PROCESE DE ÎNTĂRIRE ŞI PRODUŞI DE REACŢIE ÎN SISTEME LIANTE DE TIP ZGURĂ ACTIVATĂ ALCALIN ŞI GEOPOLIMER, CU CONŢINUT DE Pb▲

HARDENING PROCESSES AND HYDRATES IN ALKALI-ACTIVATED SLAG AND GEOPOLYMER WITH Pb CONTENT

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Beside traditionali binders – Portland cement and blended cements, the binders of type alkali activated slag (AAS) and geopolymer can represent efficient matrices for immobilization of noxious substances.

The present paper brings informations regarding the phisical and chemical processes at the hardening of alkali-activated slag and geopolymer, in which a waste with Pb content was immobilised. Geopolymer binder was prepared using a fly ash and as activator – a mixture of $Na_2O.nSiO_2$ and NaOH with silica modulus of 1.12. Alkali activated slag binders,were prepared with the same alkali-activator.

This type of binders have a good immobilization capacity of the glass waste resulted from discarded cathode ray tube (CRT) with Pb content, added in amounts corresponding to 0.18-10% Pb. The inclusion of Pb^{2+} ions into the binding systems determines a certain retarding influence on the hardening processes, the effect being more intense for shorter periods of hardening and for higher amounts of Pb. The X-ray difraction data sugests the formation of a complex aluminosilicate with Pb content.

Pe lângă lianții tradiționali – ciment portland, cimenturi mixte, lianții cu activare alcalină, fără clincher portland, de tipul LZA (liant de zgură activată alcalin) şi de tip geopolimeri pot constitui matrici eficiente pentru imobilizarea unor substanțe nocive.

Lucrarea prezentă aduce informații referitoare la procesele fizico-chimice care au loc la întărirea unor sisteme liante de tip LZA şi respectiv, geopolimer, în care s-a imobilizat un deşeu cu conținut de Pb. Liantul de tip geopolimer s-a preparat din cenuşă de termocentrală, utilizând ca activator alcalin, un amestec de $Na_2O.nSiO_2$ şi NaOH, cu un modul n al silicatului, egal cu 1,12. Acelaşi activator a fost utilizat şi pentru realizarea lianților de zgură activată alcalin.

Lianții de acest tip au dovedit o foarte bună capacitate de imobilizare a unui deşeu de sticlă rezultat din tuburile catodice scoase din utilizare (CRT) cu conținut de Pb, considerat în lianți, în proporții corespunzătoare la 0,18-10% Pb. Înglobarea Pb în sistemele liante determină o anumită frânare a proceselor de interacție, efectul fiind mai accentuat pentru perioade scurte de întărire şi proporții mai mari de Pb. Datele analizelor difractometrice sugerează formarea în unele compoziții, a unui compus complex cu Pb – hidroaluminosilicat de plumb.

Keywords: *Alkali-activated slag, geopolymer, CRT waste, reaction products, hardening processes*

1. Introduction

As a result of high energy consumption required for traditional binders manufacture based on Portland clinker (about 3000 kJ / kg), and of negative implications on the environment (the increase of greenhouse gas) [1, 2], the binders containing industrial byproducts such as slag or fly ash, or less expensive raw materials, became an objective of interest in developing of sustainable technologies, without adverse impact on the environment.

Alkali-activated slag and geopolymers prepared from industrial byproducts such as slag or fly ashes, beside the very good mechanical properties that they can develop, present a high capacity of wastes immobilization. Many researches conducted on this subject, have reported good results on the immobilization of hazardous waste with heavy metals (Cu, Zn, Cd, Cr, Pb, Ni) content, in this type of binder matrices [3 - 10].

The efficiency of waste immobilization into the hardening structure of alkali-activated slag and geopolymer type of binders depends essentially on porosity and pH of the binding matrix [2, 11]; regarding the immobilization mechanism in geopolymers, several opinions were formulated [12 - 14]:

i) metal ions are bound into geopolymers network to balance the charges;

ii) the precipitation of cations as insoluble compounds (hydroxides, silicate hydrates) may occur, followed by the physically encapsulation of its into the hardening network.

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According to literature data [14-16], the addition of more than 3% Pb^{2+} as $Pb(NO_3)_2$ in fly ash based geopolymers leads to the formation of Pb_3SiO_5, indentified by X-ray diffraction analysis at 2θ values of 29.10^0 and 33.20^0; for smaller lead amount (below 0.5% Pb^{2+}), crystalline compounds containing Pb were not identified. In alkali-activated slag binders, even 3% Pb^{2+} (introduced in the system as $Pb(NO_3)_2$) did not leads to the formation of crystalline compounds containing Pb.

