EVALUAREA COMPORTĂRII UNOR CIMENTURI COMPOZITE LA ATACUL ACIZILOR ORGANICI▲
ASSESSMENT OF THE BEHAVIOR OF COMPOSITE CEMENTS TO ORGANIC ACIDS ATTACK

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Information about the evaluation of the behavior of Portland cement to chemical attack exerted by organic acid medium is still limited and there is no standardized method for evaluation of its chemical stability. Most studies assess the durability of cement without addition (type CEM I) or containing various proportions of granulated blast furnace slag (type CEM II /A-S, CEM III/B) in contact with organic media such as acetic or lactic acid, and most refer to the behavior of cement pastes and mortars.

This paper presents data on the acid corrosion resistance of common cements type CEM II/A-V, CEM II/B-M(S-V) and CEM V/A(S-V) (according to EN 197-1) compared with that of cement without additions (type CEM I). The evolution of the mechanical strengths and weight changes of the mortar specimens, immersed into 5% solution of lactic acid, acetic acid or lactic acid + acetic acid for 180 days, combined with evolution of the pH and concentration of species presented in solution have shown that the acid corrosion resistance of cements containing granulated blast furnace slag and fly ash is higher than that of fly ash cement and sole Portland cement.

Informaţiile cu privire la evaluarea comportării cimenturilor portland faţă de atacul chimic exercitat de medii acide organice sunt încă limitate şi nu există metode standardizate de evaluare a stabilităţii lor chimice. Cele mai multe studii evaluează durabilitatea cimentului fără adaos (tip CEM I) sau conţinând proporţii variabile de zgură granulată de furnal (tip CEM II/A-S, CEM III/B) în medii organice, cum ar fi acidul acetic sau lactic, cele mai multe referindu-se la comportarea pastelor şi mortarelor de ciment.

Lucrarea prezintă date referitoare la rezistenţa la coroziune acidă a unor cimenturi uzuale tip CEM II/A-V, CEM II/B-M(S-V) şi CEM V/A (S-V) (conform EN 197-1), comparativ cu cea a unui ciment fără adaos (tip CEM I). Evoluţia rezistenţelor mecanice şi modificărilor de masă ale probelor de mortar, imersate în soluţii de 5% acid lactic, acid acetic şi respectiv acid lactic+acid acetic, timp de 180 zile, corelată cu evoluţia pH-ului soluţiilor şi concentraţiei speciilor prezente in soluţie, au aratat că rezistenţa la coroziune acidă a cimenturilor cu conţinut de zgură granulată de furnal şi cenuşă zburătoare este mai mare comparativ cu cea a cimentul cu cenuşă zburătoare şi a cimentului portland unitar.

Keywords: *composite cements, chemical stability, organic acids, mechanical strength*

1. Introduction

Concrete is the most widely used construction material in both civil and industrial constructions and it is subjected to acid attack carried out by different corrosive agents such as ground water, seawater, acid rain, industrial or agricultural waste.

Environmental protection requirements impose to the producers of cement and concrete the reducing of the natural raw materials consumption and decreasing of the CO_2 emissions in the atmosphere in the manufacturing process.

In the past decade there has been increased the production of cement with admixture (especially with granulated blast furnace slag, fly ash and its combinations) in detriment to the sole cement, thus becoming necessary to investigate the behavior of composite cements under the action of acidic corrosive environments. Most of the studies are related to the durability of cement with admixture under the inorganic acids conditions such as hydrochloric acid, sulfuric acid or nitric acid [1-5]. Currently information on the damage caused by organic acid environments is still limited and there are no standardized methods for assessing its chemical stability. The zootechnic sector produces effluents (such as liquid manure or effluent from silage fodder) containing organic acids which exert the chemical attack on the cement based materials. Also, in the cheese and dairy products processing results organic acids such as acetic or lactic acids. Therefore, it is necessary to determine the intensity of the organic acids attack on the floor / platforms or silos made of cement-based materials.

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Most studies assess the durability of sole cement (CEM I) or cements containing different proportions of granulated blastfurnace slag (GBS) such as CEM II/A-S and CEM III/B in organic media like acetic acid or lactic acid, but most concern behavior of cement pastes or mortars [1,6-12]. Evaluation of concrete's behavior containing CEM I and 30-85% ground granulated blastfurnace slag (GGBS) [11] showed that the presence of GGBS in Portland cement mortar improves its performance in environments exposed to organic and the durability increases with increasing of slag proportion in concrete. Sanchez et al. [13] provide information about the behavior of mortars containing cement with fly ash type CEM II/A-V, CEM II/B-V and CEM IV/B on the action of the combinations of organic acids present in the swine manure sludge under aerobic and anaerobic conditions. The authors of [13] noted an increase in the compressive and flexural strengths of cements exposed in both conditions, as a result of size pores reducing in the outside of specimens.

This paper aims to provide information on the chemical stability of composite cements containing fly ash and GBS type CEM II/A-V, CEM II/B-M(S-V) and CEM V/A(S-V) compared to sole cement (CEM I) to the attack exerts by one solution of 5% lactic acid, 5% acetic acid and its combinations, for 180 days.

