Unconventional Technique for Producing Borosilicate Glass Foam

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Abstract
The study aims to test an advanced technique but insufficiently valued in the world in the process of experimental manufacture of borosilicate glass foam. It is about the unconventional technique of heating solids by using the microwave radiation converted into heat. The experimental equipment on which the tests were performed was a 0.8-kW microwave oven commonly used in the household with constructive adaptations to be operational at high temperature. The adopted manufacturing recipe was composed of borosilicate glass waste with the addition of calcium carbonate, boric acid and water in different weight proportions. The material was sintered at 829-834 ºC by predominantly direct microwave heating and the optimal foamed product had characteristics similar to those manufactured by conventional techniques (apparent density of 0.33 g/cm³, thermal conductivity of 0.070 W/m•K, compressive strength of 3.1 MPa and a homogeneous microstructure with pore size between 0.7-1.0 mm). The energy efficiency of the unconventional manufacturing process was remarkable, the specific energy consumption being only 0.92 kWh/kg.

Introduction
The borosilicate glass is a type of glass containing mainly silica (SiO₂) and boron trioxide (B₂O₃). It has a very low coefficient of thermal expansion and a high resistance to thermal shock compared to any of the usual glasses. Also, it is chemically inert. The borosilicate glass is commonly used as laboratory glassware, pharmaceutical containers, optical equipment, cookware, equipment for lighting and electronics (Willsey, 2015). According to (Glass, 2019), the average chemical composition of the borosilicate glass includes: 75.0% SiO₂; 10.5% B₂O₃; 5.0% Na₂O; 7.0% Al₂O₃ and 1.5% CaO. The softening point of glass is 785-821 ºC.

According to an European Union report (Rodriguez Vieltez et al., 2011), the production of borosilicate glass in 2007 in the EU countries for the application fields mentioned above was 3.7 millions tons and the amount of borosilicate glass waste generated in the same year was 2 millions tons. Generally, glass foam using glass waste as a basic raw material has proven excellent properties such as light weight, low thermal conductivity, thermal and chemical stability, non-toxicity, satisfactory high mechanical strength, fire resistance,
resistance to rodent attack, insects, bacteria, acids, impermeability to water and steam, etc. (Scarinci et al., 2005). The borosilicate glass foam incorporates the above properties, with the mention that the resistance to acids (sulfuric acid, hydrochloric acid, etc.) is remarkable (Zhenshen, 2019) and the thermal expansion coefficient has very low values (6•10⁻⁶ K⁻¹). Due to its characteristics the borosilicate glass foam is usable in industry and civil engineering, especially as anticorrosive equipment and thermal insulating material.

The literature presents several techniques for the manufacture of borosilicate glass foam using borosilicate glass waste and different types of foaming agents and mineral additives. According to (Scarinci et al., 2005), the most commonly used foaming agent, suitable for the manufacture of borosilicate glass foam, is carbon black with a grain size below 150 μm. In order to obtain a quality foaming, an oxygen supplier is needed, such as SO₃ in the composition of glass waste (usually in green and amber soda-lime glass waste) or additions of oxides such as iron oxide (Fe₂O₃) or antimony oxide (Sb₂O₃) in the raw material. The average heating rate of the sintering/foaming process recommended in the paper (Lv et al., 2010) is 8 °C/min.

The use of borosilicate glass waste mixed with disodium phosphate (Na₂HPO₄) as a stabilizing agent, Sb₂O₃ as an additional oxygen supplier and carbon black as a foaming agent was presented in the paper (Zhai et al., 2014). The best results were obtained by sintering the powder mixture at 775 °C and the use of 1% carbon black, 6% Na₂HPO₄ and 0.9% Sb₂O₃. The bulk density had the value of 0.408 g/cm³, the porosity was 84.6%, the compressive strength reached 1.57 MPa and the water absorption was 1.57%. It has been found that by increasing the proportion of Sb₂O₃ in the glass mass, the viscosity is decreased leading to a reduction in the sintering temperature. Also, the low viscosity generates pores that come to communicate with each other. A much lower viscosity of molten glass is unfavorable for the formation of a homogeneous microstructure. Due to the presence of boron in the composition of the foam glass, a high value of compressive strength (4.4 MPa) was reached. The main crystalline phase of the foam sintered at 775 °C was sodium aluminum phosphate and to a lesser extent cristobalite.

