EXPANDING RESIDUAL CLEAR FLAT GLASS WITH COAL POWDER IN OXIDANT ATMOSPHERE OF THE OVEN USING WATER GLASS SOLUTION

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ABSTRACT: Porous material based on residual glass was non-conventionally made using anthracite as a pore-forming agent under the conditions of an oven with oxidizing atmosphere. Water glass as a protective agent for carbon particles avoiding their early oxidation and manganese dioxide (MnO_2) as an oxygen-supplier contributed to obtaining cellular glass with excellent physical, thermal, mechanical, and morphological features. The best specimen made by microwave heating at 790 °C using 1 % anthracite, 11.5 % water glass, 1.8 % MnO_2, and 5 % water allowed creating a product with excellent heat-insulating features (apparent density of 0.21 $g \cdot cm^{-3}$, heat conductivity of 0.039 $W \cdot m^{-1} \cdot K^{-1}$), and acceptable compressive resistance (2.3 MPa), the microstructural appearance being homogeneous with uniformly distributed pores with very low dimensions (0.1-0.4 mm). KEYWORDS: clear flat glass, anthracite, oxidant atmosphere oven, microwave, water glass, manganese dioxide.

1. INTRODUCTION

The current worldwide trend of manufacturing new types of construction materials qualitatively similar to that traditional, but environmentally friendly under conditions of low energy consumption, is well known. Initiated since the last decades of the last century, the technical solution of recycling residual materials (plastics, glasses, metals, paper and cardboard, etc.) for the manufacture of new materials with new added value had a beneficial contribution to cleaning the environment of huge deposits of waste, which is in a continuous growth rate as well as on saving the energy and the usual raw materials necessary for the manufacture of materials that consume large amounts of fossil fuels and emit greenhouse gases (CO_2) into the atmosphere [1].

This article is concentrated on recycling the residual glass and modern techniques for the preparation of materials with heat-insulating properties and good mechanical strength, that can be used especially in the construction sector. In principle, the main method of valorizing the bottle as fine particles consists in the inclusion of pore-forming agents, which release a gas following a chemical reaction developed at temperatures between 750-1150 °C. The thermally softened glass mass traps the gas into bubbles and the further increase in temperature and implicitly, the increase in pressure inside the gas bubble causes the expansion of the glass. Its subsequent cooling leads to forming spongelike matrix (cellular glass) simultaneously incorporating numerous exceptional properties (light weight, low heat conductivity, physical and chemical unaltered, compression strength, fire, water, vapors, corrosion, frost, the aggression of some external factors (rodents, insects, bacteria, etc.), non-toxicity, etc. These properties of the expanding glass are superior compared to those of the heat-insulating materials used in construction (expanded or extruded polystyrene) and are considered adequate for this purpose [2].

Already, the industrial production of porous bottle based on the use of high proportions of residual glass is working in several countries in Europe (Switzerland, Belgium, Austria, Germany, Czech Republic, Great Britain, Scandinavian countries, etc.), the United States, and China. The main type of recycled glass used for making the cellular bottle in industry is soda-lime-silica glass coming from post-consumer drinking bottle and flat bottle recovered from the building demolition, while the usual materials used as pore-forming agent are coal powder, carbon black, calcium carbonate, silicon carbide, and glycerin [3, 4].

Numerous research teams from all over the world are carrying out studies on an experimental-scale, testing new manufacturing techniques, new pore-supplying agents, different additives capable to increase the performance of cellular glass under conditions of economical and environmentally friendly energy consumption, the results being

disseminated through means of communication [5-11].

Conforming with data from the publisher work [12], the most frequently used pore-forming agent is carbon and carbonaceous products (coal powder, coke, graphite, carbon black, glycerin). As a result of the oxidation process, CO_2 and CO are released at relatively low temperatures (up to 800 °C) compared to the temperatures at which other agent types release gaseous compounds. The bond nature between carbon atoms influences the oxidation mechanism. In the range of relatively low temperatures (below 800 °C), the oxidation process is kinetic, the surface reaction speed between oxygen and carbon being limited.

