

# STICLA CELULARĂ CA SOLUȚIE DE INERTIZARE A UNUI MATERIAL COMPOZIT EPUIZAT FOLOSIT PENTRU ADSORBȚIA ARSENULUI DIN APE UZATE

## CELLULAR GLASS AS INERTIZATION ALTERNATIVE FOR THE EXHAUSTED COMPOSITE ADSORPTION MATERIAL RESULTED FROM THE REMOVAL OF ARSENIC FROM WASTE WATERS

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Cellular glass is an insulation material that competes with polymeric and fibrous insulators on the market, having significant advantages such as the constant insulation efficiency, fire protection, corrosion and moisture resistance and long term dimensional stability. The present paper proposes a green solution to harness the exhausted composite adsorption material resulted from the removal of arsenic from wastewaters, by vitrification using two types of common recycled glasses: window panes and cathode ray tubes (CRT). Based on the high carbon amount present in the exhausted adsorption material, it was used as porogen material for cellular glasses. The apparent porosity, determined using the saturation under vacuum method, ranged between 40.25 - 62.15%. The optimal porous microstructure, having small uniform pores (under 150  $\mu\text{m}$ ) and narrow dimensional scattering, was obtained using both exhausted adsorption material and silicon carbide. The hydrolytically stability, measured according to the ISO 719/1985, classifies all the investigated glasses in HGB1-HGB3 stability classes. The arsenic, lead and iron ions leachability were determined in accordance with the American Extraction Procedure Toxicity Test. No discernable losses of arsenic or lead (in the case of samples containing CRT recycled glass) were measured, regardless of time or environment's pH. The amount of iron extracted after 28 days was very low, ranging from 0 to 0,056 % of the total iron. The thermal conductivity of the obtained cellular glasses range from 0.092 to 0.133 W/mK, classifying them as insulators. These results confirm the viability of the proposed alternative for immobilizing the exhausted adsorption material containing arsenic together with common recycled glasses as cellular glasses having high chemical stability and good thermal conductivity with multiple economic advantages

Sticla celulară este un material izolator termic care concurează pe piață cu alte materiale polimerice sau fibroase prezentând un număr de avantaje importante față de concurenți: constanță în timp a eficienței de izolare, rezistență la foc, coroziune și umiditate și constanță dimensională. Lucrarea de față propune o soluție ecologică de imobilizare a unui material adsorbant compozit epuizat, folosit în prealabil pentru adsorbția arsenului din ape uzate, prin vitrificare alături de două deșeuri de sticlă comune: sticla de geam și respectiv de tub cinescop. Ținând cont de cantitatea ridicată de carbon prezentă în materialul adsorbant epuizat, acesta a fost utilizat ca agent porogen pentru obținerea sticlei celulare. Porozitatea aparentă, determinată prin metoda saturării sub vid, este cuprinsă între 40,25 – 62,15%. Microstructura poroasă optimă din punct de vedere al uniformității și al distribuției dimensionale (pori sub 150  $\mu\text{m}$ ) a fost obținută prin folosirea concomitentă a deșeurii de adsorbție și a carburii de siliciu ca agenți porogeni. Stabilitatea hidrolitică, măsurată conform ISO 719/1985, califică probele studiate în clasele de stabilitate HGB1-HGB3. Imobilizarea ionilor de arsen, plumb și fier în matricea vitroasă a fost determinată în conformitate cu American Extraction Procedure Toxicity Test. Nu au fost puse în evidență pierderi de arsen sau plumb (în cazul folosirii sticlei reciclate din tub cinescop) la nici un termen considerat, indiferent de pH-ul mediului chimic agresiv. Cantitatea de ioni de fier solubilizată din probele realizate după 28 zile a fost foarte redusă, fiind cuprinsă între 0 - 0,056 % din totalul fierului existent în sticle. Conductivitatea termică a sticlelor sintetizate, cuprinsă între 0,092 – 0,133 W/mK, permite clasificarea acestor materiale ca izolatori termici. Rezultatele obținute confirmă viabilitatea soluției propuse pentru imobilizarea materialului adsorbant compozit conținând arsen alături de cele două deșeuri de sticlă sub formă de sticle celulare având rezistențe chimice ridicate și bune proprietăți termoizolante, în condiții economice avantajoase.

