STABILIZAREA / SOLIDIFICAREA HIDROXIZILOR DIN NĂMOLURILE REZIDUALE INDUSTRIALE ÎN MORTARE ȘI BETOANE

STABILIZATION / SOLIDIFICATION OF INDUSTRIAL WASTE SLUDGE OF HYDROXIDES IN MORTARS AND CONCRETES

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The present work were the efficiency and mechanism of stabilization/solidification of ultimate inorganic waste (hydroxides and metal fibers sludge), in cement matrix. This waste, characterized by the standard toxicity characteristic leaching procedure (TCLP) test, showed that the concentrations (mg/kg) of heavy metals were: Ni^{2+}= 384.9, Cr^{3+} = 80.2 and Pb^{2+} = 165.6, the average concentrations of heavy metals in the raw waste far exceed those of standards required. Leaching of heavy metals from the mortar and concrete was evaluated by the Monolithic leaching test (MLT), under dynamic leaching conditions for 64 days with periodic leachate renewal in different mediums: neutral, basic, acid and sulfated, for two types of formulations selected: Mortar MD and Concrete B (25%). The mechanical strength, XRD, endogenous shrinkage and total withdrawal (with and without exchange with the surroundings) were analyzed. The amounts of heavy metals released into the sulfated medium are important compared with those observed in other mediums. The results showed that heavy metals could be effectively, immobilized in cement matrices. It was concluded that further research on the influence of the metal fibers in the retention of heavy metals is needed to improve the effectiveness of stabilization / solidification in cement matrices.

Keywords: *Hydroxides sludge, metal fibers, cementitious matrix, leaching, stabilization / solidification*

1. Introduction

Stabilization / solidification (S/S) treatment methodologies have been widely used over the past three decades, where it is now an established treatment methodology [1-3]. Such methodologies have been widely used to treat inorganic contamination successfully. S/S treatments include a wide range of similar processes that usually involve mixing inorganic cementitious binders, such as Portland cement, into the waste to transform it into a new, solid, non-leachable material [4-8]. The treated waste product encapsulates potentially hazardous contaminants, reducing contact between the waste and any potential leachant. Cement-based stabilization is the suited for inorganic wastes, in particular those containing heavy metals [9-11]. According to the literature, the heavy metals are well immobilized within pastes solidified, whereas chlorides are only partially retained. In fact, the performance of S/S depends on the extent to which its hydration reactions are affected.

Most of the binders induce high pH that promotes adsorption, precipitation, absorption / encapsulation into nano-porous calcium silicate hydrate (C-S-H) gel [12], and incorporation into crystalline components of the cement matrix [13]. In addition, it is important to note that stabilization of contaminants may depend on the chemical speciation of species and the influence of the sulfate and chloride on setting, strength development and final strength [14, 15]. Many properties were considered in correlation with leaching behavior of heavy metals in solidified materials [16].

However, long-term effectiveness and chemical durability of S/S treated materials are still, not well understood. The laboratory leaching data can simulate the behavior of waste samples under ideal, static conditions or under chemical attack. This study, therefore, aims to investigate an effective stabilization / solidification (S/S) technique and predict the durability of confinement for ultimate waste containment (cutlery unit BCR of Algerians industry), using hydraulic binders (Portland cement - CEM I) of Ain El Kebira Algeria in mortars with and without reinforcement (metallic fibers powder waste from machining of metal parts) and in the concrete. It was focused on the influence of testing conditions, the release of chemical species, amount of waste [17, 18], solvent type (acid, base, sulfate or demineralized water) and materials nature (concrete or mortars).

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The objectives are the waste characterization of the cutlery unit, the evaluation of the release of various chemical species contained in the porous matrix under different leaching conditions and the evaluation of the performance of the used method which is surface run-off water to monolithic stabilized waste. To achieve these objectives, two formulations have suggested for the S/S of waste used. To follow the long-term release of chemical species and pollutants, a monolithic leaching test (MLT) was used. In future perspective, it is interesting to study the effect of the metallic powders wastes from machining combined with hydroxide sludge on the leaching of toxic species in the (S / S) materials.

2. Experimental research

2.1. Used materials

Inorganic waste (sludge of hydroxides and metal fibers), the ordinary Portland cement (CEM I) of Ain El Kebira Algeria, standard sand bags packed 1350 ± 5g, demineralized water and four different size fractions as 0/4 mm (fine sand), 8/15mm and 15/25mm (gravel) of aggregates were used.

