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MAKING FINE-POROSITY THERMAL INSULATION-MICROWAVE ASSISTED

BY

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Abstract. Fine-porosity glass foam was made from ground recycled waste glass, using sodium hydroxide dissolved in distilled water, borax, and titanium dioxide as additives. The dry mixture was heated for sintering at 810-825°C in a constructively and operationally adapted microwave oven, heating times being varied between 27-60 min. The proportion of NaOH was increased from 4.5 to 9%, so that the NaOH/borax ratio had values between 0.75-1.50. The experiments allowed the identification of the optimal NaOH/borax ratio (1.25), so that the characteristics of the appropriate specimen were: density of 0.14 g·cm⁻³, porosity of 89.6%, heat conductivity of 0.042 W·m⁻¹·K⁻¹, and compression strength of 1.2 MPa. The pore size was very low (between 40-80 μm), obtaining a structure with predominant closed-porosity.

Keywords: glass foam, fine-porosity, microwave, NaOH, borax.

1. Introduction

Modern thermal insulation mounted on the surface of the building exterior walls has the ability to reduce the heat transfer, prevent wall wetting, and improve the thermal protection of the building as its whole. Energy saving due to

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applying an efficient heat insulation can even reach 45%. According to recent investigations, it has found that thermal insulation modernization of buildings is more effectiveness than the use of solar and wind energy as modern procedures for heating efficiency in the construction area.

Currently, they are known several modern materials and procedures with an insulating role, which have proven to be effective in this field: mineral wool, also known as mineral fiber, expanded or extruded polystyrene, polyurethane, aerogel as the lightest solid insulator, and sprayed froth with the ability to fill hard-to-reach spaces (Exploring new materials, 2024; Jelle *et al.*, 2010).

Mineral wool is usually made from minerals (such as silica, stone, rock, and glass). Making mineral wool involves melting and processing it into shaped fibers inside the insulation. This insulation type is waterproof and fireproof. Glass mineral wool withstands temperatures up to 595°C, while stone wool withstands up to 980°C (which is its melting temperature).

Polystyrene has thermal conductivity values in the range of 0.033-0.040 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$, while polyurethane has heat conductivity values within the limits of 0.020-0.030 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ (Thermal insulation, 2024), but has the disadvantage of being dangerous to human health, especially in the event of a building fire.

According to (El-Shiekh *et al.*, 2009), the main thermal insulation materials used in industrial and commercial installations are: calcium silicate, glass (as fiberglass and fiber or rock wool), expanded perlite, elastomeric foam, plastic foam, and insulating cement. Thermal conductivity values of these insulation materials are within the limits of 0.0216-0.049 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$. The lowest values correspond to urethane and isocyanurate foam blocks.

Aerogel, containing up to over 99% air, has a unique density of only 0.16 $\text{kg}\cdot\text{m}^{-3}$. It is produced from hydrophobic silica, although aerogels prepared from plastic waste are also known. Aerogels represent excellent and very promising heat insulation solutions (Hostler *et al.*, 2008). The technique of using carbon black below the pressure of heat transfer through radiation allows obtaining very low values of thermal conductivity (0.004 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$). However, commercially available aerogels with thermal conductivity below 0.013 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ at ambient pressure have been reported. The making costs of aerogels are still very high. Despite the relatively high compressive strength, these thermal insulation materials are very brittle and consequently, the tensile strength is at a low level. It has been found that the tensile strength can be improved by adding carbon fibers.

Spray froth is usually made from polyurethane, first sprayed before curing into a solid insulating layer. The froth can contain open cells as well as closed cells, which provide the best insulation.

Vacuum insulation in the form of thermal insulation panels is a state-of-the-art insulation technique, their thermal conductivity reaching values at an early age between 0.003-0.004 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ and up to 0.008 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ after about 25 years (Caps *et al.*, 2008).

Another latest generation solution is that of gas-filled panels (Mills and Zeller, 2008). This technology uses a less thermally conductive gas than air, from the group of argon, krypton, and xenon, replacing the previously mentioned vacuum. Compared to vacuum insulation panels, the gases used are less thermally insulating. The lower emissivity surfaces inside the gas-filled panels reduce the level of heat transfer through radiation. The thermal conductivity of these panels reaches higher values ($0.040 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$).

