SEPARAREA ȘI RECICLAREA DEȘEURILOR DE STICLĂ DIN TUBURI CATODICE (CRT) PRIN SPUMAREA ÎN CÂMPUL DE MICROUNDE SEPARATION AND RECYCLING OF CATHODE RAY TUBE (CRT) GLASS WASTE BY FOAMING IN MICROWAVE FIELD

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The paper presents experimental results obtained in the manufacturing process of a glass foam obtained from the recycling of glass from cathode ray tubes (CRT). The glass foam samples were performed experimentally by the sintering-foaming process. The heating technique was unconventional, the energy provided by the microwave was used as a heat source. The process is different from the conventional techniques applied both in the manufacture of industrial glass foam and in the numerous small-scale experiments known in the literature. The glass foam samples obtained were characterized by traditional methods of analysis. The main characteristics were bulk density, porosity, thermal conductivity, compressive strength, water absorption and microstructural configuration of the samples. The experimental results proved that the applied process obtained a glass foam with good characteristics (porosity of 90%, thermal conductivity of 0.042 W / m^{-1} • K^{-1} and compressive strength of 2.1 MPa) usable in construction.

Keywords: separation, recycling, glass waste, microwave field.

1. Introduction

The rapid change of the manufacture technology of the TV receivers of the last decades has made available numerous conventional devices that have been replaced with Liquid Crystal Displays and Plasma Display Panels [1]. The available devices constitute, on the one hand, a hazardous waste being included in the category of waste electrical and electronic equipment [2] and, on the other hand, a reserve of valuable materials that should be recycled. The main hazard source of this waste is the high content of lead oxide (PbO). It is especially incorporated in the funnel glass of cathode-ray tube (CRT) in the ratio range of 16-25 wt.%. The neck, which together with the funnel constitutes the back part of the CRT, is a lead-rich silicate glass containing up to 40 wt.% lead oxides. The PbO content of the pannel glass is much lower (below 1 wt.%). The three component parts of CRT represent very different weight ratios: 66 wt.% the panel, 33 wt.% the funnel and only 1 wt.% the neck,

Lucrarea prezintă rezultate experimentale obținute în procesul de fabricație a unei spume de sticlă obținută din reciclarea sticlei de la tuburi cu raze catodice(CRT).

Probele de spumă de sticlă au fost realizate experimental prin procesul de sinterizare-spumare. Tehnica de încălzire a fost neconvențională, a fost utilizată ca sursă de căldură energia furnizată de microunde. Procedeul este diferit de tehnicile convenționale aplicate atât în fabricarea spumei de sticlă industriale, cât și în numeroasele experimente la scară mică cunoscute din literatura de specialitate.

Probele de spumă de sticlă obținute au fost caracterizate prin metode tradiționale de analiză. Principalele caracteristici au fost: densitatea aparentă, porozitatea, conductivitatea termică, rezistența la compresiune, absorbția apei și configurația microstructurală a probelor.

Rezultatele experimentale au dovedit că prin procedeul aplicat s-a obținut o spumă de sticlă cu bune caracteristici (porozitate de 90%, conductivitate termică de 0,042 W/m⁻¹ • K⁻¹ și rezistență la compresiune de 2,1 MPa) utilizabile în construcții.

that it means that the real proportion of PbO in the CRT composition is about 7.6 wt.% [3]. According to [2], the average amount of PbO of a cathode ray tube glass is between 1-1.5 kg. The same bibliographic source estimated in 2012 that 2.4 million tons of CRT will still exist in Europe in 2020 in households and companies' offices.

According to [4], the recycling of CRT glass waste involves initial operations to eliminate fluorescent powders as well as metal masks and implosion band. Then, it is necessary to crush the waste followed by sorting by sieving and handpicking. It is assumed that after these waste processing operations will remain about 55% of the initial quantity of the waste and the lead content will be below 2000 ppm.

This recycled material can be used as a raw material for the manufacture of glass foams, floor tiles, bricks, or concrete.

