High-strength cellular building material prepared by direct microwave heating using industrial silicate wastes and borax

Material de construcție celular cu înaltă rezistență produs prin încălzire directă cu microunde utilizând deșeuri silicatice industriale și borax

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Rezumat. *Vitroceramica celulară a fost produsă prin sinterizare la 850-860 ºC utilizând, ca materie primă principală, cenușă zburatoare de cărbune (74-79 %), un produs secundar industrial, borax (20-25 %), ca agent de fluidizare și carbonat de calciu (1 %), ca agent de spumare clasic. Originalitatea lucrării a fost aplicarea tehnicii neconvenționale, rapidă și economică, a încălzirii directe cu microunde. Această tehnică nu este utilizată în procese similare industriale. Rezultatele au arătat rezistență foarte mare la compresiune (11,7-18,3 MPa), proprietăți termoizolante bune, adecvate pentru aplicații in construcție și consum specific de energie foarte redus (0,50-0,60 kWh/kg).*

Cuvinte cheie: vitroceramică celulară, încălzire directă cu microunde, cenușă zburătoare de cărbune, borax, rezistență la compresiune.

Abstract. *Cellular glass-ceramic was produced by sintering at 850-860 ºC using, as the main raw material, coal fly ash (74-79 %), an industrial by-product, borax (20-25 %,) as a fluxing agent and calcium carbonate (1 %), as a classic expanding agent. The originality of the work was the application of the unconventional, fast and economical technique of direct microwave heating. This technique is not used in similar industrial*

processes. The results showed very high compressive strength (11.7-18.3 MPa), good thermal insulation properties, suitable for building applications and very low specific energy consumption (0.50-0.60 kWh/kg).

Keywords: cellular glass ceramics, direct microwave heating, coal fly ash, borax, compressive strength.

1. Introduction

The global energy and ecological crisis that appeared at the end of the 20th century caused major changes in all spheres of economic activity affected by the need to reduce energy consumption by recycling industrial waste or from other types of activities.

For a long time, recycling waste resulting from the demolition or redevelopment of buildings was not a common practice. There was an exception at the end of World War II in several European countries (Germany, UK, etc.) due to the huge quantities of masonry rubble available for the manufacture of aggregates for new constructions [1, 2]. The global concern has resurfaced in recent decades with the need to reduce energy consumption for the manufacture of common building materials and reduce greenhouse gas emissions. Currently, the annual rate of masonry rubble generation is 70 million tons in the UK and 50-60 million tons in Germany [3].

Building materials containing wastes of concrete, clay brick, natural aggregate, and other mineral components were used for manufacturing lightweight aggregate [4]. The raw material was crushed, ground in a ball mill, pelletized in a disc pelletizer, fired and stabilized in a rotary kiln. Silicon carbide as an expanding material was added. The sintering temperature was 1160-1180 ºC obtained by conventional heating process. Pellets with dimensions between 2-8 mm, bulk density between 0.7-1.0 g cm^{-3}, compressive strength between 4-5 MPa, and water absorption in the range 8-15.5 vol. % were obtained at the end of process.

Another recipe for the manufacture of lightweight aggregates based on clay brick, mortar, plaster, lightweight, aerated, and normal concretes, and sand lime brick is presented in [5]. Raw material was crushed, ground, and sieved, the grain size being sub 100 μm. The adopted pore-supplying agent was silicon carbide (SiC) in a weight ratio of 3 %. The mixture was pelletized in a disc pelletizer and then conventionally heated in a rotary kiln to the optimum temperature for maximum material expansion of 1185 ºC. In terms of quality, the lightweight aggregate produced from masonry rubble was almost similar to that of lightweight expanded clay aggregate (LECA).

The processes of manufacturing porous materials (cellular glass-ceramics) using silicate industrial waste or industrial by-products, other than recycled glass waste, have facilitated applying the unconventional technique of direct microwave heating. Previous experiments performed by the Romanian company Daily Sourcing & Research SRL showed that glass (soda-lime glass type) is not suitable for direct microwave irradiation at the usual frequency of 2.45 GHz and specific powers between 1.50-1.65 kW/kg (typical for the microwave oven commonly used in the household and also in experiments presented in the literature [6-9]). The direct irradiation causes the destruction of the macrostructure of glass in its core at the

foaming temperature. Instead, the silicate industrial waste, even with glass waste, but in very low proportions, subjected to the direct microwave heating at very high rates are not structurally affected. This experimental finding allowed to successfully carry out foaming the building waste and the manufacture of cellular glass-ceramic with thermal insulation properties and high compressive strength.