2. Experimentals

2.1 Materials used

It was envisaged achievement in the laboratory conditions of the four type of cements, according to EN 197-1:2011 [14], 32.5 R strength class, namely: CEM I, CEM II/A-V containing of 20% fly ash, CEM II/B-M(S-V) containing of 20% GBS and 10% fly ash and CEM V/A(S-V) containing 20% GBS and 20% fly ash, respectively. For this purpose it used: industrial clinker of Portland cement, fly ash, GBS and gypsum, whose characteristics are shown in Table 1.

The cements were produced in a laboratory tube mill with a capacity of 20 kg/ batch, by grinding of clinker and admixtures with 5% gypsum up to a fineness, expressed as a Blaine specific surface area, which allows the development of quite similar compressive strengths after 28 days. Table 2 shows the characteristics of cement produced.

For preparation of organic acids media the following materials were used:

- a lactic acid solution of 90% concentration (high purity), manufactured by Merck;
- a glacial acetic acid (concentration of 99.8 to 100%), high purity, manufactured by Baker.

Three solutions of organic acids were prepared: one solution of 5% acetic acid, one solution of 5% lactic acid and one solution of 5% lactic + acetic acids (ratio of 1:1). The mediums were chosen in correlation with the date from literature in order to ensure a pH values in range of 2-4.5 [15, 16].

2.2. Methods used

Mortars were prepared according to EN 196-1 [17], with water/binder = 0.5 and binder/sand = 1/3. Mortar specimens with dimensions of 40x40x160 mm were kept for 24 hours in a saturated humidity condition (relative humidity of 90%), and after demoulding the specimens were stored for 27 days immersed in water. Further the samples were stored until the testing period of 1 month, 3 months and 6 months as follows:

- immersed in water - considered as reference;
- immersed in a solution of 5% lactic acid (pH 2.32);
- immersed in a solution of 5% acetic acid (pH 2.42);
- immersed in a solution of 5%lactic acid + acetic acid (1:1) (pH 3.9)

During the experiment the solutions were not changed.

Mortar specimens were visually examined before and after exposure to organic acid medium.

The compressive and flexural strengths and weights of the cement mortar specimens immersed in different exposure media were determined.

In addition, the pH and the concentrations of the important species presented in solutions (Ca, Si, Al, Fe and Mg) were monitored over time. In this paper are presented only the results on CaO variation.

For pH determination it was used a portable pH meter type THERMO ORION and for determination of the main species leaching from solution the following principle was used:

- silica insolubilization in a certain proportion of solution by hydrochloric acid attack and it separation from the major oxides by filtration;

-a gravimetric determination of silica: it makes its purity by volatilization with hydrofluoric acid and sulfuric acid; the residue is disaggregated using a mixture of sodium carbonate and sodium chloride and it dissolved in hydrochloric acid;

- the solution of the residue adds to the original filtrate and from certain proportion determines the major oxides by complexometric method.

Degradation of mortars under organic acids attack was estimated by evaluating their physical and mechanical properties for 180 days, compared to those developed in the reference medium.

Chemical stability was evaluated by the loss of compression strength and flexural strength respectively, calculated using the formula:

$$\text{Strength loss} = [(S_w - S_a)/S_w] \times 100 \quad (1)$$

where S_w –strength of mortar specimen stored in

Table 1

Chemical analysis of the raw materials used for the cements obtaining
Analiza chimică a materiilor prime utilizate pentru producerea cimenturilor

Characteristic / *Caracteristica*,%	Clinker/ *Clincher*	Silicious fly ash / *Cenusa zburătoare silicoasă*	Granulated blastfurnace slag *Zgură granulată de furnal*	Gypsum/ *Gips*
L.O.I / *P.C*	2.83	3.08	3.56	nd
CaO	62.32	5.65	42.78	nd
SiO_2	20.10	56.24	34.23	nd
Al_2O_3	6.51	22.44	11.01	nd
Fe_2O_3	4.39	8.03	1.00	nd
MgO	1.29	0.00	3.99	nd
SO_3	0.49	1.33	0.33	44.33
Na_2O	0.44	0.56	0.81	
K_2O	0.96	2.22	0.81	
Free CaO /*CaOliber*	0.33	0.00	nd	
Reactive SiO_2 /SiO_2 reactiv		52.04		
$CaSO_42H_2O$				93.40
$CaSO_4$				1.50

Table 2

Physical and mechanical characteristics of the cement obtained laboratory
Caracteristicile fizico-chimice ale cimenturilor obținute in laborator

