NOI MATRICI VITROASE PENTRU IMOBILIZAREA DEŞEURILOR DE PLUMB
NEW VITREOUS MATRIX FOR THE LEAD WASTES IMMOBILIZATION ▲

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An alternative recycling method for glass wastes (window panes, container glass and tableware) and fly ash as new glass matrix for lead waste immobilization was proposed in the following paper. The fly ash was previously used as adsorption support for the soluble lead waste from residual waters. The fly ash with 6% Pb^{2+} together with the glass wastes and borax were used as raw materials for the investigated glasses; three sets of glass samples were synthesized corresponding to weight ratios waste glass:borax:fly ash of 1:1:1, 1:0.75:1.25 and 1:0.5:1.5 respectively. The raw materials were melt for 90 minutes at 1200°C, resulting homogenous glasses. The hydrolytically and chemical stability, and lead leechability were determined according to the American Extraction Procedure Toxicity Test, ISO 719/1985 and DIN 12116/2001 respectively. The hydrolytically stability of all the studied glasses was between 11.16-17.95 µg/g R_2O soluble, qualifying them in the HGB1 stability class. The chemical stability of all glasses, characterized by the dissolution rate was 0-0.09 µg/cm²h, depending on the glass composition. The lead leechability was 0-0.07 % of the total lead in glasses, depending on the glass composition and pH of the aggressive medium. The obtained results confirm the viability of the proposed solution for lead waste immobilization with multiple economic advantages.

Lucrarea prezintă o metodă alternativă de reciclare a deşeurilor de sticlă (geam, ambalaj şi menaj) şi a cenuşii de termocentrală sub forma unor noi matrici vitroase capabile să imobilizeze deşeuri de plumb. Cenuşa de electrofiltru a fost utilizată ca material adsorbant pentru reţinerea compuşilor solubili ai plumbului din ape industriale. Deşeul rezultat, cu un conţinut de Pb^{2+} de 6%, alături de deşeurile de sticlă şi de borax s-a folosit la obţinerea matricilor vitroase; au fost realizate trei serii de probe cu un raport masic deşeu de sticlă:borax:cenuşă de 1:1:1, 1:0,75:1,25 şi 1:0,5:1,5. Topirea probelor la o temperatură de 1200°C timp de 90 minute. Pentru sticlele obţinute s-a determinat stabilitatea hidrolitică, stabilitatea chimică şi respectiv gradul de cedare al plumbului conform the American Extraction Procedure Toxicity Test, ISO 719/1985 şi DIN 12116/2001. Stabilitatea hidrolitică a tuturor sticlelor studiate se încadrează în domeniul de valori : 11.16-17.95 µg/g R_2O solubilizat, ceea ce încadrează aceste sticle în clasa de stabilitate HGB1. Stabilitatea chimică a sticlelor, măsurată prin rata de solubilizare a fost cuprinsă între 0-0,09 µg/cm²h, depinzând de compoziţia chimică. Gradul de solubilizare a plumbului s-a încadrat în domeniul de valori 0-0,07 % din plumbul total existent în sticlă, depinzând de compoziţia chimică a sticlei şi de pH-ul mediului agresiv. Rezultatele obţinute confirmă viabilitatea soluţiei propuse pentru imobilizarea deşeurilor cu plumb, cu multiple avantaje economice.

1. Introduction

The management of hazardous solid wastes represents a serious concern for environmental protection and public health reasons. In the past, fly ash was generally released into the atmosphere, but in recent years pollution control measures require that it should be captured and disposed in landfilling areas [1, 2]. The disadvantages of this method are related to the groundwater contamination and weathering problems. In the light of the latest environmental regulations, new recycling, or treatment processes must be developed and applied in order to transform ashes to non-hazardous materials [3]. Vitrification is one of the most promising waste management techniques

reducing the waste volume, destroying the residual organics and immobilizing heavy metals [4]. The economic disadvantage of vitrification may be counterbalanced by the further conversion of the glasses obtained from wastes into marketable products: glass or glass-ceramic matrix composites [5 - 7], glass fibers [8], glazes [9] or glass foams [10].

