 

# HIGH-STRENGTH POROUS AGGREGATE MADE BY MICROWAVE IRRADIATION HEATING TECHNIQUE OF CLAY AND GLASS WASTES FROM BUILDING DEMOLITION

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ABSTRACT: A high-strength porous aggregate for construction applications that require compression resistance and porosity has been experimentally made by direct microwave warming of residues from building demolition (clay brick and clear flat glass). Borax (4 wt. %) as a flux agent and silicon carbide (2 wt. %) as an expanding agent were incorporated into the starting mixture. Due to the high proportion of clay waste as an aluminum silicate material, the direct microwave warming was possible at very high warming rates (between 34-42.4 ºC/min) without affecting the structural homogeneity of the final product. The specific electricity consumption was extremely low (0.59-0.78 kWh/kg). The cellular glass features were: apparent specific gravity between 0.72-0.98 g/cm$^3$, porosity between 50.3-65.7 %, heat conductivity in the range 0.161-0.197 W/m·K, compression resistance between 6.4-8.5 MPa, and pore size below 1.1 mm.
KEYWORDS: high-strength aggregate, microwave warming, residual clay brick, residual clear flat glass, borax, silicon carbide.

## 1. INTRODUCTION

The storage of waste (plastics, glasses, metals, textiles, cardboard, etc.) and industrial by-products (metallurgical slag, red mud, and other various sludges, etc.) in large landfills has suffocated and intoxicated the soil and air for decades, the rate of generation being continuously increasing until the end of the 20th century. Waste and by-products affect the health of both people and the environment, which it contaminates. Under these conditions, the international community has recently decided on numerous measures to stop or reduce the accumulation of waste by recycling it. The global research is concerned in this direction, progressing in recent decades.

At present, the glass is one of the wastes with a very high annual generation rate, coming mainly from post-consumer drinking bottle or other container glass as well as from the demolition and renovation of buildings. The possibility of producing light weight porous materials (cellular glass) with resistance to mechanical stress, fire, moisture, corrosion, etc. using pure glass, had been discovered since the middle of the last century. With the need to recycle waste, the manufacture of cellular glass began to be practiced on an industrial scale using glass waste as a raw material. The production of cellular glass from recycled residual glass by conventional high temperature heat treatment techniques has become an usual concern since the 1980s of the 20th century. The basic principle of manufacturing this material type is well known and consists in the removal of a gas (or gaseous compound) into a material softened by heating with adequate viscosity, of the glass-based basic material powder mixture. A solid or liquid expanding agent embedded in the powder mixture releases the gas by a chemical reaction in a temperature range that includes the softening point of the residual glass. The most commonly used expanding agents are: carbon (in the form of carbon black, coal, graphite, glycerol, etc.), calcium carbonate, silicon carbide, etc. [1]. Physical, thermal, mechanical and morphological properties of cellular glasses (low apparent specific gravity, high porosity, low heat conductivity, high or at least acceptable compression resistance, homogeneous porous microstructure), resistance to fire and humidity as well as to the aggression of various agents (rodents, insects, bacteria, acids, etc.), chemical stability, non-toxicity have determined a wide area of applicability, especially in construction, both as a light weight heat insulation material and different porous shapes and as high-strength dense porous materials [1, 2]. The industrial production of glass foam developed in several European countries (Switzerland, Belgium, Austria, Germany, the Czech Republic, Scandinavian countries, etc.) as well as in the United States and China is mainly based on residual glass containing soda ($Na_2CO_3$), lime ($CaO$) and silica ($SiO_2$) (post-consumer drinking bottle or flat glass), which is the most suitable basic material for the expanding process [3]. Numerous other types of residual glass have been tested together with other industrial silicate waste (cathode ray tube, metallurgical slag, coal fly ash, oil shale by-products, quartz sand, coal gangue, copper slag, etc.) on experimental scale [4-7].

Till now, the energy source used in the cellular glass industrial manufacture as well as in the majority of small-scale experiments were conventional techniques based on burning the fossil fuels or electrical resistances. Although the electromagnetic waves power converted into heat, known since 1930-1940, proved to be a fast, "clean" and economical heating technique, it was applied only in industrial drying processes and moderate temperature warming processes as well as in household at the food preparation. In recent decades, experiments on various material types (organics, metals, glasses, etc.) have showed that these are adequate for effective warming using electromagnetic waves [8]. However, the literature does not provide information on their use in unconventional industrial processes, but to a lesser extent on some tests in the experimental stage.