Considering the relatively high costs and some disadvantages highlighted above of the latest generation techniques and materials related to the production of new thermal insulation materials, the paper's authors focused their interest on insulating products made in the form of glass foam.

According to recent literature (Liu *et al.*, 2021), the properties of glass foam depend largely on the type of pores. Closed-pore glass foam is used in buildings as a thermal insulation material, having very good performances (thermal conductivity below $0.1 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$, compression strength above 0.5 MPa, resistance to water, and long life-span. Open-pore foams (open porosity above 50%) are excellent acoustic insulation materials, are water-absorbing, and are recommended for application in subway stations and gardens.

In general, the main raw materials used in the production of glass foam are residual glass, fly ash, and metallurgical slag. The selected foaming agent and the slag powder required for the formation of the porous structure are adopted depending on the thermal characteristics of raw materials. The most commonly used foaming agent is black carbon. The characteristics of the porous structure are influenced by several factors such as the difference between the foaming temperatures of agents as well as the viscosity of material mixture melt during foaming. The particle size of materials that make up the mixture affects the uniformity of the porous structure. In the work (Liu *et al.*, 2021), the starting mixture was composed of quartz sand, barite, and borax, while calcium carbonate (CaCO_3) was added as a foaming agent. The work conclusions showed that borax contributed to the formation of a uniform porous structure and fine thermal and mechanical properties. Quartz and barite influenced the achievement of a lower density and a higher compression strength. Carbon dioxide (CO_2) resulted from CaCO_3 decomposition at high temperature has the ability to react with the graphite completing the graphite oxidation and increasing pores strength. The optimal foam performances were obtained using the combination of 1% graphite and 2% CaCO_3 at 850°C . The thermal conductivity reached $0.066 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$. The increase of CaCO_3 content influenced the appearance change of porous structure from closed porosity to open porosity.

Based on the current state of knowledge regarding the production of glass foams, several types of inorganic or organic materials are used as expanding agents. Thus, pure carbon, calcium or sodium carbonate, glycerol, silicon carbide or nitride, sugars, starch, organic wastes, etc. are frequently chosen for these processes. In general. The froths are produced under the conditions where the

recycled residual glass constitutes the required precursor. In the work (Bento *et al.*, 2013; da Silva *et al.*, 2019), sodium hydroxide (NaOH) was adopted as a foaming agent. The foams were prepared using glass waste ground in a ball mill, mixed with titanium dioxide (TiO₂) in 20% proportion and NaOH in variable proportions between 3-17%. TiO₂ was used as a hardening agent and NaOH was dissolved in water (1 g·mL⁻¹). The mixture was dried and then calcined at 800°C for 60 min with the heating rate of 15°C·min⁻¹. According to (da Silva *et al.*, 2020), the role of NaOH content is to decrease closed porosity of the foam and implicitly, its density. As a consequence, the open porosity is increasing. It has been found that borax associated with low amounts of NaOH favours the densification of foamed products.

A research team including authors of the current paper has carried out experiments for making fine porosity-glass foam using a liquid carbonic expanding agent-glycerol (Dragoescu *et al.*, 2020). The basic raw material was colourless flat glass, to which glycerol (between 1.0-1.8%), water glass (in the range of 5.3-7.5%, and distilled water (within the limits of 7.7-10%) were added in the mix. The sintering temperature varied between 810-824°C and the heating rate had values in the range of 19.1-20.3°C·min⁻¹. Physical, thermal, mechanical, and microstructural features of specimens were the following: density between 0.20-0.26 g·cm⁻³, porosity in the range of 85.5-88.2%, heat conductivity within the limits of 0.056- 0.070 W·m⁻¹·K⁻¹, compression strength in the range of 4.6-5.8 MPa, and pore size within the limits of 0.3-0.8 mm and 0.8-1.1 mm.

