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BIO-FOAMING AGENT USED FOR PREPARING CELLULAR GLASS FROM RECYCLED RESIDUAL GLASS

BY

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Abstract. Cellular glass from powders of recycled residual glass, borax as a fluxing agent, and elm leaves as a bio-foaming agent thermally treated at over 825ºC by unconventional microwave heating (according to the own technique) was experimentally manufactured. The wetted mixture was pressed into a mold and then freely inserted into the inner space of a ceramic tube of high microwave susceptible materials placed inside the microwave oven. Ground elm leaves (51 wt. %) were used for the first time as a bio-foaming agent replacing the much more expensive usual foaming agents. The results showed that the cellular glass in the optimal variant is suitable for its application as a heat-insulating building material, having the apparent density of 0.35 g/cm^3, thermal conductivity of 0.074 W/m·K, and porosity of 83.3%. Due to the choice of using borax (6 wt. %), the compressive strength of the foam was improved, the 2.5 MPa value of the optimal variant being only the lowest of the range of 2.5-5.9 MPa achieved in this experiment.

Keywords: cellular glass, residual glass, elm leaf, borax, microwave heating.

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1. Introduction

The continuous increase of waste generation in the world and the need to protect the environment against it has led in recent decades to the development of projects at an international level for recycling the waste by its reusing in order to manufacturing new products or re-introducing it into the initial manufacturing circuit. Residual glass in the form of post-consumer commercial products or those at the end of their life, such as the waste from the demolition of buildings, represents a very large amount with a continuously increasing generation rate. Recycling the glass waste as raw material in manufacturing processes of the new glass is a technologically used process, but processing the selection by colour and implicitly by quality is expensive. The orientation towards the manufacture of alternative construction materials made by foaming the residual glass is an efficient operation, especially since it is not necessary to select the waste by colour.

The principle of waste foaming is the incorporation of a foaming agent in the starting mixture and heating them to temperatures between 750-1100ºC, at which the agent releases a gas or a gaseous compound that spreads in the softened mass of waste, but remains blocked, in the form of bubbles gas. At the end of the process, by cooling, the bubbles turn into pores forming the typical structure of glass foam. Being porous, the new material has low density and also low thermal conductivity, which ensure excellent thermal insulation properties. Several types of foaming agent are known and used in the industrial production of cellular glass (black carbon, coal, graphite, calcium or sodium carbonates, dolomite, gypsum, silicon carbide, silicon nitride, glycerol) (Scarinci *et al*., 2005). Research is also very active in this area, testing various other types of cheaper foaming agent types, usually looking for waste that has the ability to foam the glass such as clam shells (Lunip *et al.*, 2016), egg shells (Fernandes *et al*., 2013), ceramic lining of used casting molds (Vancea and Lazau, 2014), polishing glass powder organic residue (Attila *et al*., 2013), water glass without other foaming agent (Hesky *et al.*, 2015; Owoeye *et al.*, 2020), propyl gallate (Qu *et al.*, 2016), foaming agents of vegetable origin such as banana leaves Arcaro *et al.*, 2016), yerba mate (da Silva *et al*., 2018), oak leaves (Paunescu *et al.*, 2020), etc.

It is important to specify that the heating techniques for foaming the glass waste applied in industrial processes as well as those used in small-scale experiments mentioned above were conventional. Some of the authors of the current paper have recently carried out unconventional heating tests by fast and economic microwave irradiation of the material subjected to foaming. Although electromagnetic waves have been known since the middle of the 20th century, their application in the world has been limited to few fields (transmissions, radar, processes of drying and low-temperature heating), the high-temperature heating being tried in extremely few cases. The group of Romanian companies Daily Sourcing & Research SRL and Cosfel Actual SRL has in its experimental base

more equipment that can operate with microwaves up to high temperature (below 1200ºC) and these have been used in several previous experiments, including in making processes of glass foam with vegetable foaming agent (Paunescu *et al.*, 2020). The own heating technique based on the microwave radiation applied in foaming the glass waste constitutes the originality of this paper together with the first use of finely ground elm leaves as an expanding agent.

The work aimed to make a glass foam with excellent thermal insulation properties (low density and low thermal conductivity), but simultaneously having an acceptable compressive strength.

Elm is a type of tree or shrub from the Ulmaceae family with a bushy crown, which grows preferably in colder areas in North America, Great Britain, Siberia, but also in China. In Europe there are three species of elm: white elm in southern areas, wych elm, more in northern areas and field elm, more in the eastern area (Caudullo and de Rigo, 2017).