A research group from Daily Sourcing & Research Company (Romania) concentrated its concerns on the experimental manufacture of expanded glass applying the nonconventional method of warming the basic material containing predominantly residual glass in the microwave field. Several tests confirmed that the direct microwave heating of a pressed powder common glass ($Na_2CO_3$-CaO-$SiO_2$) is not adequate due to the severe destruction of the structural integrity by high warming [9], being necessary applying an original technique of predominantly direct microwave heating. Unlike the soda-lime glass, the aluminum silicate material (clay, coal ash, etc.) waste has been experimentally proven to be suitable for direct microwave heating without affecting its microstructural homogeneity.

An addition of aluminum silicate glass waste in soda-lime glass waste powder for manufacturing cellular glass was tested and the experimental results were presented in [10]. The basic material included residual commercial glass and alkali-earth aluminum silicate glass waste in different proportions between 0-10 wt. % as well as silicon carbide (SiC) as an expanding agent. Conventionally heated to 950 ºC, the manufactured cellular glass registered an almost linear increase in apparent specific gravity between 0.2-0.31 g/cm$^3$ as the proportion of aluminum silicate waste increased, while the compression resistance reached the highest value (2.6 MPa) for the ratio of 5 wt. % aluminum silicate waste, followed by a slight decrease to 2.2 MPa for the proportion of 10 wt. %. The manufacturing recipe used by the authors led to the formation of reinforcing crystalline phases (wollastonite and diopside).

Other experiments performed in the microwave oven by the researchers group in the Romanian company aimed the sintering and foaming of some industrial silicate waste containing important ratios of alumina by direct heating [11]. Unlike the soda-lime glass waste, the wastes used in experiments (building waste including components of the masonry (brick, concrete, cement in high proportion between 87.3-90.8 % and coal ash between 4-9 %) could be directly heated at very high rates (around 35 ºC/min) and foamed with 3.5-5.5 % SiC. The sintering temperature varied in the range 1168-1185 ºC. The products had porosity of 60.8-70 %, heat conductivity between 0.123-0.140 W/m·K, and compression resistance between 6.0-7.3 MPa, being adequate as a filler in various types of construction that require high mechanical strength. Despite the high process temperature, the specific electricity consumption had very low values (0.96-1.01 kWh/kg).

The paper [12] written by the same authors of the present work describes results of attempts carried out for producing cellular glass by sintering/foaming at above 950 ºC using the microwave warming method by direct wave-material contact of a mixture composed of residual commercial glass (36.5-53.0 %) and residual glass of halogen lamp bulb (35-50 %) (as an alkaline earth aluminum silicate glass) as basic materials, coal ash (10 %) captured in electric filters of thermal power plants, and SiC (2-2.5 %) as an expanding agent. The features of the product obtained in the best variant were: apparent specific gravity of 0.29 g/cm$^3$, heat conductivity of 0.072 W/m·K, and compression resistance of 2.4 MPa. These features are adequate for use as a heat insulation material in building. Due to the high energy efficiency of the electromagnetic wave warming technique by direct contact, the specific electricity consumption was between 0.88-1.01 kWh/kg.

Given these previous results, the research was focused on aluminum silicate waste as a basic material and commercial residual flat glass used in the manufacture of cellular aggregate, aiming to obtain high-strength porous products, the energy consumption being economical due to the direct microwave warming efficiency.

## 2. MATERIALS AND METHODS

### 2.1 Materials

The basic material included in the making procedure of the cellular aggregate was clay recycled from the residual clay brick of the building demolition. Due to its oxide composition containing 19.2 % alumina ($Al_2O_3$) and 60.6 % silica ($SiO_2$) shown in Table 1 [13], clay is a typical aluminum silicate material, perfectly adequate for the electromagnetic wave warming by direct wave-material contact as shown by previous tests. According to [14], the addition of clay as a basic material in the making procedure of cellular glass contributes to the improvement of its mechanical properties. The other compound of the basic material was clear residual flat glass recycled from building demolition. Its oxide composition is also shown in Table 1.

