EFECTUL NATURII ACTIVATORULUI ALCALIN ASUPRA PRINCIPALELOR PROPRIETĂȚI ALE GEOPOLIMERILOR PE BAZĂ DE DEŞEURI DE STICLĂ ŞI ŞLAM ROŞU

EFFECT OF ALKALINE ACTIVATOR ON THE MAIN PROPERTIES OF GEOPOLYMERS BASED ON CULLET GLASS AND RED MUD

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In this paper are presented results regarding the synthesis and properties of geopolymers based on glass powder, with/without red mud (solid part), activated with the solution resulted in the filtration process of red mud slurry (RF). The compressive strengths achieved by these materials are below 20 MPa, smaller as compared with those assessed on the geopolymers based on the same solid components but activated with NaOH 5M solution (up to 40MPa); these results can be explained by the lower alkalinity of RF. Nevertheless, for the materials activated with RF, the substitution of glass powder with 25% red mud powder (resulted by the drying of solid part separated from red mud slurry - R), determines an increase of compressive strength values - 15-100% with reference to the one assessed on the geopolymer based only on glass powder. This increase can be explained by the ability of red mud powder (R) to continuously release alkaline ions in RF solution.

În lucrare se prezintă rezultate referitoare la sinteza şi proprietăţile unor geopolimeri pe bază de deşeu de sticlă fin măcinată, cu/fără adaos de deşeu de şlam roşu (partea solidă); aceste materiale s-au obţinut prin activarea alcalină cu soluţia rezultată prin filtratrea şlamului roşu (RF). Rezistenţele mecanice ale acestui tip de materiale au valori sub 20 MPa, spre deosebire de cele ale geopolimerilor obţinuţi din acelaşi component solid activat cu soluţie NaOH 5M (până la 40MPa); aceste rezultate pot fi explicate prin alcalinitatea mai mică a soluţiei RF. Cu toate acestea, în cazul materialelor activate cu RF, substituirea sticlei cu partea solidă separată din şlamul roşu (R), determină o creştere a valorilor rezistenţei la compresiune - cu 15-100% raportat la rezistenţa determinată pe compoziţia care conţine doar sticlă. Această creştere poate fi explicată prin capacitatea componentului solid din şlamul roşu (R) de a elibera continuu ioni alcalini în soluţia RF.

Keywords: *geopolymer, cullet glass, red mud, compressive strength, microstructure, hydrolytic stability*

1. Introduction

The recovery, re-use and recycling of a wide variety of industrial and municipal waste represents today an important research topic due to environmental, economic and legal aspects [1,2]. Some of these wastes can be regarded as valuable alternative/secondary raw materials and reused into value added products [3-5].

Waste glass can be incorporated in cement and concrete, as aggregate or supplementary cementitious material, therefore improving the sustainability of this industry [4-6]. The use of glass bottle cullet, for partial replacement of aggregate in portland cement concrete, is limited by some specific issues such as alkali silica reaction or bond strengths between cementitious matrix and glass aggregate [5,6].

Other possible use for this type of glass waste is as solid component for geopolymer materials synthesis. Geopolymers, as defined by Davidovits [7], are 3D framework structures in which tetrahedral $[SiO_4]^{4-}$ and $[AlO_4]^{5-}$ units are connected with alkali ions balancing the negative framework charge. Cyr et al. [8], used sodium or potassium hydroxide solutions to activate cullet soda-glass; the resulted geopolymer cements, achieved good compressive strengths (up to 50 MPa) when cured long periods of time (minimum 7 days) at high temperature (40-60°C); nevertheless, these materials have low durability when conserved in water, explained by the authors, by the low amount of Al (Si to Al ratio around 20) [8].

Recently, our research group reported the synthesis of geopolymers by alkali activation with NaOH solution of mixtures of waste glass and red mud [9]. Red mud slurry is a waste generated during bauxite processing; this waste has a high alkalinity (pH range from 9.7 to 12.8) and contains high amounts of Al, Na and Fe [9, 10]. In our previous researches [9, 11] we've separated by filtration the red mud slurry and used the dry solid material (R) as admixture to the glass cullet powder (in solid component formulation). In this paper we present results regarding the synthesis and properties of geopolymers based on glass cullet with/without red mud (R) activated with the solution resulted by slurry filtration (RF). To our best

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knowledge no data are available regarding the use of this solution as alkali activator in geopolymer synthesis.

2. Experimental

2.1. Materials

Soda-lime siliceous glass cullet of green and brown color (glass cullet from glass bottles production plant) was used as solid components for the synthesis of geopolymers. The oxide compositions of green and brown glasses are presented in Table 1.

The glass cullet were ground in a laboratory ball mill up to a fineness corresponding to a Blaine specific surface area of 3189 cm^2/g (brown glass) and 2981 cm^2/g (green glass). The particle size distributions of glass powders, presented in Figure 1, are almost similar, green glass (G) being slightly coarser as compared with brown glass (B).

Red mud is a sludge in which the liquid part represents around 32%. This sludge was filtrated and the resulted solution (RF) had the density of 1.015 g/cm^3, pH of 12.96 and electrical conductivity of 21.82 mS/cm. Acid neutralization capacity (ANC) of this solution (RF) was assessed by titration with HCl 0.1 N up to a pH value of 7 [10]. The ANC was 3 moles H^+/l.

The solid part separated by sludge's filtration, dried at 100°C and desegregated in a ball mill. The resulted powder (R) has a density of 1.69 g/cm^3 and Blaine specific surface area of 3216 cm^2/g.

The elemental composition of red mud powder (R) is: Fe=37.81%, Na=25.49%, Al=17.39%, Ti=5.97%, Si=5.59%, Ca=2.64%, Cr=0.25%, P=0.18%, S=0.17%, K=0.06%

The elements assessed by X-ray fluorescence in the filtrate (RF) are: Na=6.32%, Al=2.65% and K=0.05%.