Cement characteristic / *Caracteristica ciment*	Cement type /*Tip ciment*:			
	CEM I	CEM II/A-V	CEM II/B-M (S-V)	CEM V/A (S-V)
Finesses / *Finețe* (cm^2/g)	3710	4120	4050	4570
Density / *Densitate* (g/cm^3)	3.06	2.83	2.88	2,83
Initial setting time/*Timp inițial priză* (minutes)	220	270	230	250
Soudness /*Stabitate* (mm)	0.5	0.0	0.0	0.0
Compressive strength, 2 days/ *Rezistența la compresiune, 2 zile* (MPa)	17.6	14.6	12.1	11.3
Compressive strength, 28 days / *Rezistența la compresiune, 28 zile* (MPa)	39.3	47.0	39.5	41.6
Flexural strength, 2 days / *Rezistența la incovoiere, 2 zile* (MPa)	3.94	3.21	2.78	2.56
Flexural strength, 28 days / *Rezistența la incovoiere, 28 zile* (MPa)	7.17	8.98	8.82	8.87
LOI / *P.C* (%)	3.98	nd.	nd.	nd.
SO_3(%)	2.64	3.03	2.94	3.07
Cl^- (%)	0.003	0.013	0.010	0.010
Insoluble residue /*Reziduu insolubil* (%)	0.71	nd.	nd.	nd.

nd – not determined */nedeterminat*

water for t days, and the S_a–strength of mortar specimen stored in acid medium for t days.

Also, were calculated the weight loses of specimen for each medium, using the formula:

Weight loss = $[(m_i - m_t)/m_i] \times 100$ (2)

where– m_i -mass of mortar specimen before stored in acid solution, and the m_t –mass of mortar specimen stored in acid solution for t days.

3. Results and discussions

3.1 Visual examination

Visual examination of the specimens revealed the attack of organic acids solutions on CEM I by forming on the its outer side of a thin layer (about 1.5 mm) of whitish color in case of immersion in solution of lactic acid, reddish color when stored in acetic acid solution, or whitish color when stored in lactic and acetic acids solution, while keeping the specimens in water the structure is uniform, as illustrated images shown in Figure 1. It should be noted that in the case of mortar samples immersed in a solution of 5% lactic acid was observed the formation of a gelatinous surface layer (Figure 1a), probably due to the biological attack by bacteria that exercised during the curing of the specimens in sealed vessels at the room temperature. In anaerobic conditions, it is possible a partial degradation of lactic acid to acetic acid [18].

After 3 months of exposure the specimens are more degraded, showing the formation of deposits both in solution as well as on specimens as shown in the pictures from Figure 2, in which are presented the specimens immediately after removal from the solution and after cleaning and washing in running water.

Also, in the case of mortar specimens containing composite cements such us CEM II/A-V and CEM II/B-M(S-V) it is noted after one month of immersion in the acid solutions the changing of its outer surface (Figures 3 and 4) and the formation of a surface layer about 1 mm (it seems a slower chemical attack).

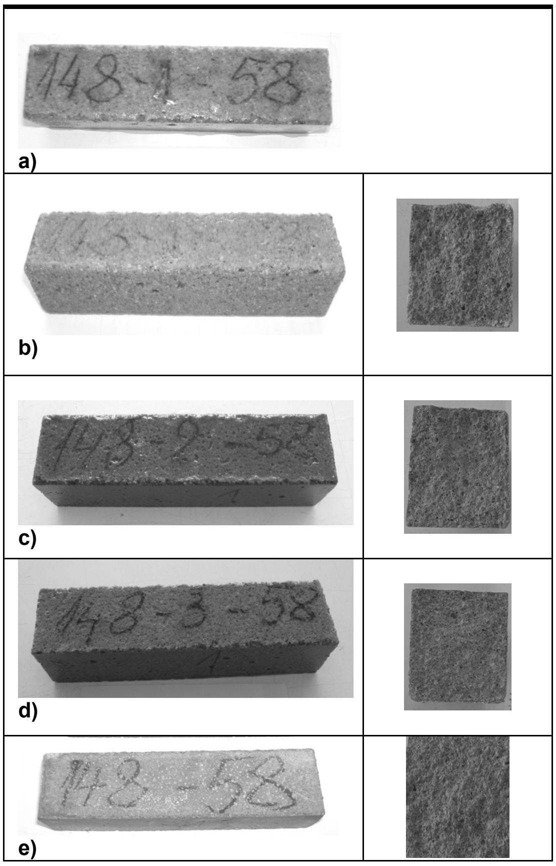

Fig. 1 - Pictures of the CEM I cement specimens after storage for 1 month in: a)lactic acid solution -before washing; b)lactic acid solution-after washing; c) acetic acid solution; d)lactic + acetic acids solution; e)water /*Imagini ale epruvetelor de ciment CEM I după păstrare 1 lună în: a)soluție acid lactic-înainte de spălare; b) soluție acid lactic-după spălare; c)soluție acid acetic; d)soluție acid lactic+acetic; e)apă.*

