

ECO-FRIENDLY SOLUTION FOR WASTES RESULTED FROM THE REMOVAL OF Cr(VI) WITH Fe⁰ IMMOBILIZATION IN GLASS BASED STONEWARE MATRIX

SOLUȚIE ECOLOGICĂ DE IMOBILIZARE A UNOR DEȘEURI PROVENITE DIN ÎNDEPĂRTAREA Cr(VI) CU Fe⁰ ÎN MATRICE CERAMIC - VITROASE

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The present paper investigates another alternative to immobilize the exhausted reactive mixtures resulted from the removal of Cr(VI) with Fe⁰ in continuous-flow system (column experiments), in glass based stoneware matrix. This study completes our previous investigations focused on vitreous matrix designed to retain the exhausted reactive mixtures [1]. In order to increase the ecological impact of the proposed solution, two types of recycled waste glasses were used: window panes and cathode ray tubes (CRT). The exhausted reactive mixtures and the two waste glasses were mixed together with the Bojidar kaolin and then pressed into cylinders having the diameter and height around 35 mm. The heat treatment was optimized considering the economically advantageous conditions at 1000°C for 90 minutes. The apparent porosity, used as compactness index for the obtained samples, range from 2.20% to 5.05% depending on the glass waste type and the amount of sand contained in the exhausted reactive mixture. The main crystalline phases confirmed by RX diffraction are wollastonite, tridymite, diopside, cristobalite and hematite. The chemical stability of the studied glass based stoneware, measured using the samples' dissolution rate after 28 days in mediums having different pH range from 0.127 to 0.778 μg/h depending on the samples' porosity and type of chemical aggression. The chromium and iron ions leachability were determined and American Extraction Procedure Toxicity Test respectively. The amounts of both investigated ions removed from the structure by the chemical attack are very low, between 0-0.238% of the total chromium and between 0-0.258% of the total iron brought by the exhausted reactive mixtures. The obtained results confirm the viability of the suggested solution for immobilizing chromium contained in the exhausted reactive mixtures together with common waste glasses and kaolin as glass based stoneware having high chemical stability with multiple economic advantages.

Lucrarea de față investighează o altă alternativă de immobilizare a amestecurilor reactive epuizate provenite din îndepărtarea Cr(VI) cu Fe⁰ în experimente pe coloană în matrice ceramic-vitroase. Acest studiu completează investigațiile anterioare asupra posibilității de a inertiza aceste deșeuri în matrice vitroase [1]. Pentru a crește impactul ecologic al soluției propuse au fost folosite două tipuri de deșeuri reciclabile de sticlă: geam și tub cinescop (CRT). Amestecurile reactive epuizate și cele două tipuri de deșeuri de sticlă au fost amestecate cu caolin de Bojidar și apoi presate sub forma unor cilindrii având diametrul și înălțimea de aproximativ 35 mm. Tratamentul termic a fost optimizat pentru respectarea unor condiții economic avantajoase la 1000°C timp de 90 minute. Porozitatea aparentă, folosită ca măsură a compactității probelor obținute, variază între 2,20% și 5,05%, în funcție de tipul deșeurii de sticlă utilizat și de cantitatea de nisip conținută în amestecurile reactive epuizate. Principalele faze cristaline puse în evidență prin difracție RX au fost wollastonitul, tridimitul, diopsidul, cristobalitul și hematitul. Stabilitatea chimică a probelor studiate, măsurată folosind gradul de solubilizare după 28 de zile în medii având diferite pH-uri s-a situat în domeniul 0,127-0,778 μg/h, în funcție de porozitatea aparentă și respectiv de tipul agresiunii chimice. Capacitatea de immobilizare a cromului și fierului au fost determinate folosind American Extraction Procedure Toxicity Test. Cantitatea extrasă în urma agresiunii chimice este foarte redusă, fiind cuprinsă între 0-0,238 % din totalul cromului și respectiv între 0-0,258% din totalul fierului aduse de amestecurile reactive epuizate. Rezultatele obținute confirmă viabilitatea soluției propuse pentru immobilizarea amestecurilor reactive epuizate conținând crom alături de două deșeuri de sticlă și caolin sub forma unor mase ceramic-vitroase cu rezistențe chimice ridicate în condiții economic avantajoase.