The main mineralogical compounds, assessed by X-ray diffraction of red mud powder (R), are: iron oxide (PDF 73-2234), iron titanium oxide (PDF 09-0317), sodium aluminum silicate ($Na(AlSiO_4)$ - PDF 81-2081), aluminum silicate hydrate $Si_2Al_2O_5(OH)_4$ - PDF 74-1023, $Na_8(Si_6Al_6O_{24})H_{0.88}(CO_3)1.44(H_2O)_2$ - PDF 77-1145 and calcium aluminum silicate hydrate (PDF 77-1994) [9].

2.2. Preparation of geopolymers

Brown glass powder (B), green glass powder (G) or mixtures of glass powder and red mud powder (R) were used as solid components for the synthesis of geopolymer materials (Table 2). The activator was NaOH 5M aqueous solution (N5) or the filtrate (RF) resulted in red mud slurry processing [11]. The liquid to solid ratio was of 0.3 or 0.25.

Table 1

Oxide composition of brown and green glass cullet / *Compoziția oxidică a deşeurilor de sticlă brună şi verde*

Material	Oxide composition (w.%) / *Oxide composition (% grav.)*								
	SiO_2	Na_2O	CaO	Al_2O_3	Fe_2O_3	MgO	K_2O	Cr_2O_3	C
Brown glass (B)	68.51	12	11.45	2.72	2.2	2.09	0.84	-	0.19
Green glass (G)	68.26	13.43	10.21	2.73	2.41	2.10	0.69	0.18	-

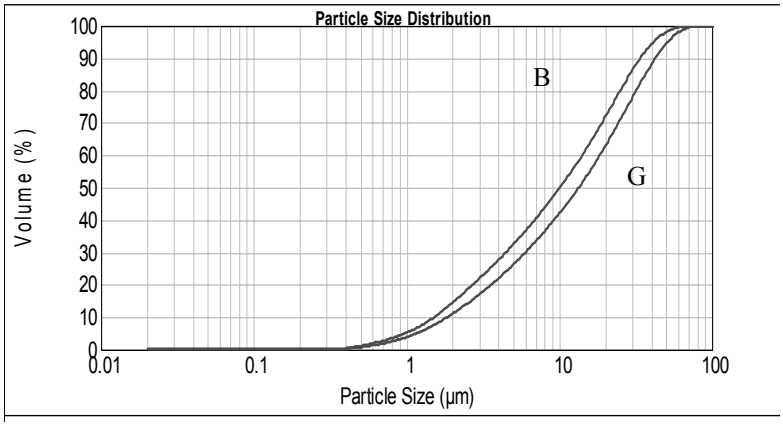

Fig.1 - Grain size distribution curves for brown (B) and green (G) glass powders/ *Distribuția granulometrică a deşeurilor de sticlă brună (B) şi verde (G) fin măcinate.*

Table 2

Formulations based on brown/green glass powders and red mud (R) with different alkali activator solution/ *Compozițiile pe bază de sticlă brună/verde fin măcinată şi partea solidă (pulbere) din deşeul de şlam roşu (R) activate cu diferite soluții alcaline*

Sample / *Proba*	Solid component (S) / *Component solid*			Liquid component (L)/*Component lichid*		Liquid to solid ratio (L/S) / *Raport lichid/solid*
	Brown glass (B) / *Sticlă bruna*	Green glass (G) / *Sticlă verde*	Red mud (R) / *Şlam rosu*	NaOH 5M	RF solution / *soluție*	
B-N5	100	-	-	✓	-	0.3
BR25-N5	75	-	25	✓	-	0.3
B-RF	100	-	-	-	✓	0.3
BR25-RF	75	-	25	-	✓	0.3/0.25
GR25-RF	75	25	-	-	✓	0.3
G-RF	100	-	-	-	✓	0.3

Mortars with binder to sand ratio of 0.5 and different values of L/S (see Table 3) were prepared, cast in rectangular molds (15x15x60mm) and vibrated for 2 minutes. The specimens were cured 3 days at 60°C (first day in mold, covered with cling film, then de-molded and stored in humid air - R.H.=90%); after that the specimens were cured in air (R.H. 65%) at 20 ±2°C.

The aggregate used for mortar preparation was siliceous sand and fulfilled the requirements of European and corresponding Romanian norm (SR EN 196-1, 2006) [12].

2.3. Methods

Chemical compositions of green and brown waste glass powders were assessed with the chemical methods described in the Romanian standard 5771/1-11/89 [13].

The Blaine specific surface area of glass powder was assessed with the method described in SR EN 196-6, 2010 [14].

Particle size distributions of glass powders were assessed with a Malvern Mastersizer 2000 laser particle granulometer.

Chemical composition of red mud was assessed by X-ray fluorescence spectrometry using an S8 Tiger Bruker).

The mineralogical composition of red mud powder was assessed by X ray diffraction (XRD) analysis using a Shimadzu XRD 6000 diffractometer. The XRD spectra were obtained using a monochromatic CuKα radiation (λ= 1.5406 Å), range 2θ from 5 to 60 degree.

SEM and EDX analyses were performed on selected pastes or mortar specimens coated with Ag, using a HITACHI S2600N microscope.

Compressive strength was assessed on mortar specimens (15x15x60 mm), using a Tonitech machine.

The durability of studied compositions was assessed by the immersion of mortar specimens cured for 7 days in demineralized water (water to solid ratio of 1.3) [8, 9]. The immersion solutions were renewed daily, the first 4 days, and then weekly up to 28 days. The solution's pH was assessed with a laboratory multi parameter PCD 6500 pH-meter.