**Keywords:** arsenic adsorption, arsenic wastes, glass recycling, cellular glass.

### 1. Introduction

Arsenic is one of the oldest poisons used in history, being known since antiquity in the form of sulphurs or arsenic acid [1,2]. The initial effects of arsenic poisoning, known as arsenicosis, usually manifests after a decade of chronic exposures to doses higher than 50  $\mu\text{g/L}$  as skin lesions, or

internal organs affections. Some studies published on the World Health Organisation (WHO) website mention that in case of chronic exposure, the risk of organs or skin cancer dangerously rises [1-5].

Arsenic contaminations occur due to both natural and anthropogenic sources. Over 200 natural arsenic minerals are mentioned [6] mainly arsenates, arsenides, arsenites, sulphides,

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sulphosalts, oxides, silicates and elemental arsenic. Anthropogenic sources of arsenic pollution are a result of the mining industry, metal smelting and alloy melting industries, pesticides, herbicides, dyes, and so on [7].

Arsenic contaminating some high depth aquifers is considered a major threat to people's health. WHO recommends a maximum of 10 µg/L arsenic content in drinking water [2,5]. Large areas of Asia (Bangladesh, India, China), North America (USA) and Eastern Europe (Hungary, Romania, Slovenia, Serbia) [4], consume water with arsenic content exceeding 80-90 µg/L, with maximum concentration ranging between 250-300 µg/L.

Toxicological impact and appropriate remediation techniques must take into account the arsenic specifications, knowing that its effects are highly species dependant. Pentavalent arsenic is 60 times less toxic than the reduced trivalent state, and organic arsenic compounds are about 100 times less toxic than inorganic arsenic compounds [8].

Many methods for arsenic removal from drinking water are described in literature: oxidation/coagulation, oxidation/precipitation [9], precipitation/filtration [9,10], electrocoagulation [9, 11], adsorption [9, 12], ion exchange [9], and membrane filtration [11]. The most commonly used method, due to its high efficiency, ease of operation and economic benefits is adsorption, using a multitude of adsorbents such as metal oxides/hydroxides [13], zeolites [14], modified active carbon [15], iron based materials [16 - 19], resins [20 - 23], zirconia [24] and so on.

The main technical drawback that hindered widespread applications of this method is that the exhausted adsorbent containing arsenic becomes a solid waste involving costs and hazard for disposal [25]. This paper proposes a green solution to harness the exhausted composite adsorbent by vitrification as non-hazardous cellular glass. The advantages offered: immobilization of hazardous ions in the glass matrix, residual organics total combustion and waste volume reduction counterbalance the main economic drawback due to the high temperature and special equipment required for vitrification [26]. From the many techniques for obtaining porous glass [27], this research propose the glass powder sintering method using the exhausted adsorbent as foaming agent together with two post-consumer recycled glasses: window panes and cathode ray tubes.

## 2. Experimental Procedure

### 2.1. Sample preparation

The composite adsorption material (CAM) was obtained starting from a mixture of starch and iron chloride corresponding to a ratio of C:Fe 10:1, mixed at 55°C and then thermally treated in inert atmosphere at 400°C resulting in a material that contains iron oxide fixed on a carbon support. This

material has an adsorption capacity, in stationary regime, of 120 µg As/g after 60 minutes.

The exhausted CAM containing arsenic was inerted by vitrification using two types of common waste glasses: window panes and cathode ray tubes (CRT). The precursor waste glasses compositions, determined by X ray fluorescence using a Niton XL 3 analyzer, are presented in Table 1.

**Table 1**  
Oxidic composition of the recycled glasses used as precursors (% weight) / *Compoziția oxidică a deșeurilor de sticlă folosite ca precursori (% greutate)*

Oxide Oxid	Window pane Sticlă geam	CRT Tub cinescop
SiO <sub>2</sub>	71.86	60.92
Na <sub>2</sub> O	13.13	8.96
K <sub>2</sub> O	0.02	7.44
CaO	9.23	0.67
MgO	5.64	0.14
BaO	-	10.80
PbO	-	8.85
Al <sub>2</sub> O <sub>3</sub>	0.08	2.07
Fe <sub>2</sub> O <sub>3</sub>	0.04	0.15

The two waste glass precursors (granulometric fraction under 0.1 mm) were prepared as presented elsewhere [28].