In the world, the concern of the researchers for the valorization of the cathode-ray tubes glass waste is manifested, especially by the manufacture

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of cellular glass with insulating properties. Both the funnel glass and the panel glass are used as raw material after the processing operations mentioned above.

What differentiates, firstly, the technical solutions adopted is the nature of the foaming agent and of the mineral additives used in the manufacturing processes.

A distinct category of foaming agents are carbonates.

According to [5], the finely ground sodium carbonate (below 63 μ m) was used in weight ratios between 6-14% in the temperature range 750-850°C. The glass waste was ground and sieved to grain size below 63 μ m. The best samples in form of pellets were obtained at 750 °C, the foaming agent ratio being 14 wt.%. The apparent density of glass foam was 0.28 g/cm³.

The work [6] mentions a manufacturing technique of cellular glass from CRT glass waste adopting calcium carbonate as a foaming agent. The sintering-foaming process occurred at 725 °C for 15-30 min.

According to [7,8], a cellular glass manufacturing recipe in the form of pellets from CRT glass waste was composed, excluding the glass waste, of dolomite and calcium carbonate as foaming agents, Na-bentonite and Ca-bentonite as pelletizing agents and a small addition of alumina to avoid aggregation of the molten particles.

Silicon carbide (SiC) is a foaming agent commonly used in cellular glass manufacturing processes [9]. The paper [10] describes a foaming process of the CRT glass waste using 4wt.% SiC with granulation below 40 μ m, cobalt tetraoxides (Co₃O₄) in ratios between 0.4-1.2 wt.% as an oxygen supply agent to intensify the foaming process as well as water and polyvinyl alcohol as binders.

SiC was also used as a foaming agent in the paper [11] together with sodium borate (borax) as a fluxing agent and titanium dioxide (TiO₂) as a stabilizing agent. The CRT glass waste (56 wt.%) was mixed with germanium tailing (40 wt.%). The mixture ratio of SiC was 1 wt.%.

Other bibliographic sources [12,13] refer to the use of SiC or TiN (titanium nitride) as alternative reducing agents in the process of manufacturing glass foam from CRT glass waste. The two agents can react at high temperature with PbO from waste by releasing gases (CO_2 or N_2) that participate in the process of foaming.

Another experimental process for the manufacture of cellular glass from CRT glass waste is described in [14]. The glass waste was mixed with 1 wt.% borax, 1 wt.% TiO₂ and successively 1-2.5 wt.% antimony trioxide (Sb₂O₃).

In recent years, a team of researchers from the Romanian company Daily Sourcing & Research has developed an experimental program for testing the manufacture of glass foam from glass waste in the microwave field.

The results were satisfactory, confirming that products like those manufactured by conventional techniques can be obtained and in addition, the specific energy consumption is very economical.

The current paper refers to experiments performed in the field of glass foam manufacture from CRT panel glass waste using the unconventional microwave heating technique. The aim of the research was to obtain a cellular glass with structural homogeneity and acceptable mechanical strength for use as an insulating material in construction, under the conditions of a very low energy consumption.

2. Methods and materials

2.1 Methods

The basic principle of the foaming process of glass waste is the incorporation in the powder raw material of a foaming agent capable to release a gas through a chemical reaction at high temperature (most often a decomposition or an oxidation). The softening point of the raw material powder mixture must be correlated with the temperature at which the gas is released, so that it meets a material with an optimum viscosity where it is blocked as bubbles and then by cooling it forms a homogeneous porous structure that characterizes a foam glass. The foaming agent as well as the mineral additions that accompany it are adopted according to the characteristics of the glass waste. Generally, there is a great diversity of foaming agents and additives, not even the manufacturing recipes used in industrial production having no standard materials for obtaining the same type of glass foam [6].