Compressive strength and mass variation (ΔCs and Δm) of mortar specimens after immersion in water were calculated with the formulas:

$$\Delta Cs = [(Csa - Csw)/Csa] \times 100\ (\%), \quad (1)$$

where Csw = compressive strength of specimens immersed in water for 28 days (MPa); Csa = compressive strength of specimens cured in air for the same time (MPa).

$$\Delta m = [(m_{1d}-m_{28d})/m_{1d}] \times 100\ (\%), \quad (2)$$

where m_{1d} = specimen's mass after 1 day of immersion in water (g); m_{28d}= specimen's mass after 28 days of immersion in water (g).

Mass and compressive strength values were the average values of at least three individual values assessed on specimens cured in similar conditions.

3. Results and discussions

3.1. Compressive strength

In Figure 2 are presented the values of compressive strength developed by B-N5 and BR25-N5 geopolymer mortars, cured the first 3 days at 60°C and then in air at 20°C for different periods of time.

For the mortars based on brown glass activated with NaOH 5M solution (B-N5), the compressive strength values increase in time, reaching almost 40 MPa after 28 days of hardening (Fig.2). The replacement of brown glass with 25% red mud powder (BR25-N5) decreases the compressive strength with 40-50%. This can be due to the lower reactivity vs. alkali solution of red mud; this waste contains crystalline compounds, with lower reactivity as compared with the vitreous phase from glass. Also, the geopolymerisation process, which determines this material's hardening, could be hindered by the high amount of iron compounds supplied by the red mud [9, 10].

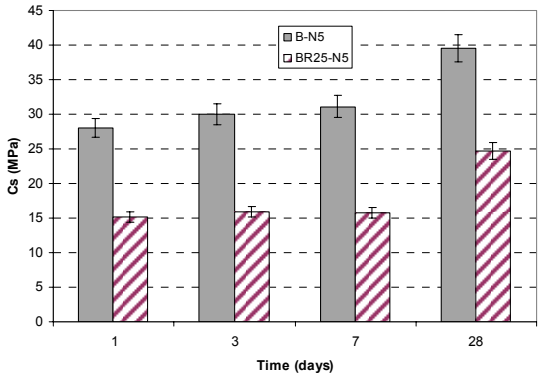

Fig.2 - Compressive strength vs. time for geopolymers based on brown glass powder (B) and mixtures of brown glass powder with 25% red mud (BR25) activated with NaOH 5M (N5)/ *Variația în timp a rezistenței la compresiune a geopolimerilor pe bază de sticlă brună (B) şi amestecuri de sticlă brună cu 25% şlam roşu (BR25) activate cu soluție NaOH 5M (N5).*

The data presented in Figure 3, shows the compressive strength values of the geopolymers obtained when using as alkali activator the solution resulted by the filtration of red mud slurry - RF; if the brown glass is alkali activated with RF is possible to obtain geopolymer mortars (B-RF) but the compressive strength values are much smaller as compared with the case when the alkali activator was NaOH 5M (Fig.2). These results can be explained by lower alkalinity of RF solution, as compared with NaOH 5M solution; according to Davidovits [7], high concentration of alkali activator solution are necessary for the dissolution of reactive phases from solid component, especially

of silicate anions, necessary for the formation of the geopolymer hardening structure.

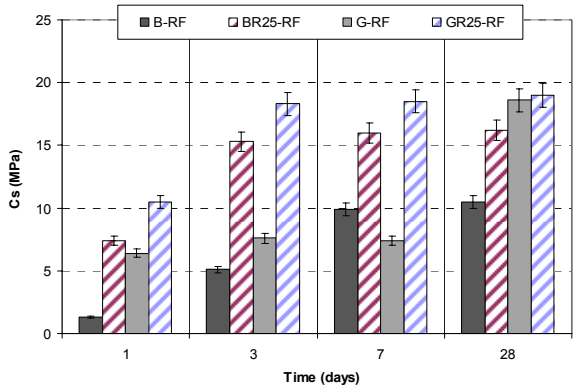

Fig.3 - Compressive strengths developed by the geopolymers based on brown glass powder (B), brown glass powder with 25% red mud powder (BR25), green glass powder (G) and green glass powder with 25% red mud powder (GR25), activated with RF solution/ *Rezistența la compresiune dezvoltată de geopolimerii pe bază de sticlă brună (B) , sticlă brună cu 25% şlam roşu (BR25), sticlă verde (G) şi sticlă verde cu 25% şlam roşu (GR25) activate cu soluție RF.*

For this type of activator solution, the replacement of brown glass with 25% red mud powder (BR25-RF) determines an increase of compressive strength values. This apparently surprising influence can be explained by the ability of red mud powder (R) to continuously release alkaline ions in RF solution; according to Grafe et al. [10] alkaline ions are continuously released in bauxite residue solution due to the dissolution of solid waste. This phenomenon explains also the well known ability of red mud to neutralize acids (ANC) [10]. The alkali ions released in RF, by the compounds present in red mud powder (R) contributes to the geopolymerisation process. Similar results, i.e. an important increase of the compressive strength achieved in the systems with red mud activated with RF, were obtained for the systems based on green glass (Fig.3).