Considering the large amount of carbon present in the exhausted CAM, it was used as porogen material together with silicon carbide (ELSIC 98, SC Elsid SA, granulometric fraction under 0.1 mm) to obtain porous glasses.

The exhausted CAM, dried at 105°C for 24 hours, was mixed together with silicon carbide and recycled glass precursors, the batch composition being presented in Table 2, based on

**Table 2**  
Batch composition of the studied porous glasses (weight ratio) / *Compoziția amestecului de materii prime [raport gravimetric]*

Sample Proba	Recycled glass Deșeu sticlă reciclat		Porogen agent Agent porogen	
	Window pane Sticlă geam	CRT Tub cine- scop	SiC	Exhausted CAM Material compozit adsorbant epuizat
1G	80	0	20	0
2G			15	5
3G			10	10
4G			5	15
5G			0	20
1E	0	80	20	0
2E			15	5
3E			10	10
4E			5	15
5E			0	20

the optimal ratio waste of glass:porogen agent 80:20, established elsewhere [29].

The raw materials, mixed together with 2% PVA as binder were uniaxial pressed at 40 MPa into cylindrical samples having the diameter and height around 35 and 100 mm respectively. The samples, previously dried at 80°C for 12 hours, were heat treated for 20 minutes at 900°C in an electrical furnace. After a fast cooling from the foaming temperature to 600°C, in order to freeze the porous structure, the obtained cellular glasses were annealed at 500°C to remove stress and then quickly cooled to room temperature.

### 2.2. Characterization methods

The apparent porosity of the obtained cellular glasses was determined using the saturation under vacuum method using water as working liquid.

Structural morphology of the porous glasses was analyzed by SEM using a QUANTA FEG 250 microscope.

The hydrolytic stability of the glass foams was determined according to ISO 719-1985, using 2 grams of glass, having particles size less than 500 µm, kept for 60 min in 50 mL de-ionized water at 98°C as described elsewhere [30].

The chemical stability of the cellular glasses was investigated by measuring the ion extraction using leaching tests performed according to the American Extraction Procedure Toxicity Test leaching procedure [31]. Three aggressive solutions having pH 5.5, 7.0 and 8.5 respectively were used, analysis being performed after 7, 14 and 28 days, prepared by taking 2.5% v/v glacial acetic acid in water and by adding concentrated ammonia solution until the desired pH value was reached. The lixivate was chemically analyzed after the considered times using an ICP-MS Aurora M90 Bruker.

The thermal conductivity of the obtained foam glasses was measured using a Transient Line Source Thermtest TLS-100 equipped with the 50 mm sensor.

## 3. Results and discussion

### 3.1 Cellular glasses apparent porosity

The apparent porosities of the obtained cellular glasses are presented in Table 3.

The obtained values, ranging between 40.25 to 62.15% are comparable to those mentioned by previous papers [29, 30]. Using CRT recycled glass containing PbO, leads to less porous materials due to a lower viscosity of the glass melt that is able to fill the pores at treatment temperature.

The influence of the exhausted waste used as porogen agent upon the glass apparent porosity is illustrated in Figure 1.

**Table 3**  
Apparent porosity of the studied porous glasses  
*Porozitatea aparentă a sticlelor celulare sintetizate*

Sample Proba	P <sub>ap</sub> [%]	Sample Proba	P <sub>ap</sub> [%]
1G	54.35	1E	48.55
2G	59.22	2E	52.16
3G	62.15	3E	50.33
4G	57.75	4E	47.15
5G	51.10	5E	40.25