Theoretically, a glass waste would not be suitable for foaming under the influence of microwave radiation due to the very high content of silica which is a microwave transparent material at low temperatures (below 500 °C), becoming highly susceptible only after this temperature is reached [18]. This theory published in 1997 has decisively influenced the future attitude of industrial producers and research teams on the possible use of microwave energy in the glass industry [6,19]. Practically, it has been proven that certain contaminants in the glass composition (e.g. Fe₂O₃, Cr₂O₃, etc.) or additives in the used powder mixture (e.g. SiC, binders, etc.), even in very small proportions, have an opposing influence allowing an efficient microwave heating starting at room temperature [19,20].

The main dielectric properties of the glass (electrical conductivity, dielectric loss factor) are largely temperature dependent. As the temperature increases, the capacity of the microwave transparent materials improves rapidly [21 - 24]. Unlike the conventional heating, in the case of using the microwave energy the heating initiation occurs in the core of material, so that it will generate itself the heat. The heating is thus more volumetric and can be very fast and selective.

The microwave heating eliminates the need to consume energy for heating the walls and the vault of furnace or other its massive components, significantly reducing the energy consumption [22 - 25]. It should be mentioned that there are some peculiarities of the powder mixtures used in the manufacture of cellular glass which favour the of Thus. absorption microwaves. high concentrations of alkali metal oxides (especially Na₂O and K₂O) in these mixtures allow efficient heating in the microwave field due to the correlation between the high electrical conductivity of the material and the absorption of microwaves [23].

The CRT panel glass waste used as raw material contains high proportions of Na₂O and K₂O. Also, the use of borax as a fluxing agent contributes by its Na₂O content to increase the efficiency of microwave heating [26].

Previous tests performed by the authors showed that, excepting the aluminosilicate wastes that were subjected without problems to the direct microwave heating, the other tested types of glass waste could not be exposed to the direct microwave irradiation without their internal structure being severely damaged [20]. Thus, it was concluded that the glass-based material should be protected with a circular screen of a high microwave susceptible material based on SiC.

Depending on the thickness of the screen, it was possible to obtain a significant decrease of the effect of the direct contact between the microwave field and the glass, part of the microwave radiation being absorbed by this ceramic screen. The optimum range of 3.5-5 mm ceramic screen thickness was experimentally determined. Thus, a mixed heating (partially direct and partially indirect) was the solution adopted in numerous experiments carried out in the last three years which proved to be efficient.

The equipment used for experiments was a 0.8 kW-microwave oven commonly used in household operating with electromagnetic waves at a frequency of 2.45 Hz corresponding to a wavelength of 12.2 cm and an energy of $1.02 \cdot 10^{-5}$ eV [26,27]. The oven was adapted for operation at high temperature (up to 1200 °C).

The constructive scheme of the equipment in Fig. 1[16] shows the positioning of the pressed material and the other components in the microwave oven.

2.2 Materials

The materials used in experiments were panel glass CRT waste, calcium carbonate, sodium borate (borax) and antimony trioxide mixed as a fine powder, the binder used for the mixture pressing being the water. The glass waste comes from recycling the glass panel of the cathode-ray tube of the TV receivers. It was crushed, ground in a ball mill and sieved below 32 μ m, and compacted with a mini press at 2 atm.

The wet powder material was previously pressed with a GEKO type hydraulic press, in a cylindrical metal mold with a removable wall and then released to a diameter of approx. 65 mm and a height of approx. 50 mm.

The initial volume of each sample was calculated.

The volume increase index was determined by dividing the volume value of the foamed sample by the initial value of the raw sample volume.

The chemical composition (wt.%) of this waste contains 61.04% SiO₂; 2.37% Al₂O₃; 7.74% Na₂O; 7.11% K₂O; 0.32% MgO; 0.84% CaO; 7.82% SrO; 9.66% BaO; 1.52% ZrO₂; 0.34% TiO₂; 0.18% Fe₂O₃; 0.20% ZnO; 0.23% PbO and 0.43% Sb₂O₃ [15].