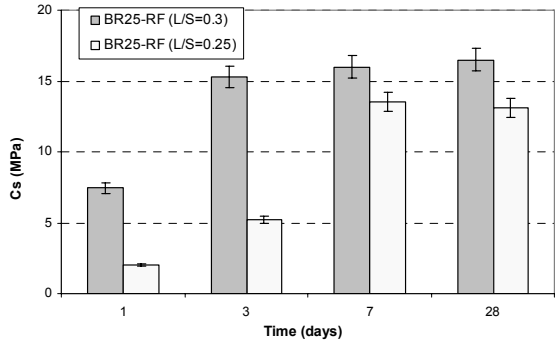

Fig.4 - Influence of liquid to solid ratio (L/S) on the compressive strength values developed by the geopolymers based on brown glass powder with 25% red mud powder (BR25), activated RF solution, cured for 3 days at 60°C and then in air at 20°C/ *Influența raportului lichid/solid (L/S) asupra valorilor rezistenței la compresiune dezvoltată de geopolimerii pe bază de sticlă brună cu 25% şlam roşu (BR25), activate cu soluție RF şi păstrate primele 3 zile în aer la 60°C şi apoi în aer la 20°C.*

The substitution of fine brown glass powder with red mud powder (R) in the composition of geopolymers mortars, increases their workability in fresh state; therefore, is also possible to prepare mortars with a lower liquid to solid ratio (0.25) (Fig. 4). In this case the values of compressive strengths (especially those recorded at early ages - 1 and 3 days) decrease. This is probably due to the reduction of dissolution process of alkali phases from solid waste (R) in the lower amount of RF solution.

3.2. Microstructure

The microstructure of geopolymer paste, resulted by the alkaline activation of brown glass with NaOH 5M solution, can be assessed in the SEM images presented in Figures 5 a,b. As it can be seen, glass grains (B in Fig.5b) are embedded in a continuous matrix consisting mostly from sodium silicate (aluminate) hydrates [9]. The EDX analyses presented in Figures 5c,d confirm the presence of Na in a higher amount in the binding matrix as compared with the brown glass grain (B).

The microstructure of geopolymer obtained by the alkali activation with NaOH 5M solution of the brown glass with 25% red mud (Fig.6) consists also from a continuous matrix; the spherical pores with diameters around 100 μm, resulted most probably from the air entrained during the mixing of precursors. In these pores, one can notice the presence of coarse particles (see arrows), which can be attributed to red mud [9]. These particles represent unchanged minerals originally present in bauxite [15] and have a low reactivity vs. the alkaline solution.

The microstructure of the geopolymer BR25-RF (Fig. 7) is less compact as compared with the one assessed for the specimens activated with NaOH 5M solution (Figs. 5 and 6). One can notice rectangular glass grains partially connected with a loose phase consisting mainly from sodium silicate (aluminate) hydrates - as suggested by the EDX analysis presented in Figure 7c. This microstructure correlates well with the lower values of compressive strength assessed on this composition (as compared with those assessed on the compositions activated with NaOH 5M).

3.3. Hydrolytic stability of synthesized geopolymers

Previous results obtained on geopolymers synthesized by alkali activation with NaOH solution of green glass with/without red mud addition, showed a high sensitivity of these compositions when immersed in water [9]. In this paper the hydrolytic stability was also assessed by compressive strength and mass changes of mortar specimens immersed in demineralized water for 28 days.