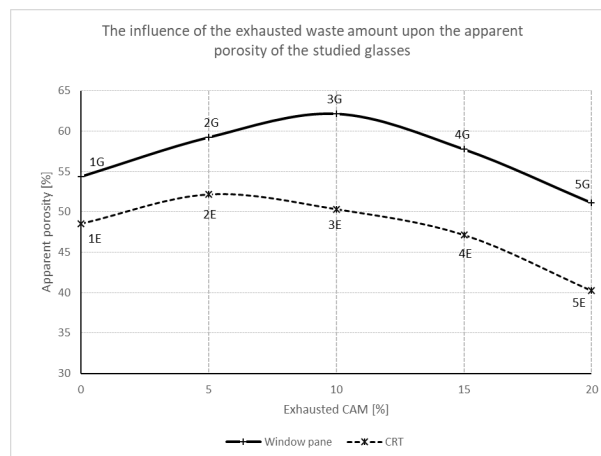


Fig. 1 - The effect of the exhausted CAM upon the apparent porosity of the cellular glasses / *Efectul adaosului de material compozit adsorbant epuizat asupra porozității aparente a sticlelor celulare studiate.*

Both glass groups present a similar evolution of the apparent porosity towards the amount of exhausted adsorption material used to replace SiC as foaming agent. After an initial positive trend, the porosity drops, even below the initial value obtained when only SiC is used. The optimal ratio exhausted CAM:SiC is 1:1 for glasses obtained from recycled window panes and 1:3 when CRT glass was used. Higher amounts of exhausted CAM leads to large quantities of carbon unable to generate CO<sub>2</sub> under the considered thermal treatment conditions, as it is presented in Figure 2 for samples 5G and 5E.

The porous structure of the samples 1G and 1E, containing only SiC as foaming agent and samples 3G and 2E having the highest apparent porosity are illustrated in Figure 3.

Using only SiC as foaming agent generates an inhomogeneous microstructure with irregular larger pores (100-500 µm) having a high dimensional scattering. The samples 3G and 2E containing both SiC and exhausted CAM have smaller pores (under 150 µm) and a narrower dimensional scattering. Using window panes as precursor leads to a more homogenous microstructure with smaller pores comparing to that obtained from CRT recycled glass. This behavior is caused by the higher CRT glass melt fluidity at the treatment temperature, leading to the coalescence of smaller pores into larger one.