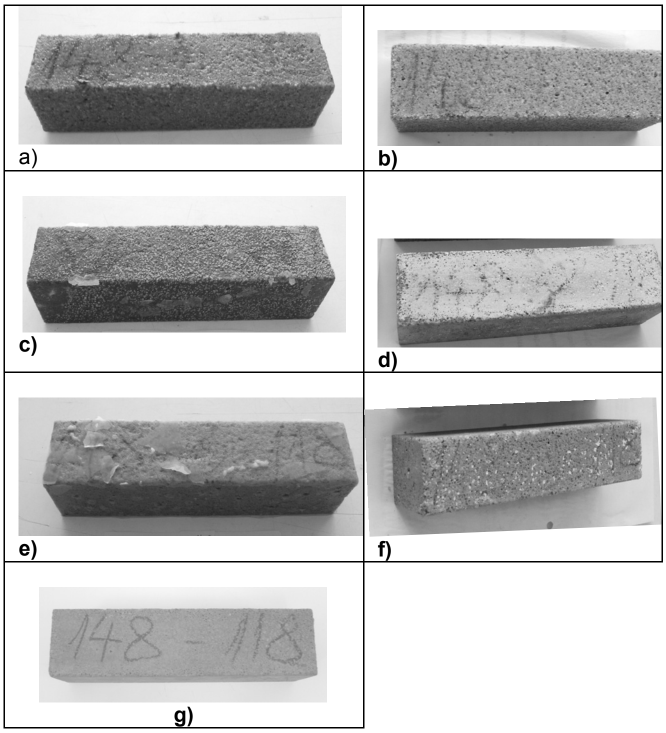

Fig. 2 - Pictures of the CEM I cement specimens after storage for 3 months in: a) lactic acid solution -before washing; b) lactic acid solution-after washing; c) acetic acid solution -before washing; d) acetic acid solution- after washing; e)lactic+acetic acids solution- before washing; f) lactic+acetic acids solution- after washing; g) water / *Imagini ale epruvetelor de ciment CEM I după păstrare 3 luni în: a) soluție acid lactic-înainte de spălare; b)soluție acid lactic- după spălare; c) soluție acid acetic- înainte de spălare; d) soluție acid acetic- după spălare; e) soluție acid lactic+acetic- înainte de spălare; f) soluție acid lactic+acetic- după spălare; g)apă.*

3.2 Variation of mechanical strengths

The compression and flexural strengths' evolution of the specimens preserved in acid solutions up to 180 days compared with those of reference conditions (stored in water) is shown in Figure 5.

There is a decrease of the compression strength when specimens are exposed in organic acid media compared to the reference (cured in water). This may be due, according to the data available in the literature, to acid corrosion process manifested by decalcification of existing hydrates in hardened cement paste [19] and formation on the surface of the test specimen of a corroded layer having a advanced porosity [20], whose thickness varies depending on the rate of diffusion of the acid through corrosion layer and the reaction rate of the acid with the cement compounds.

The losses of compressive strength after 6 months of exposure to organic acid solutions have different values, depending on the nature of the organic acid and the cement composition. Thus, the mortar prepared with CEM I shows a decrease in the compressive strength of 23.2% when immersed in lactic acid and mixture of acetic and lactic acids, respectively, and of 25.9% in the case of acetic acid attack. The mortar containing CEM II/A-V has comparable losses of compressive strength (21.3-21.8%) to the attack of lactic acid and acetic acid respectively, and slightly lower (17.3%) to the combined attack of the two organic acids. When using CEM II/B-M(S-V) are obtained the smallest loss of the compression strength in case of the specimens exposed to lactic acid solution attack (11.5%), while values are almost twice under the action of the acetic acid solution (20.4%).

Test specimens containing CEM V/A(S-V) showed the losses of compressive strength between of 14.4% and 21.2%, with a minimum recorded value in case of its storing in a solution of lactic and acetic acids, and a maximum value under acetic acid storage. The slightly low value of the loss strength in case of lactic acid could be explained by the fact that gelatinous slime layer deposited on the surface of specimens acted as a barrier, slowing to some extent the acid attack.

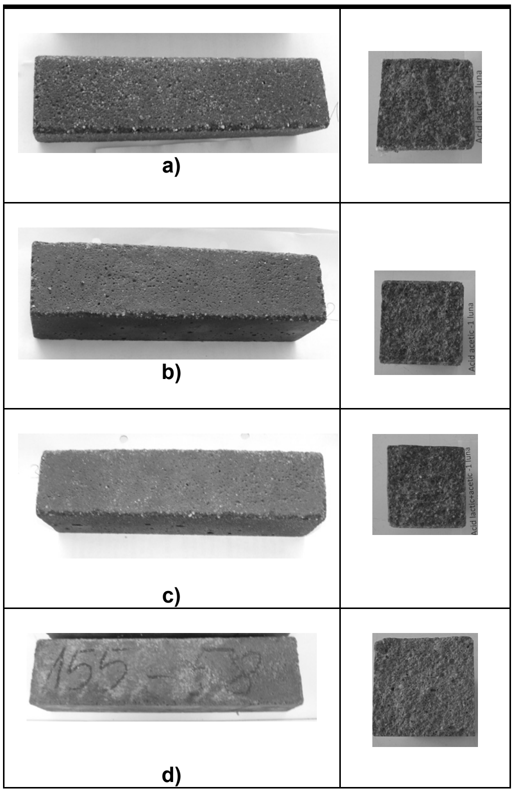

Fig. 3 - Pictures of the CEM II/A-V cement specimens after storage for one months: a)in lactic acid solution b) in acetic acid solution c) in lactic + acetic acids solution ;d) in water/ *Imagini ale epruvetelor de ciment CEM II/A-V după păstrare 1 lună în: a) soluție acid lactic; b) soluție acid acetic; c) soluție acid lactic+acetic; d) apă.*