On the other hand glass wastes attract great interest in the recycling concept [11 - 13]. Using recycled glass has major economical and ecological advantages:
- conserves natural resources, every ton of glass that is recycled saves more than a ton of the raw materials needed to create new glass [14];

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- decrease the energy used for the melting process, the waste glass cullets acting like flux in the raw materials mixture;
- less CO_2 is emitted into the atmosphere: each tone of cullet used in the furnaces enables CO_2 emissions to be reduced by around 500 kg [15];
- waste volume is reduced allowing as much domestic waste as possible to be recycled, reducing the amount sent to landfill sites [16].

This work presents the application of the vitrification process to lead-rich ashes resulted from the purification of residual waters containing soluble lead using various types of glass wastes.

2. Experimental

2.1. Sample preparation

The recycled glasses used for the following synthesis were container glass, tableware and window panes. The compositions of the precursor glasses are presented in Table 1.

The composition of the fly ash released by CET Timişoara thermal power plant is presented in Table 2.

The fly ash, having a specific BETH surface of 6.3 m^2/g, was previously used as adsorption support for the soluble lead waste from residual waters containing 200 mg/L Pb^{2+}. The adsorption conditions were:

- residual water: pH = 5;
- shaking time: 60 minutes;
- solid:liquid ratio 1:10.

The lead retention efficiency of the process was over 99%, the resulting fly ash contains 6 % Pb^{2+}.

In order to reduce the melting temperature borax was used as flux. The weight ratios waste glass:borax:fly ash for the studied glasses are shown in Table 3. The oxidic composition of the investigated glasses are presented in Table 4.

The recycled glasses powders, containing the granulometric fraction under 0.1 mm, together with the fly ash and borax were weighed to the desired compositions, mixed and ground in a porcelain mortar. The mixture was melted for 90 minutes at 1200°C in an electrical furnace with SiC heating elements. The melt was then press-quenched between two stainless steel blocks.

Table 1

Oxidic composition of the recycled glasses used as precursors /*Compoziţia oxidică a deşeurilor de sticlă reciclate utilizate*

Recycled glass / *Deşeu de sticlă*	Composition / *Compoziţie* (weight % / *% gravimetrice*)						
	SiO_2	Na_2O	K_2O	MgO	CaO	Al_2O_3	Fe_2O_3
Container glass / *Sticlă ambalaj*	72	13	-	1	12	2	–
Tableware / *Sticlă menaj*	75.9	14.46	1.21	–	7.43	1	–
Window pane / *Sticlă geam*	71.86	13.13	0.02	5.64	9.23	0.08	0.04

Table 2

Oxidic composition of the fly ash /*Compoziţia oxidică a cenuşii de termocentrală*

Oxide / *Oxid*	Composition / *Compoziţie* (weight % / *% gravimetrice*)						
	SiO_2	Na_2O	K_2O	MgO	CaO	Al_2O_3	Fe_2O_3
Fly ash / *Cenuşă termocentrală*	46.2	6.23	4.17	3.3	8.6	23.2	8.1

Table 3

Composition of the studied glasses
Compoziţia sticlelor sintetizate

Sample *Probă*	Recycled glass / *Deşeu de sticlă*			$Na_2B_4O_7$	Fly ash *Cenuşă*
	Tableware *Sticlă menaj*	Container glass *Sticlă ambalaj*	Window pane *Sticlă geam*		
S1	1	-	-	1	1
S2	-	1	-	1	1
S3	-	-	1	1	1
S4	1	-	-	0.75	1.25
S5	-	1	-	0.75	1.25
S6	-	-	1	0.75	1.25
S7	1	-	-	0.50	1.50
S8	-	1	-	0.50	1.50
S9	-	-	1	0.50	1.50

Oxidic composition of the studied glasses/ *Compoziţia oxidică a sticlelor studiate*

Sample *Probă*	Composition / *Compoziţie* (weight % / *% gravimetrice*)								
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	B_2O_3	PbO
S1	47.20	9.36	3.13	6.20	0.04	14.27	2.08	14.17	2.32
S2	46.05	9.04	3.16	8.03	0.05	13.82	1.62	14.28	2.34
S3	45.64	9.00	3.15	6.89	0.10	13.76	1.62	14.17	2.32
S4	49.14	11.03	3.72	6.69	0.05	12.66	2.36	10.11	2.76
S5	47.89	11.08	3.74	8.40	0.06	12.17	1.92	10.15	2.77
S6	47.66	10.70	3.74	7.35	0.12	12.17	1.92	10.11	2.76
S7	50.92	12.56	4.26	7.13	0.06	11.19	2.62	6.42	3.16
S8	49.90	12.30	4.29	8.79	0.07	10.76	2.21	6.47	3.18
S9	49.50	12.24	4.28	7.76	0.13	10.73	2.20	6.42	3.16

2.2. Characterization methods

The density of the glasses was measured using the picnometric method using isopropylic alcohol as working liquid.