Keywords: glass based stoneware, waste glasses, glass recycling, chromium wastes.

1. Introduction

The actual industrial development leads to an increase in environmental pollution. [2, 3]. Heavy metals, resulted from smelting, mining or other different industries are persistent and cannot be degraded naturally [4, 5].

Chromium is one of the most harmful pollutant present in industrial effluents generated

from dyeing, painting, tanning, ceramics, explosives, wood processing and paper industry [6]. The common wastewaters can contain both trivalent Cr(III) and hexavalent Cr(VI) forms, presenting various chemical properties and effects upon the environment [7]. The hexavalent chromium is the most toxic one generating mutagenic and cytogenetic effects on living organisms [8].

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Conventional methods for Cr(VI) wastewaters treatment are precipitation [9], reduction [10], membrane technology [11], ion exchange [12], electrochemical treatment [13] and adsorption [14, 15].

In recent years, notable interest has been paid out to the use of Fe⁰ for the removal of Cr(VI) from wastewaters [16]. One of the most important issues of any treatment technology is the resulted waste from the specific process.

This paper proposes an alternative way to recycle the exhausted reactive mixtures resulted from the removal of Cr(VI) with Fe⁰ in continuous flow system (column experiments), as precursor for glass based stoneware. The column filling used for the removal of Cr(VI) was composed of Fe⁰ and sand, in based on recent theoretical studies indicating that mixing Fe⁰ with non-expansive materials will sustain the Fe⁰ - systems efficiency filter [17].

Glass based stoneware are suitable materials for toxic elements immobilization based on the double barrier protection. The resulted physical and chemical properties of the composite matrix is difficult to obtain for matrices based only on glass or only on ceramic [18, 19].

By using waste glasses as precursors for the glass based stoneware synthesis there are multiple economic and environmental advantages by conserving the natural resources, saving important quantities of raw materials and decreasing the specific energy consumption and the CO₂ emissions [20]. The glass recycling process has its major drawback due to the expenses of the separation process of the glass scraps from other materials, especially for the cathode ray tubes (CRT) wastes, containing large amounts of heavy metals [21, 22].

Converting wastes to new, marketable products such as glass-ceramics [23], foam glasses [24, 25], bulk glasses [26, 27], glazes [28], packing materials in fractionation columns [29], glass materials for heat transfer processes [30], fillers in bubble columns for polymerization or esterification processes [31, 32], etc. is considered a promising method of protecting the environment and promoting sustainable development.

2. Experimental Procedure

2.1. Sample preparation

The inertization of the exhausted reactive mixtures was realized using kaolin together with two types of common waste glasses: window panes and cathode ray tubes (CRT).

The exhausted reactive mixtures resulted from the removal of Cr(VI) with Fe⁰ in continuous-flow column experiments carried out in batch system. The composition and the corresponding retained chromium for the exhausted reactive mixtures are presented elsewhere [1]. The main components of this waste are hexavalent chromium, iron and sand particles having different sizes.

The Bojidar kaolin, with a known composition [33] has two main roles: binder in the shaping phase and structure former for the glass based stoneware matrix.

The compositions of the precursor waste glasses, determined by X ray fluorescence using a Niton XL 3 analyzer, are presented in Table 1

In order to be used as glass precursor, the two waste glasses were grinded (granulometric fraction under 0.1 mm), dried and sieved.

The exhausted reactive mixtures were dried at 105°C for 24 hours and then mixed together with the kaolin and the waste glass precursors, the batch composition being presented in Table 2.