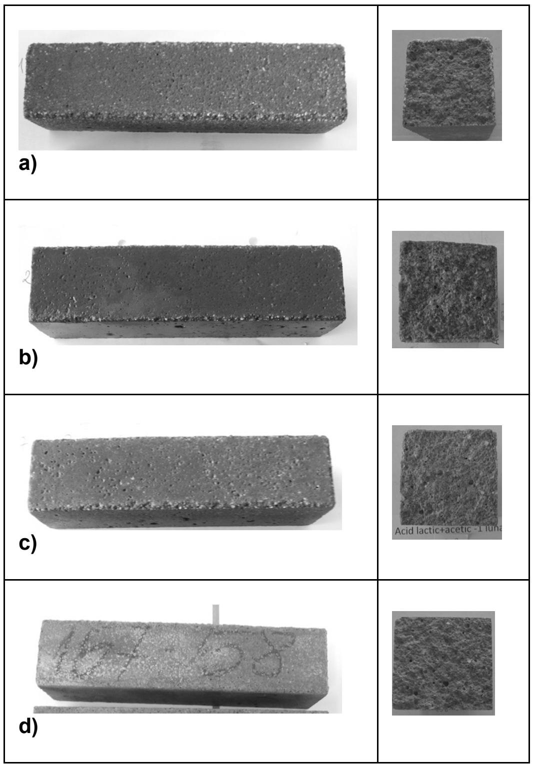

Fig. 4 - Pictures of the CEM II/B-M (S-V) cement specimens after storage for one months: a)in lactic acid solution b) in acetic acid solution c) in lactic + acetic acids solution; d) in water *Imagini ale epruvetelor de ciment CEM II/B-M (S-V) după păstrare 1 lună în: a) soluție acid lactic; b) soluție acid acetic; c) soluție acid lactic+acetic; d) apă.*

Depending on the type of cement used in mortar, the chemical stability of specimens exposed to the corrosive effect of 5% lactic acid solution decreases in the series: CEM II/B-M(S-V) > CEM V/A(S-V) > CEM II/A-V > CEM I. The same hierarchy is established in case of the 5% acetic acid solution attack, but its effect appears to be stronger than of lactic acid. The stability of cement mortars against the combined action of the two organic acids decreases in the series: CEM V/A(S-V) > CEM II/B-M (S-V) = CEM II/A-V > CEM I. The better chemical stability of the composite cements is due to the higher degree of substitution of clinker with cementitious admixtures and the formation of small amounts of $Ca(OH)_2$ during the cement hydration available for reaction with the organic acid to form water-soluble salts of calcium- lactate and acetate.

The flexural strength's evolution of the cements exposed to acid media is slightly different from that of the compressive strength (Figure 5).

For example, the cement type CEM I shows an increase of the flexural strength in the first month of exposure in acid media, then there is a decrease up to 6 months, registering the highest loss of the flexural strength (24.2% in acetic acid solution, 9.8% in lactic acid solution and 5.45% in lactic acid + acetic acid solution, respectively). The cement type CEM II/A-V exhibits a continuous increase of the flexural strength up to 3 months, after that it decreases. A different behavior in time of the flexural strength compared to the compression strength was observed by Sanchez [13] in the case of the cements with fly ash addition type CEM II/A-V, CEM II/B-V and CEM IV/B.

3.4 Variation of weight

Weight losses of the mortar specimens prepared with CEM I, CEM II/A-V, CEM II/B-M(S-V) and CEM V/A(S-V) are shown in Figure 6.

It is noted that slight variations occur in the weight of the test specimens, generally less than 2% for all cement considered, although there are significant loss of mechanical strength after 6 months of exposure to acid solutions attack (up to ca. 25% in the case of cement type CEM I). This may be an indication that this determination is not a very sensitive measure for the assessment of corrosion of macro-specimens cement, at least in the short and medium terms.

Values slightly higher of the weight loss recorded for specimens exposed to the action of lactic acid indicate its strongest attack against acetic acid and their combination.