According to the paper [6], experiments aiming at the manufacture by direct microwave heating of a high-strength aggregate included in the raw material composition clay waste from construction (70.5-83.4 %), coal fly ash (9 %), colored glass waste (2.6-15.5 %), SiC (5 %) as a pore-supplying agent, and water (20-28 %) as a binder. The temperature of the sintering process was between 1055-1150 ºC reached in 24-32 min due to the very high heating rate (35.3-43.1 ºC/min). The thermal insulation properties of the cellular glass-ceramic (apparent density between 0.60-0.69 g cm^{-3} and heat conductivity between 0.100-0.116 W $(mK)^{-1}$) were satisfactory and the compressive strength had high values (7.8-8.3 MPa). The water absorption was within normal limits (9.3-17.5 vol. %) taking into account that the starting material contained a high percentage of clay.

Aggregate made only from clay waste (75-83 %) and coal ash (15-23 %) as raw material, SiC (2 %) as a pore-supplying agent and water (25 %) as a binder by sintering at 1115-1145 ºC in a microwave oven by direct irradiation [7] led to the production of a material with properties suitable for high-strength aggregate having the apparent density between 0.50-0.68 g cm^{-3}, heat conductivity in the range 0.078-0.095 W $(mK)^{-1}$, compressive strength between 3.8-7.5 MPa, water absorption between 11.9-12.8 vol. %, and pores with high dimensions (up to 2.5 mm), reaching even 5 mm in the case of variant that used the maximum proportion of coal ash.

Aggregate manufacturing recipe including masonry rubble (85.6-90.8 %) and coal ash (4-9 %) as raw material, SiC (3.5-5.5 %) as a pore-supplying agent, and water (18 %) as a binder was applied in [3], the direct irradiation heating at 1168-1185 ºC with very high heating rates between 33.9-35.9 ºC/min leading to products with an apparent density between 0.75-0.98 g cm^{-3}, heat conductivity between 0.123-0.140 W $(mK)^{-1}$, compressive strength between 6.0-7.3 MPa, and coarser porosity with pore size between 1.2-4.5 mm.

Other work [8] using masonry rubble (88.5-93.0 %) and coal fly ash (4-7 %) as raw material, SiC (3.0-4.5 %) as a pore-supplying agent and water addition (18 %) as a binder applied the same technique of direct microwave heating at 1160-1170 ºC, the heating rate varying between 34.0-38.5 ºC/min. The thermal insulation properties of the specimens were better than the paper [3], the apparent density being between 0.40-0.54 g cm^{-3}, the thermal conductivity between 0.079-0.096 W $(mK)^{-1}$, and the porosity between 74.3-81.0 %, while the compressive strength had high values (5.5-7.0 MPa). Pore size was low reaching a maximum of 1.5 mm.

Work [9] using clay brick from demolished building waste (78.5-84.6 %) and clear residual flat glass (9.4-15.5 %) as raw material, borax (4 %) as a fluxing agent, SiC (2 %) as a pore-supplying agent, and water addition (15 %) as a binder presents a technique of manufacturing the high-strength aggregate by direct microwave

irradiation, reaching high heating rates (34.0-42.4 ºC/min). The sintering and foaming temperature range was 1080-1143 ºC. The thermal insulation properties indicated a denser material: apparent density between 0.72-0.98 g cm^{-3}, porosity between 53.3-65.7 %, and thermal conductivity in the range 0.161-0.197 W $(mK)^{-1}$. The compressive strength reached high values between 6.4-8.5 MPa. Pore size was low, the maximum value reaching 1.1 mm.

Very high mechanical strength cellular glass-ceramic was manufactured by direct microwave heating at 853 ºC using very high weight ratio of coal fly ash (82 %), calcium carbonate (5 %) as an expanding agent, sodium carbonate (13 %) as a fluxing agent, and water addition (10 %) as a binder [10]. The thermal insulation properties had high values (apparent density of 1.44 g cm^{-3} and heat conductivity of 0.281 W $(mK)^{-1}$), while the compressive strength reached a very high value (41.3 MPa). The application domain of this porous glass-ceramic may include construction types that require high mechanical stress (road and railway construction, bridge abutments, foundations, drainages, sports grounds, etc.).