The same Romanian authors has tested the production of a glass foam characterized by high mechanical strength and fine porosity (Paunescu *et al.*, 2021). In this experiment, a solid foaming agent (0.8-1.1% CaCO₃) and a liquid one (1% glycerol) have been simultaneously used, except colourless residual glass, sodium silicate, kaolin, and water addition. The peculiarity of the experiment has been the unconventional microwave heating, which facilitated very high heating rates (between 21.9-23.9°C·min⁻¹). The sintering/foaming temperature had values in the range of 834-841°C. The characteristics of the foamed products were: apparent density between 0.26-0.30 g·cm⁻³, porosity in the range of 85.71-87.62%, heat conductivity within the limits of 0.060-0.069 W·m⁻¹·K⁻¹, compression strength between 7.0-7.5 MPa, and pore size within the limits of 0.10-0.35 mm and 0.40-0.65 mm.

This work aimed at manufacturing fine porosity materials by foaming finely ground glass waste under the conditions of finding optimal correlations between the proportions of NaOH and borax used in the material mixture, so that the apparent density as well as open and respectively, closed porosity of the foamed products meet the requirements.

2. Methods and Materials

According to the literature (da Silva *et al.*, 2020), it was found that the addition of NaOH in the glass foam manufacturing process contributes to reducing the density and closed porosity of the foam, in the same time with increasing the open porosity. On the other hand, borax in association with a relatively low consumption of NaOH contributes to increasing the density and closed porosity. Weight ratio of the two components of raw material should be experimentally established, which is what the current work has achieved.

The experiment described in this paper was carried out in the experimental base of the company Daily Sourcing & Research (Romania). An 800 W-microwave oven, usually used in households for food preparation, but constructively and operationally adapted by the paper's authors, constituted the thermal equipment applied for heating the raw material mixture (Fig. 1a). The equipment originality was represented by the differentiated distribution of the pressed material heating, predominantly direct and partly indirect due to positioning a SiC and Si₃N₄-ceramic tube with the wall thickness of 2.5 mm (Fig. 1b), having the role of protective screen between the microwave emission source and the material specimen placed in the middle of existing space on a metal support. In this way, the very strong effect of direct radiation on the material subjected to heating was diminished, part of the energy potential incorporated in the electromagnetic waves being absorbed in the ceramic tube wall. The inner surface of the tube, strongly heated by the conversion of wave power into heat, transferred heat to the sample through thermal radiation, thus ensuring mixed heating, predominantly direct and partially indirect (Paunescu *et al.*, 2017).

The peculiarity of direct microwave heating is completely opposite to conventional heating, being initiated in the core of the irradiated material, where the wave power is converted into heat (Jones *et al.*, 2002). For this reason, the heat volumetrically propagates from the inside to the outside (Kitchen *et al.*, 2014), that is completely opposite to traditional heating. Therefore, the outer surface of the ceramic tube was protected with ceramic fiber mattresses to avoid heat escaping outside the system.



Fig. 1 – Experimental microwave equipment.
a – 800 W-microwave oven; b – protective ceramic tube.

The preparation of glass foam involved the mechanical processing of recycled waste glass by crushing and grinding in a ball mill. In this experiment, colourless post-consumer drinking bottle assortments were chosen. The grinding process was carried out for a sufficiently long time (about 6 hours) to obtain very fine grain sizes (below 80 μm). Fine sodium tetraborate powder $\text{Na}_2[\text{B}_4\text{O}_5(\text{OH})_4] \cdot 8\text{H}_2\text{O}$ (grain size below 50 μm) available on the market as well as titanium dioxide (TiO_2), a chemically inert powder, were also added to the starting mixture, acting as a fluxing agent and respectively, as a hardening agent for the made glass foam. Also, various weight proportions of sodium hydroxide (NaOH) dissolved in distilled water in the proportion of 1 g per mL of water were introduced into the mixture.

The next steps in the manufacture of fine-porosity glass foam were drying the mixture in a laboratory electric oven and then heating to 810-825°C with an average heating rate of 13.4-29.3°C·min⁻¹ in the microwave oven described above.

The materials chosen in this experiment for the production of fine-porosity glass foam were: colourless post-consumer drinking bottle, borax, titanium dioxide (TiO_2), sodium hydroxide (NaOH), and distilled water for dissolving the NaOH powder.