The utilize of carbonaceous products in the bottle expanding process in the oxygen-rich medium of the furnace creates serious problems due to the tendency of carbon particles to oxidize at too low temperatures, at which the physico-chemical conditions for capturing the gas bubbles released from the reaction in the glass mass are not yet fulfilled. Currently, solving this problem is done using the neutral atmosphere in the oven by blowing argon or nitrogen. The method is expensive, implying, in addition to the use of the mentioned gases, the need to ensure a proper tightness of the working space. In order to delay the process of premature carbon oxidation, a method was found to introduce the aqueous solution of water glass (sodium silicate-Na_2SiO_3) into the starting mixture, which has the ability to envelop fine carbon particles, protecting them from contact with the oxidizing atmosphere. This method is currently applied in process applying glycerin ($C_3H_8O_3$) as a pore-forming material, associated with the liquid water glass for isolating carbon particles released at a relatively low temperature during the thermal decomposition of glycerin [6, 12].

Another method to avoid premature carbon oxidation was tested by Østergaard et al. [13] by adding sodium or potassium-based phosphates (Na_3PO_4/K_3PO_4) as froth stabilizers to the material mixture. The method was applied in the expanding process of CRT panel glass under the conditions of using coal powder as a pore-forming material and Mn_3O_4 as an oxygen-providing material. The process temperature decreased with the increase in the added alkaline phosphate content.

According to [14], manganese dioxide (MnO_2) is frequently used in association with other expanding agents (carbon, SiC, and TiN), being an excellent oxygen-supplier agent for intensifying the expansion process of glass. In the mentioned paper, MnO_2 (7 wt. %) was used as an expanding agent without the participation of another agent. Cathode-ray-tube (CRT) panel glass represented the residual glass as raw material. According to the measurements, the MnO_2 decomposition starts below 500 °C increasing in intensity up to 590 °C, while the Mn_2O_3 decomposition starts at 650 °C increasing in intensity up to about 800 °C. Practically, during the Mn_2O_3 decomposition, the process of glass expansion takes place, the pores tending to become open. The cellular glass had the denseness in the range of 0.25-0.59 $g \cdot cm^{-3}$, porousness reached 79-92 %, as well as the thermal conductivity varied within the limits of 0.053-0.066 $W \cdot m^{-1} \cdot K^{-1}$. It was found that the porousness decreases with the increase of temperature and time. The cell dimension had values within the limits of 0.02-4 mm. The heating process took place in a conventional oven.

Kőnig et al. [15] used 1 wt. % charcoal (lightweight type of carbon black residue) with the grain size in the range of 15-27 μm and MnO_2 (7.2 wt. %) as an oxygen-providing material for making cellular glass from CRT panel glass. The optimal sintering/expanding temperature was 800 °C and the foamed product had excellent thermal insulation properties (denseness of 0.13 $g \cdot cm^{-3}$ and heat conductivity of 0.042 $W \cdot m^{-1} \cdot K^{-1}$).

The effect of Sb_2O_3 content on the resistance abilities of boron-silicate cellular bottles sintered at relatively small temperature (775 °C) was investigated by Zhai et al. [16]. Experimental results showed that expanding with carbon black (1 wt. %) and 0.9 wt. % Sb_2O_3 added to the mixture led to obtaining much more homogeneous microstructure, high pore size, lower bulk density, low water-absorbing, and satisfactory compression resistance.

Heating methods used in all experiments noted above were conventional. Several experiments presented below were performed by Daily Sourcing & Research SRL and Cosfel Actual SRL companies (Romania) using own microwave heating equipment. In the paper [17], a building material obtained by expanding the residual glass (post-consumer drinking glass) was made with anthracite (0.9 wt. %) using the effect of microwave irradiation. The addition to the starting mixture of 5.7 wt. % disodium phosphate (Na_2HPO_4) and water-additing (12 wt. %), as well as their sintering at 810 °C with high heating rate led to obtaining porous product with denseness of 0.27 $g \cdot cm^{-3}$, heat conductivity of 0.053 $W \cdot m^{-1} \cdot K^{-1}$, compression resistance of 2.7 MPa and cell dimension in the range of 0.1-0.5 mm. The electricity consumption was very little (0.66-0.75 kWh/kg).