Table 1

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

Oxide	SiO ₂	Na ₂ O	K ₂ O	CaO	MgO	BaO	PbO	Al ₂ O ₃	Fe ₂ O ₃
Window pane waste glass	71.86	13.13	0.02	9.23	5.64	-	-	0.08	0.04
CRT waste glass	60.92	8.96	7.44	0.67	0.14	10.80	8.85	2.07	0.15

Table 2

Batch composition of the studied materials (weight ratio) / Compoziția amestecului de materii prime [raport gravimetric]

Sample	Column	Bojidar Kaolin	Exhausted reactive mixture	Glass waste	
				Window pane	CRT
I.1G-C	I-1	1	1	1	-
I.2G-C	I-2	1	1	1	-
I.3G-C	I-3	1	1	1	-
I.4G-C	I-4	1	1	1	-
I.5G-C	I-5	1	1	1	-
I.1C-C	I-1	1	1	-	1
I.2C-C	I-2	1	1	-	1
I.3C-C	I-3	1	1	-	1
I.4C-C	I-4	1	1	-	1
I.5C-C	I-5	1	1	-	1

The raw materials were mixed together and pressed into cylinders having the diameter and height around 35 mm using a uniaxial Greisinger Electronics M.P 150 D press using 6 tf.

Two different exhausted reactive mixtures:kaolin:waste glass ratios were initially considered: 1:1:1 and 1:1:2. The glass based stoneware samples obtained using the last ratio, after the thermal treatment presented important dimensional and shape deviations, due to the high amount of liquid phase generated by the large glass quantity and therefore they were excluded from the further investigations.

Based on the previous data, conducting the heat treatment at temperatures lower than 1000°C leads to samples having high apparent porosity and consequently low immobilization capabilities for chromium. Therefore the thermal treatment, optimized considering the economically advantageous conditions, was conducted at 1000°C in order to assure a fluid melt generated from the glass precursors, able to fill the pores in the ceramic matrix.

2.2 Characterization methods

The apparent porosity of the obtained samples was measured using the liquid saturation method under vacuum with water as working liquid.

The microporous structure of the obtained materials was analyzed by SEM, using an Quanta FEG 250 microscope.

The phase composition of the studied glass samples was determined using a Rigaku Ultima 4 diffractometer.

The chemical stability of the samples was investigated by measuring the dissolution rate of the samples immersed in three extraction mediums having pH 5.5, 7.0 and 8.5 respectively for 28 days. The buffer solutions from 5.5, 7.0 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). The volume of the utilized solutions of 100 mL was maintained constant during the considered determination time at a constant temperature of 20 ± 2°C. After 28 days the samples were dried for 6 hours at 110°C until they reach constant mass. The dissolution rate of the glass samples is expressed as weight loss in time as it is presented by the following equation:

$$Dr = \frac{\Delta m}{t} = \frac{m_i - m_f}{t} \quad [\mu\text{g/h}]$$

where: m_i is the initial sample mass, m_f the final sample mass and t represents the considered experimental time of 28 days.

The chromium and iron immobilization capacity of the studied glasses was investigated by

measuring the chromium ions extraction using leaching tests performed according to the American Extraction Procedure Toxicity Test [34]. Three extraction mediums having pH 5.5, 7.0 and 8.5 respectively were used, analysis being performed after 1, 14 and 28 days. Two grams of each sample were taken and shaken with 250 ml of ammonia–acetate buffer solution for different time periods at a constant temperature of 20 ± 2°C. The chromium concentration in the extraction mediums was measured using a Bruker Aurora M90 Inductively Coupled Plasma Mass Spectrometer.

3. Results and Discussion

3.1. Samples compactness

The compactness of the obtained samples, characterized using the apparent porosity is presented in Table 3.

Table 3
Apparent porosity of the obtained samples [%]
Porozitatea aparentă a probelor sintetizate [%]

Sample	P _{ap} [%]	Sample	P _{ap} [%]
I.1G-C	2.55	I.1C-C	2.20
I.2G-C	2.85	I.2C-C	2.70
I.3G-C	3.35	I.3C-C	3.15
I.4G-C	4.45	I.4C-C	4.05
I.5G-C	5.05	I.5C-C	4.65

The values for the apparent porosity range from 2.20% to 5.05% depending on the glass waste type. The CRT glass, more fluid than the window panes waste glass due to its composition, generates higher amounts of liquid phase at the heat treatment temperature, able to fill the available pores, and a lower porosity for the corresponding glass based stoneware samples. This behavior is confirmed by the SEM images obtained using a Quanta FEG 250 microscope, presented in the Figure 1 for two representative samples.