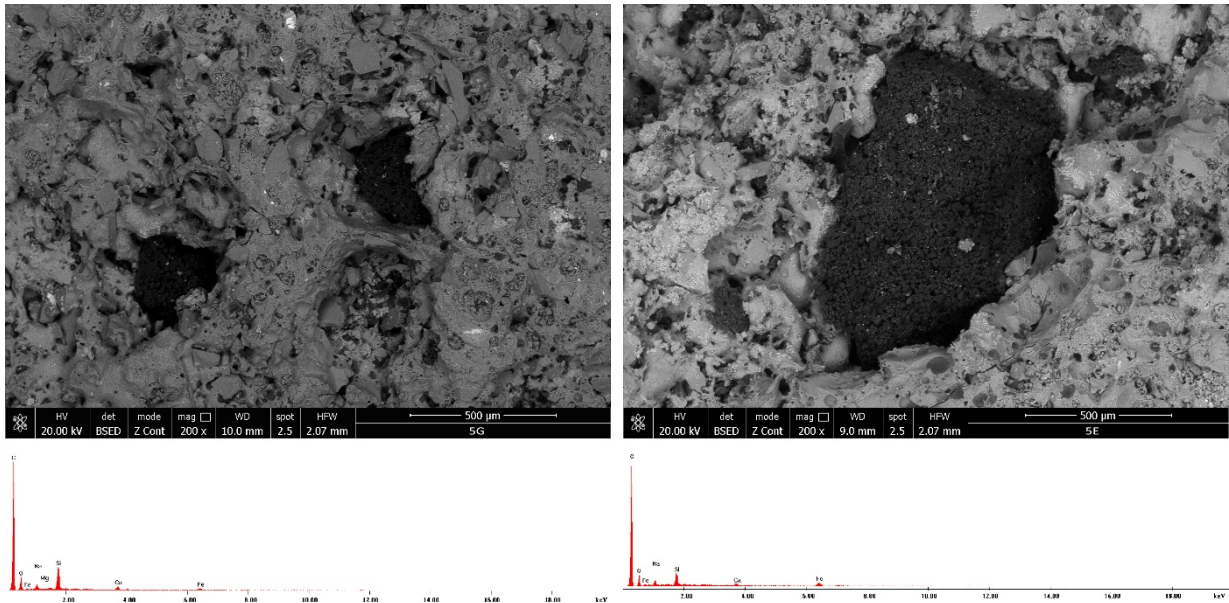


Fig 2 - SEM images of unoxidized carbon particles in samples 5G and 5E / Imagini microscopice SEM ale particulelor de carbon neoxidat din probele 5G și respectiv 5E.

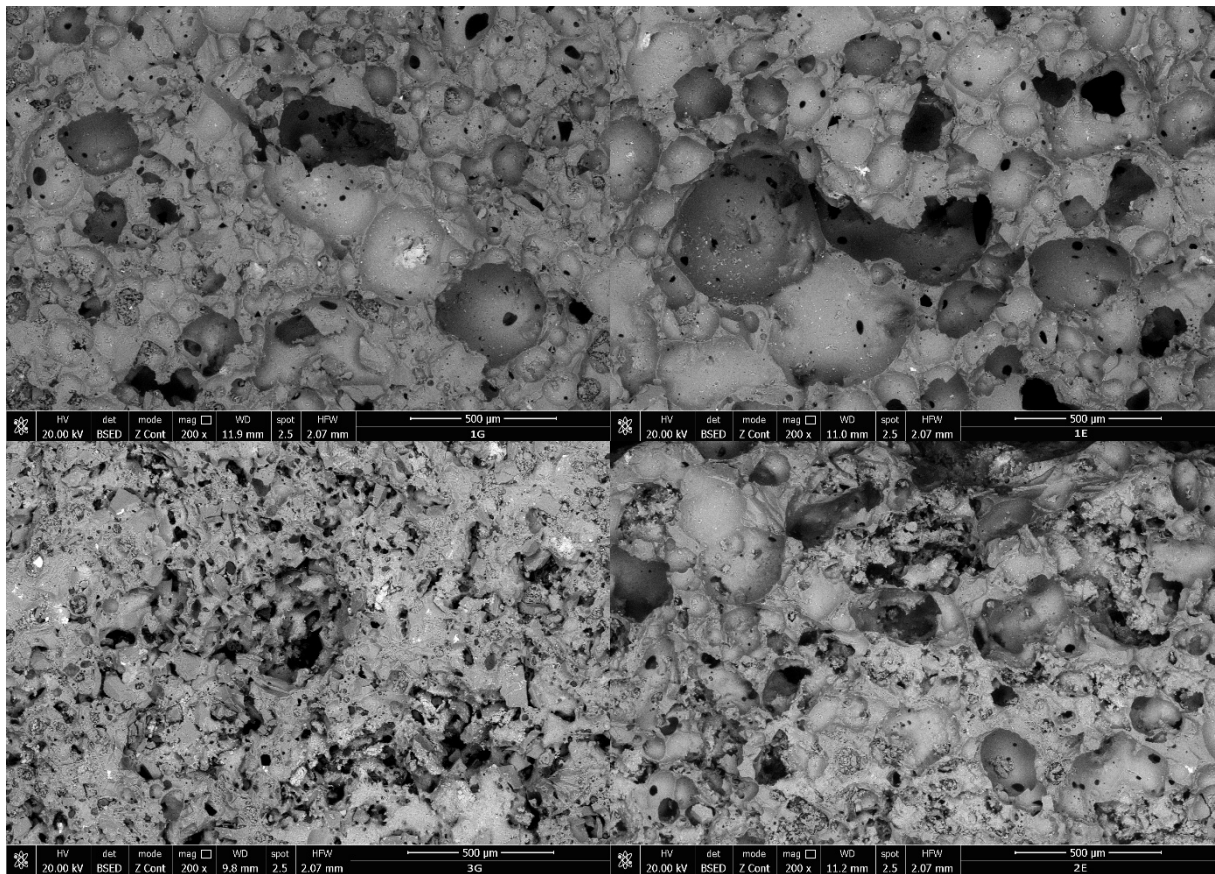


Fig. 3 - SEM images of samples 1G, 1E, 3G and 2E / Imagini de microscopie SEM ale probelor 1G, 1E, 3G și 2E.