By sintering at 1200 °C for 30 min, a glass foam was manufactured of borosilicate glass waste (91%), carbon black (0.9%) and Sb₂O₃ (8.1%) (Borosilicate, 2015). The product had a bulk density of 0.5 g/cm³, the water absorption of 0.4% and the average thermal expansion coefficient of 9.22•10⁻⁶ K⁻¹. The porous microstructure was homogeneous with pore size between 0.2-1.0 mm.

The research of the influence of Sb₂O₃ addition in the mass of borosilicate glass waste on the properties of glass foam obtained by sintering at 1500 °C is presented in Du, 2007. Low apparent density of 0.3 g/cm³ and high mechanical strength were obtained for a Sb₂O₃ addition of 0.6%. According to (Fan & Song, 2002), the increase of the porosity by 10-15% and of the mechanical strength by 20% can be achieved with 0.2-0.3% Sb₂O₃ addition.

Sintering at temperatures up to 900 °C of a borosilicate glass waste with an organic binder as a foaming agent has been experimentally researched and presented in the literature (Taurino et al., 2014). The crystallization process of the powder mixture began at 845 °C and was completed at 900 °C. The main crystalline phases of the glass foam were wollastonite and cristobalite. The apparent density of the product had the value of 0.5 g/cm³ and the porosity was between 78-79%. The product microstructure was homogeneous with closed pores, suitable for the use in civil engineering as a thermal insulating material.
The paper (Bayer, 1980) showed that experiments on the manufacture of borosilicate glass foam using as foaming agents silicon carbide (SiC) or silicon nitride (Si3N4), are very effective for foaming processes at over 950 °C. These agents allowed the control of the foamed product microstructure and the pore size. The crystallization tendency of glass powder to cristobalite could be inhibited by the addition of alumina-based materials. The production of foaming gases occurred at 950-1150 °C by the oxidation of SiC or Si3N4 in the oxidizing atmosphere of the oven.

The use of calcium carbonate (CaCO3) and boric acid (H3BO3) in the manufacturing process of borosilicate glass foam was presented in the paper (Liu et al., 2012). The weight ratio of CaCO3 was low (between 1-2%) and that of H3BO3 was 3%. The experimental results showed that the samples microstructure was homogeneous with pores with average dimension of 1 mm. The foaming effect of glass waste with CaCO3 was strongly enhanced with the increase of H3BO3 proportion. However, an excess of CaCO3 led to an increase of the apparent density and a reduction of pore size.

All the works presented above include manufacturing processes of borosilicate glass foam performed by conventional heat treatment methods. The literature also contains several works on the production of borosilicate glass foam using the unconventional method of microwave heating. The production of a reinforced borosilicate glass foam with nickel-based alloy fibres was performed on a microwave equipment operating at a frequency of 2.45 GHz (Veronesi et al., 2018). The power of the microwave generator could be varied between 300-3000 W. A silicon carbide disc was used as an auxiliary microwave absorber. The experimental results showed that a maximum volumetric ratio (10%) of metal fibres can improve the homogeneity of pores distribution in the material microstructure. The samples manufactured with 10% metal fibres using the SiC microwave absorber were the best. Probably, the fibres acted as nucleating agents for pore generation. The sintering time of the raw material was very short: below 3 min. The high porosity of the porous material combined with the mechanical strength of the metal fibres led to a composite with high resistance to thermal shock suitable for thermal protection systems.