Both samples present a microporous structure characterized by a relatively uniform distribution of the pores in the ceramic matrix with a narrow dimensional spectra, all pores having micrometric dimensions.

The influence of the sand upon the apparent porosity of the ceramic samples is presented in the Figure 2.

For both sets the apparent porosity decrease when the sand amount raise due to the replacement of a porogen material - the kaolin by a non-porous one - the sand.

3.2. Phase composition

The main crystalline phases confirmed by the X ray diffraction spectra presented in Figure 3 are cristobalite, tridymite, wollastonite, diopside and hematite.

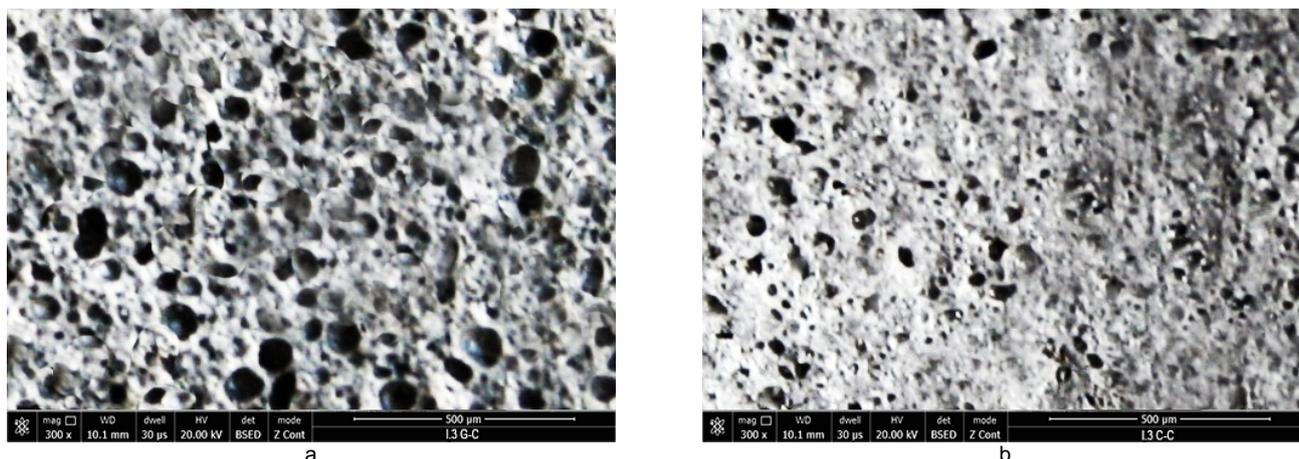


Fig. 1 - Microporous structure of a - I.3G-C and b - I.3C-C samples / Structura microporoasă a probelor a - I.3G-C și b - I.3C-C.

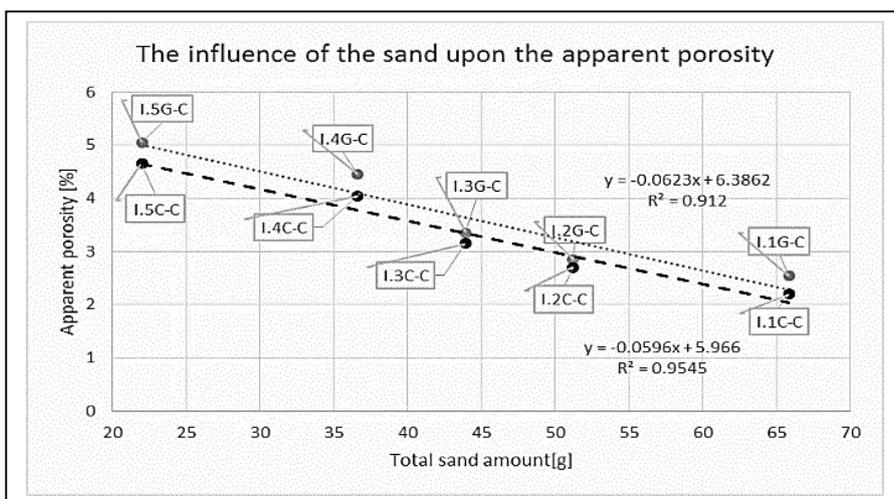


Fig. 2 - Influence of the sand brought by the exhausted reactive mixtures upon the apparent porosity of the samples / Influența nisipului adus cu amestecul reactiv epuizat asupra porozității aparente a probelor.