### 3.2 Hydrolytic stability of the cellular glasses

The resistance of the synthesized glass foams toward water aggressivity, measured according to ISO 719-1985 is presented in Table 4 together with the corresponding stability class.

The investigated cellular glasses belong to the stability classes HGB1-HGB3, comparable to that of the precursors' glasses [32, 33]. The effect of the glass porosity upon the hydrolytic stability is illustrated in Figure 4.

**Table 4**

The hydrolytic stability and the corresponding stability class of the investigated cellular glasses

*Stabilitatea hidrolitică și clasa corespunzătoare de stabilitate a sticlelor celulare sintetizate*

Sample / Proba	Alkali oxide leached / Oxid alcalin solubilizat [μg/g glass]	Stability class / Clasa de stabilitate
1G	55,65	HGB2
2G	67,20	HGB3
3G	78,10	HGB3
4G	65,45	HGB3
5G	50,75	HGB2
1E	38,50	HGB2
2E	45,45	HGB2
3E	42,05	HGB2
4E	36,35	HGB2
5E	30,25	HGB1

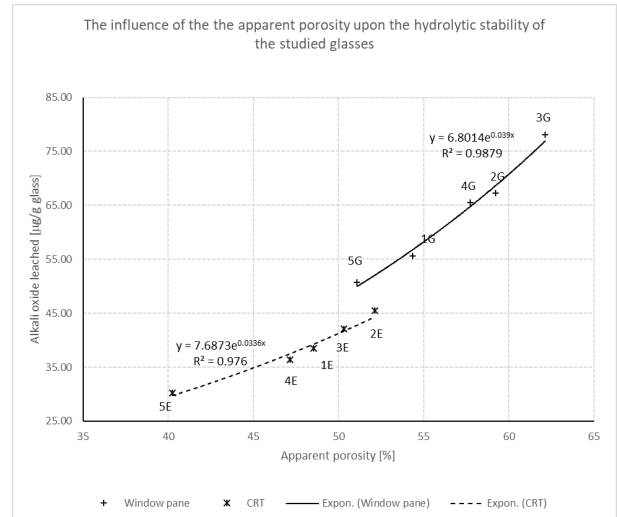


Fig. 4 - The effect of apparent porosity upon the alkali oxide extracted from the cellular glasses / *Efectul porozității aparente asupra cantității de oxid alcalin solubilizat din sticlele celulare studiate.*

Both glass groups show an exponential variation of the alkali leachability with glass apparent porosity, due to the increase in the glass surface exposed to the water's chemical attack. The grinding process imposed by ISO 719-1985 leads to a structure having more open pores and therefore being more susceptible to water aggression.