The present paper brings information regarding the hardening processes and reaction products in alkali-activated slag and geopolymer, in which the active component of the system (slag or fly ash) was substituted up to 22%, with CRT glass waste. The formation of reaction products, was studied by X-ray diffraction analysis, FTIR spectroscopy and scanning electron microscopy (SEM)

2. Experimental

2.1. Materials

The materials used in experiments were: a type F fly ash (Deva), a type C fly ash (Arad) and a granulated blast furnace slag (GBFS). Compositional characteristics and the grinding finesses are shown in Table 1.

The mineralogical composotion of these materials was assessed by X-ray diffraction (Fig.1): mulite, hematite, quartz and anortite - in fly ashes and melilites, dicalcium silicate and merwinite - in GBFS.

The CRT glass waste with Pb content of 15.45%, was used also in previous researches that focused on it immobilization in binder matrices type of Portland cement, slag cement and alkali-activated slag [7], was obtained by fine grinding (up to specific surface area Ssp = 3010 cm^2/g) of a mixture consisting of glass from discarded monitors and TV sets. The X-ray diffaction data of the waste, shows a halo characteristic for the vitrous structure of glass, on the entire 2θ domain (Figure 1).

Alkali activator was a mixture of NaSil/NaOH solution with SiO_2/Na_2O ratio of 1.12. The composition of the binding systems studied is shown in Table 2. In the geopolymer binders group, two reference compositions – G(A), based on Arad fly ash and G(D), based on Deva fly ash were prepared and studied. Pb from CRT glass waste, in small amounts, 0.18 – 0.74%, was added in fly ash C (Arad) based geopolymer (G 0.18– G 0.74), and in higher amounts 2 – 10%, it was added in fly ash F (Deva) based geopolymer (G 2– G 10) - Table 2.

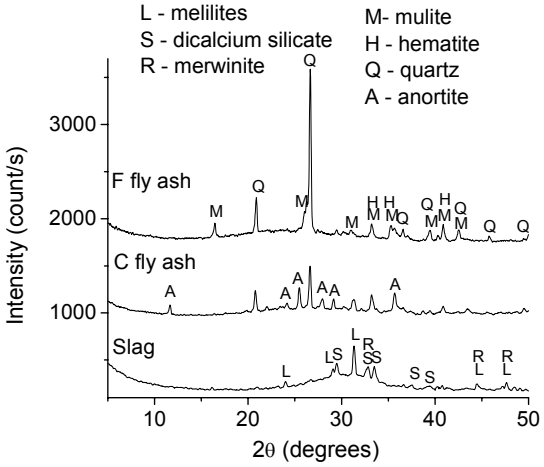


Fig.1 - X-ray patterns of the solid materials/ *Difractograme ale materialelor folosite.*

2.2. Methods

For investigations regarding to the reaction products resulted in the hardening processes in the studied systems, pastes with compositional characteristics corresponding to table 2, were prepared and thermally treated at 60^0C, for 24 hours, in covered molds after that, for the next 27 days, the geopolymer binders were kept in air, at room temperature (20 ± 2^oC) and the LZA were kept in humid air (R.H= 95%), at room temperature. After 1 day, 7 and 28 days, the

Table 1

Compositional and fineness characteristics of the used materials / *Caracteristici compoziţionale şi de finețe ale materialelor utilizate*

Chemical compounds *Componenți chimici* (%)	Granulated blast furnace slag (GBFS) *Zgură granulată de furnal*	Deva fly ash *Cenuşă de Deva*	Arad fly ash *Cenuşă de Arad*
Loss on ignition *Pierdere prin calcinare*	3.76	2.69	6.03
SiO_2	35.43	51.5	40.41
Al_2O_3	11.25	23.68	19.02
Fe_2O_3	0.60	8.93	13.82
CaO	42.85	6.69	12.5
MgO	3.997	1.99	2.59
SO_3	0.09	1.10	5.43
Minor compounds *Compuşi minori*	1.86	2.68	-
Specific surface area *Suprafaţa specifică* (cm^2/g)	2441	3022	3617

