUSION

Generally, the glass foams are attractive as insulating materials in construction due to their suitable physical and mechanical features. It was found that the glass foam manufacture using clear flat glass waste is much more difficult compared to the use of bottle glass waste.

The tests conducted on an adapted 0.8 kW domestic microwave oven in the Romanian company Daily Sourcing & Research aimed to reduce the macrostructural homogeneity of the glass foams, the main disadvantage of the manufacturing process using clear flat glass waste.

The solution used was the intensification of the direct microwave heating compared to the indirect heating, obtained by the reduction of the wall thickness of the silicon carbide enclosure, which contains the powder mixture of raw material.

Using coal ash (9.5 – 10.5%) and silicon carbide (2.5 – 3.5%)the foaming temperature range was reduced from over 1000 ºC up to 978 – 992 ºC, the process duration was diminished from 102 – 107 min to 71 – 75 min and the heating speed has reached 13.1 –13.6 ºC/ min compared to speeds of only 9.3 – 9.7 ºC/ min obtained without the intensification of the direct microwave heating.

The significant improvement of the macrostructural homogeneity of the foamed products ensured better physical and mechanical features.

The use of microwave field in the foaming process of clear flat glass waste constitutes a recommended solution for obtaining porous materials with superior physical, mechanical and morphological features

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