Fig. 1 - Experimental microwave equipment/ Cuptor experimental cu microunde[16]

a – overall image of the microwave equipment; b – constructive scheme of the equipment: 1 –microwave oven; 2 – ceramic tube; 3 – ceramic lid; 4 – metal plate; 5 – pressed material; 6 – metal support; 7 – ceramic fiber mattress; 8 – waveguide; 9 – radiation pyrometer.
 a - imaginea de ansamblu a echipamentului cu microunde; b - schema constructivă a echipamentului; 1 - cuptor cu microunde; 2 - tub ceramic; 3 - capac ceramic; 4 - placă metalică; 5 - material presat; 6 - suport metalic; 7 - saltea din fibră ceramică; 8 - ghid de undă; 9 - pirometru radiații.

Та	ble	1

Variant	CRT panel glass	Calcium carbonate	Sodium borate	Antimony trioxide	Water addition	
	waste wt.%	wt.%	(borax) wt.%	wt.%	wt.%	
1	89.0		4.5	1.7		
2	89.3	4.8	4.1	1.8	8.0	
3	89.6		3.7	1.9		
4	89.9		3.3	2.0]	

Composition of the studied samples /Compozitia probelor studiate

Calcium carbonate used as a foaming agent had a grain size below 6.3 µm. The borax purchased from the market with a grain size < 400 µm was ground in the ball mill at the grain dimension below 63 µm. It was used in experiments as a fluxing agent due to the rich content in Na₂O.

The antimony trioxide was used at a very fine granulation (below 10 µm) due to its significant influence to reducing the glass foam density [14]. As a binder water was used.

The recipe for the manufacture of glass foam from glass waste from the CRT panel (in four variants presented in Table 1) was adopted in accordance with the information in the literature and based on the authors' own experience.

Excepting the glass-based raw material (between 89.0-89.9 wt.%), they are used calcium carbonate as a foaming agent whose proportion was kept constant at 4.8 wt.%, borax with ratios between 3.5-4.5 wt.%, antimony trioxide used between 1.7-2.0 wt.% and a supplementary amount of water as a binder in a constant ratio of 8 wt.%.

The pressed powder material was deposited freely on a metal plate placed on a bed composed of ceramic fiber mattresses. The material was protected by a ceramic tube made of a mixture of SiC and Si₃N₄ (80/20) with a diameter of 125 mm, a wall thickness of 5 mm and a height of 100 mm, which was placed on the same ceramic bed at the base of the oven. A lid of the same material as the tube was placed above.

Considering the way in which the heating of the material is initiated (in its core) and the fact that the heat propagates from the inside to the peripheral areas, a very great importance of the thermal protection with the ceramic fiber mattresses of the outer surface of the tube and the lid was given. The microwave field was distributed by a single waveguide placed on one of the side walls of the oven.

The temperature control of the material during the heating process was carried out with a radiation pyrometer placed above the oven, the visualization of the material being done through holes provided in the upper metal wall of the oven, the ceramic lid and the ceramic fiber mattresses deposited on the lid, on the axis of viewing the pyrometer.

The electricity consumption related to the microwave heating of each glass foam sample in the experiments was metered. By dividing to the amount of glass foam produced, the specific energy consumption was calculated.

2.3 Characterization of the glass foam samples

The glass foam samples experimentally made by the sintering-foaming process of the CRT glass waste were characterized by traditional analysis methods. The main features were apparent density, porosity, thermal conductivity, compressive strength, water absorption and microstructural configuration of the samples.

The apparent density was measured by the gravimetric method [23].

The porosity was calculated by the comparison method [24] between the porous sample density and the density of the same material type in compact state (melted and then cooled to room temperature).

The determining method of the thermal conductivity [25] was the measurement of the thermal flow that crosses a sample of standard dimensions (50 mm-thickness) placed between two metal plates, one heated and protected with insulating material and the other cooled.