2. Methods and Materials

The own method of microwave heating of the glass waste-based material mixture was established taking into account that the direct contact intensity of the microwave field emitted with the usual frequency of 2.45 GHz and the magnetron power of 800 W against the material is excessively strong causing destruction of the internal structure of glass foam. Decreasing the heating rate from over 40ºC/min to adequate values of 20-25ºC/min was achieved by placing between the issuing source and material of a ceramic tube made of a mixture of silicon carbide and silicon nitride in the 80/20 ratio, also provided with a lid made of the same material, with the outer diameter of 125 mm, height of 100 mm and wall thickness of 2.5 mm. The homogeneous mixture of the material, previously pressed in a metal mold with removable walls, was freely placed after its removing from the mold in the inner space of the tube on a metal support placed on the heat-insulating bed of ceramic fiber mattresses at the base of the microwave oven. Microwave heating has the peculiarity of being initiated in the material core by converting the power of waves into heat. This volumetrically propagates (Jones *et al.*, 2002) towards the peripheral areas (in the opposite direction compared to the conventional heating). That is why it is essential to effectively thermal protect the outer surfaces of tube and lid with ceramic fiber mattress resistant to 1200ºC to avoid heat loss outside the system.

Silicon carbide and silicon nitride in the composition of the ceramic tube are materials highly susceptible to microwaves, which absorb radiation efficiently. The thickness of the tube of 2.5 mm was experimentally determined by the authors (Axinte *et al.*, 2019), such as to allow the predominantly direct heating, through which the waves penetrate the ceramic wall and come into direct contact with the glass powder. Partially, the waves are absorbed in the mass of the tube wall, which quickly heat up through the same conversion of the wave

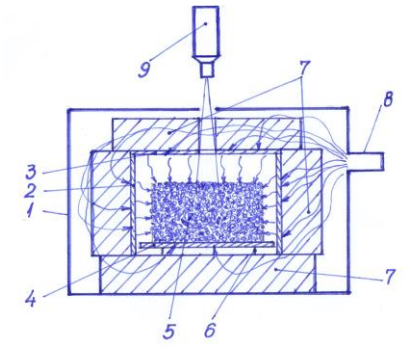
power into heat mentioned above. The wall heat is then transferred by thermal radiation to the glass.

The radiation pyrometer mounted above the oven in its central axis ensured the temperature control of material, being visualized through the holes provided in the upper metal wall of the oven, the ceramic lid and the ceramic fiber mattress that protects the lid.

Choosing a bio-foaming agent (of vegetable origin) is a cheap solution compared to the mineral agents noted above. The carbon in the composition of tree leaves is an excellent agent contributing to the expansion of raw material powder as a result of its oxidation reaction (at 750-900ºC) and the release of carbon dioxide and carbon monoxide, which remain trapped in the form of gas bubbles.

The microwave equipment and its constructive and functional scheme are shown in Fig. 1.

a



b

Fig. 1 – Microwave equipment and its constructive and functional scheme
a – overall microwave equipment; b – constructive and functional scheme:
1 – 800 W-microwave oven; 2 – ceramic tube; 3 – ceramic lid; 4 – metal plate;
5 – pressed powder material; 6 – metal support; 7 – thermal insulation;
8 – waveguide; 9 – radiation pyrometer.

Materials used in this experiment were recycled glass waste (50% colourless, 25% green, and 25% amber) and sodium borate (borax) as raw material as well as ground elm leaves as a foaming agent. The glass waste was selected from post-consumer drinking bottles in the three colours mentioned above with the oxide composition indicated in Table 1. The waste was washed and dried at 110ºC, then broken and ground in a ball mill. After sieving, the grain size below 80 μm was retained for the experiment. The high Na_2O content (30.8%) of borax was decisive for its choice as a fluxing agent in the current experiment. Na_2O is the most important and well-known fluxing material mainly for the glass industry (Bray, 2001). Also, borax contains high proportions of boron in the form of boric oxide (69.2%) according to (Borax, 2016).