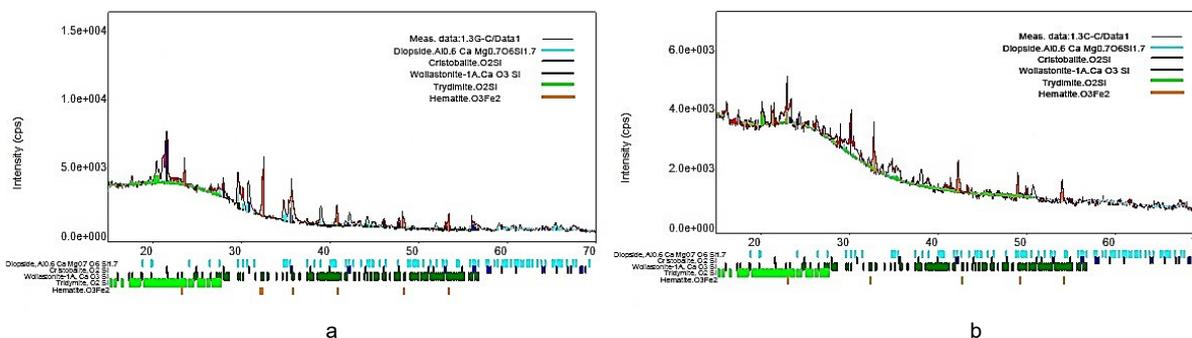


Fig. 3 - Diffraction spectra of the samples a - I.3G-C and b - I.3C-C / Spectrele de difracție ale probelor a - I.3G-C și b - I.3C-C.

The cristobalite is generated by the recrystallization process of the amorphous SiO₂ while the tridymite is generated by the sand. The presence of hematite can be assigned to the oxidation during the heat treatment of the complex iron mixed hydroxides and oxyhydroxides of Fe(III) precipitated in the columns [35]. The diopside is generated by the reaction of the amorphous SiO₂ resulted from the thermal decomposition of the kaolinite with CaO and MgO from the glass melt.

3.3 Glass based stoneware chemical stability

The values for the chemical stability,

measured by the samples' dissolution rate are summarized in the Table 4.

The influence of the apparent porosity upon the samples' chemical stability is presented in the Figure 4.

The increase of the apparent porosity leads to a rise of the dissolution rate values and a reduction of the chemical stability for all the investigated samples due to the higher specific surface exposed to the chemical aggression. As it can be observed in the Figure 4, all the dependences are quasilinear, the correlation coefficient R² ranging from 0.9439 to 0.9958.

Table 4

Dissolution rate values [μg/h] for the studied samples / Gradul de solubilizare [μg/h] pentru probele sintetizate

Glass based stoneware samples having windows panes waste glasses as precursor				Glass based stoneware samples having CRT waste glasses as precursor			
Sample	D _r [μg/h]			Sample	D _r [μg/h]		
	pH=5.5	pH=7.0	pH=8.5		pH=5.5	pH=7.0	pH=8.5
I.1G-C	0.144	0.138	0.425	I.1C-C	0.132	0.127	0.311
I.2G-C	0.151	0.148	0.468	I.2C-C	0.14	0.133	0.382
I.3G-C	0.162	0.158	0.521	I.3C-C	0.155	0.153	0.475
I.4G-C	0.172	0.172	0.665	I.4C-C	0.165	0.159	0.589
I.5G-C	0.189	0.185	0.778	I.5C-C	0.177	0.175	0.688