Tabele 2

Binding pastes compositions / *Compoziții ale pastelor liante*

Binder indicative *Indicativ liant*	Solid components / *Componenți solizi* (%)					Liquid *Lichid*	Liquid/solid ratio** *Raport lichid/solid*	Pb brought by waste *Pb adus de deşeu* (%)
	Arad fly ash *Cenuşă de Arad*	Deva fly ash *Cenuşă de Deva*	GBFS	Glass waste *Deşeu de sticlă*	NaOH			
G(A)	54	-	-	-	8.6	37.4% NaSil*	0.45	-
G 0.18	53.4	-	-	0.6	8.6	37.4% NaSil*	0.45	0.18
G 0.37	52.7	-	-	1.3	8.6	37.4% NaSil*	0.45	0.37
G 0.74	51.5	-	-	2.5	8.6	37.4% NaSil*	0.45	0.74
G(D)	-	54	-	-	8.6	37.4% NaSil*	0.45	-
G 2	-	47.7	-	6.3	8.6	37.4% NaSil*	0.45	2
G 4	-	42.6	-	11.4	8.6	37.4% NaSil*	0.45	4
G 10	-	32	-	22	8.6	37.4% NaSil*	0.45	10
LZA	-	-	54	-	8.6	37.4% NaSil*	0.45	-
LZA 0.18	-	-	53.4	0.6	8.6	37.4% NaSil*	0.45	0.18
LZA 0.37	-	-	52.7	1.3	8.6	37.4% NaSil*	0.45	0.37
LZA 0.74	-	-	51.5	2.5	8.6	37.4% NaSil*	0.45	0.74
LZA 2	-	-	47.7	6.3	8.6	37.4% NaSil*	0.45	2
LZA 4	-	-	42.6	11.4	8.6	37.4% NaSil*	0.45	4
LZA 10	-	-	32	22	8.6	37.4% NaSil*	0.45	10

*- activator concentration reported to binding mixture, reprezenting Na_2O in NaSil+NaOH mixture, was 15.5% / *concentrația activatorului raportat la amestecul liant, reprezentand Na_2O în amestecul NaSil+NaOH, a fost 15.5%*

** - the liquid has been consisting by the water brough by NaSil solution and the water supplementary added for preparing the pastes; the solid represented the fly ash/slag, NaOH and solid NaSil / *lichidul a constat în apa adusă de soluția de NaSil şi apa adăugată suplimentar la prepararea pastelor; solidul a constat din cenuşă/zgură de furnal, NaOH şi NaSil solid*

hydration was stopped and the powder samples resulted by the fine grinding were analysed by:

- X-ray diffraction, using a X-ray diffractometer type of Shimadzu XRD 6000;
- FT IR spectroscopy, using a spectrophotometer type of ShimadzuFTIR 8400;
- scanning electron microscopy (SEM), using a HITACHI S2600N microscope.

3. Results and discussions

X-ray diffraction analyses of LZA binders are presented in Figure 2.

The data suggests, in the initial heat treatment conditions, a intense hydration of slag in absence of Pb (LZA), which developped in time, up to 28 days (Fig. 2a). The peaks in 2θ domain at 28-32°, corresponding to the silicate hydrates with calcium and magnezium - $(C,M)_xSH_y$ and silicate hydrates with calcium and sodium - $(C\ N)_nSH_y$, with a higher crystalisation degree, explained by the formation under initially heat treatment conditions, sustain this ideea.

Addition of 0.18 (even up to 4%) Pb, brought by CRT glass waste, did not affected the slag hydration. The intensities of peaks in 2θ domain above mentioned, corresponding to the silicate hydrate with calcium and magnesium / sodium, confirm the slag hydration. Some low intensity peaks occured at 2θ values of 12.6° and 24°, on LZA 0.18 diffraction spectrum (Fig. 2b), could suggest the presence of hydrotalcite ($Mg_6Al_2CO_3OH_{16}{\cdot}4H_2O$) and analcime ($Na(Si_2AlO_6)H_2O$) type of hydrates [17]. The increase of Pb proportion up to 10%, corresponding to 22% CRT waste of total mass - LZA 10 (see Table 2), determines a retardation of hydration processes; the smaller intensity peaks in 2θ domain at 28-32° (Fig. 2c), sustain this ideea. For a longer period of hardening (28 days), the retardation influence on hydration processes is attenuate; the hydrates evolves quantititatively and structurally, as is suggested by the diffraction data for this period, in the Figure 2c compared with Figure 2a.

The diffraction data of geopolymer binders whitout/with CRT glass waste content, are shown in Figures 3 and 4. For geopolymer binders based on Arad and Deva fly ashes, whitout CRT waste, G(A) and G(D), the reaction products formed, with a low degree of crystallization, are sodium silicate hydrates – hydroxisodalite (the peaks at 2θ value of 24^0, 31.8^0, 36.8^0) - Figs. 3a and 4a, with the mention that for C fly ash based geopolymer, the hydroxisodalite is identified after 28 days only. This could be explain by the lower reactivity of this fly ash, compared with C fly ash (Arad), correlated