2.2. Mortars' preparation

The preparation of mortars is generally, based on the NFEN196-1 standard. The material is prepared in a mixer of 3 Kg. First, the standard sand, fiber and waste are mixed at medium speed for 10 min to obtain a homogeneous mixture. The cement then was added and the mixture was mixed for a few minutes. The addition of demineralized water was considered for to avoid the addition heavy metals in trace amounts. Then, the mortars made are molded for prepare test samples (three samples for each test) with dimensions of 40 mm × 40 mm × 160 mm for mechanical strengths testing at 7 and 28 days and for measurement of the dimensional changes (shrinkage and swelling).

Specimens of 40 mm × 40 mm × 40 mm were made for preparation of monolithic blocks for MLT tests. The S/S materials manufactured were protected as much as possible confronted with the ambient air to outsides (carbonation phenomenon) because it contributes in the reduction of porosity of the S/S materials in the outer surface, which changes the dynamic of elements release. Formulations made up in this study are presented in Table 1A. The choice of the percentage of waste sludge of hydroxide at 25 % is based on conclusions of search [19].

2.2.1. Concretes' preparation

The concretes were prepared according to the standard FN-P-18-400. The preparation of the concrete samples was based on the method of Dreux Gorris, which is the more answered after many formulations of concrete based fibers that were tested on site. The concrete mixing was, carried out in a concrete mixer of 30-liter capacity. The waste was introduced at the same time with granular component. The specimens produced were cylinders of 160 mm × 320 mm, for the compressive strength test, Samples of 25 mm × 60 mm were made for preparation of monolithic blocks for MLT test and prismatic samples of 70 mm × 70 mm × 280 mm equipped with studs to enable measurement of the dimensional changes (shrinkage and swelling) of the concrete.

Table 1

. **Mix proportion of mortar and concrete mixtures**

(A) mortar formulation					
Designation	MT	MD	MDF 10	MDF 20	MDF 30
Sand (g)	1350	1350	1350	1350	1350
Cement (g)	450	450	450	450	450
Water (g)	225	225	225	225	225
Waste (g)	0	112.5	112.5	112.5	112.5
Rapport (%) Waste/Cement	0	25	25	25	25
Fiber powder(g)	0	0	45	90	135
Rapport (%) Fiber /Cement	0	0	10	20	30

(B) concrete formulation		
Specimens	B (0%)	B (25%)
Sand (0/4) (Kg)	845.62	845.62
Gravel (8/15) (Kg)	478.12	478.12
Gravel (15/25) (Kg)	514.88	514.88
Cement (kg)	350	350
Water (kg)	175	175
Waste (kg)	0	87.5
Rapport (%) Waste/Cement	0	25

2.3 Testing methods

2.3.1. X-Ray Diffraction (XRD) analysis

XRD examinations were performed to determine the microstructure of the waste (sludge of hydroxides and metal fibers). The small amount of powder samples are scanned by an X-Ray diffractometer with an angle of 2θ. The X-Rays are scattered by atoms in a pattern that indicates lattice spacing of elements present in the material analyzed. Once the X-ray analysis is completed, the spectra are recorded and analyzed for the specimens using, the XPERT-PROF PANALYTICAL diffractometer.

2.3.2. Toxicity Characteristic Leaching Procedure (TCLP)

Leaching tests were performed according to toxicity characteristic leaching procedure (TCLP) U.S.EPA-1311 method to determine metals mobility under natural worst case conditions (USEPA, 1992). The liquid/solid ratio was 20 L/kg. The mixture was stirred for an 18-hour, period at a rate of 30±2 rpm and then filtered with a 0.45 µm filter. The pH of the solidified/stabilized materials were measured and decreased by adding nitric acid to be less than tow. The concentrations of Pb^{2+}, Ni^{2+} and Cr^{3+} were determined using flame atomic absorption spectrometry (FAAS) Aurora Instruments AI 1200. The flame generated by the combustion of acetylene in the presence of air. The wavelengths used in the measurement of concentrations of Pb^{2+}, Ni^{2+} and Cr^{3+} are 283.3, 232 and 357.9 nm, respectively.

2.3.3.Mechanical strength

Compressive strengths were measured with a press of type universal testing machine (IBERTEST) with capacity 200 KN, piloted by computer. For each mixture, three specimens were tested according to the NF EN 196-1 standard.

2.3.4. Shrinkage and swelling tests

For shrinkage measurement, the mixtures were cast in prismatic molds of 40 mm × 40 mm × 160 mm for mortar and 7 × 7 × 28 cm^3 for concrete and covered with a moist tissue to prevent any evaporation at early age. At 1 day of age, the mixtures are unmolded and prepared for shrinkage and swelling measurements. They were provided with an adhesive band at both avoid the ends to edge effects desiccation. For the autogenous shrinkage measurement, the samples were covered completely with a thin layer of bitumen surrounded by an adhesive band to ensure no water exchange with the external environment. During shrinkage measurement, samples were placed in a frame for shrinkage measurement equipped with a micrometer precision comparator. For each mixture, the measured values of shrinkage were done for two samples and average values were reported. For the measurement of the swelling, the same experimental protocol is used in emerging the specimens in distilled water (saturated atmosphere), swelling and autogenous shrinkage tests were performed at ages, 1, 3, 7, 9, 14, 21, 28, 60, 90,120 and 150 days.