In another work [18], Paunescu et al. used carbon black (1 wt. %), Na_2HPO_4 (5.9 wt. %), Sb_2O_3 (0.8 wt.%), and water-additing (10 wt. %) for expanding boro-silicate residual bottle in an adapted microwave oven. The optimal process temperature was 790 °C and the average heating rate reached 24.8 °C/min. Characteristics of the best specimen were: denseness of 0.34 $g \cdot cm^{-3}$, porousness of 84.5 %, heat conductivity of 0.060 $W \cdot m^{-1} \cdot K^{-1}$, compression resistance of 2.2 MPa, cell dimension within the limits of 0.4-0.7 mm, and electricity consumption of 0.68 kWh/kg.

This paper aimed at the testing production of porous bottle from residual clear flat glass adopting a pore-forming agent from the carbonaceous material category (anthracite powder), an aqueous solution of water glass (sodium silicate) for covering the surface of carbon grains to avoid its early-oxidation, and MnO_2 as an oxygen-providing agent. The process took place in a microwave oven adapted to operate at high temperature under the conditions of oxidizing atmosphere, being original considering that, except for previous experiments of authors of this work, no other similar technique for manufacturing cellular glass with carbonic expanding agent did not use microwave heating in oxidizing atmosphere.

2. METHODS AND MATERIALS

2.1 Methods

The expanding technique of bottle in the oxidizing atmosphere of the experimental equipment, in which anthracite is used as a carbonic agent, water glass as a protective agent for enveloping carbon particles, and manganese oxides have different roles as an oxygen supplier and expanding agent, includes several specific peculiarities.

The use of manganese oxides under the conditions of heating in oxidizing atmosphere of the oven and the use of carbonic expanding agents are important particularities of the process. The reduction of manganese oxides takes place successively in different periods of the glass expansion process starting at temperatures above 450 °C.

The reduction of manganese oxides begins with the transformation of MnO_2 into Mn_2O_3 and Mn_2O_3 into Mn_3O_4 at temperatures above 450 °C, then these two phases are reduced by each carbon or carbon monoxide in the Mn-C-O system [19].

$$2MnO_2 = Mn_2O_3 + 0.5O_2 \quad (1)$$

$$3Mn_2O_3 = 2Mn_3O_4 + 0.5O_2 \quad (2)$$

$$Mn_3O_4 = 3MnO + 0.5O_2 \quad (3)$$

Reaction (1) that takes place under the sintering temperature range exhibits an unwanted impact on the expansion process, because carbon burns with oxygen in the oven atmosphere. The product of reaction (2) serves as an expanding agent in the oxygen-rich medium. Mn_3O_4 can become an oxygen-providing material (reaction 3) in an atmosphere without oxygen favouring the carbon oxidation during the expansion process [20].

The role of water glass solution (sodium silicate) was mentioned above, protecting the carbon particles from premature and ineffective oxidation for the process.

The basic method applied in the making process of foam bottle with carbonic expanding agent in the oxidizing atmosphere of the oven was the use of electromagnetic wave heating (as a non-conventional method) in the authors' team own version of predominantly direct heating combined with partially indirect heating [21].

Under the conditions that electromagnetic waves were discovered at the middle of the 20th century and their application was successfully carried out since that period in communications and radars, until now, the special ability of this wave type in high temperature heating processes has not been exploited on industrial scale. Microwave applications are known in various industrial treatments of drying, tempering, defrosting, thermal processes at low and moderate level in the fields of medical waste, rubber vulcanization, treatment of sewage sludges, chemical residues, ceramics, polymers, composites, etc. [22-25].