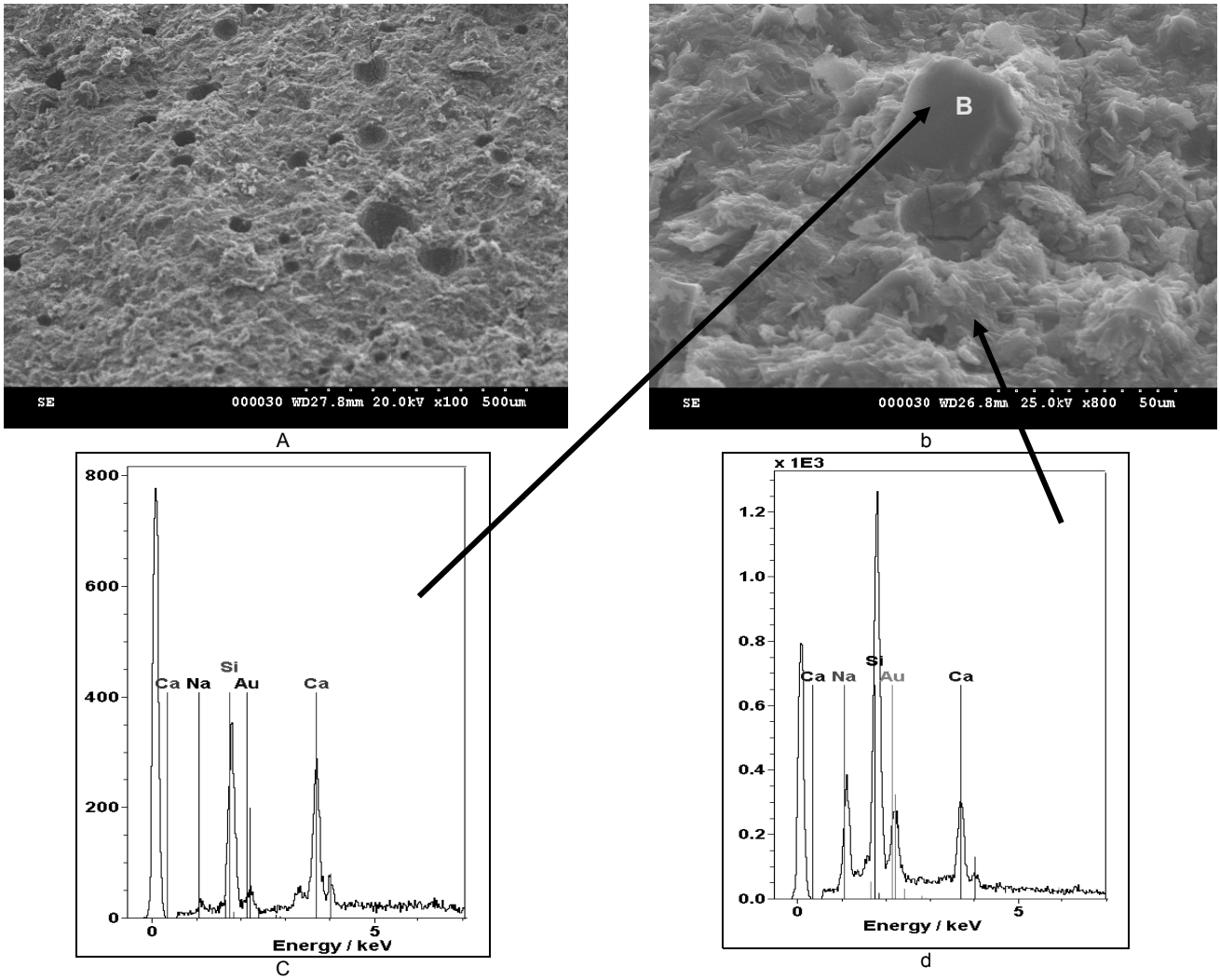

Fig.5 - SEM micrographs (a,b) and EDX spectra (c,d) of B-N5 specimen after 7 days of curing / *Imagini de microscopie electronică de baleiaj (a,b) şi spectroscopie de raze X cu dispersie de energie (c,d) a probei B-N5 după 7 zile de întărire.*

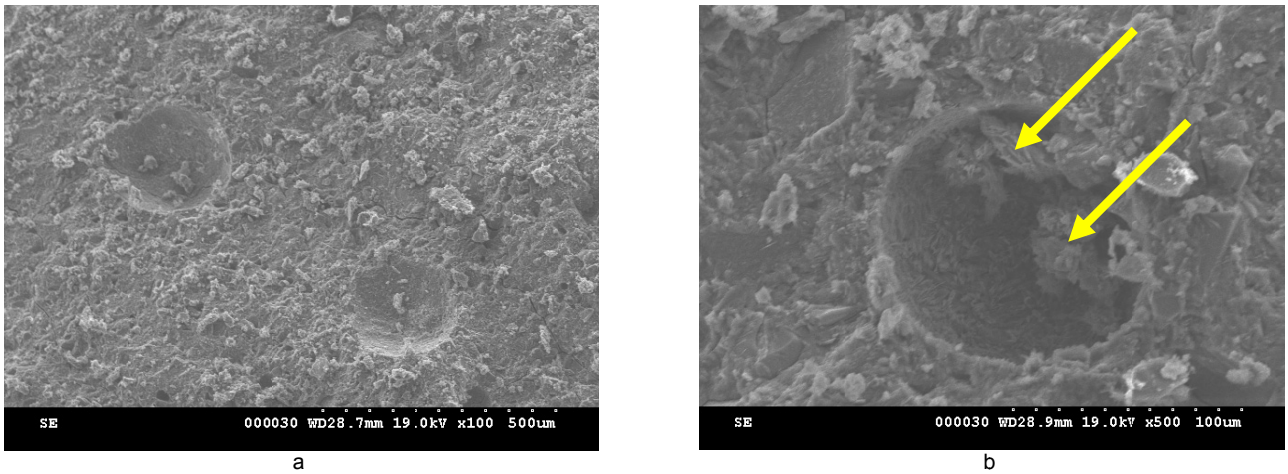

Fig.6 - SEM micrographs of BR25-N5 specimen after 7 days of curing / *Imagini obținute prin microscopie electronică de baleiaj a probei BR25-N5 după 7 zile de întărire.*