2.3.5. Monolithic Leaching Test (MLT)

A monolithic block of known dimensions was placed in contact with a leachate. The leachate renewed at contact times, was selected to be able to determine the intensity and dynamic release of elements by analyzing the leachates obtained. A liquid / solid surface ratio of 10 cm^3/cm^2 was maintained constant at each solution renewal. Monolithic cubes subjected to leaching were of 40 mm × 40 mm × 40 mm.

For each material, the sample was immersed in demineralized water in polyethylene container (Fig. 1), which was hermetically closed to prevent air penetration (CO_2) and water evaporation during the test. The experiment performed at room temperature of (23±1C°) and protected from light. For each material, the test was performed in duplicate to observe the reproductibility of the results. The solutions renewed after 0.25; 0.75: 1; 2; 5: 7; 20 and 28 days, giving a total of 64 days of continuous leaching. We obtained eight solutions to analyze after filtration (0.45 µm) to determine the concentrations of different elements. The results of MLT are flows of chemical species followed (nickel (Ni), chromium (Cr) and lead (Pb)); these results were presented graphically as a function of contact time or average time calculated by using the formula:

$$T_i = \left[\frac{\sqrt{t_i} + \sqrt{t_{i+1}}}{2} \right]^2$$

Were t_i are the durations of each leach sequence.

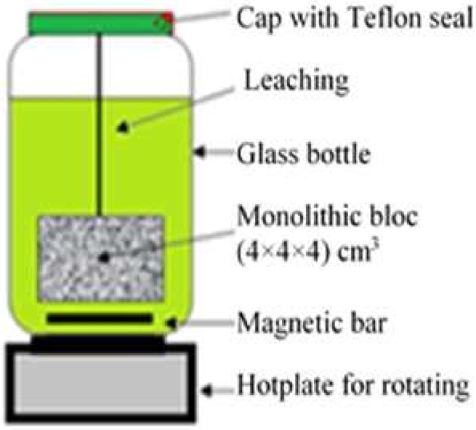

Fig. 1 - The scheme of the monolithic block leaching test.

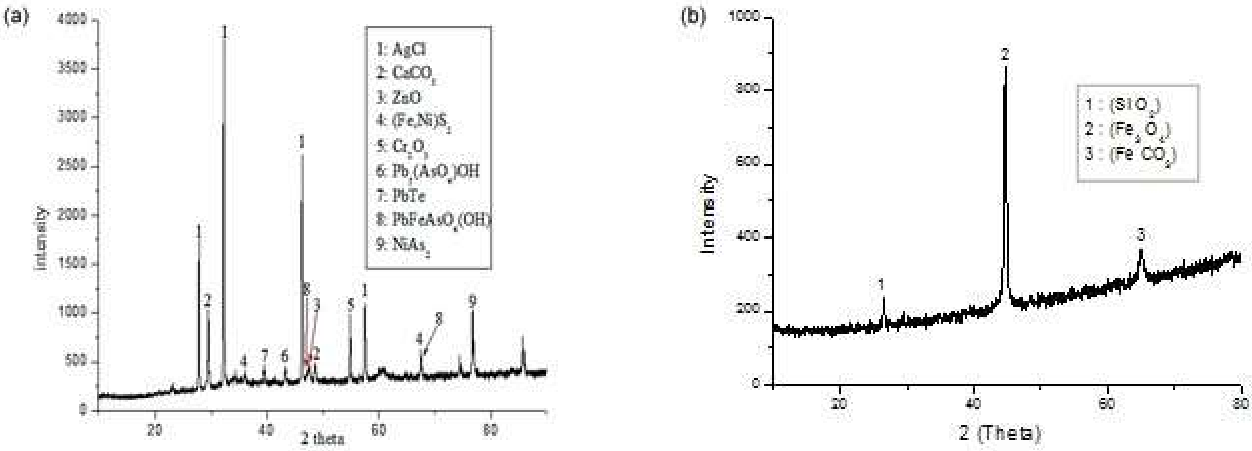

Fig. 2 - X-ray diffraction patterns: (a) raw waste of the cutlery unit and (b) fiber powder recovery machining of metal parts waste.