The compressive strength was determined using a TA.XTplus Texture Analyzer (ASTM C552-17) and the thermal conductivity was measured by the heat-flow meter method (ASTM E1225-04) similar to the transient hot line method TC3000 of XIATECH Chinese technique [19]. The water absorption was determined by the water immersion for 24 hours method (ASTM D570). An ASONA 100X Zoom Smartphone Digital Microscope was used to investigate the microstructural configuration of the glass foam samples.

An own device was used to determine the compressive strength by developing an axial pressing force generated with a hydraulically operated piston.

The last pressing force axially applied to the sample before to crack was considered the compressive strength value.

The tested sample had a cylindrical shape with the diameter of 80 mm and the height of 70 mm. The water absorption of the glass foam sample was measured by the traditional method of its water immersion (ASTM D 570). The microstructure of the foam glass samples was observed with a Smartphone Digital Microscope, that allowed the determination of the pore size(using the digital microscope scale) and the homogeneity of their distribution.

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Finicipali parametri funcționali al procesului de fabricație a spumei de sucia									
Variant	Dry raw	Sintering-	Heating	Average rat	e, °C/min	Index of	Specific		
	material/ cellular glass amount g	foaming temperature	time	Heating	Cooling	volume growth	energy consumption		
	Ŭ	°C	min				kWh/kg		
1	508/493	780	30.0	25.3	6.8	1.55	0.71		
2	508/496	786	30,5	25.1	6.4	1.65	0.72		
3	508/494	793	32.5	23.8	6.7	1.70	0.77		
4	508/496	800	34.5	22.6	6.6	1.80	0.81		

Main functional parameters of the manufacturing process of glass foam

Table 2



Fig. 2 - Dependence on apparent density on sample porosity /Dependența densității aparente de porozitatea probei.



Fig. 3 - Dependence of thermal conductivity on sample porosity /Dependenta conductivității termice de porozitatea probei.



Fig. 4 - Dependence of water absorption on sample porosity /Dependența absorbției apei de porozitatea probei.

3. Results and discussion

The main functional parameters of the manufacturing process of glass foam and the physical, mechanical, and morphological features of the samples were shown in Table 2 and Figures 2-4.

The basic functional parameters of the process (sintering-foaming temperature and heating time) were experimentally determined by visualizing the upper surface of each sample.

The stabilization of the sintering temperature measured with the radiation pyrometer at a maximum value and the beginning of its slight decrease was the indication of completing the foaming process. The duration of the heating process was timed between the beginning of the magnetrons supply with electricity and the end of the process.

The functional parameters and the samples features noted between 1-4 correspond to the four compositions of the experimental variants.

From the analysis of data from the literature, excepting the foaming processes of CRT glass waste that used SiC as a foaming agent whose optimum temperatures were higher (880-1050 °C), the processes in which CaCO₃ or Na₂CO₃ constituted the foaming agent the required temperatures were significantly lower (below 800 °C).

A similar situation was obtained in the experiments performed under the influence of microwave radiation, according to the data in Table 2. The temperature values were between 780-800 °C.

The influence of the mixed microwave heating technique was manifested by the high heating rate of over 22.6 °C/min, reaching a maximum of 25.3 °C/min.

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Thus, the duration of the sintering-foaming process was low (below 34.5 min) leading to achieving very economical specific energy consumptions (between 0.71-0.81 kWh/kg). In principle, the low specific energy consumption of glass foam manufacture in the microwave field is a confirmation of the superior efficiency of the unconventional technique compared to the conventional techniques.

However, the fact that the literature does not provide information about this functional parameter, that defines the energy efficiency of the manufacturing processes, makes difficult a comparison between the two heating techniques.

The only bibliographic source that provides very elusive information about the energy consumption of glass foam manufacturing processes is [19], but it refers to a reported average consumption (100 kWh/m³, i.e. about 0.8-0.85 kWh/kg) of one of the greatest world companies (Misapor) with a wide range of foamed products.

Moreover, according to [17], an industrial scale-microwave equipment (high power) could be up to 25% more energy efficient compared to a domestic microwave oven of the type used in experiments.