The hydrolytic stability of the glasses 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. 25 mL of the obtained solution was titrated against 0.01 mol/l HCl solution. The volume of HCl needed for neutralization is recorded in order to express the equivalent Na_2O extracted.

The lead immobilization capacity of the studied glasses was investigated by measuring the lead ions extraction using leaching tests performed according to the American Extraction Procedure Toxicity Test [17]. This procedure uses three extraction mediums having pH 5.5, 7 and 8.5 respectively, analysis being performed after 1, 14 and 28 days. The lead concentration in the extraction mediums was measured using a Varian SpectrAA 110 atomic spectrometer fitted with the Mark 7 flame atomization system.

The chemical stability, expressed by the dissolution rate $Dr = \dfrac{\Delta m}{S \cdot t}$, where: Δm represents the weight loss at the time *t* and S the total surface of the sample, was determined using the same conditions as the leechabilty tests. The buffer solutions from 5.5, 7 and 8.5 pH were 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 pH of the solution was measured using a digital pH meter (Type E-500). 2 g each of the glasses were taken and shaken with 250 ml of ammonia–acetate buffer solution for different time periods at a constant temperature of 20 ± 2°C.

3. Results and discussions

3.1. Glasses densities

The glass density reflects the structural changes in the structure of glasses, beeing affected by structural softening/compactness, changes in geometrical configuration, coordination number, cross-link density and the dimensions of interstitial spaces in the structure. The density values for the studied glasses are shown in Table 5.

Density of the studied glasses
Densitatea sticlelor studiate

Sample / *Probă*	Density / *Densitate* [g/cm^3]
S1	2.63
S2	2.64
S3	2.64
S4	2.70
S5	2.70
S6	2.71
S7	2.75
S8	2.77
S9	2.75

The density of a glass is mainly determined by its composition [18]. The influence of the PbO amount upon the glass density is presented in Figure 1.

The increase in the density can be related to there placement of B_2O_3 and Na_2O with an oxide of greater molecular mass, PbO [19]. A good correlation between the concentration of PbO and the density of the glasses can be established.

3.2. The hydrolytic stability of the glasses

The resistance of the studied glasses toward water aggresivity was analyzed according with ISO 719-1985, the results being summarized in Table 6.

All the investigated glasses belong to the hydrolytic class HGB1. A favorable effect of the ash amount upon the glass stability can be observed. In order to correlate the obtained values with the glass composition the effect of water upon the oxides contained in the studied glasses was expressed as:
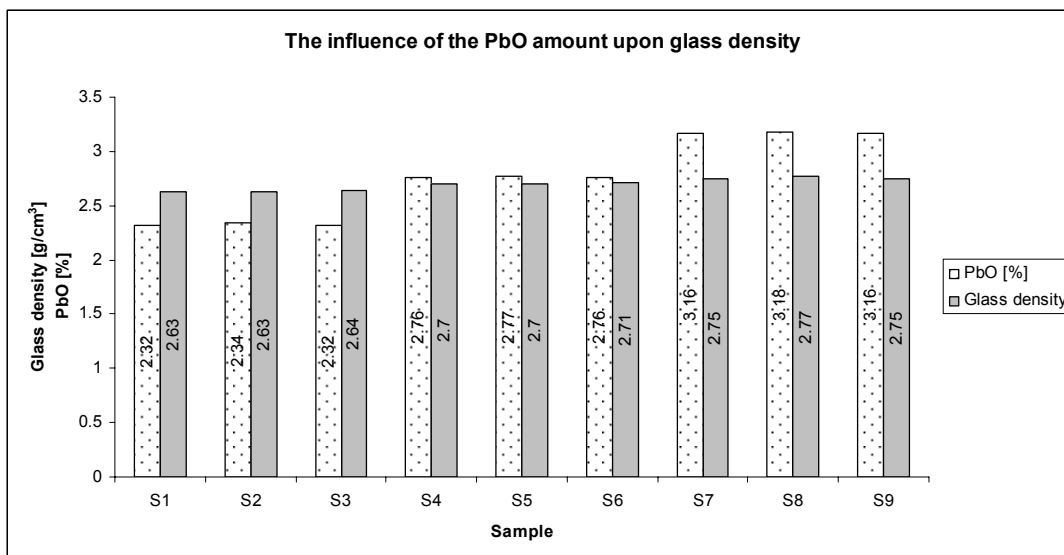
Fig. 1 - Influence of the PbO amount upon the density of the glasses / *Influența cantității de PbO asupra denstății sticlelor.*