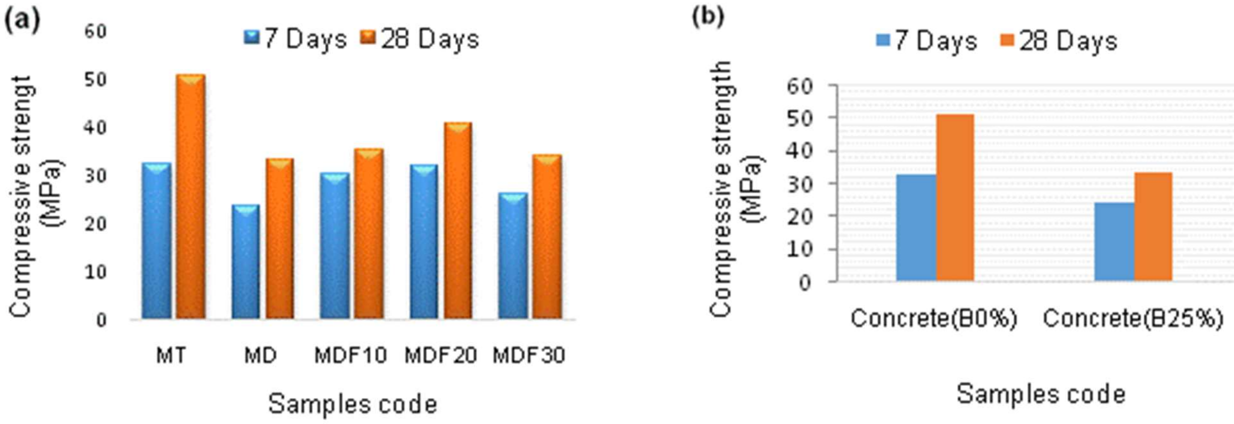

Fig. 3 - Mechanical strengths: (a) mortars; (b) concretes.

3. Results and discussions

3.1. Characterization of the waste (cutlery unit BCR of Algerians industry)

The XRD pattern showed the presence of complex phases as is shown in Figure 2(a), containing the chemical elements such as Ni, Cr and Pb. We conclude that this is classified as hazardous waste. To this effect, the waste of the cutlery unit must be Stabilized/Solidified by hydraulic binder to reduce its pollution and its toxicity.

3.2. Characterization of the fiber powder recovery machining of metal parts Waste

Crystallized mineralogical phases were detected at different diffraction angles values namely: 1: SiO2 (2θ=26.43°), 2: Fe_3O_4 (2θ= 82.61°), 3: $FeCO_3$ (2θ= 99.12° and 116.81°), as is shown in Figure 2(b).

3.3. Toxicity Characteristic Leaching Procedure (TCLP) of the waste (cutlery unit of industry)

The raw waste of the cutlery unit was characterized by the TCLP test. The results shown that the concentrations (mg/kg) were: Ni^{2+}= 384.9, Cr^{3+} = 80.2 and Pb^{2+} = 165.6; the average concentrations of heavy metals in the raw waste were far exceed those of standards required: 50 mg/kg for Ni^{2+}, $Cr^{3+}$and Pb^{2+} (French Association for Standardization [AFNOR]) [20]. Mineralogy is the main tool for understanding the state of coalescence of heavy metals in cement matrixes.

3.4. Behavior of Materials with solidified / stabilized waste

3.4.1. Mechanical strength

Compressive strength of solidified/stabilized materials were determined for 7 and 28 days of curing. Figure 3 (a and b), shows the evolution of mechanical strengths of mortars and concretes as a function of times (days). In general, after 28 days of curing samples showed higher strengths than at 7 days of curing. This observation is common in studies and experiments dealing with concretes or mortars strength reported in literature [21, 22]. In this study, the results obtained for materials confirm this behavior. Mechanical strengths of S/S materials are higher than those recommended by the XP X31-211 standard, which is equal to 1 MPa. The results show that the mortar has a better resistance than the concrete; this is due to the presence of gravel in the concrete which increases its porosity and reduces the resistance.

It observed that the materials without waste have higher strengths at 28 days of curing; it is in the order of 50 MPa for mortars and 37.5 MPa for the concrete compared to materials with 25% waste.

It appeared that the strength of solidified/stabilized materials decreases. This is due to:

(1) The nature and the quantity of waste,

(2) The quantity of cement available for hydration (excesses of the quantity of waste compared to the quantity of cement, the waste absorbs a quantity of water destined for the hydration of cement)

It is important to note that the authors Maria Georgescu et al [23] found that in the presence of waste (fly ash) in high proportion (up to 50%), there was a significant reduction in compressive strengths in mortars.