**Table 1.** Oxide components of basic material

| Basic material | Oxide components (wt. %) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | $SiO_2$ | $Al_2O_3$ | CaO | MgO | $Fe_2O_3$ | $Na_2O$ | $K_2O$ | $TiO_2$ |
| Residual clay brick | 60.6 | 19.2 | 2.6 | 2.9 | 8.1 | 1.2 | 3.9 | 1.3 |
| Clear residual flat glass | 71.1 | 1.3 | 9.3 | 3.9 | 0.2 | 14.2 | | - |

The residual clay brick was cleaned of the inherent adhesions of mortar and then broken into small lumps. Their grinding takes place in a ball mill and finally the granules with dimensions between 32-80 μm were selected following the sieving process.

The residual flat glass was washed for cleaning, and broken in a crusher. The grinding process was performed in a ball mill. The grain sizes lower than 80 μm were selected after sieving.

The materials preparing was carried out in Bilmetal Industries Company.

An important addition to the starting mixture was sodium borate with the formula $Na_2B_4O_7$ known as borax. Its theoretical composition includes 30.8 % $Na_2O$ and 69.2 % $B_2O_3$. Due to the high content of sodium oxide ($Na_2O$), which is one of the main fluxing materials, borax has the role of reducing the softening point of the silicate mixture subjected to the foaming process. On the other hand, the high content of pure boron (about 11 %) in the composition of borax contributes to improving the mechanical properties of expanded material [16, 17]. Borax was bought from the market with the granulation below 400 μm and the grinding operation in a laboratory electric device was required to reduce its granulation below 130 μm.

SiC as an expanding agent was commercially bought at a grain size below 10 μm. Its use in experimental tests was performed without further procedures.

### 2.2 Methods

In principle, the method of expansion a silicate material is based on incorporating an expanding agent into its powder mass and warming the material mixture so that its beginning the softening temperature to be below the warming temperature value. An adequate correlation between the softening point and the temperature range in which the expansion gas is released as a result of the chemical reaction should exist, allowing blocking the gas bubbles in the material volume and subsequently the formation of the specific structure [1].

The method of expanding the material by conventional heating follows the laws of thermodynamics and is well known. The method of direct microwave warming completely complies with other laws. The microwave is not a source of energy, but only a carrier of energy. By its contact with a susceptible material, the microwave power is converted into heat. The initiation of this process takes place in the center of the heated material, which becomes the hottest point of the material body and the heat transfer takes place from inside to outside, being in exactly the opposite direction compared to the conventional heating [18, 19].

The adopted expanding agent (SiC) oxidizes at temperatures of above 900 ºC [20] using air from the oven oxidizing atmosphere and releases $CO_2$ and CO (in the gaseous state) as well as $SiO_2$ and SiO (in the solid state, which enters into the composition of the molten material) through two possible chemical reactions. According to [1, 21], these reactions take place in the temperature range 950-1150 ºC.

The experimental equipment used in the test (Figure 1) was a domestic 800 W-electromagnetic wave oven, constructively adapted for operation up to 1200 ºC. The metal walls of the oven were not thermally insulated, instead the pressed powder material was highly thermally protected with thick layers of ceramic fiber mattress resistant to 1200 ºC. The material containing the wet mixture of basic material and expanding agent was previously introduced in a removable cylindrical metal mold and axially pressed at about 10 MPa, then it was removed and freely deposited into the oven on a metal plate supported by a metal support 15-20 mm above the layer of ceramic fiber mattresses at the bottom of the oven. The material warming during the experiment was monitored with a Pyrovar type pyrometer (measuring domain: 600-2000 ºC) fixed on a support at 350-400 mm above the oven, whose upper metal wall had a 30 mm-hole for viewing.