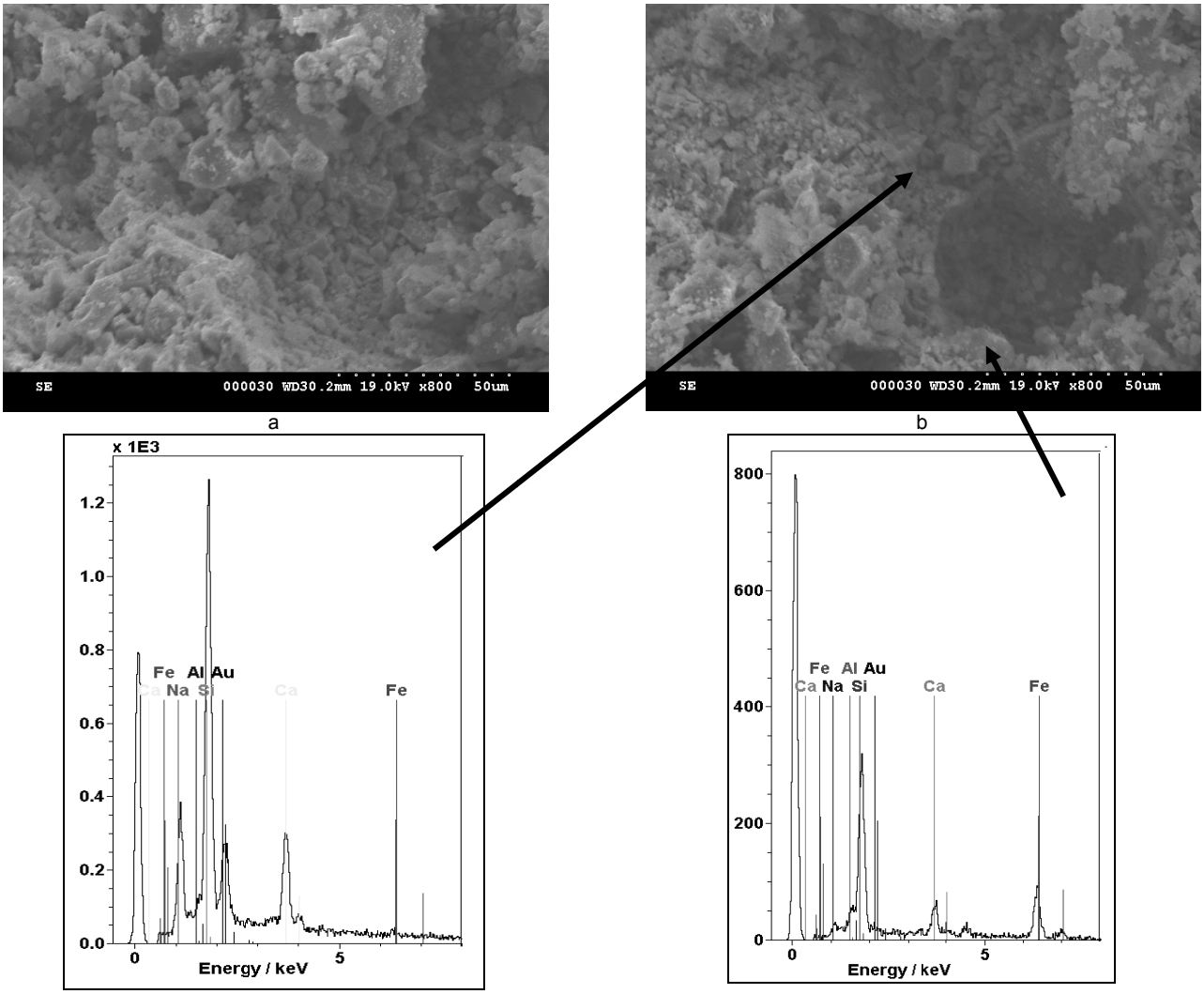

Fig.7 - SEM micrographs (a,b) and EDX analysis (c,d) of BR25-RF specimen after 7 days of curing / *Imagini de microscopie electronică de baleiaj (a,b) şi spectroscopie de raze X cu dispersie de energie (c,d) a probei BR25- RF5 după 7 zile de întărire.*

Although the visual aspect of the mortar specimens (based on brown glass with/without red mud) was not altered after 28 days of immersion in water (Figs. 8 and 9) the compressive strength values recorded an important decrease (32 up to 55% with reference to the compressive strength value of the specimen cured the same time in air) – Figure 10.

For the mortar specimens immersed in water for 28 days is recorded also a mass loss (Fig. 10). This mass loss was calculated with reference to the specimen's mass determined after 1 day of immersion in water. This value was selected as reference, due to the fact that a mass increase was noticed after the first day of immersion in water (mainly due to the specimen's open

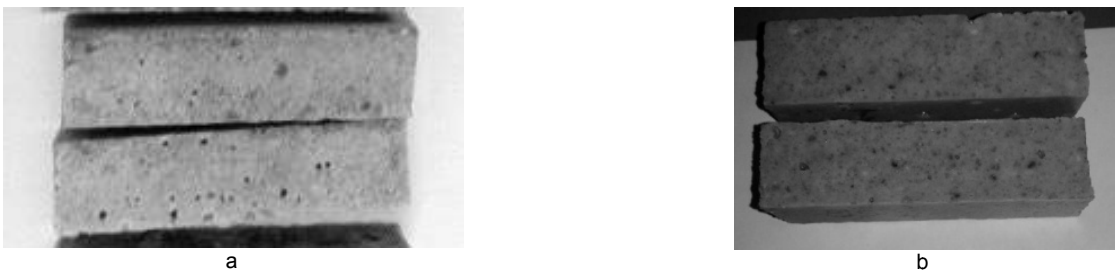

Fig. 8 - Visual aspect of B-N5: a) before immersion test; b) after immersion in water for 28 days / *Aspectul vizual al B-N5:a) înainte de testul de imersie; b) după imersarea în apă timp de 28 zile.*

A b

Fig. 9 - Visual aspect of BR25-N5: a) before immersion test; b) after immersion in water for 28 days / *Aspectul vizual al BR25-N5: a) înainte de testul de imersie; b) după imersarea în apă timp de 28 zile.*

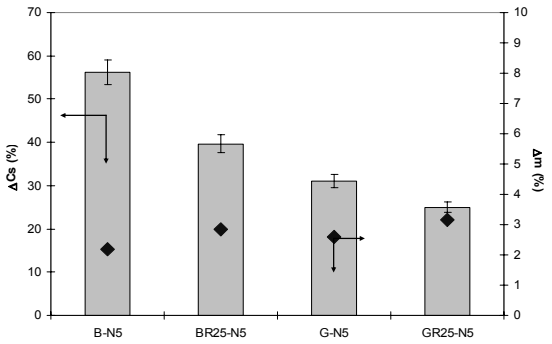

Fig. 10 - Compressive strength loss (∆Cs) and mass loss (∆m) after 28 days of immersion in water / *Pierderea de rezistența la compresiune (∆Cs) şi de masa (∆m) după 28 de zile de imersare a probelor în apă.*

porosity), followed by a continuous mass decrease up to 28 days of immersion [9]. These results suggest a continuous dissolution of geopolymers obtained by alkali activation of brown and green glasses with/without red mud additions.

The continuous dissolution of these materials is confirmed also by the high pH value of the solutions in which the mortar specimens were immersed and kept different periods of time (Table 3); it should be kept in mind that immersion solution (demineralized water) was renewed daily in the first 4 days and then weekly up to 28 days.

The microstructure of mortar specimens immersed for 28 days in demineralized water is presented in Figures. 11 and 12.

As it can be noticed, the matrix which connected the glass grains in B-N5 specimen (see also Figs. 5 a and 5 b) is severely deteriorated and numerous grains are visible, along with a phase with low crystallinity (see dotted circle in Fig. 11b). A similar aspect was assessed by SEM for geopolymers synthesized from cathode ray tubes waste glass activated with NaOH or KOH solutions [16].