Table 1
Oxide composition of recycled residual glass specimens

Oxide composition	Glass waste type (wt. %)		
	Colourless	Green	Amber
SiO_2	71.7	71.8	71.1
Al_2O_3	1.9	1.9	2.0
CaO	12.0	11.8	12.1
Fe_2O_3	-	-	0.2
MgO	1.0	1.2	1.1
Na_2O	13.3	13.1	13.3
K_2O	-	0.1	0.1
Cr_2O_3	0.05	0.09	-
SO_3	-	-	0.05
Other oxides	0.05	0.01	0.05

It is known from the literature (Paunescu *et al*., 2018) the favorable effect of the use of boron on the mechanical strength of glass foam and this property was interesting in the case of this experiment to obtain a foamed product with higher strength. Borax was commercially purchased with a grain size below 400 μm. Processed by grinding in a usual electric device, its grain size was reduced below 100 μm.

The elm leaves (selected from freshly fallen leaves from the tree, avoiding the dead leaves) were chemically analyzed identifying the carbon, hydrogen, and nitrogen content in their composition with Perkin-Elmer CHN 2400 analyzer. Also, ash, volatile materials, solids, and fixed carbon contents were determined according to ASTM E1755-01 (2020), ASTM E872-82 (2006), and ASTM E1756-08 (2008). The determining results are presented in Table 2.

Table 2
Dry composition of elm leaf

Volatile solids (wt. %)	Fixed carbon (wt. %)	Ash (wt. %)	Carbon (wt. %)	Hydrogen (wt. %)	Nitrogen (wt. %)
74.3	13.0	6.4	41.1	5.7	0.8

Biomass components such as lignocellulosic and carbon fractions indicating the organic matter presence is revealed by the high amount of volatile solids. According to the literature (Demirbas, 2004), volatiles decompose by heating at 80-140ºC into gaseous compounds such as light hydrocarbons (methane, ethane, propane, and butane), carbon mono- and dioxide as well as tar, which up to 600ºC decompose almost completely in elementary components. The heating rate and the temperature level have a favorable role on this process.

Before mixing with the residual glass powder and borax, the elm leaves were dried by heating at 110ºC and repeatedly ground in a laboratory grinder to

a grain size below 500 μm. The mixing of the three solid components was carried out in an electrically operated mixer for 3 min. Then, the mixture was wetted with water as a binder and moderately pressed in a cylindrical metal mold. The compacted material was removed from the mold and placed freely in the microwave oven according to the details presented in the method description.

Usual characterization methods were applied to identify the features of glass foam specimens. The apparent density was measured by the gravimetric method (Manual, 1999) and porosity was determined by the comparing method between the apparent density and the compact material density (true density) after melting and cooling (Anovitz and Cole, 2005). The thermal conductivity measurement was made with a flat steady-state heat-flow meter according to the method developed by Yue *et al.*, 2006 and the compressive strength was determined with 10 kN-hydraulic axial press machines according to EN 826:2013. Test of water absorption for 24 hours was made by the method of specimen immersion in water using the procedure UNI EN 1338:2004 recommended by Andreola *et al.*, 2013. The microstructural appearance of glass foam specimens was investigated with the Biological Microscope MT5000 model with captured image, 1000 x magnification and using the X-ray diffractometer Bruker-AXS D8 Advance with CuKα radiation according to EN 13925-2: 2003, the XRD examination was made. The characterization methods of glass foam specimens mentioned above were applied to existing equipment in Metallurgical Research Institute SA, University "Politehnica" of Bucharest, Daily Sourcing & Research SRL and Cosfel Actual SRL.

3. Results and Discussion

Four experimental variants were adopted, the solid mixture containing variable proportions of fine powders of glass waste, borax, and elm leaves. Considering the experimental results previously obtained under the conditions of using banana leaves (Arcaro *et al.*, 2016) and oak leaves (Paunescu *et al.*, 2020), the proportions of the vegetable foaming agent were significantly higher compared to the proportions of the mineral foaming agents usually industrially or experimentally used in the world (1-2 wt. %, reaching a maximum of 5 wt. % in the case of Na_2CO_3 according to Scarinci *et al*.,2005). Thus, the adopted range of elm leaves values for the tests was 30-51 wt. %. Borax as a fluxing agent and at the same time, an additional material that contributes to increasing the mechanical strength of the foam, was used at a constant value (6 wt. %). Raw material (recycled residual glass) completed the amount of the solid mixture (between 43-64 wt. %). Water addition (13 wt. % in all variants) had the role of a binder to facilitate the cold mixture pressing. The composition distribution of variants tested in the current experiment is shown in Table 3.