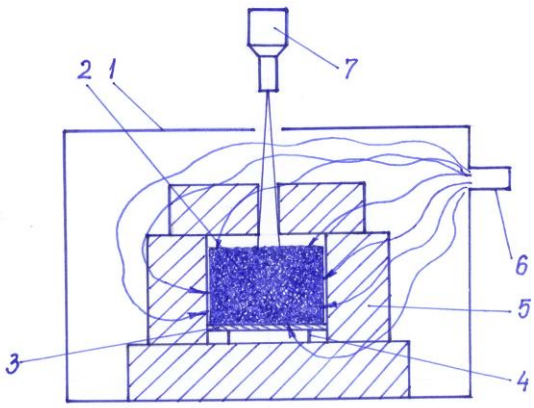



**Figure 1.** Composition and operation scheme of the test equipment
1 – 800 W-oven; 2 – pressed material; 3 – metal plate; 4 – metal support; 5 – thermal insulation; 6 – waveguide; 7 – pyrometer.

The determination of the features of cellular glass samples was carried out using common techniques of analysis. The apparent specific gravity was determined by the gravimetric method [22] and the porosity was measured and calculated by the comparing technique of the "true" (in compact state) density and apparent specific gravity [23]. The compression resistance was measured with a TA.XTplus Texture analyzer and the heat conductivity by the guarded-comparative-longitudinal heat flow method (ASTM E1225-04). The water absorption was based on the water immersion procedure (ASTM D570) and the samples microstructure configuration was investigated with a Smartphone Digital Microscope.

## 3. RESULTS AND DISCUSSION

### 3.1 Results

In order to experimentally manufacturing the high strength porous aggregate using residual clay brick and clear residual flat glass as basic materials, borax as a flux agent, and SiC as an expanding agent in the electromagnetic wave oven presented above, four experimental variants (shown in Table 2) were adopted.

The weight proportions of borax, SiC, and water addition were kept constant in all variants at the following values: 4.0; 2.0; and 15.0 wt. %, respectively. Residual clay brick (78.5-84.6 %) and residual flat glass (9.4-15.5 %) proportions were variable, the ratio between the two raw materials being in the range 5.06-9.

**Table 2.** Experimental variants tested by microwave irradiation

| Variant | Residual clay brick (wt. %) | Clear residual flat glass (wt. %) | Borax (wt. %) | SiC (wt. %) | Water addition (wt. %) |
|---|---|---|---|---|---|
| 1 | 84.6 | 9.4 | 4.0 | 2.0 | 15.0 |
| 2 | 83.0 | 11.0 | 4.0 | 2.0 | 15.0 |
| 3 | 80.8 | 13.2 | 4.0 | 2.0 | 15.0 |
| 4 | 78.5 | 15.5 | 4.0 | 2.0 | 15.0 |

The operation data of the warming process by microwave irradiation are presented centralized in Table 3.

**Table 3.** Operation data of the manufacturing procedure

| Variant | Dry basic material/ cellular glass amount (g) | Sintering/expanding temperature (ºC) | Warming time (min) | Medium rate (ºC/min) | | Index of volume increasing | Specific electricity consumption (kWh/kg) |
|---|---|---|---|---|---|---|---|
| | | | | Warming | Cooling | | |
| 1 | 450/438 | 1143 | 33 | 34.0 | 5.3 | 1.39 | 0.78 |
| 2 | 450/436 | 1119 | 30 | 36.6 | 5.2 | 1.45 | 0.72 |
| 3 | 450/437 | 1098 | 27 | 39.9 | 5.4 | 1.50 | 0.64 |
| 4 | 450/439 | 1080 | 25 | 42.4 | 5.1 | 1.68 | 0.59 |

In conformity with the data in Table 3, the dry raw material amount was kept constant (450 g) in all variants. Practically, the process operation data were influenced only by the ratio between residual clay brick and clear residual flat glass, which had the highest value (9) in the first variant and the lowest value (5.06) in variant 4, the other variants having intermediate values (7.54 and 6.12, respectively).

The influence of clay on the process temperature was obvious, the highest temperature (1143 ºC) corresponding to the maximum proportion of clay mixed with glass waste (variant 1). By decreasing the proportion of clay, the temperature of the sintering/foaming process is also reduced, reaching the minimum value (1080 ºC) in variant 4.