Other experiments were recently performed in the field of borosilicate glass foam production using the microwave radiation as an energy source (Paunescu et al., 2019) in the Romanian company Daily Sourcing & Research. The experiments were performed on a 0.8 kW-microwave oven of the type commonly used in the household adapted for high temperature operation. Several types of foaming agent and mineral additives were successively used: SiC (3%) as a foaming agent and coal fly ash (9.1%); CaCO3 (1.3%) as a foaming agent; activated carbon (1%) as a foaming agent and Na2HPO4 (6.2%). The sintering/foaming processes occurred at 970, 830 and 820 °C respectively. The best variant in terms of material quality and specific energy consumption was those made with activated carbon and Na2HPO4. Its main characteristics were: apparent density of 0.34 g/cm3, thermal conductivity of 0.055 W/m•K and compressive strength of 2.5 MPa. The product microstructure was homogeneous, with pore size between 1.0-2.5 mm. The specific energy consumption of the heating process was 2.84 kWh/kg, the value being relatively high due to the small amount of raw material (250.6 g) compared to the available power of the oven.

The research in the glass foam manufacturing field focused in the last four years on the unconventional technique application of the microwave radiation in the Romanian company Daily Sourcing & Research has shown on an low experimental scale an energy efficiency at
least similar to that of the industrial production. The microwave heating technique is almost unused in the glass industry.

The paper aimed to manufacture a borosilicate glass foam with physical, mechanical and microstructural characteristics superior to those previously obtained, under improved conditions of microwave irradiation, in order to reduce the specific energy consumption below 1 kWh/kg.

**Methods**

The manufacturing technique of borosilicate glass foam involves two ways of approaching this problem: one with an energetic character and a technological one. In terms of energy, the adopted solution is the predominantly direct microwave heating based on the existence of a ceramic screen made of a high microwave-susceptible material (SiC + Si3N4) placed between the microwave emitting source and the material subjected to heating. The screen is a ceramic tube with the outer diameter of 125 mm, the height of 100 mm and the wall thickness of 2.5 mm. The material previously pressed into a mold and then released is placed freely in the inner space of the tube on a metal plate. The ceramic tube and the metal plate that supports the pressed material are deposited on a bed of ceramic fibre mattresses at the base of the microwave oven. The upper opening of the tube is covered with a lid made of the same material. The tube and the lid are thermally protected with ceramic fibre mattresses to avoid the heat loss to the outside. It should be noted that the microwave heating mode is completely different from the conventional heating, the thermal process being initiated in the core of the material, after which the heat flow propagates from the inside to the peripheral areas. The use of the ceramic tube with a wall thickness of 2.5 mm allows the predominant penetration of the waves, which act directly on the material. However, part of the microwave field is absorbed into the mass of the tube wall, which heats up rapidly and intensely, becoming a secondary emission source of thermal energy to the material by radiation. The control of the heating process of the material was carried out with a radiation pyrometer mounted above the oven at about 400 mm. To visualize the upper surface of the heated material, the upper sheet wall of the oven was provided with a 30 mm-diameter hole and also similar holes were made in the lid and the ceramic fibre protection layer of the lid. In Figure 1 the main components of the experimental microwave equipment are shown.

![Figure 1. Experimental microwave equipment](image)

A – constructive scheme of the experimental equipment: 1 – 0.8 kW-microwave oven; 2 – ceramic tube; 3 – ceramic lid; 4 – metal plate; 5 – pressed material mixture; 6 – metal support; 7 – ceramic fibre mattress; 8 – waveguide; 9 – radiation pyrometer; B – overall image of the microwave equipment; C – SiC + Si3N4 ceramic tube; D - ceramic fibre thermal protection of tube and lid.

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In terms of the manufacturing technology, the solution of using CaCO₃ as a foaming agent together with H₃BO₃ as a mineral additive was adopted. The basic principle of foaming glass waste with CaCO₃ is its decomposition with the release of a gas (CO₂) in the viscous mass of the thermally softened glass, which is blocked in the form of bubbles. By cooling, the gas bubbles will form a porous structure specific to the glass foam. Calcium oxide (CaO) also resulting from the decomposition process enters in the composition of molten glass. The chemical decomposition reaction of CaCO₃ is as follows:

\[ \text{CaCO}_3 = \text{CaO} + \text{CO}_2 \quad (1) \]