Analysing the data in Table 3, it is remarkable the optimal combination between the physical (apparent density, porosity, thermal conductivity) and mechanical (compressive



Fig. 5 - Images of cross section of the samples/*Imagini ale secțiunii transversale a probelor* a – sample 1, heated at 780 °C; b – sample 2, heated at 786 °C; c – sample 3, heated at 793 °C; d – sample 4, heated at 800 °C./a - proba 1, încălzită la 780 °C; b - proba 2, încălzită la 786 °C; c - proba 3, încălzită la 793 °C; d - proba 4, încălzită la 800 °C.



Fig. 6 - Pictures of the sample's microstructure/*Imagini ale microstructurii probelor* a – sample 1, heated at 780 °C; b – sample 2, heated at 786 °C; c – sample 3, heated at 793 °C; d – sample 4, heated at 800 °C. / a - proba 1, încălzită la 780 °C; b - proba 2, încălzită la 786 °C; c - proba 3, încălzită la 793 °C; d - proba 4, încălzită la 800 °C.

strength) characteristics of the glass foam samples. The porosity is very high (between 85.5-92.0%) allowing a very low thermal conductivity, ideal for an excellent thermal insulation of the material. The compressive strength has more than acceptable values for using the glass foam as an insulating material in construction. Also, the water absorption varied between 0.2-0.8%, (the water absorption has very low values), that it means the impermeability of the material.

The physical and mechanical characteristics of the samples should be analyzed also in correlation with morphological ones. Images of cross section of the samples are presented in Fig. 5 and pictures of the sample's microstructure are shown in Fig. 6.

Analysing the pictures in Fig. 6, a phenomenon that is common for the thermal treatment of the CRT glass waste at higher temperature (over 800 °C) is observed. As the temperature and the pore size increase, the molten glass flows from the pore wall in struts, resulting pores with thinner walls. At certain critical thicknesses the pore wall breaks and the neighboring pores join.

The thickness of the vertical films of molten glass and the wall of the intact pore were determined at 86-200 μ m and the critical thickness of the rupture was calculated at 1-20 μ m, in accordance with the information in the literature[15,22] and based on the authors' own experience.

The conditions of forming a partially open pore structure are created. The pore size increases and negatively influences the compressive strength of the material. Fig. 6c indicates an incipient stage of this phenomenon corresponding to the foaming at 793 °C and Fig. 6d shows a slightly more advanced stage of the formation of the open pores corresponding to the foaming at 800 °C. In these conditions, the glass foam sample made according to variant 2 seems to be the optimal sample.

So, using 4.8 wt.% CaCO₃, 4.1 wt.% borax, 1.8% Sb₂O₃, 8% water addition and a heat treatment of the glass-based mixture at 786 °C, a porous material with apparent density of 0.20 g/cm³, porosity of 90%, thermal conductivity of 0.042 Wm⁻ ¹·K⁻¹ and compressive strength of 2.1 MPa can be obtained. The water absorption is practically zero and the porous microstructure is fine with closed pores having very low size between 0.4-0.8 mm.

4. Conclusions

A glass foam with good characteristics of insulating material (porosity of 90%, thermal conductivity of 0.042 W/m⁻¹·K⁻¹ and compressive strength of 2.1 MPa) usable in construction was experimentally made from recycled CRT panel glass waste by microwave heating technique at 786 °C on an adapted domestic oven.

This advanced method completely unused in the industrial manufacturing processes of glass foam and also in the numerous known tests performed on an experimental scale (only sporadically) proved to have an excellent energy efficiency (specific consumption of 0.72 kWh/kg) by comparison with the only information provided in the literature (0.8-0.85 kWh/kg) as the average consumption reported by one of the greatest world companies (Misapor) with a wide variety of glass foam products.

Excepting the use of the unconventional processing technique, the originality of the research presented in the paper consists in the application of the mixed microwave heating technique (partially direct and partially indirect) to cancel the destructive effect on the internal structure of glass-based material of the direct microwave heating.

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