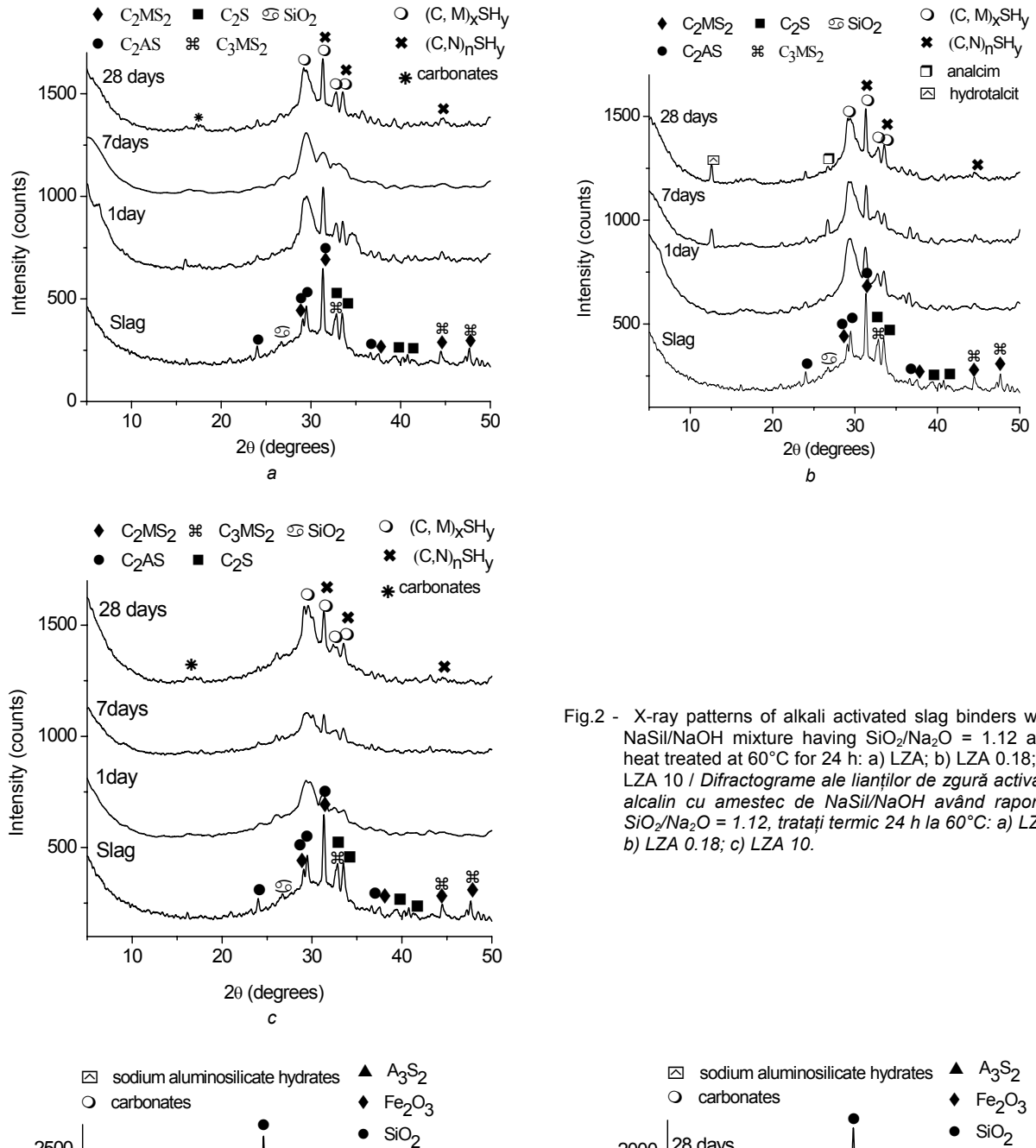


Fig.2 - X-ray patterns of alkali activated slag binders with NaSil/NaOH mixture having SiO_2/Na_2O = 1.12 and heat treated at 60°C for 24 h: a) LZA; b) LZA 0.18; c) LZA 10 / *Difractograme ale lianților de zgură activată alcalin cu amestec de NaSil/NaOH având raportul SiO_2/Na_2O = 1.12, tratați termic 24 h la 60°C: a) LZA; b) LZA 0.18; c) LZA 10.*