Recent work [11] has highlighted a new technique for the manufacture of cellular glass-ceramic by the direct overfiring of coal fly ash at relatively low temperature due to the addition of borax without the use of a usual pore-supplying agent. When the critical point of the densification temperature is exceeded, the glass-ceramic foam porosity increases and the pore dimension also increases, being the basis of the porous glass-ceramic manufacture by direct overfiring. By adding borax the boron-oxygen bond can change the structure of quartz and the network of amorphous vitreous phase in coal fly ash. According to [12], self-expansion of coal fly ash could occur at the sintering temperature, crystals being able to precipitate in molten to form pores. The experimental results showed that the overfiring temperature of coal ash has a high value (about 1190 ºC). The use of borax as a fluxing agent was the solution adopted for the significant decrease of the process temperature. The above-mentioned work [11] used coal fly ash/borax mixtures in ratios from 90/10 to 70/30. The borax proportion influenced bulk density, porosity, and flexural strength values. It has been experimentally found that with the increase of borax addition and process temperature, the viscosity of vitreous phase and the bulk density gradually decrease and the porosity gradually increases. Borax ratio of 15 % led to the temperature decrease to 1000 ºC, the 20 % ratio allowed the temperature decrease to 925 ºC, while the 25 % ratio allowed reaching the low temperature of 822 ºC.

In view of the previous experimental results exposed above, the present work aimed to produce by sintering at a relatively low temperature a porous glass-ceramic material using coal fly ash, borax as a fluxing agent and calcium carbonate ($CaCO_3$) as a pore-supplying agent under the conditions of applying the unconventional microwave fast heating technique. The originality of the work is the use of the unconventional technique of direct microwave heating, practically not applied in the world in cellular glass-ceramic manufacturing processes and to a very small extent previously tested by the authors of this paper.

2. Materials and methods

The main raw material used in this experiment was coal fly ash, an industrial by-product of the thermal power stations. The material has been provided by the Paroseni station from Romania having the following chemical composition: 46.5 % SiO_2, 23.7 % Al_2O_3, 7.9 % CaO, 3.2 % MgO, 6.0 % Na_2O, 4.1 % K_2O, 8.2 % Fe_2O_3, and 0.4 % other oxides. The coal ash has been taken over with the grain size below 250 μm and has been mechanically processed in an electric grinding device up to grain dimensions below 100 μm.

Because Na_2O is known as an excellent fluxing material and sodium tetraborate decahydrate ($Na_2B_4O_7 \cdot 10H_2O$), known as borax, contains 30.8 % Na_2O [13, 14], this material was adopted as a fluxing agent in experiments. Also, its high ratio of boron in the form of B_2O_3 creates the borax ability to significantly increase the mechanical strength of the expanded product. Borax was purchased from the market as a crystalline white powder.

Calcium carbonate ($CaCO_3$) one of the most effective pore-making agents having a fine grain size (below 5 μm) has been also used in the starting mixture.

The adopted method was mainly based on the ability of coal fly ash to expand itself by the liquid-phase direct sintering process forming numerous pores in the molten material mixture including borax to decrease the process temperature due to its fluxing property. The addition of $CaCO_3$ as a pore-supplying agent contributes to the foaming process. $CaCO_3$ decomposes in the temperature range 750-900 ºC releasing CO_2 as gas bubbles [15-17] according to reaction (1), which are blocked in the molten and turn in a pore network after cooling.

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

The equipment used in this experiment was a 0.8 kW-microwave oven of the household type for food preparation, but constructively adapted for operation at much higher temperatures (up to 1200 ºC). The direct microwave heating of the pressed powder mixture applied in this experiment has a special peculiarity by comparison with the conventional heating processes. The unconventional heating is initiated in the core of the irradiated material, where the microwave power is converted into heat. Thus, the highest temperature level is reached in the middle of the material and the propagation of heat takes place volumetrically from inside to outside [18, 19], i.e. inversely to its propagation in conventional techniques. Under these conditions, the thermal protection of the irradiated material with 1200 ºC-resistant ceramic fiber mattresses is essential. Although the metal walls of the microwave oven were not protected, the heat loss outside was kept low (below 70 ºC) despite the very high temperature of the thermal process (around 1000 ºC). Pyrovar-radiation pyrometer mounted above the oven in its central axis allowed monitoring the evolution of the temperature of irradiated material by viewing it through 30 mm-holes provided in the upper metal wall of the oven and in the ceramic fiber mattress that protects the upper

area of the hot material. The constructive and functional scheme of the experimental equipment is shown in Fig. 1.