As mentioned above, recently, Daily Sourcing & Research and Cosfel Actual companies have designed and experimentally used microwave heating equipment for different types of silicate waste [18, 21]. Considering that it was found that the recycled commercial bottle powder (soda-lime-silica bottle) is not adequate for directly microwave heating, the technical solution of using a crucible or cylindrical tube of highly electromagnetic wave absorption material procured from China was chosen for protecting the heated material avoiding the negative effect at the structural level of the direct heating under the microwave irradiation. The intensity of this field was practically sufficiently reduced, the waves completely penetrating through the thin wall (2.5 mm) of the crucible (tube) and coming into direct contact with the material. Thus, the fast and efficient heating is performed without affecting the quality of the material structure.

The components and operational principle of the experimental microwave plant is presented in Figure 1. Several aspects on characteristics and peculiarities of the 0.8 kW-electromagnetic wave equipment were detailed in other previous papers [26, 27] published by authors of the current work.

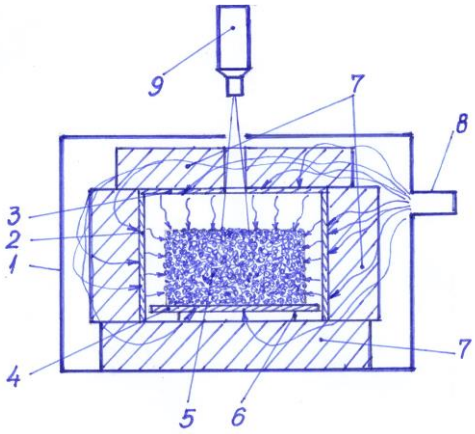

Figure 1. Composition and operational principle of the plant
1 – oven; 2 – tube; 3 – lid; 4 – metallic plate; 5 – material for heating; 6 – support; 7 – thermal protection; 8 – waveguide; 9 – pyrometer.

2.2 Materials

Material components adopted for this experiment were: residual clear flat bottle as raw material, anthracite powder as a pore-forming material, water glass (sodium silicate) as an covering material for protecting carbon grains, and manganese dioxide (MnO_2) as an oxygen-providing material.

The residual flat bottle was recycled from window shards. The waste was washed, dried, crushed, ground in a ball mill, and sieved, the grain size under 90 µm being selected. The chemical composition of clear flat bottle was as follows: 72.8 % SiO_2, 9.0 % CaO, 3.8 % MgO, 14.1 % Na_2O, 0.03 % K_2O, 0.15 % Al_2O_3, 0.1 % Fe_2O_3, and 0.02 % TiO_2.

Anthracite with the chemical formula $C_{15}H_{11}O^-$ [28] is a hard coal containing about 94 % carbon. Anthracite was chosen as a pore-making material in the starting mixture of the experiment, although its use in the oxygen-rich atmosphere of the plant would normally negatively influence the expansion process of the bottle powder. The coal was crushed, ground in the ball mill, and sieved. The grain size of the fine powder was chosen below 50 µm.

The water glass content influences the expansion process of the bottle-based mixture, increasing the optimal temperature for forming the cellular matrix along with the increase of the water glass proportion. On the other hand, the crystallization during the expanding process affects this process by weakening it and also favouring the formation of open pores [29]. Water glass (sodium silicate) is a compound including Na_2O and SiO_2 that forms a glassy solid soluble in water. In this experiment, water glass was used as a 40 % commercially available aqueous solution, having binding and adhesive abilities.

Manganese dioxide (MnO_2) was used into the mixture as an oxygen-providing material, being purchased from the market (95 % concentration) at a grain size less than 50 µm.

Three manufacturing recipes (R1-R3) were chosen in this work (Table 1) to investigate the influence of different components of the wet mixture on physical, mechanical, thermal, and morphological features of achieved cellular products.

Table 1. Composition of wet manufacturing recipes

Composition	**Recipe (wt. %)**		
	R1	R2	R3
Clear flat glass	87.7	85.8	83.8
Anthracite	1.0	1.0	1.0
MnO_2	1.8	2.2	3.0
Water glass	11.5	15.0	18.5
Dilution water	5.0	7.0	9.0

2.3 Method for characteristic investigation of specimens

Conventional methods were chosen for examining the characteristics of cellular glasses fabricated in this experiment. Archimedes' method (ASTM D792-20) was adopted for measuring apparent density and porosity according to the paper [30] recommendation. For determining heat conductivity values the heat-flow method (ASTM E1225-04) [31] was used. The compressive resistance could be evaluated with the TA.XTplus C Texture analyzer with the rate range of 0.01-40 mm/s. The method of immersion under water of samples was the procedure by which the absorbing-water capacity of specimens was determined (ASTM D570). Microstructural features of cellular glass samples were investigated using ASONA 100X Zoom Smartphone Microscope.