Table 6

The hydrolytic stability and the corresponding hydrolytic class
Stabilitatea hidrolitică şi clasa de stabilitate corespunzătoare

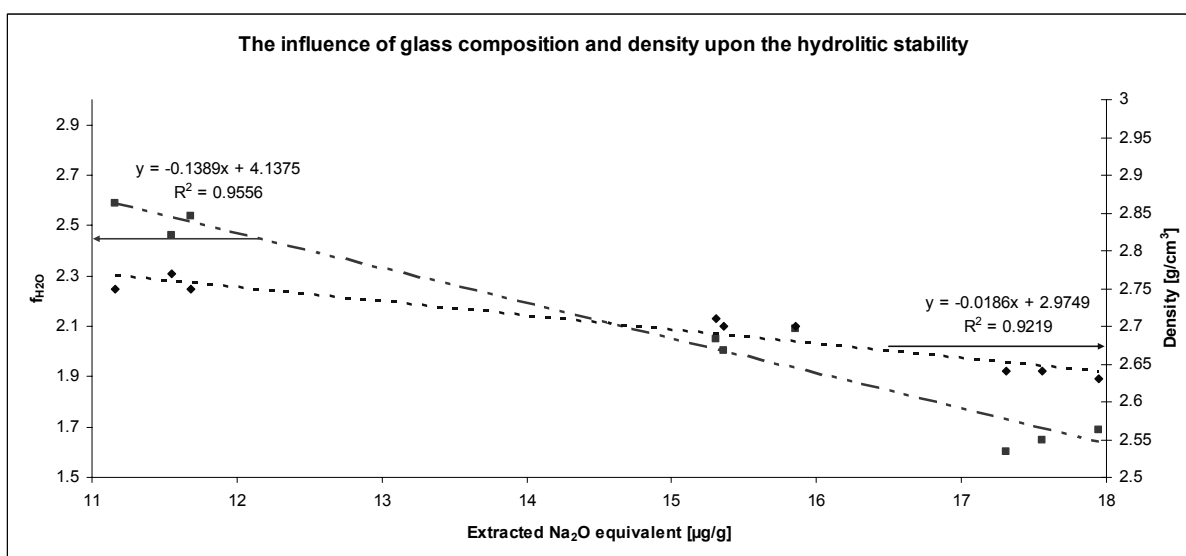| Sample /*Probă*	Extracted Na$_2$O equivalent [μg/g] *Echivalent Na$_2$O solubilizat*	Hydrolytic class *Clasa de stabilitate*
S1	17.95	HGB1
S2	17.31	HGB1
S3	17.56	HGB1
S4	15.85	HGB1
S5	15.36	HGB1
S6	15.31	HGB1
S7	11.16	HGB1
S8	11.55	HGB1
S9	11.68	HGB1

Fig. 2 - The effect of the glass composition and density upon their hydroyitic stability / *Efectul compoziției şi respectiv densității sticlelor asupra stabilității hidrolitice a acestora.*

$$f_{H2O} = \frac{\%SiO_2 + \%Al_2O_3 + \%Fe_2O_3 + \%PbO}{\%Na_2O + \%K_2O + \%MgO + \%CaO + \%B_2O_3}$$

The influence of the glass structure, reflected by the density values and the glass composition upon the hydrolytic stability is showed in Figure 2.

A quasilinear dependence, having linear squared regression coefficients over 0.9, can be observed for both compositional factor f_{H2O} and density upon glass hydrolytic stability. The glasses with higher concentration of oxides less sensible to water attack, containig more fly ash and therefore more SiO_2 and Al_2O_3 (samples S6-S9) and lower quantities of alkaline and earth alcaline oxides, have higher densities, a more compact structure,

less susceptible to be leeched by water.