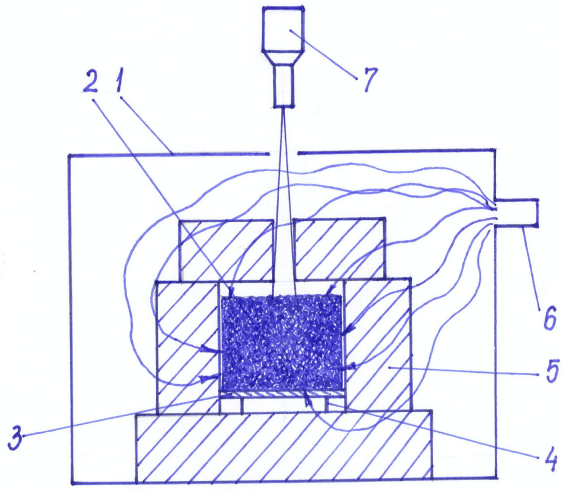

Fig. 1. Constructive and functional scheme of the experimental equipment
1 – microwave oven; 2 – pressed powder mixture; 3 – metal plate; 4 – metal support;
5 – ceramic fiber heat protection; 6 – waveguide; 7 – radiation pyrometer.

The following methods were applied to characterize the cellular glass-ceramic specimens. The gravimetric method [20] was used to determine the apparent density and the method of comparing the "true" density and the apparent density [21] was used to calculate the porosity of the specimens. Determining the heat conductivity was performed by 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. To identify the crystalline phases of the porous materials, the XRD technique (EN 13925-2:2003) was applied using X-ray diffractometer Bruker-AXS D8 Advance with CuKα radiation.

3. Results and discussion

Four experimental variants containing coal fly ash, borax, $CaCO_3$, and water addition as a binder were adopted to test manufacturing the high-strength cellular glass-ceramic. The $CaCO_3$ weight proportion was kept constant at 1.0 %, while coal ash/borax ratio was modified from 87/12 up to 74/25. The composition of experimental variants is shown in Table 1.

Table 1

Composition of experimental variants

Variant	Coal fly ash (wt. %)	Borax (wt. %)	$CaCO_3$ (wt. %)	Water addition (wt. %)
1	87.0	12.0	1.0	10.0
2	83.0	16.0	1.0	10.0
3	79.0	20.0	1.0	10.0
4	74.0	25.0	1.0	10.0

The main functional parameters of the sintering/expanding process are presented in Table 2.

Table 2

Functional parameters of the heat process

Parameter	Variant 1	Variant 2	Variant 3	Variant 4
Dry raw material/cellular glass-ceramic amount (g)	430/ 418.6	430/ 419.0	430/ 418.7	430/ 418.5
Sintering/foaming temperature (ºC)	830	840	850	860
Heating time (min)	20	21	22	24
Average rate (ºC/min) · heating · cooling	40.5 5.3	39.0 5.4	37.7 5.2	35.0 5.4
Index of volume growth	1.30	1.45	1.60	1.85
Specific energy consumption (kWh/kg)	0.50	0.52	0.55	0.60

According to the data in Table 2, constant amount (430 g) of solid starting material was used in this experiment. The method consisted in simultaneous increase of the weight ratio of borax (Table 1) and heating temperature from 830 to 860 ºC. The exceptional energy efficiency of the heating process by direct microwave irradiation of the pressed material led to very short durations (20-24 min) and heating rates between 35.0-40.5 ºC/min, much higher compared to the rates commonly used in conventional processes [15]. The volume of irradiated material has increased by foaming the coal ash powder and forming the pore network by 30-85 %. Variant 1 with 12 % borax and the process temperature of 830 ºC produced a dense structure, while variant 4 with 25 % borax simultaneously with reaching the temperature of 860 ºC generated a porous structure with larger pore dimensions. In terms of energy, the manufacturing process of cellular glass-ceramic using coal fly ash as the main raw material, borax, and $CaCO_3$ were extremely economical in all four variants, the specific energy consumption values being very low (0.50-0.60 kWh/kg) at a lower level compared to the most efficient industrial processes.