The nonconventional technique of direct electromagnetic wave warming of the mixture has proven an excellent energy efficiency, the warming rate reaching extreme values (34.0-42.4 ºC/min), well above the normal level of the conventional warming (10-15 ºC/min) commonly practiced in the industrial making the cellular glass. Also, the range of very economical values of specific electricity consumption (0.59-0.78 kWh/kg) is remarkable. Unlike similar experiments where the proportion of commercial residual glass ($Na_2CO_3$-CaO-$SiO_2$) is predominant compared to other silicate waste added to the mixture and the wave warming by direct contact is impossible without severe destruction in the glass-based material structure, if the predominant raw material is an aluminum silicate material (e.g. clay), the ultra-fast warming process does not affect its structure, according to the images in Figure 2, which shows the appearance of the samples in cross section.

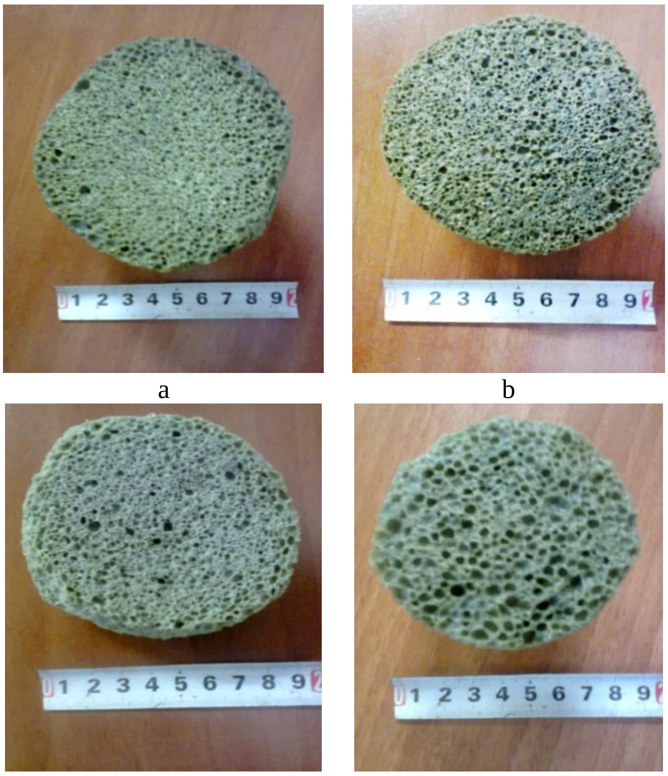



**Figure 2.** Appearance of cellular glass samples in cross section
a – variant 1; b – variant 2; c – variant 3; d – variant 4.

The images in Figure 2 indicate a dense structure with very low pore size in variant 1 (with the highest ratio of residual clay) and the progressive decrease of the dense property of the expanded material towards the samples manufactured in variants 2-4.

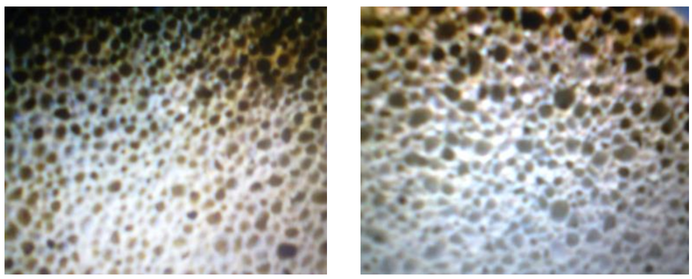
Applying the characterization methods of expanded products made in this experiment, the main physical, thermal, mechanical and microstructural features of aggregate were determined, being presented in Table 4.