3.3. The lead immobilization in the glass matrix

The leaching test had a duration of 1 day, 14 days and 28 days respectively, after which the solid and the liquid were separated by filtration. The lixiviate was chemically analysed using a Varian SpectrAA 110 atomic absorption spectrophotometer. The results are presented in Figure 3.
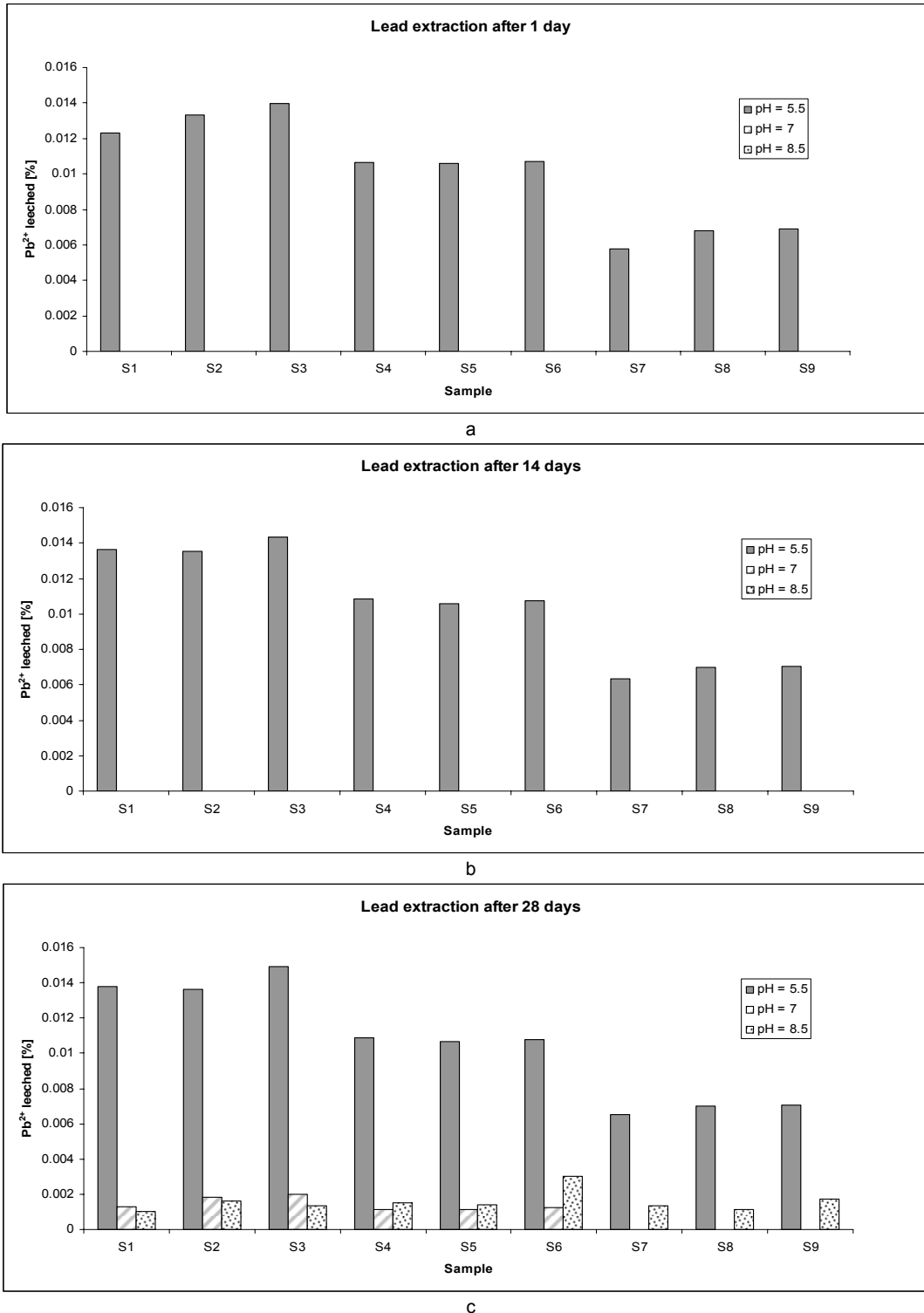

a

b

c

Fig. 3 - Lead ions leachability for the investigated glasses at: / *Solubilizarea ionilor de Pb²⁺ din sticlele studiate la:* 1 day / *zi,* 14 days / *zile,* 28 days / *zile.*

The lixiviation values for Pb^{2+} are under 0.015 % for all samples. The pH of the attack solution influence the lead leechability, the most aggressive medium beeing the acid one for all the studied periods of time. The neutral and basic mediums provoke a very limited lead dissolution (less than 0.002%) only after 28 days. This behaviour can be explain based on the glass composition and the susceptibility of different oxides to the attack of mediums withh different pH.