The colourless post-consumer drinking bottle was recovered from the huge drinking bottle reserve. It was washed, dried, broken, ground and sieved to select grain size below 80 μm . The chemical composition of this type of glass, previously determined (Dragoescu *et al.*, 2018) includes: 71.7% SiO_2 , 12.0% CaO , 13.3% Na_2O , 1.9% Al_2O_3 , 1.0% MgO .

Borax favourably influences the fluxing and contributes to reducing the temperature of the glass melt. Also, it can control the dependence between temperature, viscosity, and surface tension to obtain an optimal glass fiber. It has been experimentally found that borax associated with small amounts of NaOH favours increasing the density of foamed products. This particularity of borax was considered interesting in the current experimental conditions. Borax was commercially purchased being in fine powder state (under 50 μm).

TiO_2 was adopted in the material mixture due to its contribution to hardening the glass foam. In recent literature (Jiang *et al.*, 2024), TiO_2 effects were researched on crystallization, microstructure and pore morphology as well as phases composition. Increasing the TiO_2 content in the raw material leads to a change in the thermal stability of the glass-based foamed product, which initially decreases and then increases. Also, TiO_2 favours crystallization under the conditions of a very low content, then at higher contents it inhibits this process. Thus, TiO_2 -fine powder was used in the mixture in a constant weight proportion of 8%.

NaOH has a well-known role in reducing the density and, implicitly, the closed-porosity of glass froths. As mentioned above, (da Silva *et al.*, 2019) identified that borax introduced together with NaOH can normalize the ratio

between closed and open porosity, leading to the desired properties of the glass froth. In this experiment, various NaOH contents (between 9-18 g) were tried, maintaining constant the borax content at 12 g (6%).

Four experimental versions for producing the glass froth were tried using components noted above. Table 1 contains the amounts utilized under the conditions where all versions benefited from the same total amount of 200 g.

Table 1
Composition of the material mixture versions

Material	Version 1	Version 2	Version 3	Version 4
Colourless post-consumer drinking bottle (g)	154	148	142	136
Borax (g)	12	12	12	12
Titanium dioxide (g)	16	16	16	16
Sodium hydroxide (g)	9	12	15	18
Distilled water (g)	9	12	15	18
Total mixture (g)	200	200	200	200

The determining methods of physical, thermal, mechanical, and microstructural features of glass foam specimens were in general those commonly applied for this foamed material type. Apparent density was determined using Archimedes' principle in accordance with the ASTM C373 standard. ISO 18754:2020 was applied to measure the porosity. The compression strength of fine porosity-glass foam samples was measured with a hydraulically operated compression testing machine with the pressing capacity of 105 MPa, according to the ASTM C133-97 (2015) standard. The water-absorption of samples was identified in accordance with ASTM C373-18 standard by their immersion under water. Microstructural appearance of specimens was investigated with Biological Microscope MT5000 model.

3. Results and Discussion

According to the data in Table 1, in this experiment, versions in which the ratio between NaOH and borax varied within the following limits: 0.75, 1.00, 1.25 and 1.50 were tried.

In the four versions, the operational parameters of the making process of glass froth had the following values exposed in Table 2.

According to Table 2, the experiment on fine-porosity glass foam making aimed to examine influencing the duration of sintering/foaming process, heating rate of raw material as well as NaOH/borax ratio on the porous product

characteristics. Thus, the duration of the heating process was significantly increasing between the four versions (between 27-60 min), the heating rate was decreasing from 29.3 to 13.4°C·min⁻¹, and the NaOH/borax ratio increased from 0.75 to 1.50.

Table 2
Operational parameters of the thermal process

Version	Raw material/glass foam amount (g)	Sintering/foaming temperature (°C)	Heating time (min)	Average rate (°C·min ⁻¹)	
				Heating	Cooling
1	200/188	810	27	29.3	5.0
2	200/190	815	38	20.9	5.1
3	200/188	820	48	16.7	5.0
4	200/189	825	60	13.4	5.1

The physical, heat, mechanical, and microstructural features of the glass foam specimens are presented in Table 3.