**Table 4**. Main features of expanded products

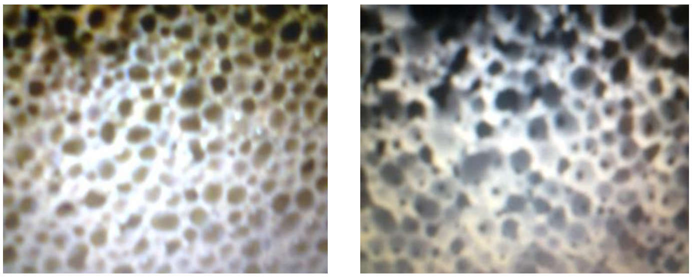
| Feature | Variant 1 | Variant 2 | Variant 3 | Variant 4 |
|---|---|---|---|---|
| Apparent specific gravity (g/cm$^3$) | 0.98 | 0.90 | 0.83 | 0.72 |
| Porosity (%) | 53.3 | 57.1 | 60.5 | 65.7 |
| Heat conductivity (W/m·K) | 0.197 | 0.189 | 0.174 | 0.161 |
| Compression resistance (MPa) | 8.5 | 7.8 | 7.2 | 6.4 |
| Water absorption (vol. %) | 7.1 | 6.6 | 6.5 | 6.0 |
| Pore size (mm) | 0.1-0.7 | 0.2-0.7 | 0.3-0.9 | 0.4-1.1 |

Examining the feature values of expanded materials it can concluded that the products made of wastes from building demolition (residual clay brick and clear residual flat glass) are porous dense materials with relative high apparent specific gravity (0.72-0.98 g/cm$^3$), but with acceptable heat conductivity (between 0.161-0.197 W/m·K) to have heat insulation properties. In the same time, the expanded product has high values of compression resistance (6.4-8.5 MPa) corresponding the requirement of the heat insulation materials usable in construction applications where the compression resistance is a main request. Water absorption between 6-7.1 vol. % represents usual values for a material containing high proportions of clay.

The microstructural peculiarity of expanded product samples is shown in Figure 3.



a b



c d ___ **2 mm**

**Figure 3.** Microstructural peculiarity of expanded product samples
a – variant 1; b – variant 2; c – variant 3; d – variant 4.

According to the pictures in Figure 3, the four expanded product samples manufactured by wave irradiation have good microstructural homogeneity, clearly non-affected by the very high microwave warming rates. Pore size has the following values: between 0.1-0.7 mm (variant 1), between 0.2-0.7 mm (variant 2), between 0.3-0.9 mm (variant 3), and between 0.4-1.1 mm (variant 4).

3.2 Discussion

Previous tests have shown that the direct microwave heating of a powder mixture, in which glass has a predominant proportion, severely affects the macro and microstructural homogeneity of the heated foaming material. This conclusion is valid in the case of the oven equipped with an 800 W-microwave generator and operating at the usual frequency of 2.45 GHz. The useful capacity of the oven allows the loading of a pressed powder material of maximum 600 g. The heating rate can exceed 40 ºC/min, by comparison with the conventional process of expanding the glass-based mixture for which the recommended rate is 10-15 ºC/min (in some cases even reaching 25 ºC/min), according to the literature [1, 21].

Experiments on the same microwave oven in Daily Sourcing & Research have shown that a mixture of aluminum silicate materials (masonry rubble and coal fly ash) reacts differently to microwave irradiation through direct microwave warming. These experiments have proven that the sintering/expanding process takes place normally without affecting the structure of the material, despite the very high warming rate [24]. This conclusion was the basis of the research whose results were presented above.

## 4. CONCLUSION

The objective of the paper was to produce a high-strength porous aggregate by direct microwave irradiation of a pressed mixture containing demolition residues (clay brick and clear flat glass), borax (4 %) as a flux agent and SiC (2 %) as an expanding agent. In this mixture the predominant weight proportion (78.5-84.6%) was that of clay brick waste, which is an aluminum silicate material. Previous experiments with direct microwave heating of this type of material have shown that the very high warming rate does not affect the integrity and homogeneity of its structure. Due to the high content of clay the sintering/expanding process temperature was very high (1080-1143 ºC), although borax had the role to reduce the softening point of mixture. The high warming rate (34-42.4 ºC/min) significantly decreased the process time (25-33 min) and the specific electricity consumption value (0.59-0.78 kWh/kg).

In terms of quality, the manufactured aggregate had excellent mechanical properties, its compression resistance being between 6.4-8.5 MPa. Being a dense material its apparent specific gravity was quite high (0.72-0.98 g/cm$^3$), but the heat conductivity had acceptable values for a material with heat insulation properties (0.161-0.197 W/m·K). The microstructural peculiarity of cellular aggregate samples has shown even repartition of pores (having low size), the microstructural homogeneity being a characteristic of all samples.

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