The samples S1-S3, having more alkaline and earth alkaline oxides comparing to the other samples, are more sensitives to the acid aggresion, the fragility of the attacked structure allowing the Pb^{2+} extraction from the glass matrix. The lowest lixivations, regardless of the medium pH and attack time, were for the samples S6-S9, suggesting the positive effect of ash for the lead immobilization.
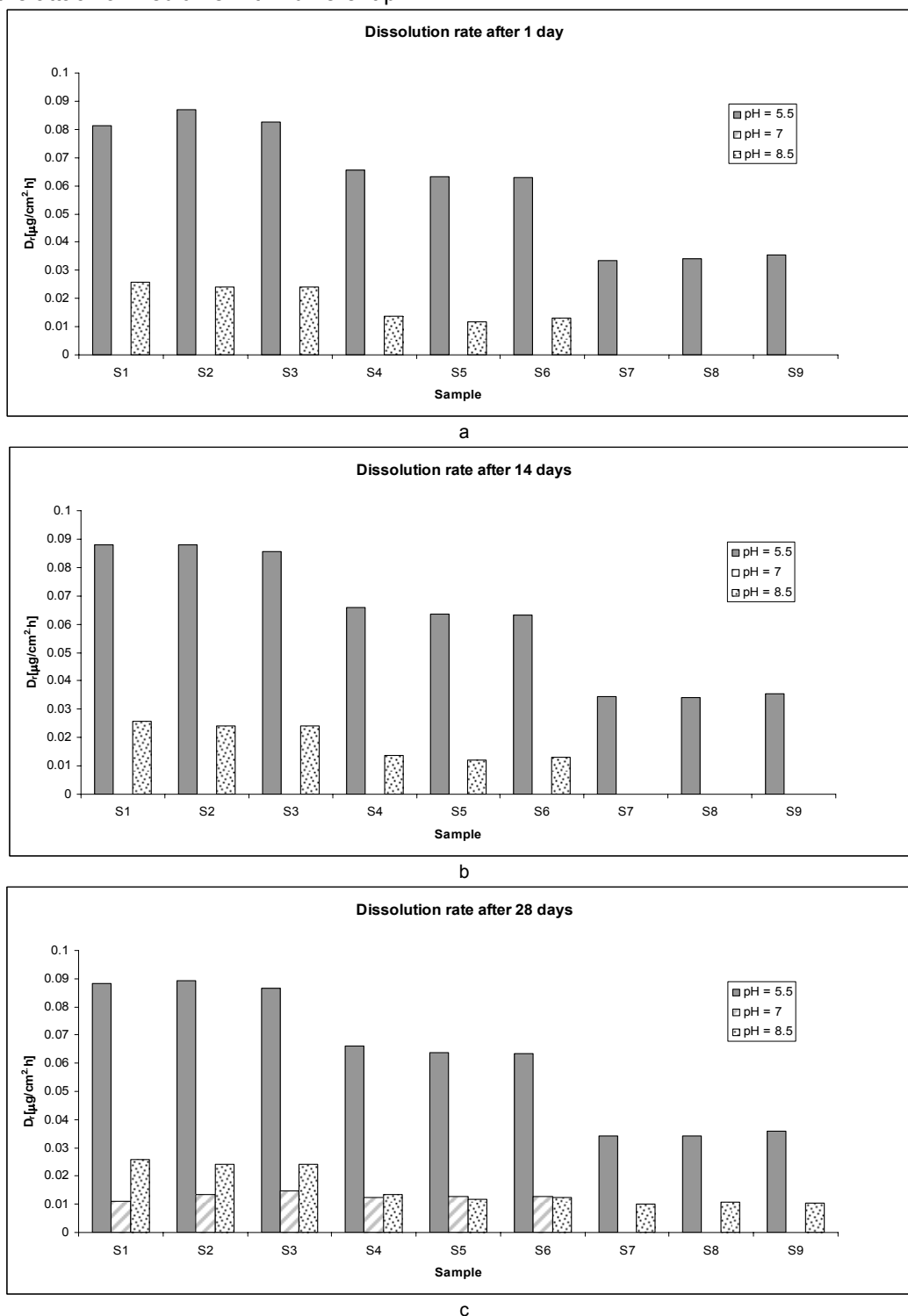