For improving the quality of our material, a study was performed on the compositions of mortars with waste and metal fiber powder. From these results, we can conclude that the MDF20 mortar is the best composition that gives good compressive strengths; we compare these results with those of the mortar MD waste and without fiber MT.

3.4.2. Influence of the waste on the total and endogenous shrinkage of the stabilized / solidified materials

The endogenous shrinkage and the total shrinkage for mortars and concretes compositions were analyzed. Figure 4(a, b) shows that, whatever the nature of the sample, the curves of the withdrawal have approximately the same shape and exhibit the same behavior along the test duration. The waste containers of the compositions MD, B(25%) developed a shrinkage (endogenous and total) less than that developed by the control samples (MT, B(0%) at the age of 150 days.

A comparative study shows that the amplitudes of the deformations obtained at 28 days for the two types of compositions containing waste, have low endogenous shrinkage compared to shrinkage recorded for materials without waste. However, this is explained by the fact that the addition of the waste to the mixture increases the reactivity of calcium (Ca^{2+}) because it is substituted by (Pb^{2+}, Ni^{2+} and Cr^{3+} contained in the waste), after which the quantity of (Ca^{2+}) reacts with the atmosphere (CO_2) to cause the formation of calcite in the capillary pores. The deposition of calcite in these pores causes low capillary depressions, the latter causes a compression in the solid skeleton of the formulation, and therefore there will be less shrinkage. It shown as concrete develop less shrinkage compared to mortars, this explained by the fact that the addition of aggregates in concretes reduces the pore size causing a low shrinkage. It has been observed that the total shrinkage is greater than the autogenous shrinkage; this is due to the evaporation of water, which is

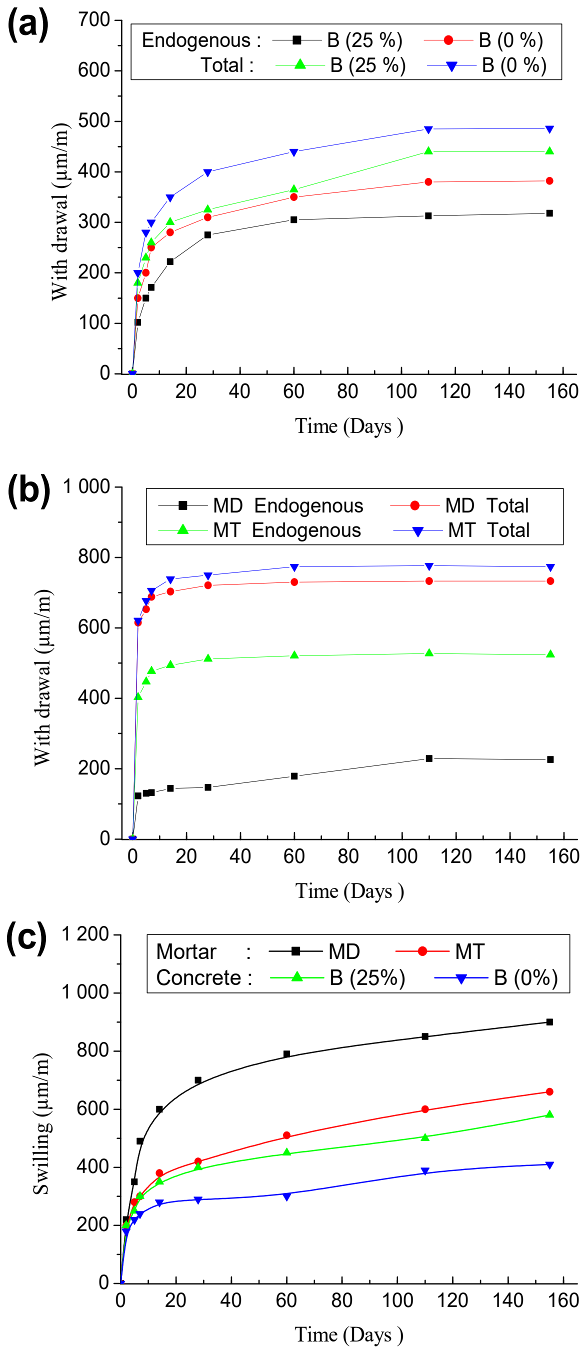

Fig. 4 - Endogenous and total shrinkage (a) concrete B (0% and 25%); (b) mortars MT and MD; (c) influence of the addition of the waste on the swelling.

porous in the networks due to the effect of temperature (exchange with the surrounding environment). After 28 days of hardening the shrinkage stabilized. It observed that the addition of waste reduces the shrinkage; our study confirms the results obtained in literature [24, 26]. It could be conclude that the addition of the waste from the Algerian cutlery industrial unit (BCR) in cement matrices reduces the shrinkage rate, which could consider an advantageous gain limiting cracking of the blocks after their hardening before putting them in the final waste storage centers.