Table 3
Composition of experimental variants

Variant	Glass waste (wt. %)	Borax (wt. %)	Elm leaves (wt. %)	Water addition (wt. %)
1	64	6	30	13
2	57	6	37	13
3	50	6	44	13
4	43	6	51	13

Considering the decomposition process at 80-140ºC of volatiles into gaseous compounds and then, up to 600ºC into elementary components, the heating rate was kept at low values (below 5ºC/min) up to 600ºC by periodically stopping the oven energy supply. Above this temperature, the heating rate had the maximum available level of over 27ºC/min until the material expansion occurs, which can be observed by stopping the tendency of the material temperature to rise as measured by the pyrometer and the slow start of its decrease.

The main functional parameters of the making process of glass foam are presented in Table 4.

Table 4
Main functional parameters of the process

Parameter	Variant 1	Variant 2	Variant 3	Variant 4
Dry/wet raw material amount (g)	400/452	400/452	400/452	400/452
Foaming temperature (ºC)	825	829	832	835
Heating duration (min)	47	49	52	56
Average rate (ºC/min) - heating - cooling	 17.1 5.2	 16.5 5.2	 15.6 5.3	 14.6 5.1
Glass foam amount (g)	380	376	379	380
Index of volume growth	1.40	1.50	1.60	1.75
Specific consumption of electricity (kWh/kg)	1.29	1.36	1.43	1.54

According to the data in Table 4, the amount of solid materials forming the starting mixture was kept at the constant value of 400 g and the amount of wet materials after the addition of water as a binder was 452 g for all the variants tested. The temperature at which the expansion process was finished varied between 825ºC corresponding to variant 1, in which the proportion of elm leaves was minimal (30 wt. %) and 835ºC corresponding to variant 4 having the highest proportion of leaves (51 wt. %). The duration of the heating process increased from 47 min (variant 1) to 56 min (variant 4). Keeping the low limit of the heating

rate (below 5ºC/min) up to 600ºC decisively influenced the average value of the heating rate of the process in all variants. The highest average rate was that recorded in variant 1 of 17.1ºC/min and the lowest was recorded in variant 4 of 14.6ºC/min. The average free cooling rate of the cellular glass was relatively constant (5.1-5.3ºC/min). The specific consumption of electricity for activating the oven magnetron was determined by reading the meter index, divided to the amount of made glass foam (between 376-380 g). According to the data in Table 4, the specific energy consumption had low values between 1.29-1.54 kWh/kg. The increase in volume by expansion was evaluated by comparing the volume of the foamed material with the initial volume of the compacted mixture. The physical appearance of the glass foam specimens is shown in Fig. 2.

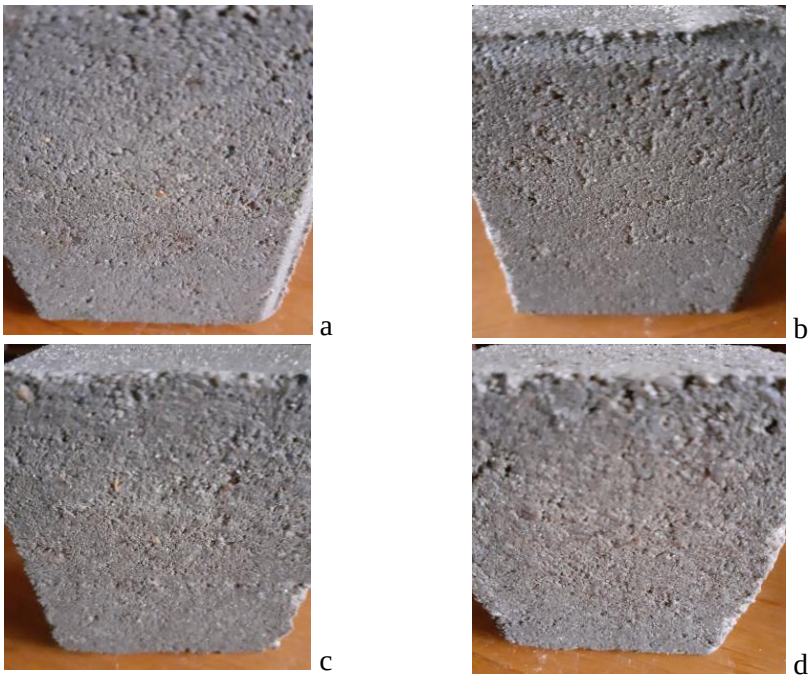


Fig. 2 − Appearance of the glass foam specimens
a – variant 1; b – variant 2; c – variant 3; d – variant 4.