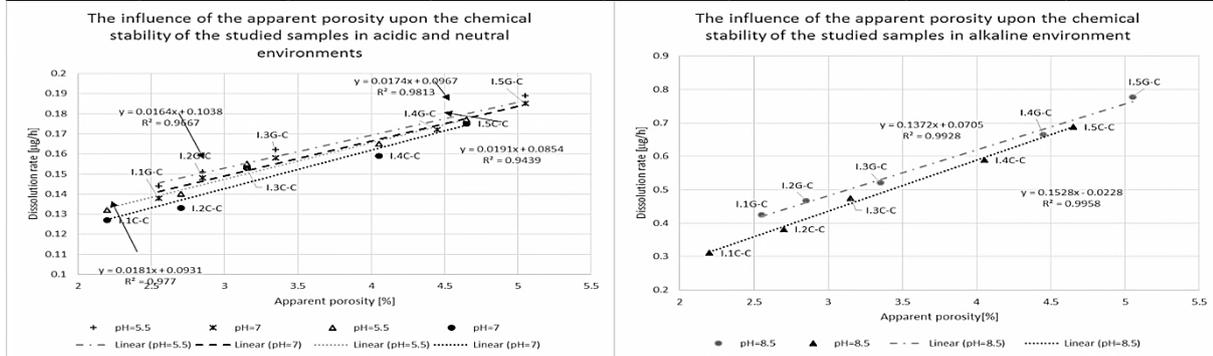


Fig. 4 - Apparent porosity influence upon the chemical stability of the samples / Influența porozității aparente asupra stabilității chimice a probelor sintetizate.

3.4. Chromium and iron ions immobilization in the glass based stoneware matrices

The chromium and iron immobilization capacity of the studied samples were measured according to the American Extraction Procedure Toxicity Test as it was presented elsewhere [1].

The lixiviation values calculated as percentage of chromium leached from the total amount introduced by the exhausted reactive mixtures are illustrated in the Figure 5.

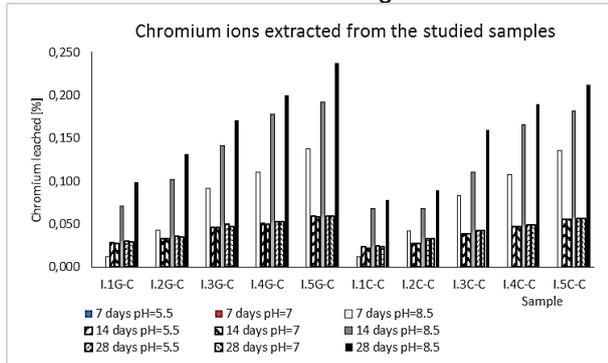


Fig. 5 - Chromium ions losses from the studied samples / Pierderile de ioni de crom din masele studiate.

The chromium ions lixiviation values from all the investigated samples, regardless the environment's pH and the considered time are very low, ranging between 0-0.238% of the total chromium brought by the exhausted reactive mixtures. The samples show no discernable losses of chromium after 7 days at pH=5.5 and pH=7.0. The matrices presents a higher sensibility to alkaline aggression manifested as low chromium losses.

All the obtained samples show a better

chromium immobilization in acid and neutral environments compared to that in alkaline environment, due to the passivation effect of the SiO₂ enriched materials' surface.

The CRT waste glass as precursor leads to lesser chromium losses based on lower porosity of the matrices caused by the higher fluidity of these glass melt compared to the one generated by the windows panes wastes.

The described behavior for the chromium losses in all the studied samples suggests that the chromium ions are immobilized by the glass phase, more sensible to the alkaline aggression comparing to the ceramic phases.

The iron extracted from the investigated samples, expressed as percentage of iron ions leached from the total iron introduced by the exhausted reactive mixtures are illustrated in the Figure 6.