3.4.3. Effect of the waste on the swelling of the composite materials (mortars, concretes)

The swelling ratio of this material followed the same rate for both formulations, this shows that the addition of aggregate (gravel) sand has no influence on the swelling. The curves illustrated in (Fig. 4c), show that the swelling is particularly important in materials containing waste. This can explained as follows. The amount of waste added in the formulations prevents the complete hydration of cement and contributes to the increase of the porosity of the material after its curing. The immersion of the samples in the water (swelling test) gives rise to the hydration of cement particles which remain anhydrous. The chemical reaction caused by the contact of the C_3A and H_2O particles in the presence of the gypsum forms the sulfoalumlinates (ettringite) which causes swelling under the effect of the ettringite needles thrusts formed in the pores of the material.

We observed that the rate of swelling of the concrete is lower compared to the mortars; this is due to the fact that the addition of aggregates (gravel) reduces the porosity and permeability of the concrete and consequently there will be less swelling. The contact of the cementitious materials containing the waste with the water increases the swelling rate and can induce leaching of large quantities of heavy metals into the surrounding environment. For this purpose, the use of leaching tests is inevitable for proper evaluation of the swelling phenomenon.

3.5. Behavior of Materials containing waste at leaching test (MLT)

Leaching results on monolith, obtained for the monitored chemical, species illustrated in (Fig. 5 (a-f)), shows the variation of flows leached based on the average time for 64 days of leaching in four different media neutral, basic, acid and sulfated, for 2 types of selected compositions mortar MD (25 %) and concrete B (25 %). Generally, whatever the nature of the media is, we observe the beginnings of diffusional type of release, or the slope of the curve is -0.5 [25]; the fact is due to the concentration gradients between pore water of binding material very busy and aggressive solution, no charge, create a transfer ion diffusion. This release results in the significant contribution of the monolithic outer surface of the block to leaching. Then the leached flow decreases due to exhaustion of the free waste phenomenon, which is on the outer surface of the monolithic block.

The release of these metals continues, to decrease and becomes controlled, by variations of the resulting concentrations of diffusion and chemical reactions of dissolution-precipitation (chemical equilibrium conditions). According to Figure 5 (a-f), it found that the total flow leached species followed in the sulfated medium are very