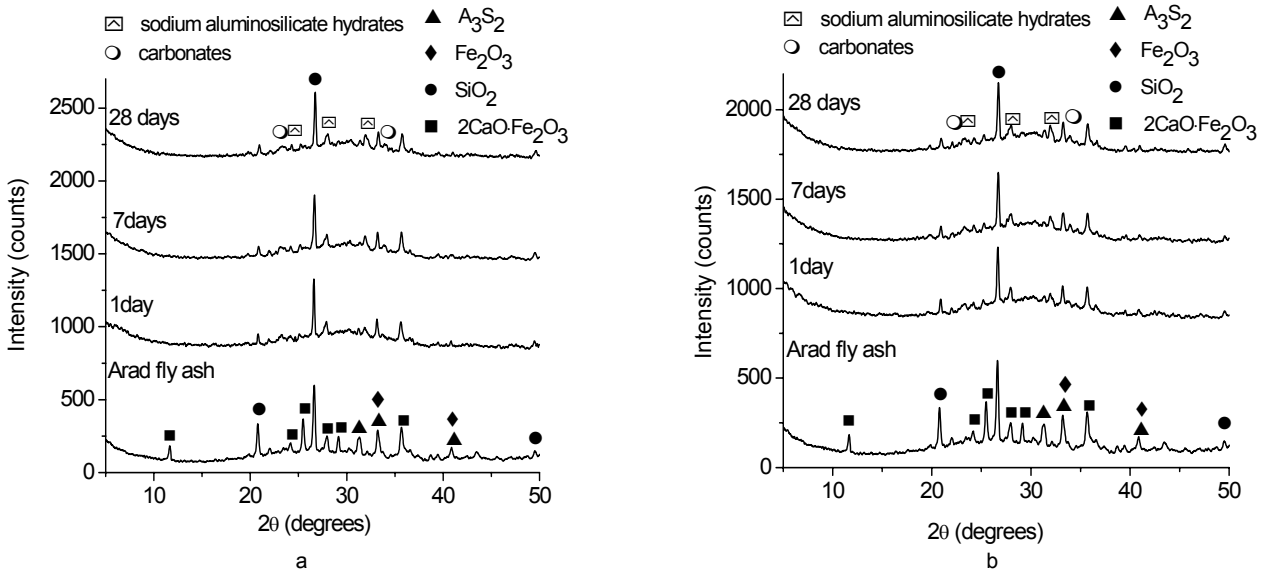


Fig.3 - X-ray patterns of geopolymers based on C fly ash: a) G(A); b) G 0.74 / *Difractograme ale lianților de tip geopolimer obținuți din cenuşă Arad: a) G(A); b) G 0,74.*

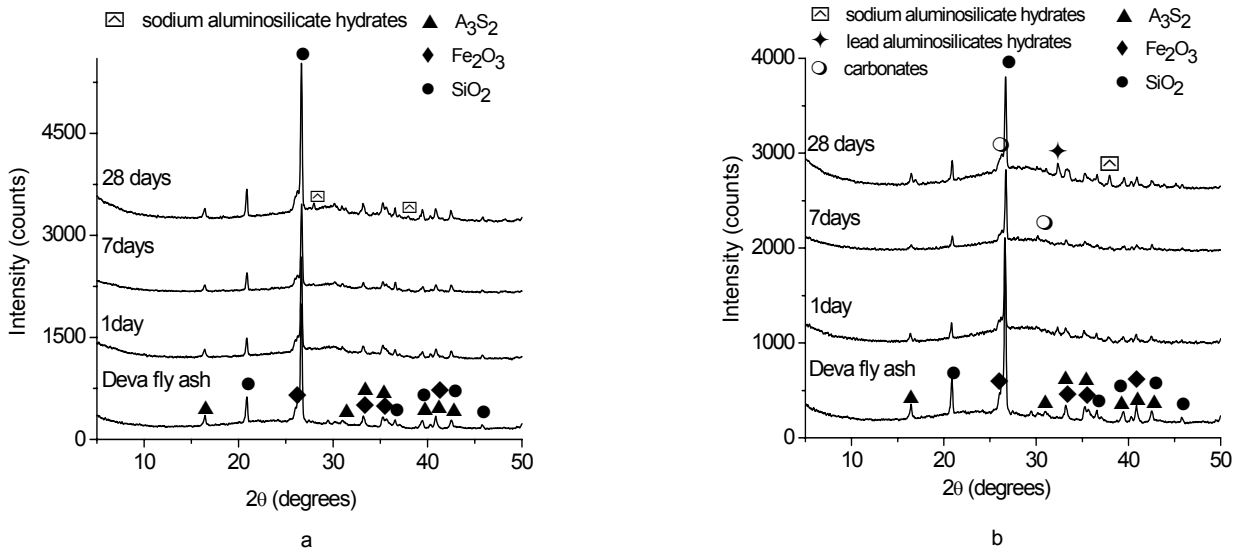


Fig.4 - X-ray patterns of geopolymers based on F fly ash: a) G(D); b) G 10 / *Difractograme ale lianților de tip geopolimer obținuți din cenuşa Deva: a) G(D); b) G 10.*