The analysis of the microstructural peculiarities of glass foams in the four experimental variants was carried out by examining the specimens section under the microscope. Images of the microstructural appearance of specimens are shown in Fig. 3. According to the images, the distribution of the closed cells that compose the microstructures in the four variants is uniform. Their size is variable, falling within the following value ranges: 0.1-0.4 mm (variant 1), 0.2-0.6 mm (variant 2), 0.3-0.8 mm (variant 3), and 0.3-1.0 mm (variant 4).

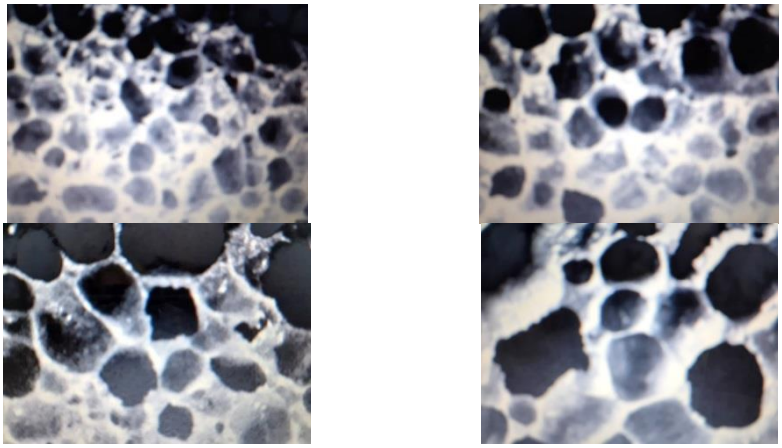


Fig. 3 − Images of microstructural configuration of cellular glass specimens
a – variant 1; b – variant 2; c – variant 3; d – variant 4.

The results of determining the physical, thermal, mechanical and microstructural characteristics of the glass foam specimens using the investigation methods mentioned above are centralized in Table 5.

Table 5
Characteristics of glass foam specimens

Vari-ant	Apparent density (g/cm^3)	Porosity (%)	Thermal conductivity (W/m·K)	Compressive strength (MPa)	Water absorption (vol. %)	Pore size (mm)
1	0.87	58.6	0.180	5.9	1.2	0.1-0.4
2	0.68	67.6	0.137	4.7	1.0	0.2-0.6
3	0.51	75.7	0.103	3.6	0.9	0.3-0.8
4	0.35	83.3	0.074	2.5	0.8	0.3-1.0

The mechanical features of the glass foam samples were significantly influenced by the addition of 6 wt. % borax. Thus, the compressive strength values were over 2.5 MPa reaching the highest level (5.9 MPa) in the case of variant 1 prepared with the lowest proportion of elm leaves as foaming agent (30 wt. %).

Due to the expansion effect of material, the foam apparent density was reduced from 0.87 g/cm^3 in the case of variant 1 to 0.35 g/cm^3 in the case of variant 4, which benefited from the highest proportion of foaming agent (51 wt. %). Implicitly, the glass foam porosity increased from variant 1 to variant 4 (in the range of 58.6-83.3%) and the thermal conductivity decreased from 0.180 W/m·K to 0.074 W/m·K. The water absorption test by immersion in water of specimens showed that the porous material is absorbent only to a very small extent (0.8-1.2 vol. %).

The XRD pattern of cellular glass made from glass waste, borax and vegetable foaming agent at 835 ºC highlighted the presence of crystalline phase peaks representing wollastonite, quartz, and cristobalite (Fig. 4).

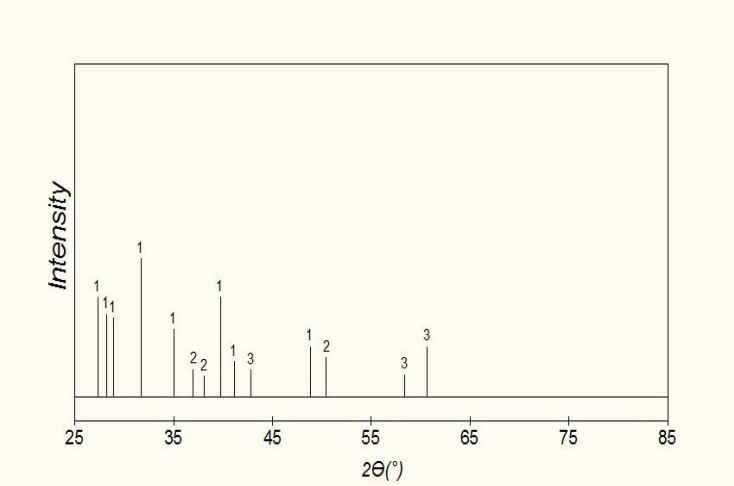


Fig. 4 − XRD pattern of cellular glass at 835ºC:
1 – wollastonite; 2 – quartz; 3 – cristobalite.