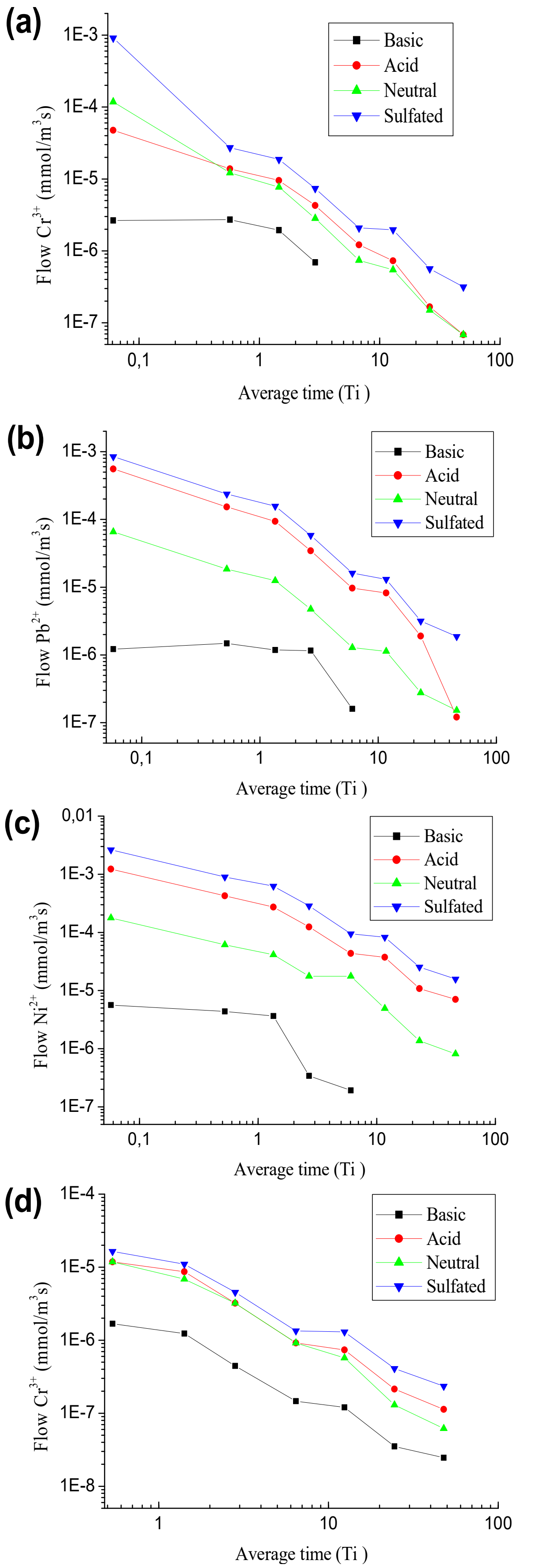

Fig. 5 – continues on next page

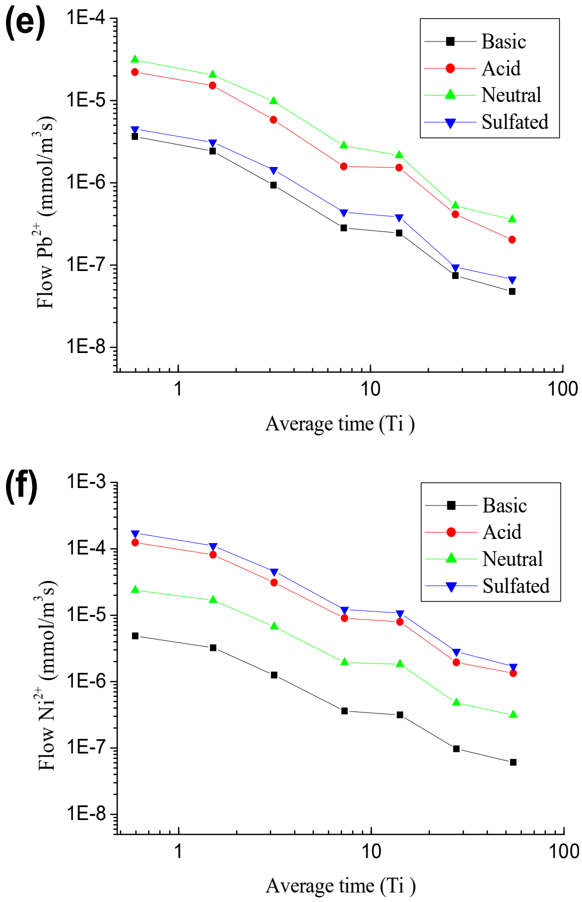

Fig. 5 - Cr^{3}, Pb^{2} and Ni^{2+} release as a function of average time (Ti): (a, b and c) for mortar MD (25%), (d, e and f) for Concrete B (25%).

important in comparison with those released into the acidic, neutral and the basic media. The flow evolved leached in diminishing order illustrated as follows: sulfated, acidic, neutral and basic media. This is due to the ionic strength of the solution sulfated compared with other leachate and the high content of sulphates (SO_4) in the sulphated medium compared to other media. The excess of the sulphates in contact with the tricalcium aluminate (C_3A) causes a swelling which favors the release. When the blocks are in contact with the sulfated solution several phenomena may performed, at the first there is dissolution of the portlandite then, increase of the volumes of the pores and at the end the sulphates penetrate intensively inside the matrix, incoming sulfates react with the ions in the pore solution, hydrated cement phases and anhydrous cement grains. These reactions generate precipitation of secondary species namely ettringite, gypsum, visual cracks and inducing the appearance of crystallization pressures. However, the deterioration of the mortar was more important relative to the concrete; this is due to the addition of gravel, which slows the diffusion of sulphates inside the block and therefore decreasing the rate of degradation of the concrete.

The increase in porosity is increasing the reactive surface (solid - solution) and thereby the release of pollutants will be more important. Figure 6 shows that in the case of a basic medium, the amount of heavy metals leached were very low; this is due to the basicity of the material, similar to that of the leaching. However, the amount of heavy metals in neutral and acidic media was very important in relation to the basic medium, this is due to the dissolution of most of the hydrates which fixes heavy metals from the waste and which becomes unstable when the pH of the solution becomes not basic. The total leached flow of heavy metals in the MD (25%) composition is higher than those leached of composition B (25%).This is due to the presence of gravel in concrete, which slows the diffusion of heavy metals to the leachate.

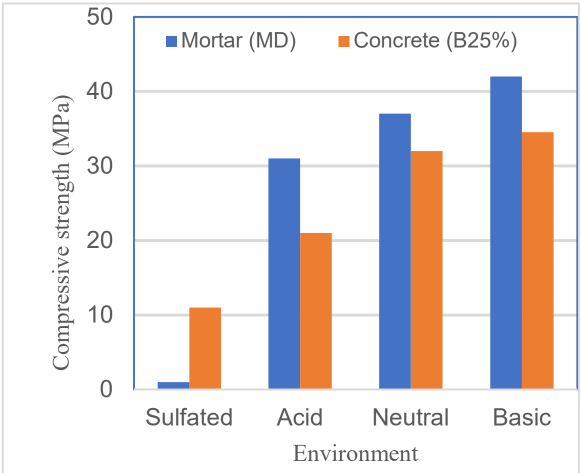

Fig. 6 - Compressive strength of monolithic blocks leached for 64 days.