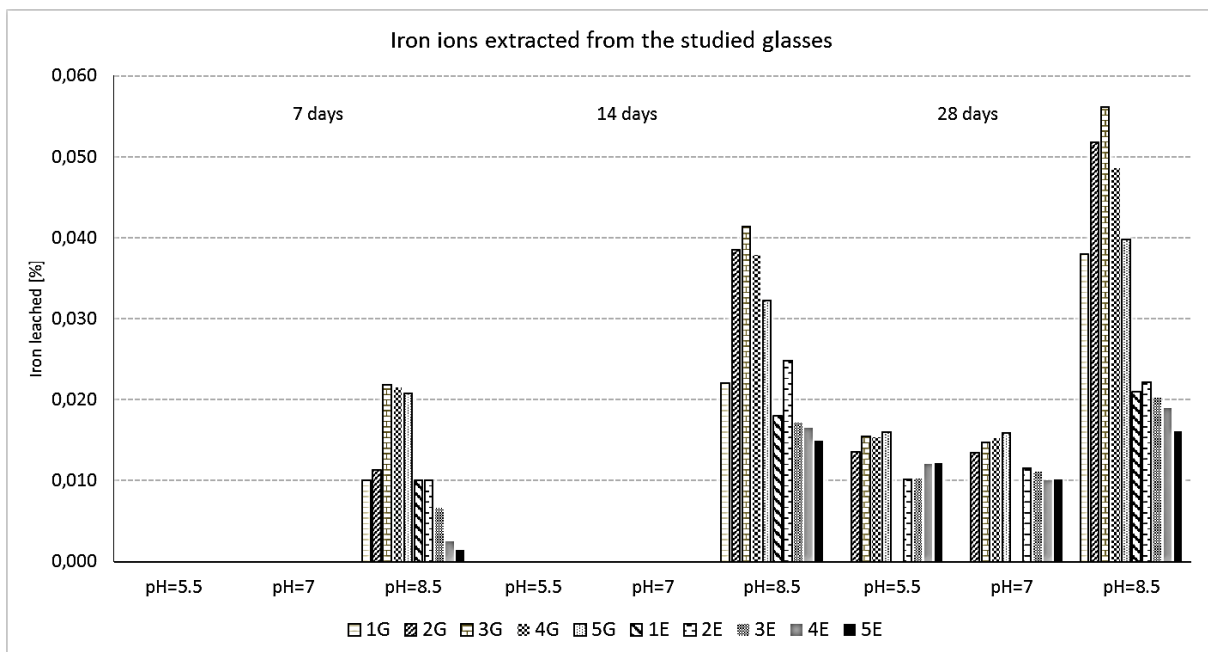


Fig.5 - Iron ions leached from the studied cellular glasses / *Pierderile de ioni de fier din sticlele celulare studiate.*

### 3.3 The ions immobilization in the cellular glasses

The vitrification effect upon the ions immobilization in the glass matrix was studied for arsenic, lead and iron ions. Three chemical aggressive environments were used for the three considered periods of time (7, 14 and 28 days). The glass ability to retain the mentioned ions was expressed as a percentage measured in lixivate relative to the total ion amount in the glass.

No discernable losses of arsenic or lead (in the case of samples 1E to 5E containing CRT recycled glass) were measured, regardless of time or environment's pH.

Iron ions amount leached from the investigated cellular glasses under the mentioned conditions is presented in Figure 5.

No record of iron ions losses were mentioned after 7 and 14 days when glasses are exposed to the acid and neutral environments due to the superficial passivation effect. Very small amounts of iron (less than 0,016%) are leached after 28 days of exposure to these environments.

The alkaline medium has a deeper effect upon the glass stability and therefore the iron immobilization due to the alkali destructive effect upon the glass network. Iron losses appear at all terms and range between 0,010 to 0,022 % after 7 days and 0,016 to 0,056 % after 28 days.

Using CRT as recycled glass precursor leads to more stable glasses and a higher iron immobilization effect, due to better encapsulation of iron oxide in the glass matrix comparing to windows panes glass.

### 3.4 Thermal conductivity of the cellular glasses

The measured values for the thermal conductivity of the synthesized foam glasses are presented in Table 5.

**Table 5**  
The thermal conductivity of the investigated cellular glasses  
*Conductivitatea termică a sticlelor celulare sintetizate*

Sample / Proba	$\lambda$ [W/mK]	Sample / Proba	$\lambda$ [W/mK]
1G	0,113	1E	0,121
2G	0,101	2E	0,115
3G	0,092	3E	0,118
4G	0,105	4E	0,125
5G	0,116	5E	0,133

All cellular glasses have thermal conductivities ranging from 0.092 to 0.133 W/mK, between the corresponding values of the two main phases present in their structure: glass, having  $\lambda = 0.6-1.38$  W/mK and air in the pores, having  $\lambda = 0.023$  W/mK. The measured values of the thermal conductivity classify the synthesized materials as insulators having  $\lambda < 0.25$  W/mK but are higher comparing to the industrial foam glass having

$\lambda = 0.043-0.048$  W/mK.

The influence of the apparent porosity upon the thermal conductivity of the obtained foam glasses is illustrated in Figure 6.