The immersion for 28 days in demineralized water of the specimen with red mud (BR25-N5) determines also an important degradation of the binding matrix (see Figs 11 c,d as compared with Figs. 6 a,b).

A similar degradation of specimens based on green glass powder was assessed by SEM analysis (Fig. 12); for G-N5 mortar the binding matrix (M) has a high porosity and is loosely connected to the glass grains (G).

The microstructure of the specimen with red mud (GR25-N5) is slightly different; one can notice (Fig. 12 c,d) two phases - an amorphous phase (A) which partially cover rectangular crystals (see arrows). As previously presented [9], these crystalline deposits can be formed by the crystallization of amorphous phases (specific for sodium silicates hydrates [17]) when the aluminium content increase (due to the substitution of green glass with 25% red mud); when the mortar specimens are immersed for 28 days in water, the amount of amorphous phase decreases, due to the leaching phenomenon.

The results obtained in this study pointed out the possibility to completely re-use the red mud slurry (solid and liquid part) in the synthesis of geopolymers based on waste glass powder. Nevertheless, the performances of these materials, from the aspects of compressive strength and hydrolytic stability, are below those reported in other studies for geopolymers obtained by alkaline activation with sodium hydroxide solution of fly ash and red mud mixtures [18] or glass powder and fly ash mixtures [16,19].

Table 3

pH values of the solution in which the mortar specimens were immersed different periods of time / *Valorile pH-ului soluțiilor în care au fost imersate probele pentru diferite perioade de timp*

Sample / *Probă*	pH of the leachate after different period of immersion time: *pH-ul soluției de imersare după diferite perioade de timp:*							
	1 day / *1 zi*	2 days / *2 zile*	3 days / *3 zile*	4 days / *4 zile*	7 days / *7 zile*	14 days / *14 zile*	21 days / *21 zile*	28 days / *28 zile*
B-N5	11.4	11.83	11.64	11.37	11.53	11.63	11.68	11.69
BR25-N5	11.63	11.66	11.52	11.25	11.34	11.71	11.64	11.9
G-N5	11.69	11.64	11.64	11.6	11.81	11.7	11.64	11.55
GR25-N5	11.85	11.81	11.73	11.76	11.89	12.25	11.85	11.73

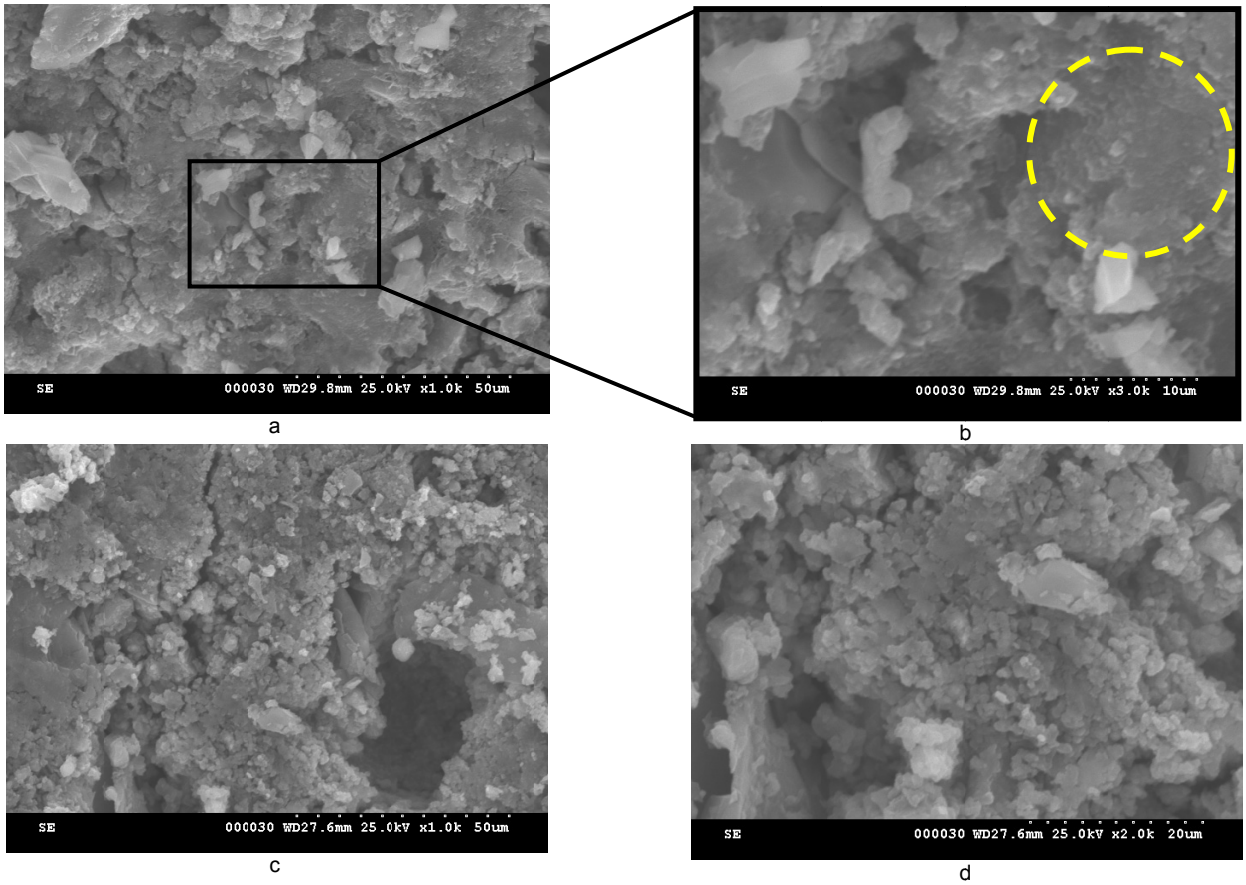

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Fig. 11 - SEM micrographs of mortar specimens immersed 28 days in demineralized water a,b-B-N5, c,d- BR25-N5/ *Imagini obținute prin microscopie de baleiaj ale probelor de mortar după 28 de zile de imersare în apă a,b-B-N5, c,d-BR25-N5.*