According to (Ducman, 2004; Karundasa et al., 2019), the reaction (1) occurs at temperatures in the range 900-1150 °C. By heating at low temperature (above 300 °C) boric acid is gradually dehydrated forming pyroboric acid (H₂B₄O₇), which around 330 °C decomposes into boron trioxides (B₂O₃) and water vapor (Balci et al., 2012).

\[ \text{H}_2\text{B}_4\text{O}_7 = 2\text{B}_2\text{O}_3 + \text{H}_2\text{O} \quad (2) \]

B₂O₃ thus formed which is an anhydrous microcrystalline material enters in the molten glass composition at over 900 °C increasing the B₂O₃ weight ratio and contributing to the increase of thermal shock resistance, chemical resistance and mechanical strength of glass foam. The materials used in experiments were borosilicate glass waste, calcium carbonate as a foaming agent and boric acid as a mineral additive. The chemical composition of the borosilicate glass waste including mainly laboratory glassware waste was: 71.5% SiO₂; 12.7% B₂O₃; 2.6% Al₂O₃; 5.8% Na₂O; 0.2% K₂O; 4.0% CaO; 2.2% MgO; 0.3% Fe₂O₃; 0.7% TiO₂. The glass waste was broken, ground in a ball mill and sieved at a grain size below 100 μm. The commercial calcium carbonate with a grain size below 40 μm was used without any further processing. The commercial boric acid in the form of small crystals was ground in an electrical laboratory device and sieved at the grain size below 36 μm.

Based on the own experience (Paunescu et al., 2019) and information from the literature, four experimental variants were adopted for the manufacture of borosilicate glass foam including: borosilicate glass waste (between 95.1-96.0 wt.%), calcium carbonate (between 1.2-1.5 wt.%), boric acid (between 2.8-3.4 wt.%) and a constant addition of water of 10 wt.%. The composition of the four variants is shown in Table 1. As mentioned above, the heating technique adopted was unconventional being used microwave radiation as an energy source.

### Table 1. Experimental variants of raw material

| Raw materials                  | Variant 1 | Variant 2 | Variant 3 | Variant 4 |
|-------------------------------|-----------|-----------|-----------|-----------|
| Borosilicate glass waste, wt.%| 96.0      | 95.7      | 95.4      | 95.1      |
| Calcium carbonate, wt.%       | 1.2       | 1.3       | 1.4       | 1.5       |
| Boric acid, wt.%              | 2.8       | 3.0       | 3.2       | 3.4       |
| Water addition, wt.%          | 10.0      | 10.0      | 10.0      | 10.0      |

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**Result and Discussion**

The experiments were performed on the 0.8 kW-microwave oven described above in the four variants indicated in Table 1. The quantities of dry and wet raw material (536 g and 590 g, respectively) were kept constant for each variant. Also, the microwave emission was kept constant and the power dissipated by the single magnetron of the oven in the system (around 700 W) was not changed throughout the experiment. Table 2 shows the variation of the functional parameters determined for each experimental variant. The temperature of the heated material was measured with the radiation pyrometer beginning at about 750 °C (taking into account its measuring domain). The indication of the pyrometer that the temperature rise curve has almost flattened and the growth has stagnated is the signal that the foaming process has reached its maximum level. The beginning of the slight decrease of the temperature value is the indication that the foaming occurred in the whole mass of the material and reached its upper area. At this point, the power supply to the magnetron was stopped. Keeping it in operation would mean overheating the material with negative effects on the microstructure and its physical and mechanical properties.

| Parameter                  | Variant 1 | Variant 2 | Variant 3 | Variant 4 |
|----------------------------|-----------|-----------|-----------|-----------|
| Dry/wet raw material amount, g | 536/590   | 536/590   | 536/590   | 536/590   |
| Sintering/foaming temperature, °C | 829       | 830       | 832       | 834       |
| Heating time, min           | 39.5      | 40        | 41        | 43        |
| Average rate, °C/min        |           |           |           |           |
| -Heating                   | 20.4      | 20.3      | 19.8      | 18.9      |
| -Cooling                   | 5.8       | 5.6       | 5.5       | 5.7       |
| Index of volume growth      | 1.80      | 1.85      | 1.95      | 2.20      |
| Glass foam amount, G        | 520       | 522       | 519       | 520       |
| Specific energy consumption, kWh/kg | 0.89   | 0.90       | 0.92       | 0.96       |