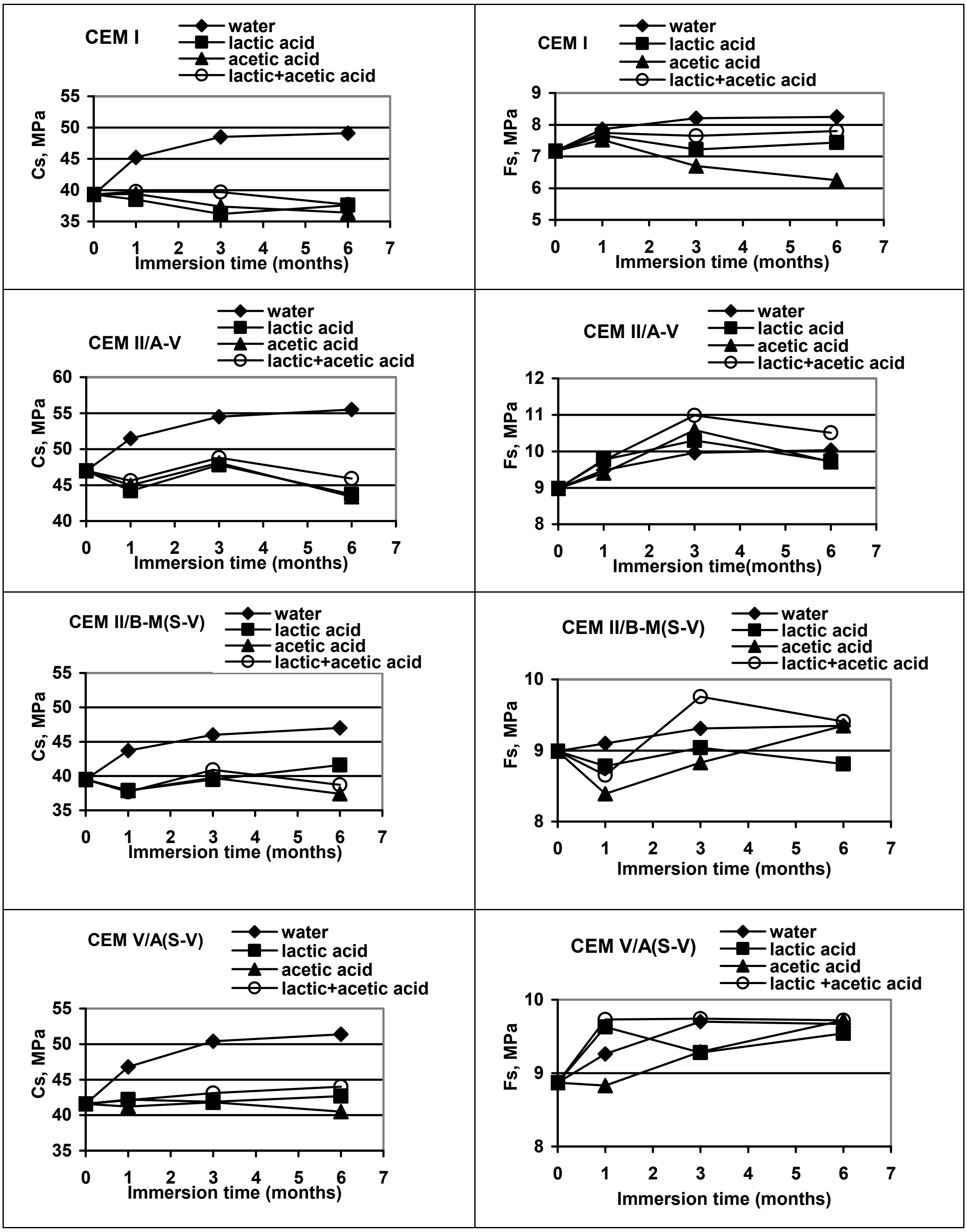

Fig. 5 - Evolution of the compressive and flexural strengths of the mortar specimens prepared with CEM I, CEM II/A-V, CEM II/B-M(S-V) and CEMV/A(S-V) cured up to 6 months in organic acids solutions and in water (reference) / *Evoluția rezistențelor la compresiune şi la încovoiere ale epruvetelor de mortar preparate cu ciment CEM I, CEM II/A-V, CEM II/B-M(S-V) şi CEM V/A(S-V) păstrate pînă la 6 luni în soluții acide organice şi în apă (mediu de referință).*

3.5. Variation of pH and Ca^{2+} leached in acid solutions

Figures 7a-c show the variation vs. time of the pH of acid solutions in which has been immersed the mortar specimens based on the studied four types of cement. Figures 7d-f illustrate the evolution of CaO concentration in organic acid solutions (the dissolution rate of Ca is considered as the most relevant factor of the durability of binders compared with other species leaching as Si, Al and Fe [7]).