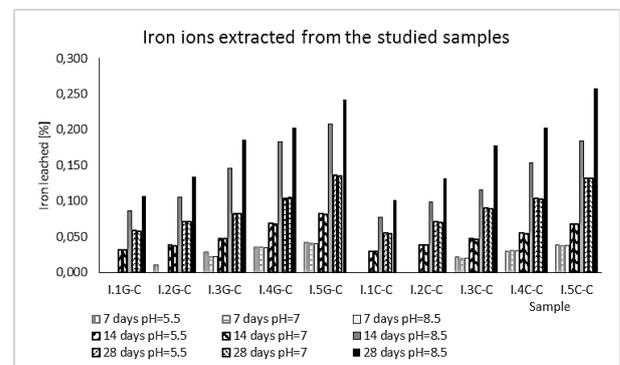


Fig. 6 - Iron ions losses from the studied samples / Pierderile de ioni de fier din masele studiate.

The iron ions losses from all the investigated samples regardless the environment's pH and the considered time are very low, ranging between 0-0.258%.

The sample I.1G-C, I.1C-C and I.2C-C show a perfect immobilization of the iron ions after 7 days in acidic and neutral mediums. Increasing the iron content and simultaneously decreasing the sand amount in the exhausted reactive mixtures leads to a lower encapsulation of the hematite in the matrices and therefore a higher sensitivity to chemical aggression.

The passivation effect caused by the leaching of the alkali oxides from the samples' surface at lower pH values generates a better iron immobilization towards acid and neutral chemical attack.

4. Conclusions

This study proposes an alternative solution to recycle the exhausted reactive mixtures resulted from the removal of Cr(VI) with Fe⁰ in continuous flow system (column experiments), together with two common waste glasses as glass based stoneware materials.

The compactness of the obtained samples, characterized using the apparent porosity, range between 2.20% to 5.05% depending on the glass waste type used as precursor. The more fusible CRT glass generates larger amounts of liquid phase at the heat treatment temperature, that fill the available pores, generating lower porosities for the corresponding samples. The influence of the sand/kaolin ratio upon the apparent porosity of the ceramic samples show a quasilinear behavior for both sets of samples.

The main crystalline phases, confirmed by the X-ray diffraction, are cristobalite, tridymite, wollastonite, diopside and hematite.

The chemical stability of the investigated glass based stoneware, measured by the samples' dissolution rate range from 0.127 to 0.778 μg/h. A quasilinear dependence on the apparent porosity was highlighted due to the higher specific surface exposed to the chemical aggression.

The synthesized materials present a very good chromium immobilization. Only the alkaline environment generates low chromium extraction, less than 0.238% after 28 days. The CRT waste glass is more favorable as precursor compared to the window pane recycled glass based on its effect upon the apparent porosity of the structure.

The general behavior of the investigated samples towards chemical aggression suggests that chromium ions are mainly located in the glass phase, more sensitive to the alkaline attack comparing to the ceramic phases.

The investigated samples show a good iron immobilization regardless the environment's pH, the lixiviation values after 28 days ranging

between 0-0.258%. Increasing the ratio Fe⁰/sand particles in the exhausted reactive mixtures generates a lower encapsulation of the hematite in the matrices and therefore a higher sensitivity to chemical aggression.

The obtained results confirm the viability of the proposed solution for immobilizing chromium contained in the exhausted reactive mixtures together with common waste glasses in glass based stoneware materials having good chemical stability with multiple economic advantages.

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Secția de științe medicale

Secția de Științe chimice

Secția de științe economice

REINDUSTRIALIZAREA ROMÂNIEI A DOUA DEZBATERE NAȚIONALĂ DEDICATĂ CENTENARULUI MARI UNIRI,

a avut loc în Aula Academiei Române, în ziua de 8 iunie 2018

Au fost prezentate lucrările:

Acad. VICTOR VOICU, Vicepreședinte al Academiei Române – *Cuvânt de deschidere*

Prof. PETRE T. FRANGOPOL, Membru de onoare al Academiei Române

Importanța Educației și cercetării în reindustrializarea României

Prof. univ. dr. ing. GHEORGHE IVĂNUȘ

Efectele benefice ale chimiei petrolului și gazelor naturale

Acad. NICOLAE A. ANASTASIU

Resursele minerale ale României, bogății nevalorificate

Prof. univ. dr. TUDOREL ANDREI

Comerțul exterior al României cu produsele românești

Dr. Ing. DORU PUȘCAȘU

Reindustrializarea domeniului materialelor oxidice pentru construcții din România