Analyzing the data in Table 2, it is observed that the values of the final temperature of the sintering/foaming process are in a very small range (829-834 °C). The same observation is valid also for the duration of the heating process (between 39.5-43 min). Unlike the average heating rate reported in the literature [6] of 8 °C/min, the range of average heating rate values performed in the current experiments was 18.9-20.4 °C/min. The index of volume growth of foamed materials had values between 1.80-2.20, the highest volume increase corresponding to the highest ratios of CaCO₃ (1.5%) and H₃BO₃ (3.4%). The most remarkable performance of the borosilicate glass foam manufacturing process with the addition of CaCO₃ and H₃BO₃ mixed with glass waste and using the microwave radiation as an energy source was the specific energy consumption in very low limits (between 0.89-0.96 kWh/kg), which demonstrates the high energy efficiency of the unconventional
technique application by comparison with the conventional techniques still commonly used in the glass industry.

The determination of the main physical, thermal, mechanical and microstructural characteristics of experimentally produced borosilicate glass foams was performed by well-known methods. The apparent density was measured by the gravimetric method (Manual, 1999) and the porosity was calculated by the method of comparing the true and apparent density (Anovitz & Cole, 2005). The thermal conductivity was determined by the guarded-comparative-longitudinal heat flow (ASTM E1225-04 standard) and the compressive strength was measured using a Stable Micro Systems TA XT Plus Texture Analyzer. The water absorption was determined by the water immersion method (ASTM D570 standard) and the glass foam microstructure was examined with a Smartphone Digital Microscope. The physical, thermal, mechanical and microstructural characteristics of the borosilicate glass foam samples are shown in Table 3.

Table 3. Physical, thermal, mechanical and microstructural characteristics of the glass foam samples

| Variant | Apparent density g/cm³ | Porosity % | Thermal conductivity W/m-K | Compression strength MPa | Water absorption % | Pore size mm |
|---------|-------------------------|------------|-----------------------------|--------------------------|-------------------|-------------|
| 1       | 0.43                    | 80.45      | 0.087                       | 2.9                      | 1.8               | 0.1-0.3     |
| 2       | 0.38                    | 82.73      | 0.079                       | 3.0                      | 2.7               | 0.3-0.6     |
| 3       | 0.33                    | 84.98      | 0.070                       | 3.1                      | 3.0               | 0.7-1.0     |
| 4       | 0.31                    | 85.91      | 0.067                       | 3.0                      | 3.2               | 0.7-1.4     |

The data in Table 3 show that the products made by the four experimental variants had high values of compressive strength (between 2.9-3.1 MPa). The main role for this high mechanical characteristic belongs to the addition of boric acid which releases B₂O₃, whose favorable influence on the mechanical strength of glass foams is known. B₂O₃ also favors the foaming process, so that higher proportions of CaCO₃ and H₃BO₃ simultaneously, lead to a high compressive strength under high porosity conditions. The apparent density of the products was relatively low (0.31-0.43 g/cm³), with the lowest value corresponding to the highest proportion of CaCO₃ (variant 4). The porosity was high (80.45-85.91%), the highest value belonging to variant 4. Like the apparent density, the thermal conductivity had low values (0.067-0.087 W/m·K), variant 4 having the lowest value. The water absorption of the porous products has relatively low values (between 1.8-3.2%).

Figure 2 presents pictures of the four foamed products and microstructural images of the glass foam samples are shown in Figure 3.
Figure 2. Pictures of the borosilicate glass foam samples
A – sample 1 heated at 829 °C; B – sample 2 heated at 830 °C;
C – sample 3 heated at 832 °C; D – sample 4 heated at 834 °C.