a

b

c

Fig. 4 - Chemical stability of the investigated glasses at: / *Stabilitatea chimică a sticlelor studiate la:*
1 day / *zi,* 14 days / *zile,* 28 days / *zile.*

3.4. The chemical stability of glasses

The resistance of the investigated glasses towards the chemical aggresivity was tested according to DIN 12116/2001, the results beeing presented in figure 4.

The dissolution rates used to characterize the chemical stability are under 0-0.09 $\mu g/cm^2h$ for all samples. The most aggresive medium was the acid one (pH = 5.5), that attacks the alkaline and earth alkaline oxides from the glass matrix. The neutral medium have a very weak effect upon the long term glass stability (D_r less than 0.015 $\mu g/cm^2h$). The basic medium (pH = 8.5) influence the chemical stability of the glasses by attacking the B_2O_3 oxide from the glass matrix, having dissolution rates lower than 0.026 $\mu g/cm^2h$. The higher amount of alkaline and earth alkaline oxides from the glasses S1-S3 generates a higher sensitivity for all the three considered periods of time. The higher chemical resistances, regardless of the medium pH and attack time, were for the samples S6-S9, confirming the positive effect of ash for the chemical resistance of glasses.

4. Conclusions

The vitrification process can be applied to lead-rich ashes resulted from the purification of residual waters containing soluble lead using different types of glass wastes: tableware, container glasses and window panes.

Three different glass waste:borax:fly ash weight ratio were used to prepare the studied glasses: 1:1:1, 1:0.75:1.25 and 1:0.5:1.5 respectively in order to establish the maximum amount of fly ash that can be added to the glass matrix, using the economic criterion of the lower melting temperature. Higher amounts of fly ash generate incomplete fused glasses or partially sintered materials without practical interest.

The hydrolytic stability of the investigated glasses qualifies them as HGB1 glasses having a very good resistance toward water aggresivity, according to ISO 719-1985. A good correlation between compositional and structural factors upon the glass stability was established.

The lead immobilization capacity of the studied glasses was characterised by measuring the Pb^{2+} lixiviation according to the American Extraction Procedure Toxicity Test. The very low values of the leaching rate for the Pb^{2+}, under 0.015 %, qualifies all the glasses as good lead immobilization materials.

All the studied glasses had a very good chemical stability according to DIN 12116/2001. The low values of the dissolution rate for all glasses make them suitable for industrial manufacturing.

The most stable glasses were obtained using the higher amount of fly ash (glass waste:borax:fly ash weight ratio of 1:0.5:1.5), due to the improvement of the glass matrix based on the SiO_2 brought by this. The corresponding glasses S6-S9 had the lowest alkaline extraction (under 11.70 $\mu g/g$), the best lead immobilization effect (lixiviation values for Pb^{2+} are under 0.002% after 28 days) and the highest chemical stability (having dissolution rates under 0.037 $\mu g/cm^2h$ after 28 days). The obtained results show no meaningful differences on the behaviour of the three waste glasses used as precursors.

The obtained results confirm the viability of the proposed solution for immobilizing lead wastes into glasses using glass wastes as precursor materials having very good chemical stability with multiple economic advantages.