3. RESULTS AND DISCUSSION

3. Results

Operational parameters of cellular glass production in the technical conditions mentioned above were determined during the process and are presented in Table 2. The wet quantity of basic material separately prepared in the three versions of the manufacturing recipe was kept at a constant value (500 g).

Table 2. Operational parameters of the making process

Parameter	Recipe		
	R1	R2	R3
Wet basic material/cellular bottle quantity (g)	500/ 410	500/ 412	500/ 411
Sintering/expanding temperature (°C)	790	793	800
Heating time (min)	26	27	30
Heating rate (°C/min)	29.6	28.6	26.0
Cooling rate (°C/min)	5.4	5.5	5.4
Specific electricity consumption (kWh/kg)	0.66	0.68	0.76

The analysis of the data in Table 2 indicates, on the one hand, the undesirable influence of increasing the water glass content above a certain limit (in the case of this experiment, above 15 wt. %), slowing down the expansion process of residual glass with anthracite. The required temperature of this process tends to increase more sharply, reaching 800 °C in the case of the R3-manufacturing recipe containing 18.5 % water glass. On the other hand, the data presented in Table 2 reveals the special effect in energy terms of applying the preponderantly direct electromagnetic wave heating method compared to conventional heating methods. The heating rate reached an extremely high value (29.6 °C/min) in the case of the R1 recipe, but the other values (28.6 and 26.0 °C/min, respectively) are also very high, allowing the reduction of heating time to 26-30 min. As a result, the value level of the specific electricity consumption was very reduced, in particularly in the case of R1 and R2 (0.66 and 0.68 kWh/kg, respectively). To avoid the formation of structural cracks due to internal stresses, the cooling rates were kept within the limits proposed in international works [2], around 5.5 °C/min.

Results of investigating the important features of cellular bottle specimens are presented in Table 3.

Table 3. Characteristics of cellular bottle specimens

Manufacturing recipe	Denseness ($g \cdot cm^{-3}$)	Porousness (%)	Thermal conductivity ($W \cdot m^{-1} \cdot K^{-1}$)	Compressive resistance (MPa)	Absorbing-water (vol. %)	Pore size (mm)
R1	0.21	90.0	0.039	2.3	1.8	0.1-0.4
R2	0.24	88.6	0.046	2.0	1.6	0.2-0.5
R3	0.37	82.4	0.067	1.6	1.3	0.4-0.9

The aspect of cellular bottle samples produced in oxygen-rich atmosphere of the oven from recycled clear flat bottle with anthracite as a pore-forming agent, water glass as a covering material for the carbon grains, and MnO_2 as an oxygen-providing material is shown in Figure 2, while the microstructural features of these specimens are presented in Figure 3.

a

b

c

Figure 2. Aspect of cellular bottle specimens corresponding to the three manufacturing recipes
a – R1; b – R2; c – R3.

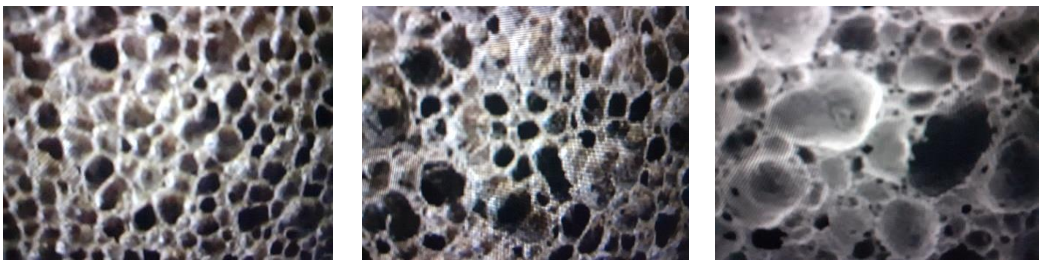

Figure 3. Microstructural features of cellular bottle samples
a – R1; b – R2; c – R3.