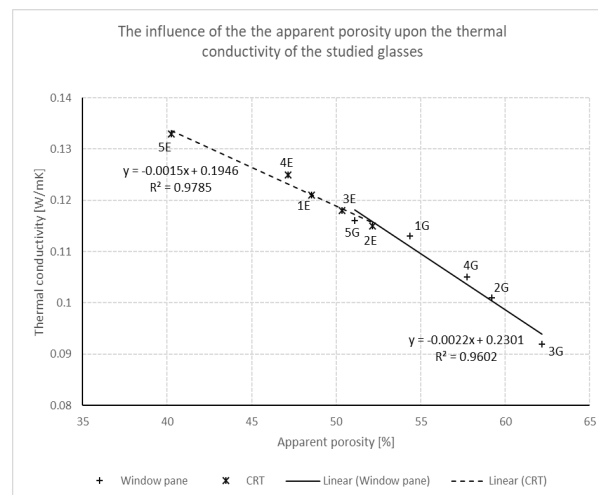


Fig. 6 - The effect of apparent porosity upon the thermal conductivity of the cellular glasses / *Efectul porozității aparente asupra conductivității termice a sticlelor celulare studiate.*

The thermal conductivity of both glass groups decrease quasilinear with porosity growth. Using windows panes recycled glass leads to cellular glasses having better insulating effect comparing to those obtained from CRT recycled glass.

## 4. Conclusions

The current paper proposes an ecologically efficient solution to use two common recycled glasses: window panes and cathode ray tubes together with silicon carbide and an exhausted adsorption composite material, previously used to retain arsenic from waste waters for the cellular glass synthesis.

The thermal treatment used for the foaming process was conducted at 900°C for 20 minutes, based on economically advantageous premises. The optimal ratio recycled glass:foaming agent of 80:20 [%] was previously established. Multiple SiC: exhausted adsorption composite material ratio were tested in order to obtain cellular glasses having good insulating properties and best immobilization capacities for arsenic and iron brought with the exhausted CAM.

The apparent porosity of the obtained cellular glasses ranges between 40.25 to 62.15%, being comparable to those mentioned in previous papers. CRT recycled glass containing PbO leads to less porous foam glasses due to lower viscosity of the glass melts that are able to fill the open pores during the foaming process. The optimal ratio of exhausted CAM:SiC is 1:1 for glasses obtained from recycled window panes and 1:3 for recycled CRT. Using higher amounts of exhausted CAM generate large carbon quantities unable to

oxidize to CO<sub>2</sub> under the considered thermal treatment conditions and consequently decrease the porogen effect of the CAM.

The microporous structure of the foamed glasses depends on the exhausted CAM:SiC ratio. Samples 3G and 2E considered optimal, have pores under 150 μm and a narrow dimensional scattering. Using window panes as precursor generates a more homogenous microstructure with smaller pores compared to that obtained from CRT recycled glass.

The hydrolytic stability of the obtained foam glasses correspond to the stability classes from HGB1 to HGB3 according to ISO 719-1985. An exponential dependence of the alkali leachability with glass apparent porosity was established based on the increase in the glass surface exposed to the water chemical attack.

The ions immobilization effect in the glass matrix was investigated for lead generated by CRT recycled glass and for arsenic and iron brought by the exhausted CAM. No discernable losses of arsenic or lead were recorded, regardless of time or environment's pH. The amount of iron ions extracted from all the studied foam glasses regardless the environment's pH and the considered time is very low, between 0-0.056% of the total iron brought by the exhausted CAM. The higher iron ions losses are generated by the alkaline environment that creates a continuous destabilizing attack on the vitreous matrix. CRT glass precursor have a positive effect upon the iron immobilization based on a better encapsulation of the iron oxide in the glass matrix.

The thermal conductivity of the studied cellular glasses ranges from 0.092 to 0.133 W/mK, classifying them as insulators having  $\lambda < 0.25\text{W/mK}$ . Foam glasses obtained from windows panes precursor have better insulating properties compared to those obtained from CRT recycled glass.

These results confirm the viability of the proposed alternative of recycling common glass wastes together with an exhausted adsorption material containing arsenic for synthesizing cellular glasses with improved chemical and insulating properties.

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