Figure 3. Microstructural images of the glass foam samples
A – sample 1; B – sample 2; C – sample 3; D – sample 4.
According to the pictures in Figure 3, the microstructures of the four samples are homogeneous with an uniform distribution of closed spherical pores with low dimensions that vary depending on the manufacturing recipe. Sample 1 made with the lowest weight proportions of CaCO3 (1.2%) and H3BO3 (2.8%) has the lowest pore size (between 0.1-0.3 mm). The pore dimensions have an increase tendency with the increase of the foaming agent and additive weight proportions. Sample 2 has pores with dimensions between 0.3-0.6 mm, sample 3 has pores with dimensions in the range 0.7-1.0 mm and sample 4 manufactured with the highest weight proportions of CaCO3 (1.5%) and H3BO3 (3.4%) has pores with dimensions between 0.7-1.4 mm.

Analyzing the obtained experimental results and the requirements for the use of the foamed product in construction as a thermal insulating material with high mechanical strength, the sample manufactured by variant 3 (95.4% glass waste, 1.4% CaCO3, 3.2% H3BO3 and 10% water) was adopted as optimal. Sintered and foamed at 832 ºC by microwave heating with the average heating rate of 19.8 ºC/min the foamed product had a specific energy consumption of 0.92 kWh/kg. The glass foam characteristics were: apparent density of 0.33 g/cm3, porosity of 84.98%, thermal conductivity of 0.070 W/m•K, compressive strength of 3.1 MPa, water absorption of 3% and pore size (uniformly distributed) between 0.7-1.0 mm.

Numerous experiments presented in the literature have tested different manufacturing recipes of borosilicate glass foam determining the characteristics of the obtained products. Carbon black, silicon carbide, silicon nitride and calcium carbonate were used as foaming agents, the sintering temperature varying between 820-1200 ºC depending on the foaming agent nature, but also the nature of the various additives added to the raw material mixture. Generally, the apparent density of the foamed products were in the range 0.30-0.50 g/cm3 and the compressive strength had relatively high values reaching oven 4.4 MPa in the case of using carbon black, Na2HPO4 and Sb2O3 (Zhai et al., 2014). Most experiments were performed using the conventional heating technique, whose specific energy consumptions were not mentioned in the literature. The Romanian company Daily Sourcing & Research experimentally produced borosilicate glass foam using as foaming agent successively SiC, CaCO3 and activated carbon by sintering at 970, 830 and 820 ºC, respectively (Paunescu et al., 2019). The heating technique was unconventional based on the microwave radiation converted into heat. The characteristics of the products were similar to those of the products made by conventional techniques, but the specific energy consumption was low (2.62 kWh/kg in case of CaCO3 use and 2.84 kWh/kg in case of activated carbon use) being negatively influenced only by the small amount of raw material compared to the real heating capacity of the oven.

Comparing the experimental results obtained in this work it can be concluded that in terms of quality the foamed products were similar to those previously made (conventional or unconventional), but in terms of energy the manufacturing processes were much more efficient, the specific energy consumption of the optimal sample being only 0.92 kWh/kg.

Conclusion

An advanced but insufficiently valued technique in the world has been tested in the experimental manufacturing process of borosilicate glass foam. It is about the unconventional technique of heating solids by using the microwave radiation converted into heat. For this purpose, a 0.8 kW-microwave oven commonly used in the household in food preparation adapted for high temperature operation was the experimental microwave equipment that facilitated the production of glass foam. A known manufacturing recipe in the literature
composed of borosilicate glass waste, calcium carbonate (1.2-1.5 wt.%), boric acid (2.8-3.4 wt.%), and water addition (10 wt.%) forming a pressed powder mixture was adopted. The material was sintered at temperatures between 829-834 °C and was predominantly direct microwave heated using a high microwave susceptibility ceramic tube with a wall thickness of 2.5 mm. The optimal foamed product had an apparent density of 0.33 g/cm³, porosity of 84.98%, thermal conductivity of 0.070 W/m•K, compressive strength of 3.1 MPa, water absorption of 3% and pore size (uniformly distributed) between 0.7-1.0 mm. The manufacturing process had a very high energy efficiency the specific energy consumption being only 0.92 kWh/kg. In terms of quality the foamed products were similar to those previously made, but in terms of energy the process efficiency was much higher compared to the manufacturing processes (conventional or unconventional) known in the literature.

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