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NOUTĂŢI / NEWS

Nano-scale reaction processes at the interface between apatite and aqueous lead

Synthetic hydroxyapatite ($Ca_5(PO_4)_3OH$, HAP) powder and a bulk single crystal of natural fluorapatite (($Ca_{4.915}Na_{0.014})(P_{3.029}Si_{0.010})O_{12}(F_{0.930}Cl_{0.098})$, FAP), which was mechanically polished normal to the c axis, have been reacted with Pb nitrate solutions (2×10^{-3} mol l^{-1}) at an initial pH of 5.0 at room temperature and examined by high-resolution transmission electron microscopy (HRTEM). In the experiment using HAP powder crystals, secondary Pb phosphate, hydroxypyromorphite ($Pb_5(PO_4)_3OH$, HPY), forms epitaxially on the tip of the HAP crystal parallel to the a or b axis. Two types of mechanisms are observed in the HPY formation: (i) Whisker formation, which refers to an epitaxial growth of HPY needle-like nanocrystallites on the tip of the HAP crystal parallel to the a or b axis from the solution supersaturated with respect to HPY, and (ii) pseudomorphism, which is a direct replacement of HAP by HPY concurrent with the HAP dissolution at the interface. The HPY pseudomorph further grows as large as ~10 µm for 30 days by consuming the HPY whisker nanocrystallites following the Ostwald step rule. Nucleation of HPY on the specific crystalline faces, which presumably have lower interfacial energy, predominantly governs the kinetics and mechanisms of Pb immobilization by HAP nanoparticles. Such mechanisms can be applicable to nanoscale pseudomorphisms occurring on submicron-sized particles. In the experiment using bulk FAP, cross-sectional TEM demonstrates preferential leaching of Ca from the surface, and a secondary Pb-phosphate, chlorpyromorphite ($Pb_5(PO_4)_3Cl$), precipitates without growing epitaxially on the FAP surface. There is no evidence, under the experimental conditions of this study, of a cation exchange mechanism between Pb and Ca maintaining apatite structure (i.e., bulk diffusion of Pb into HAP crystal). The results obtained in the present study underscore the importance of the interface-driven mechanism in the interaction between aqueous toxic metals and apatite structured minerals.

Procesele la scară nano la interfaţa dintre apatită şi plumb în soluţie apoasă

Pulberea de hidroxiapatită sintetică ($Ca_5(PO_4)_3OH$, HAP) şi masa unui singur cristal de fluoroapatită naturală (($Ca_{4,915}Na_{0,014})(P_{3,029}Si_{0,010})O_{12}(F_{0,930}Cl_{0,098})$, FAP), care a fost polisat mecanic normal la axa c, au reacţionat cu soluţii de azotat de plumb (2×10^{-3} mol l^{-1}) la un pH iniţial de 5, la temperatura camerei şi au fost examinaţi prin microscopie electronică de înaltă rezoluţie (HRTEM). În experimentul care foloseşte cristale de pulbere de HAP, fosfat secundar de plumb, hidroxipiromorfitul ($Pb_5(PO_4)_3OH$, HPY se formează epitaxial pe vârful cristalelor de HAP, paralel cu axele a sau b. Se observă două tipuri de mecanisme la formarea lui HPY: (i) formarea unor mănunchiuri care se referă la creşterea epitaxială a nanocristalitelor aciculare de HPY pe vârful cristalului HAP, paralel cu axele a sau b, din soluţiile suprasaturate cu HPY şi (ii) pseudomorfism, care este un proces de înlocuire directă a HAP cu HPY, concurent cu dizolvarea lui HAP la interfaţă. Pseudomorful HPY mai creşte până la ~10 µm timp de 30 zile, consumând nanocristalitele din mănunchiurile HPY, urmând regula etapei lui Ostwald. Nucleaţia lui HPY pe feţele cristaline specifice, care se presupune că are energie interfacială mai mică, guvernează predominant cinetica şi mecanismele de imobilizare a Pb de către nanoparticulele de HAP. Astfel de mecanisme pot fi aplicate pseudomorfismului la nanoscară, care are loc pe particulele de dimensiune submicronică. În experimentul care utilizează o cantitate solidă de FAP, microscopia TEM în secţiune demonstrează dizolvarea preferenţială a Ca de pe suprafaţă şi precipitarea unui fosfat de Pb secundar, clorpiromorfit ($Pb_5(PO_4)_3Cl$), fără creştere epitaxială pe suprafaţa FAP. Nu există o dovadă, în condiţiile experimentale ale acestui studiu, cu privire la un mecanism de schimb cationic între Pb şi structura apatitică care menţine Ca (difuzia în masă a Pb în cristalele de HAP). Rezultatele obţinute în prezentul studiu subliniază importanţa mecanismului generat de interacţia dintre metalele toxice apoase şi mineralele cu structură de apatită.

Material prelucrat de:/ *Material worked by: Alina Melinescu,* Sursa /*Source: Chemical Geology 340 (2013) 121–130*