Images of the cross section-appearance of cellular glass-ceramic specimens are shown in Fig. 2.

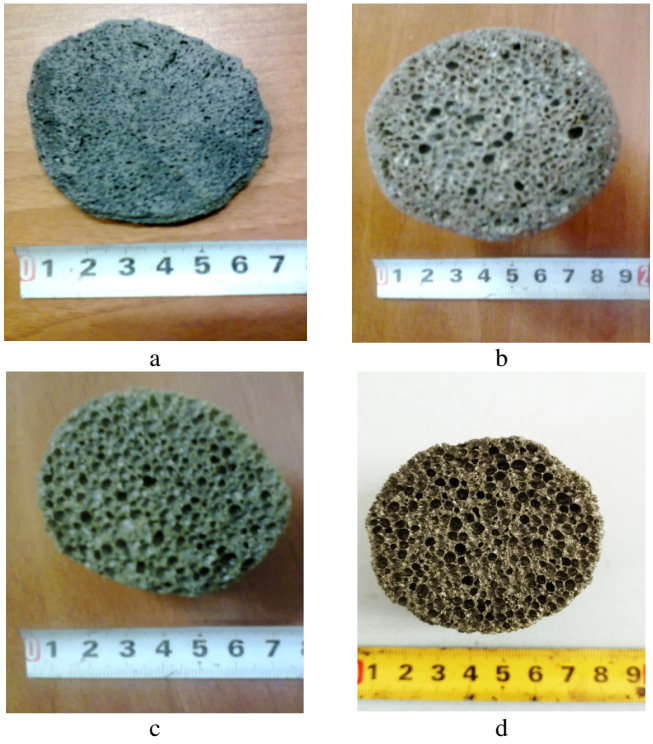

Fig. 2. Cross section-appearance of specimens
a – specimen 1 heated at 830 ºC; b – specimen 2 heated at 840 ºC;
c – specimen 3 heated at 850 ºC; d – specimen 4 heated at 860 ºC.

The physical, thermal, mechanical, and morphological characteristics of specimens determined by the analysis methods mentioned above are indicated in Table 3.

Table 3

Physical, thermal, mechanical, and morphological features

Variant	Apparent density (g cm^{-3})	Porosity (%)	Heat conductivity [W $(mK)^{-1}$]	Compressive strength (MPa)	Water absorption (vol. %)	Pore size (mm)
1	1.10	47.6	0.228	29.7	1.2	0.15-0.50
2	0.96	54.3	0.198	23.0	1.1	0.25-0.65
3	0.80	61.9	0.172	18.3	0.9	0.40-1.00
4	0.65	69.0	0.140	11.7	0.7	0.60-1.20

The data in Table 3 show remarkable mechanical features, the specimen compressive strength being in the range 11.7-29.7 MPa. Unfortunately, the thermal insulation properties of the material are affected especially in the case of variants 1 and 2, which due to the dense character of their macrostructure have high values of apparent density (0.96-1.10 g cm^{-3}) and heat conductivity [0.198-0228 W $(mK)^{-1}$] as

well as quite low values of porosity (47.6-54.3 %). The values of the physical-thermal characteristics that define the thermal insulation properties of specimens 3 and 4 (density between 0.65-0.80 g cm^{-3}, heat conductivity between 0.140-0.172 W $(mK)^{-1}$, and porosity between 61.9-69.0 %) are acceptable. These physical-thermal characteristics together with the compressive strength values and the low level of water absorption (below 0.9 vol. %) constitute excellent performances of cellular glass-ceramic materials made by the technology presented in this work.

The microstructural homogeneity of specimens, shown in Fig. 3, is also a physical feature favourable for the thermal insulator role of the material.