Table 3
Physical, thermal, mechanical, and microstructural features

Version	Apparent density (g·cm ⁻³)	Porosity (%)	Heat conductivity (W·m ⁻¹ ·K ⁻¹)	Compression strength (MPa)	Water abs. (%)	Pore size (µm)
1	0.22	87.7	0.052	1.5	2.8	15-70
2	0.18	88.9	0.048	1.4	2.5	20-75
3	0.14	89.6	0.042	1.2	2.4	40-80
4	0.09	90.8	0.039	0.9	2.1	30-110

Results presented in Table 3 showed that increasing addition of NaOH aqueous solution in combination with the use of borax at a constant value contributed to the sharp decrease of the apparent density (from 0.22 to 0.09 g·cm⁻³) of the glass foam. This decrease of density was relatively tempered by the presence of borax, whose effect is the opposite, favouring the densification of the foamed product. At higher proportions of NaOH mixed with borax (in our experiment, at a NaOH/borax ratio of 1.50), the density decreased greatly and was due to the increase in open porosity. As a consequence of the reduced density values, the heat conductivity of porous specimens was also reduced (between 0.039-0.052 W·m⁻¹·K⁻¹). The constant addition of TiO₂ contributed to maintaining a satisfactory level of the compression strength (within the limits of 0.9-1.5 MPa).

Images of the fine-porosity glass foam specimens are presented in Fig. 2 and microstructural appearance of these is shown in Fig. 3.

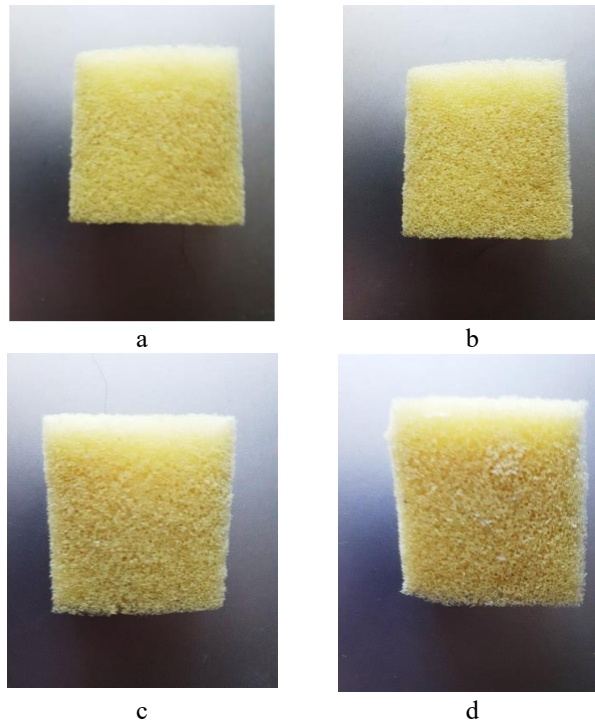


Fig. 2 – Images of the fine-porosity glass foam specimens
a – version 1; b – version 2; c – version 3; d – version 4.

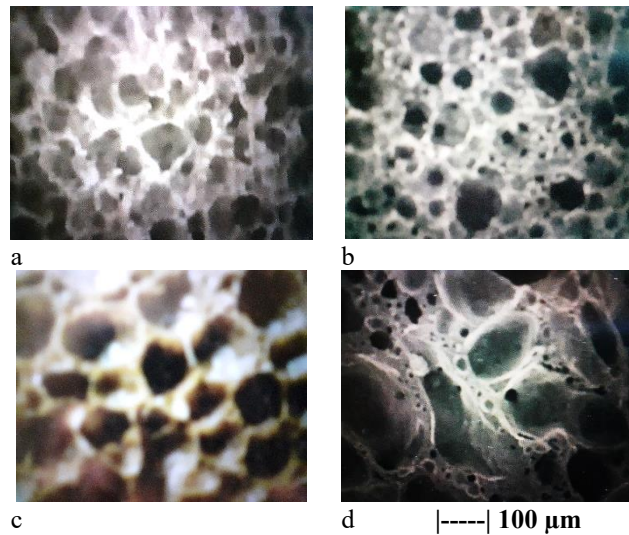


Fig. 3 – Microstructural aspect of the fine-porosity glass foam samples
a – version 1; b – version 2; c – version 3; d – version 4.