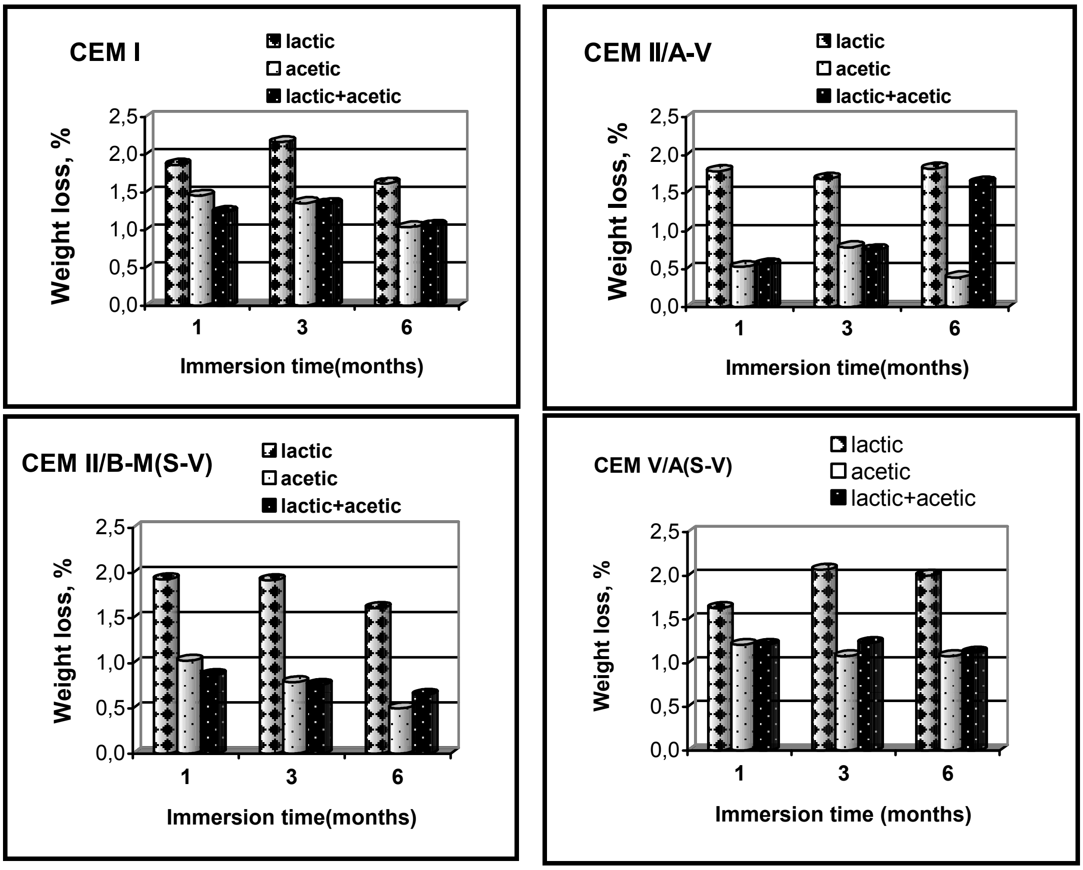

Fig.6 Variation in time of the weigth loss of mortar specimens prepared with CEM I, CEM II/A-V, CEM II/B-M(S-V) and CEM V/A(S-V)/ *Variația in timp a pierderii de masă a epruvetelor de mortar preparat cu ciment CEM I, CEM II/A-V, CEM II/B-M (S-V) şi CEM V/A.*