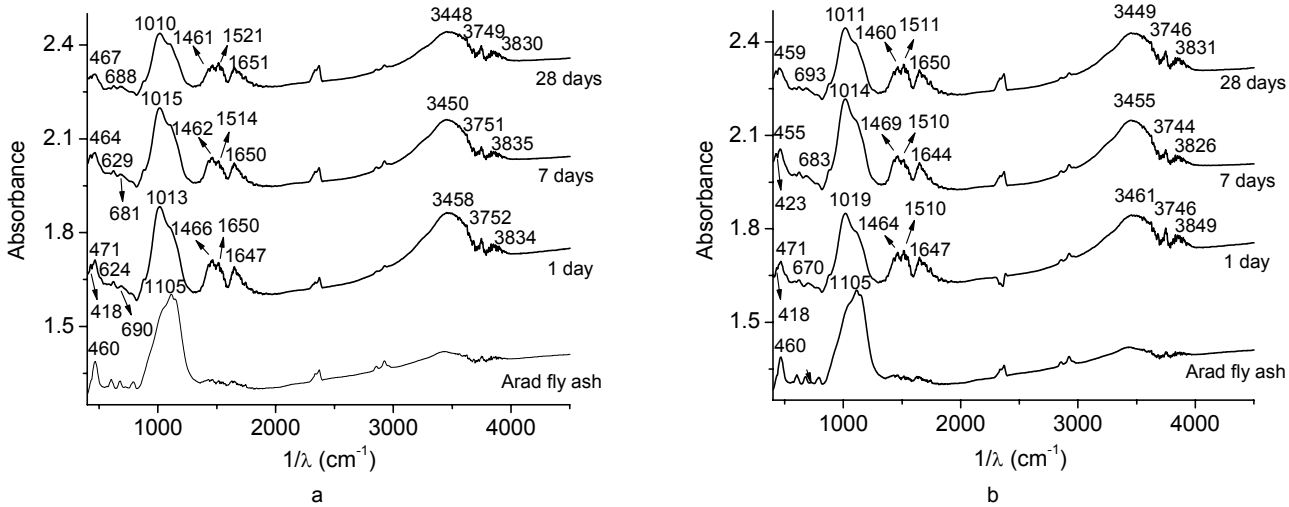


Fig.5. - FTIR spectra of the geopolymers hardened up to 28 days: a) G(A); b) G 0.74/ *Spectre FTIR ale lianților de tip geopolimer după perioade de întărire de până la 28 zile: a) G(A); b) G 0,74.*

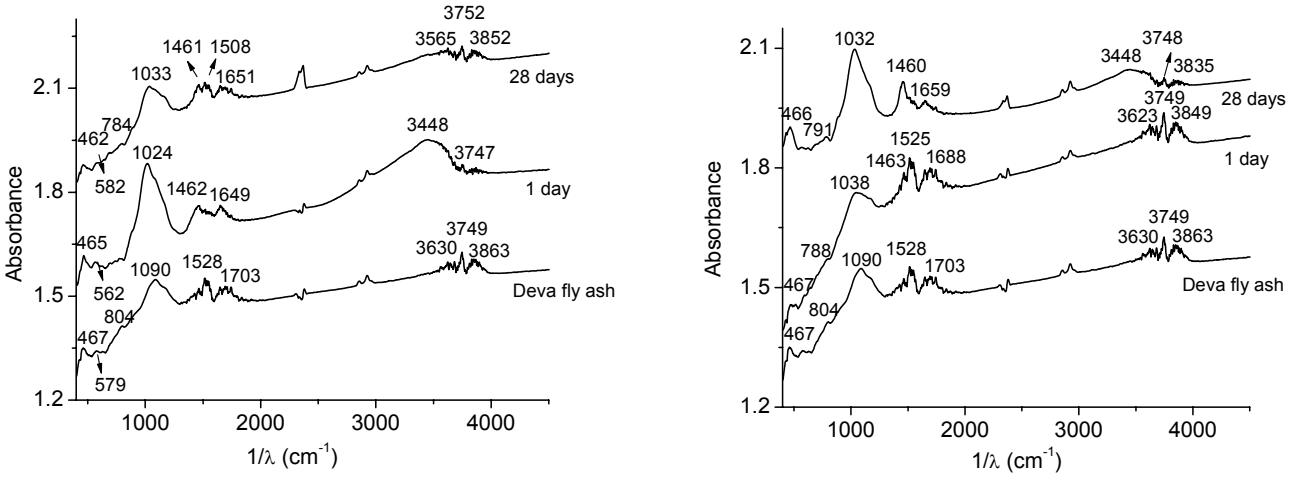


Fig.6 - FTIR spectra of the geopolymers hardened up to 28 days: a) G(D); b) G 10 / *Spectre FTIR ale lianților de tip geopolimer după perioade de întărire de până la 28 zile: a) G(D); b) G 10.*

with theirs structural and compositional characteristics [18].