Concording with Table 3, cellular bottle specimens have the typical characteristics of porous products with excellent thermal insulating abilities (reduced denseness, little thermal conductivity, and high porousness). Manufacturing with a carbonic expanding agent (anthracite) in the oxygen-rich medium of the plant constituted a special challenge due to the tendency of carbon particles to prematurely oxidize, long before reaching the required thermal parameters (temperature and viscosity) of the bottle dust. The use of water glass and MnO_2 proved to be viable in this experiment, contributing to ensuring the required quality of cellular products. Among the three tested manufacturing recipes, R1 and R2 showed excellent characteristics, while R3 exhibited some inadequate features. The microstructural peculiarities observed in Figure 3 were decisive, indicating exceeding the optimal content of water glass in the material mixture. Thus, the sample made with the R3 recipe, in which the water glass content reached 18.5 %, had an inhomogeneous microstructure with larger pores compared to the other specimens and, in addition, with tendency of neighbouring cells to intercommunicate through their common wall. Usually, this type of microstructure decreases the material apparent density, but at the same time it also decreases its compression resistance.

3.2 Discussion

Coal and carbonaceous products are the most frequently used materials as pore-forming agents [2] in the manufacturing processes of foamed bottle from waste bottle. The heat process takes place at low temperature (around 800 °C). However, there is a major impediment caused by the early onset of carbon oxidation (below 500 °C). If the oven in which the glass expanding process takes place has an oxidizing atmosphere, the carbon can burn through oxidation using the oxygen in the oven atmosphere. That is why the use of carbon is associated with ensuring an inert atmosphere in the oven by blowing nitrogen or argon. Creating the inert atmosphere into the oven involves additional production costs. Recently, the involvement of water glass proved to be a viable solution due to its ability in liquid state to protect carbon particles, preventing them from oxidizing with the oxygen in the oxidizing atmosphere. In particular, in case of utilizing glycerin as a carbonic liquid foaming material, water glass is introduced into the mixture.

The coal-water glass-MnO_2 combination used in the oven with oxidizing atmosphere has not been practically used until now, being one of originality elements of this paper.

Applying the electromagnetic waves in residual glass expansion processes was done by the authors' article in numerous other works. The tests were performed in all cases in microwave ovens with oxidizing atmosphere, but in general, the expanding agent was not of carbonic origin, except for glycerin in association with water glass. A very recent work was published in 2023 [17] including tests in which the mixture was composed of post-consumer drinking glass, anthracite (0.9-1.1 %) as a pore-forming material, Na_2HPO_4 (5.7-6.3 %) as a froth stabilizer material to prevent early oxidation of carbon, and water (12 %). The best version had excellent properties (denseness of 0.27 $g \cdot cm^{-3}$, heat conductivity of 0.053 $W \cdot m^{-1} \cdot K^{-1}$, compression resistance of 2.7 MPa, and cell size within the limits of 0.1-0.5 mm). Manufacturing recipes adopted in the current paper differed from the previous works carried out in the microwave field, being tested for the first time.

4. CONCLUSION

The original and effective solution to difficulties related to the glass expansion with a carbonic agent (anthracite) in oxidizing atmosphere of the oven was applied in this work. The use of water glass (11.5 wt. %) and MnO_2 (1.8 wt. %) protected the carbon particles from early oxidation and then provided the oxygen necessary for its oxidation. Non-conventional microwave heating in the preponderantly direct and partly indirect version was also another element of originality. Thermal insulation properties of cellular glass (denseness of 0.21 $g \cdot cm^{-3}$, heat conductivity of 0.039 $W \cdot m^{-1} \cdot K^{-1}$,

and porousness of 90.0 %) were excellent and the compression strength reached an acceptable level (2.3 MPa). The manufacturing process of cellular glass had a remarkable energy effectiveness (specific electricity consumption of 0.66 kWh/kg).

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