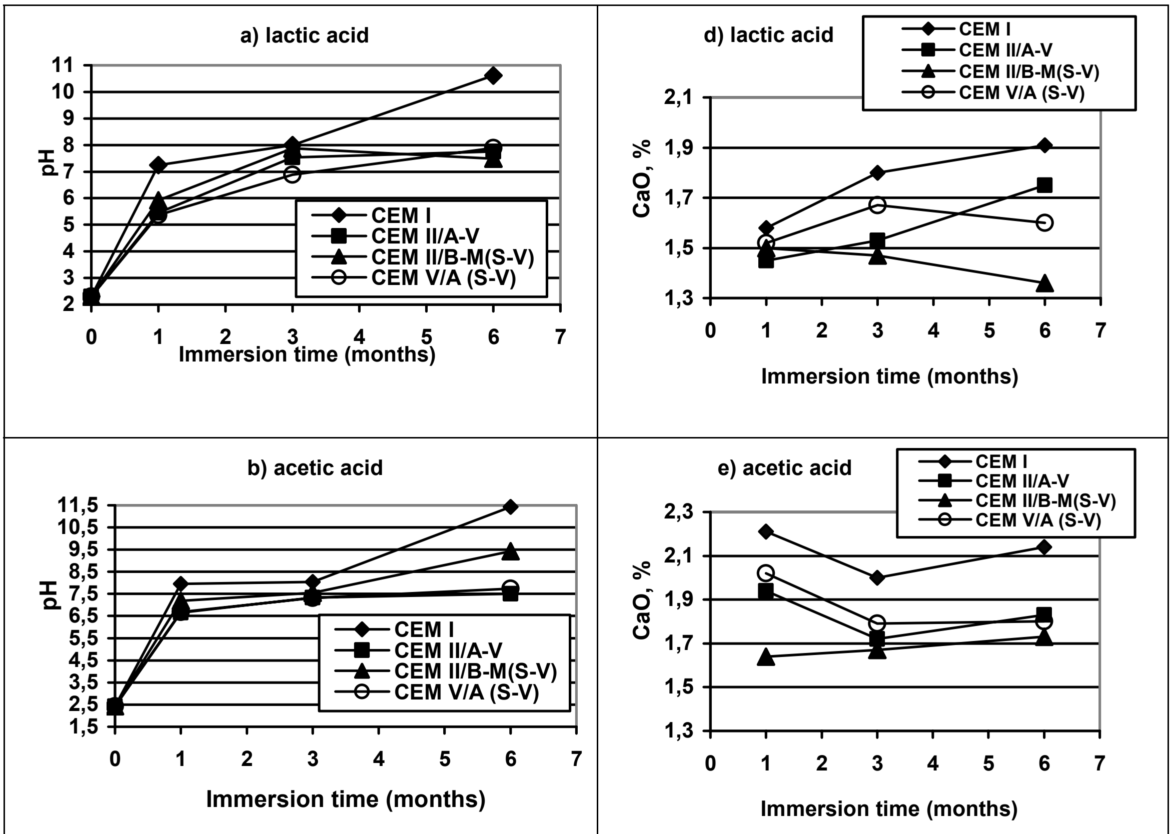

Fig. 7 se continuă pe pagina următoare/ *continues on next page*

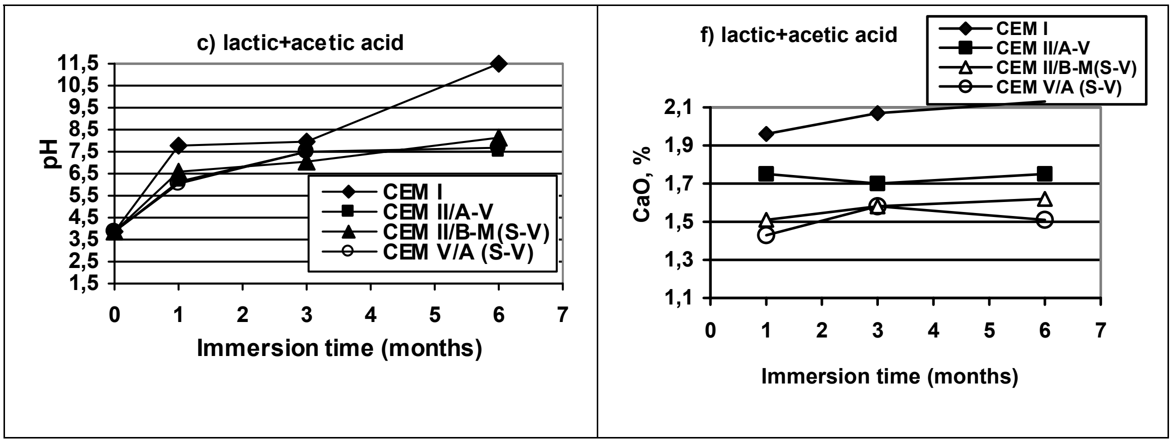

Fig. 7 – Change in time of the solutions` pH in which were immersed the cement samples (a-c) and of the CaO concentration into acidic solutions (d- f). / *Variația în timp a pH-ului soluțiilor în care au fost imersate epruvetele de ciment (a-c) şi a concentrației de CaO din soluțiile acide (d-f).*

It is noticed the highest variation of the pH from 3 to 6 months in the case of the mortar specimens containing CEM I immersed in acid solutions (from 7.5 - 8.0 to 10.6 - 11.5 units), indicating a reduced stability of the cement in acid media due to higher amount of $Ca(OH)_2$ released during cement hydration and its availability for reaction with the acid to form water-soluble salts of calcium lactate or calcium acetate and a higher amount of C-S-H exposed of the decalcification. Composite cements have a lower increase of pH values for solutions containing lactic acid (up to 7.5-8.5) and acetic acid (up to 9.5) respectively, being more stable than CEM I.

The variation in time of the calcium oxide concentration in the acid solutions is generally discontinuous, with more intense period of solubilisation of calcium oxide, followed by a slight decline.

It has been found in all test specimens of mortar immersed in organic acid solutions up to 6 months the formation of a white precipitate in large quantity which resulted in a pH increase of the solution.

4. Conclusions

Based on the experimental data obtained in this work it can draw the following conclusions:

- The losses of the compressive strength after 6 months of exposure to the attack of organic acid solutions have different values, depending on the nature of the organic acid and the cement composition. The highest loss of compressive strength is recorded for the mortar prepared with CEM I, namely 23.2 % in the case of immersion in lactic acid and in a mixture of lactic acid + acetic acid solutions and 25.9 % in acetic acid solution, respectively.
- The weight losses of the specimens are in generally below 2%, for all cements considered, although losses of compression strengths are significant after 6 months of exposure to acid solutions attack (up to approx. 25% for CEM I). This may be an indication that this determination is not a very sensitive measure for the assessment of the cement's corrosion by macro-specimens at least in the short and medium terms.
- The pH values of the acid solutions where composite cements specimens were immersed for 6 months are in the range of about 7.5 - 9.5, compared to 10.6 - 11.5 in the case of the CEM I specimens.
- The obtained results allowed a classification of chemical resistance of mortars to the organic acid attack depending on the type of binder and the strength of the acid. The mortars prepared with CEM II/B-M(S-V) and CEM V/A(S-V) showed a better chemical stability compared with CEM II/A-V and CEM I, which is due to the higher degree of substitution of clinker with cementitious admixtures and the formation of small amounts of Ca $(OH)_2$ during the cement hydration available for reaction with the organic acid to form water-soluble salts of calcium-lactate and acetate.
- The attack exerted by the acetic acid solution seems to be higher than of lactic acid solution, though the corrosive effects of lactic acid is known to be more intense. A possible explanation for this behavior could be the biological degradation of lactic acid with the formation of a gelatinous layer on the surface of the mortar's specimens which hinders to some extent the attack of lactic acid, correlated with the partial conversion of lactic acid to acetic acid. The intensity of lactic acid attack is close to that of acetic acid, while the combination of the two acids is lower aggressiveness.

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