Addition of 0.74% Pb in G 0.74 binder, obtained from fly ash C, do not cause significant changes in the crystallization degree of the reaction products (Fig. 3b); they remain with low crystallization degrees, the small specific peaks sustaining this ideea. In the fly ash F based geopolymer with 10% Pb content brough by CRT glass waste - G 10 - the formation of a complex compound of Pb i.e. lead aluminosilicate hydrates, their presence is suggested by the low intensity peak at 2θ value of 32.4^0 (Fig. 4b).

FTIR analyses, performed on the geopolymers based on Arad and Deva fly ashes, hardened up to 28 days, are shown in Figures 5 and 6.

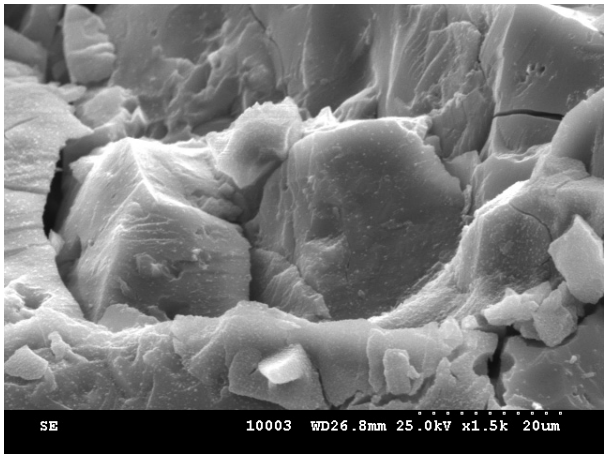
The shifting of some absorbtion bands or appearance of new bands on the geopolymers spectra, compared with fly ashes spectrum, suggests the formation of some typical compounds for geopolymer binders. Thus, the absorption band of fly ashes, at 1090 cm^{-1} (in fly ash F) or at 1105 cm^{-1} (in fly ash C), is shifted, for all periods of time, to smaller wave numbers, i.e. 1010 up to 1038cm^{-1}. This band has been assigned to asymetric stretching of Si-O and Al-O bonds from the structural groups of alumino-silicates [16, 19, 20]. The absorbtion bands between 400 – 420 cm^{-1} have been assigned to vibration bonds of poly(sialate) network, formed around opened pores [20]. The absorbtion band at ~ 460 cm^{-1}, assigned to the in plane deformed of Al-O and Si-O bonds, is well outlined and more widely for fly ashes spectra and is smaller on the geopolymer spectra (Figs. 5a and 6a); this is due to the consumption of silica and alumina in geopolymerization process [21]. The peaks from spectral domain at 1460 – 1520 cm^{-1} are assigned to C-O bonds in sodium (bi)carbonate, which is formed as secondary compound in this binder [21].

The absorption bands at ~ 1647-1680 cm^{-1} are assigned to H-OH vibration bonds; for H-O vibration bonds of H_2O molecules absorbed on the particle's surface or retained within polymeric gel are assigned the absorption bands from 3200-3600 cm^{-1} [21- 23].

The Pb presence (0.74%), brough by CRT glass waste, do not cause important changes in IR spectra of fly ash C based geopolymers (Fig. 5b). Addition of 10% Pb, brought by CRT glass waste, into F fly ash based geopolymer (G10) causes some changes of IR spectra (Fig. 6b), suggesting an influence on the geopolymerization and hardening processes of this type of binders. So, the absorbtion band at ~ 1020 - 1030 cm^{-1} (Figs. 6a, 6b), has a smaller amplitude for binder containing Pb, G 10, which allows to consider the formation of a smaller quantities of geopolymeric compounds; for longer curring periods (up to 28 days, in air, at room temperature) this band is amplified (Fig. 6b), suggesting the continuous formation of the geopolymeric compounds in such conditions. These data are in good correlation with the information provided for this type of binders in an other paper [24].

Scanning electron microscopy (SEM) analysis performed on LZA binders, presented in Figure 7, reveal some morfological characteristics of reaction products formed during the hardening process.

On the Figure 7a, are present plaques, possibly hexagonal, that can be assigned to the hydrogarnet type of compounds with natrium embedding (black marked zone). The presence of calcium and magnesium silicate hydrates in these binders, hardened for 28 days, is suggested, in Figure 7a, by some formations with irregular outline (white marked zone). Often, can be seen also, the presence of micro cracks (marked with arrows) possibly caused by the high reaction kinetics correlated with the initial heat treatment at 60^0C for 24 hours [4].



a

b

Fig.7 - SEM micrographs of alkali activated binder: LZA (a) and LZA 10 (b), after 28 days of hardening / *Imagini SEM realizate pe liantul de zgură activată alcalin: LZA (a) şi LZA 10 (b) după 28 zile de întărire*.