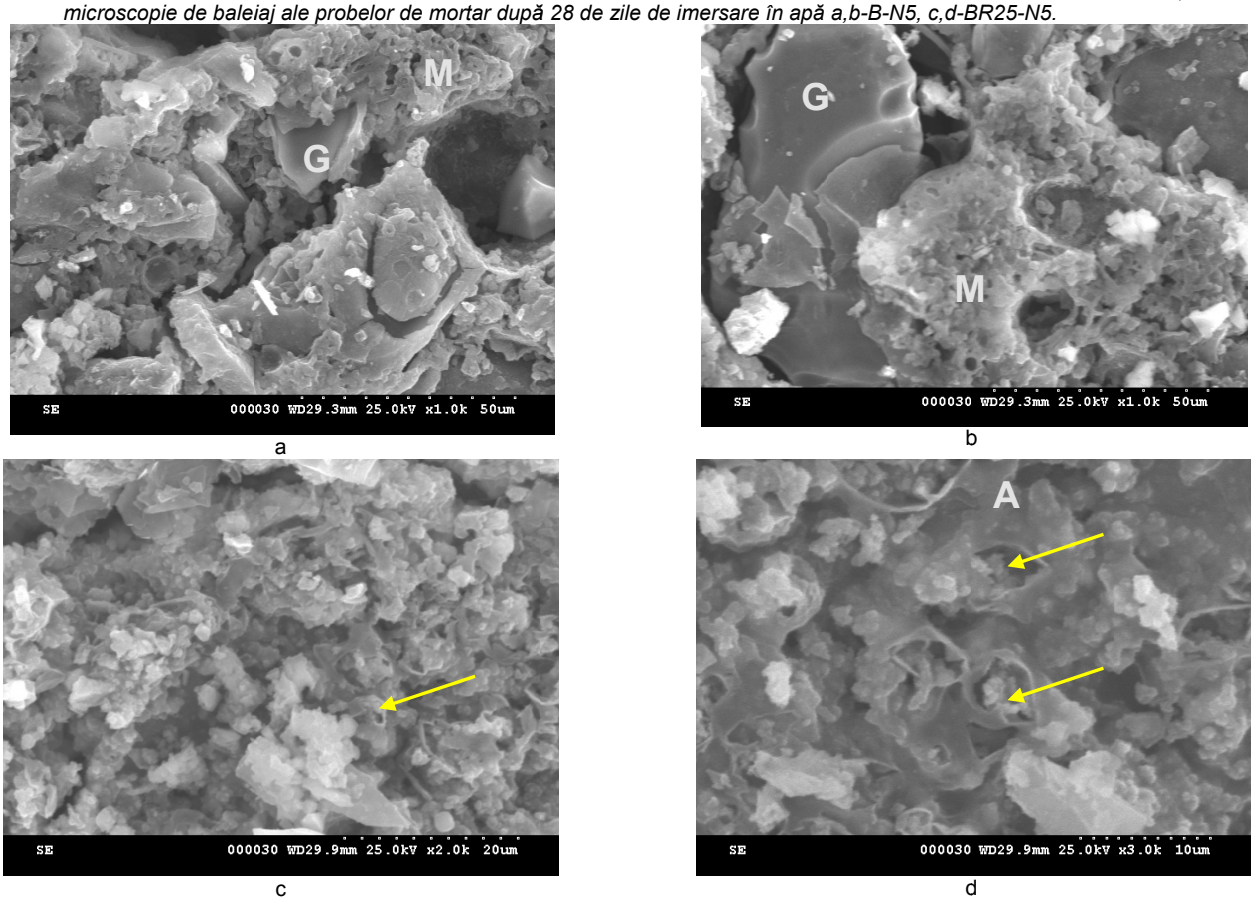

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Fig. 12 - SEM micrographs of mortar specimens immersed 28 days in demineralized water a,b-G-N5, c,d- GR25-N5/ *Imagini obținute prin microscopie de baleiaj* ale *probelor de mortar după 28 de zile de imersare în apă a,b-G-N5, c,d-GR25-N5.*

Therefore, our future studies will focus on geopolymers formulations which will include also fly ash.

4. Conclusions

Based on the results obtained in this study, one can formulate the following conclusions:

- Geopolymers can be produced from finely ground bottle glass waste, using as alkali activator solution the filtrate (RF) separated from the red mud slurry. Nevertheless the compressive strengths achieved by these materials are smaller (with 50-70% as compared with those obtained for the materials based on the same solid components in which the activator solution was NaOH 5M); these results can be explained by the lower alkalinity of RF, as compared with NaOH 5M solution;

- For the mortars in which the liquid component is NaOH 5M solution, the substitution of brown glass powder with 25% red mud powder (resulted by the drying of solid part separated by filtration from red mud slurry), determines a decrease with 20-50% of compressive strength values. When the alkali activator is RF, the replacement of glass powder with 25% red mud powder determines an increase (with 15-100%) of the compressive strength values; this opposite influence (as compared with the system activated with NaOH solution) can be explained by the ability of red mud powder (R) to continuously release alkaline ions in RF solution. Similar results were obtained for the systems based on green glass.

- The immersion of these materials in demineralized water (up to 28 days) determines the leaching of sodium silicate (aluminate) hydrates which are the main components of the binding matrix.

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