General investigation of the surface of foam glass samples according to Fig. 2 showed that all of them are characterized by fine porosity.

According to some remarks in the literature, the idea that increasing the NaOH content in the manufacture of glass foam can lead to obtaining structures with open porosity was also confirmed in the pictures in Fig. 3 (especially in picture "d"). This type of inhomogeneous structure causes, on the one hand, a strong reduction of the material density, but, on the other hand, an uneven and undesirable circulating way of the heat flow.

The pore sizes of specimens were very low starting with those corresponding to version 1 (between 15-70 μm). The size values were increasing in the case of version 2 (between 20-75 μm) and version 3 (between 40-80 μm). The experimental version 4 produced pores with dimensions in the range of 30-110 μm , characterized by an open-porosity structure.

The analysis of the glass foam specimens produced in this experiment led to the selection of version 3 as the optimal version. The sample corresponding to version 4 was excluded due to its open-porosity structure despite its excellent physio-thermal characteristics. Among the other specimens, the sample made in version 3 is the best: apparent density of $0.14 \text{ g}\cdot\text{cm}^{-3}$, porosity of 89.6%, heat conductivity of $0.042 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$, compression strength of 1.2 MPa, and pore size between 40-80 μm .

The material composition of mixture for making the optimal specimen included: 7.5% NaOH, 6% borax as well as 8% TiO_2 . According to experimental results, the maximum allowable proportion of NaOH addition was 7.5 % and the optimal NaOH/borax ratio was 1.25.

4. Conclusions

The work aimed to create a fine-porosity glass foam from ground recycled glass waste, using NaOH dissolved in distilled water, borax, and titanium dioxide. The aqueous NaOH solution had the role of reducing the density of the foamed product and at the same time, reducing the closed-porosity. The experimental optimization of the physical-thermal characteristics of the glass foam aiming at obtaining a very low density and a structure predominantly consisting of closed-pores in relation to open ones was one of the original priority objectives of the study. Also, adopting the own unconventional microwave heating method by using a protective screen (ceramic tube) made of SiC and Si_3N_4 with a wall thickness of 2.5 mm to temper the direct irradiation of the heated material was another element of originality. Four experimental versions were tested, where the NaOH/borax ratio was varied between 0.75-2.50. The sintering temperature was within the limits of 810-825°C, the heating times being in the range of 27-60 min. The optimal version, using 7.5% NaOH, 6% borax, and 8% TiO_2 , allowed to obtain the following features of the glass foam: apparent density of $0.14 \text{ g}\cdot\text{cm}^{-3}$, porosity of 89.6%, heat conductivity of $0.042 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$,

compression strength of 1.2 MPa, and pore size between 40-80 μm . According to the experimental results, the maximum permissible proportion of NaOH was 7.5% and the optimal NaOH/borax ratio was 1.25.

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FABRICAREA TERMOIZOLAȚIEI CU POROZITATE FINĂ ASISTATĂ DE MICROUNDRE

(Rezumat)

Spuma de sticlă cu porozitate fină s-a realizat din sticlă reziduală reciclată măcinată, utilizând ca adaosuri hidroxid de sodiu dizolvat în apă distilată, borax și dioxid de titan. Amestecul uscat a fost încălzit pentru sinterizare la 810-825°C într-un cuptor cu microundre adaptat constructiv și funcțional, duratele de încălzire fiind variate între 27-60 min. Proporția NaOH a fost crescută de la 4,5 la 9%, astfel încât raportul NaOH/borax să aibă valori între 0,75-1,50. Experimentele au permis identificarea raportului optim NaOH/borax (1,25), astfel încât caracteristicile specimenului adecvat să fie: densitatea de 0,14 g·cm⁻³, porozitatea de 89,6%, conductivitatea termică de 0,042 W·m⁻¹·K⁻¹ și rezistența la compresiune de 1,2 MPa. Dimensiunea porilor a fost foarte redusă (între 40-80 μm), obținându-se o structură predominant cu porozitate închisă.