For the binder with 10% Pb, brought by CRT waste, LZA10, the SEM micrograph in Figure 7b reveals the presence of some glass grain (C) embedded in the geopolymer matrix (B), and the presence of prismatic stretched formations (A), suggesting the presence of some natrium carbonates (Na_2CO_3 or $NaHCO_3$), identified also, on XRD spectra.

In Figure 8 is shown the SEM micrograph of the fly ash C based geopolymer, with 0.74% Pb (G 0.74).

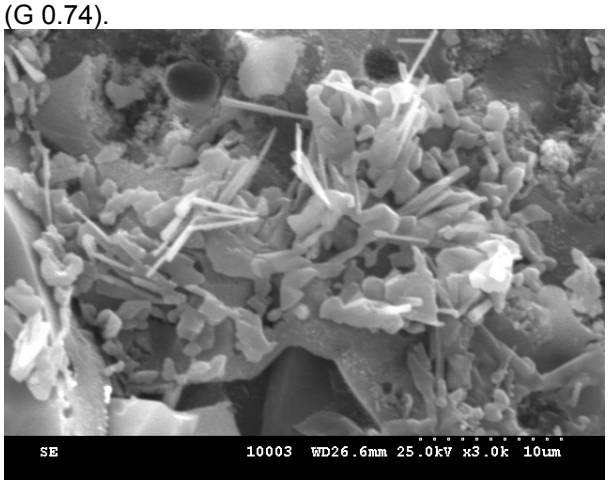


Fig.8 - SEM micrograph of G 0.74 geopolymer, hardened up to 28 days / *Imagine SEM a liantului de tip geopolimer G 0,74, întărit 28 zile*.

This image reveals spherical cavities in which, the fly ash grains were, before specimens fracture, surrounded by an amorphous phase. This phase can be formed by the alkaline aluminous–silicate hydrates, type of zeolite. The prismatic crystallized formations can be Na_2CO_3 or $NaHCO_3$, compounds identified also on XRD patterns.

4. Conclusions

The hardening of alkali-activated slag and geopolymer is determined by the hydration and geopolymerization processes (respectively), intensified in both binding systems by the initial thermal treatment at 60^0C.

The XRD and SEM analyses suggests the formation in the LZA systems of the following hydrates: silicate hydrates with calcium and magnesium - $(C, M)_xSH_y$ and silicate hydrates with calcium and sodium - $(C, N)_nSH_y$.

The main reaction product formed in geopolymers is a gel; sodium alumino-silicate hydrates (hydroxisodalite) was detected also by XRD analyses.

The influence of Pb brought by CRT glass waste on LZA and geopolymer binders depends on the nature of the binder and the Pb amount considered:

- small amounts of Pb (0.18%) does not affect the hydration process of slag; the peaks at 2θ value between 28 - 32°, corresponding to magnesium/natrium calcium silicate hydrates sustain this ideea; the small peaks at 2θ value of 12.6° and 24°, on the LZA 0.18 pattern, suggest the formation of hydrotalcite and analcime;
- high amounts of Pb (10%) caused a slower hydration of slag, even under the initial heat treatment conditions; the peak's intensity of the specific hydrates are lower but, in time, up to 28 days, this effect is attenuated.

The FTIR spectra of geopolymer type binder with 10% Pb content, suggests, by the smaller amplitudes of the characteristic bands, the formation of smaller quantities of geopolymeric compounds, as result of a retarding effect of Pb on the hardening processes.

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MANIFESTĂRI ŞTIINȚIFICE / SCIENTIFIC EVENTS

ICCS13 Intl Congress on Concrete Sustainability
27-29 May 2013, Tokyo, Japan

TOPICS:

1) Environmental impact reduction technologies

- Materials, - Construction, - Repair & rehabilitation, - Demolition, - Reuse and recycling, - CO_2-uptake
- Thermal mass, - Environmental engineering structures, - CO_2 capturing

2) Sustainability aspects in durability

3) Environmental design, evaluation, and systems

- Design systems, - Carbon accounting, - Carbon footprint, - Building information modeling
- Evaluation tools, - LCA, - Development of evaluation indices, - Codes, - Standards
- Specifications, - Guidelines

4) Social & economic aspects

- Resources management, - built environment, - aesthetics, - LCC

5) Case studies of sustainable concrete materials and structures

6) Other related topics

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