Table 2

Rate leaching of heavy metals in different media for 64 days

Samples	Environment	Cr^{3+} (%)	Ni^{2+} (%)	Pb^{2+} (%)
Mortar MD (25%)	sulfate	11.85	28.67	23.01
	acid	8.23	17.33	14.42
	neutral	4.65	10.24	7.63
	basic	0.84	2.23	1.61
Concrete B (25%)	sulfate	8.66	19.41	14.66
	acid	3.92	13.07	10.53
	neutral	2.66	6.72	4.72
	basic	0.23	1.81	1.12

Small amounts of chemical species, leached after 64 days (which are equivalent to 96 years of release) are shown in Table 2. These results confirm the effectiveness of the S / S process using hydraulic binders, in trapping of heavy metals contained into the waste unit BCR.

The results recorded in Table 2, show that: the amounts of heavy metals leached after 64 days are still lower compared to leachate from the raw waste of BCR unit, which proves the effectiveness of treatment stabilization / solidification at the using hydraulic binders. The leached amounts of chromium are very low compared to other metals in the four media and for both materials types. This can be explained by its chemical nature which is poorly soluble; the amount of heavy metals released into the sulfated medium is more important compared with those observed in other media. This clearly shows the brutal effect of sulfates in the deterioration of binding materials and the release of toxic heavy metals into the environment. To confirm the deterioration of the materials submitted to the sulfated medium and evaluate their degradation in terms of compressive strength, the tests performed on monolithic blocks recovered after 64 days of leaching. The results are illustrated in the Figure 6.

4. Conclusions

The aim of this study was to investigate the effectiveness of the stabilized / solidified process of inorganic waste (hydroxide sludge and metal fibers) containing heavy metals in cement matrices. The main conclusions can be summarized as follows:

According to the TCLP test, the waste characterization results of the cutlery unit BCR of Algerians industry show that it has a polluting nature (rich in heavy metals, particularly nickel); its concentration far exceeds those required by standard, hence the fact that stabilization / solidification is recommended.

The results of the mechanical strength (compressive) are acceptable because they meet the current standard and are influenced by the presence of pollutants (type and content). Thus, it appears that the doped materials by waste have lower strengths compared to standard materials (without waste). Then, there it was observed that strength at 28 days of hardening is about 50 MPa for mortars and 37.5 MPa for concrete compared to materials with 25% waste.

Immersion studies of materials in the water, promotes the swelling phenomenon in the binding matrix; there is induced a release of large amounts of toxic metals into the surrounding environment.
The use of leaching tests to determine the intensity and dynamic release of the elements is necessary for evaluating the efficiency of the stabilization by hydraulic binders.

The use of the leaching test on monolith (MLT) in neutral (pure water), sulfated, acidic and basic media allowed the determination of the parameters controlling the release of chemical species. Tests MLT, conducted on mortar and concrete shown for the sulfated medium: the large flows of heavy metals release (nickel, chromium and lead), compared to other study areas. The literature [27] confirms this. The material consumes some sulfates of the external solution (sulfates diffusion inwards of the binding matrix).This phenomenon leads to the precipitation of secondary species, creating cracks in the block, which accelerates the degradation of composites materials and the release of toxic pollutants into the surrounding environment in large quantities.

The optimal material selected after our investigation is the concrete. This choice is, motivated by the benefits and optimum parameters presented by this.

The amounts of heavy metals leached after 64 days are still, lower compared to leachate from the raw waste of BCR unit in environment study, which proves the effectiveness of stabilization / solidification treatment by using of hydraulic binders.

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MANIFESTĂRI ȘTIINȚIFICE / SCIENTIFIC EVENTS

3rd ICDC (International Congress on Durability of Concrete) from 22nd to 25th October 2017 in the city of Adelaide, Australia. The venue is the Adelaide Convention Centre.

The ICDC durability program will include topics such as:

- Durability design
- Durability planning
- Exposure assessment
- Deterioration mechanisms
- Good practice
- Modelling of deterioration processes
- Performance of existing structures
- Concrete penetrability
- Cracking and crack control
- Maturity and matched curing
- Sampling and laboratory tests
- In service inspection and testing
- Structure health monitoring
- Concrete Repair
- Cathodic Protection
- Quality assurance

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