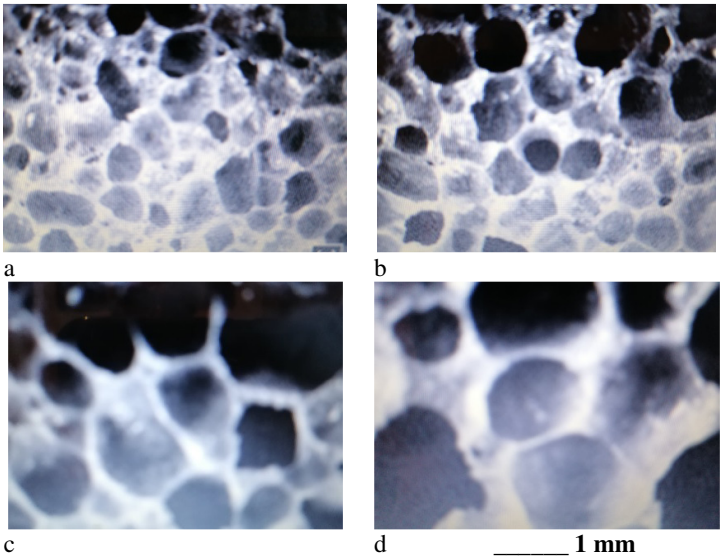

Fig. 3. Microstructural appearance of cellular glass-ceramic specimens
a – specimen 1; b – specimen 2; c – specimen 3; d – specimen 4.

According to the pictures in Fig. 3, the pore size varied between the four experimental variants. With the increase of the weight proportion of borax and the process temperature, the pore size increased, reaching in variant 4 the range of values 0.60-1.20 mm. The other ranges of values corresponding to variants 1-3 are indicated in Table 3.

According to [22], glass-ceramics are fine microstructure-materials containing at least one crystalline phase making by the controlled crystallization. Various silicate-based wastes such as coal ash, metallurgical slag, fly ash and filter dusts from waste incinerators, red mud from zinc hydrometallurgy, sludges, etc. are adequate for production of glass-ceramics. The crystalline phases of the cellular glass-ceramic manufactured in this experiment were identified by the XRD technique. It has been observed that proportions of borax between 12-20 % lead to obtaining specimens having as main crystalline phase quartz and to a lesser extent anorthite and mullite. In the case of specimen 4 with 25 % borax, practically the only crystalline phase identified was quartz.

Manufacturing recipe of cellular glass-ceramic adopted by authors replaced recycled glass waste with an industrial by-product (coal fly ash) resulting as a residue from coal burning in thermal power stations. Coal fly ash has the ability to expand itself at the sintering temperature, borax significantly reducing the melting temperature. $CaCO_3$ as an usual expanding agent was supplementary used contributing also to pores forming. Applying this recipe had as a result manufacturing porous product with high compressive strength. On the other hand, the making process of this type of material made by rapid and economical unconventional direct microwave heating allowed to reach excessively high heating rates, well above the limit recommended in the literature (10-15 ºC/min) [15], without affecting the microstructural homogeneity of the product. It should be mentioned that the direct microwave heating which in the case of using glass waste as a raw material was not suitable, being instead perfectly adequate in the case of other industrial silicate waste (e.g. coal ash) [23].

4. Conclusions

The current work aimed at the experimental manufacture of cellular glass-ceramic using coal fly ash, an industrial by-product, as the main raw material, borax as a fluxing agent, and calcium carbonate (1 %) as a classic expanding agent. The replacement of recycled glass waste commonly used as silicate waste with coal ash was based on its ability to expand itself at the sintering temperature, significantly reduced due to the addition of borax (between 12-25 %). The originality of the paper was applying the unconventional technique of direct microwave heating not used in industrial processes, where less fast and less economical conventional methods are preferred. The method consisted in simultaneous increasing the weight ratio of borax and heating temperature from 830 to 860 ºC. The exceptional energy efficiency of the heating process by direct microwave irradiation of the pressed material led to heating rates between 35.0-40.5 ºC/min, much higher compared to the rates commonly used in conventional processes. Of the four variants tested, the variants prepared from 20-25 % borax, 74-79 % coal ash, 1 % $CaCO_3$, and 10 % water addition were considered optimal, the mixture being sintered at 850-860 ºC. The results indicated very high compressive strength (11.7-18.3 MPa) and good thermal insulation properties (apparent density of 0.65-0.80 g cm^{-3}, heat conductivity of 0.140-0.172 W $(mK)^{-1}$, porosity of 61.9-69.0 %) suitable for use as thermal insulation material in construction domains that require high resistance to mechanical stress. The specific energy consumption values were very low (0.50-0.60 kWh/kg) at a lower level compared to the most efficient industrial processes.

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