The analysis of the experimental results carried out in order to produce glass foam using elm leaves as a bio-foaming agent led to the conclusion that the optimal variant is variant 4, in which the proportion of the foaming agent was the highest (51 wt. %). Although the compressive strength had the lowest value compared to the other variants, it was still high enough (2.5 MPa) to be suitable for the use of glass foam in building applications. On the other hand, the characteristics that influence the thermal insulation properties of the material, *i.e.* apparent density, thermal conductivity, and porosity had excellent values: 0.35 g/cm^3, 0.074 W/m·K, and 83.3%, respectively. Water absorption had a very low value of only 0.8 vol. %.

The comparison with another glass foam manufactured according to the literature (Arcaro *et al*., 2016) from waste glass and banana leaves (30-50 wt. %) by conventional heating between 700-850ºC showed that the ranges of porosity values (58.5-87.5%), the compressive strength (1.17-3.50 MPa), and the thermal conductivity (0.06-0.15 W/m·K) include the optimal values of the current experiment, but the range of compressive strength is inferior in value compared to the limits of this characteristic in the tested variants in this experiment (2.5-5.9 MPa).

Also, there is a good similarity between the experimental results of this work and those reported in (Paunescu *et al.*, 2020) regarding the manufacture of glass foam using oak leaves, except for the compressive strength values which are only between 1.2-3.4 MPa.

4. Conclusions

Experimental production of cellular glass using residual materials such as recycled glass waste (43-64 wt. %) and elm leaves as a bio-foaming agent (30-51 wt. %) as well as sodium borate also known as borax (6 wt. %) with the role of fluxing agent and water addition as a binder (13 wt. %) was performed by grinding the solids, their mixing, watering, and pressing in the form of a compacted material, which was heated to over 825ºC in an adapted microwave oven for high temperature operation. The own technique of predominantly direct and partially indirect microwave heating by means of a high microwave susceptible ceramic tube placed between the wave emission source and the compacted material as well as the first use of elm leaves as a foaming agent were the original elements of the work.

Four experimental variants were tested under the conditions of varying the weight proportions of glass waste and elm leaves within the limits shown above. By increasing the proportion of the foaming agent, the process temperature increased from 825 to 835ºC, the apparent density was reduced from 0.87 to 0.35 g/cm^3, the porosity increased from 58.6 to 83.3%, the thermal conductivity decreased from 0.180 to 0.074 W/m·K, and the compressive strength decreased from 5.9 to 2.5 MPa. Taking into account the results of experiment, variant 4 with the best thermal insulation properties, despite the minimum value of the compressive strength (2.5 MPa), which is still high enough, was adopted as the optimal variant. The glass foam made in the optimal variant is suitable for its application as a heat-insulating building material.

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BIO-AGENT DE SPUMARE UTILIZAT PENTRU FABRICAREA STICLEI CELULARE DIN DEȘEU DE STICLĂ RECICLAT

(Rezumat)

Sticlă celulară din pulberi de deșeu de sticlă reciclat, borax ca agent de fluidizare și frunze de ulm ca bio-agent de spumare, tratate termic la peste 825ºC prin încălzire neconvențională cu microunde (conform tehnicii proprii), a fost fabricată experimental. Amestecul umezit a fost presat într-o matriță și apoi introdus liber în spațiul interior al unui tub ceramic din materiale înalt susceptibile la microunde plasat într-un cuptor cu microunde. Frunze de ulm măcinate (51%) au fost utilizate în premieră ca bio-agent de spumare înlocuind agenți de spumare uzuali mult mai scumpi. Rezultatele au arătat că sticla celulară în varianta sa optimă este adecvată pentru aplicarea ca material de construcție termoizolant, având densitatea aparentă de 0,35 g/cm^3, conductivitatea termică de 0,074 W/m·K și porozitatea de 83,3%. Datorită alegerii utilizării boraxului (6%), rezistența la compresiune a spumei s-a îmbunătățit, valoarea de 2,5 MPa a variantei optime fiind doar cea mai mică a intervalului 2,5-5,9 MPa realizat în acest experiment.