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DESIGN OF A NOVEL SYSTEM ALLOWING THE SELECTION OF AN ADEQUATE BINDER FOR SOLIDIFICATION/STABILIZATION OF WASTES

J. Pera, G. Thevenin, and M. Chabannet
U.R.G.C. Matériaux, INSA de Lyon, Villeurbanne, France

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ABSTRACT

Literature review shows that there is a lack of complete and consistent data on waste-binder interactions. Few links exist between research on the Solidification/Stabilization (S/S) mechanisms and the formulation of binders for immobilization. Therefore, a twofold program was developed allowing both to be done in parallel. This protocol had two targets: understanding the mechanisms involved in the S/S of heavy metals for each type of binder and rating the binder capacity for fixing heavy metals. The experimental procedure relies on two substrates: 1) the study of suspensions by means of conductimetry, XRD, FTIR, DTA, SEM-EDXA, ICP, ion chromatography, and colorimetry; and 2) the study of pure pastes for leaching tests and microstructural characterization (XRD, FTIR, DTA, SEM-EDXA). Results confirm whether the pollutant modifies the hydration or not, give access to the extent of "binder stabilization" without taking into account the solidification part, and elucidate the mechanisms involved. © *1997 Elsevier Science Ltd*

Introduction

Numerous investigations on the stabilization of metal-containing wastes using solidification/stabilization (S/S) systems have been carried out (1–11). They cover the basic understanding on the mechanisms of metal fixation in cement based S/S systems. Other studies used calorimetry, nuclear magnetic resonance (NMR) scanning electron microscopy (SEM), and transmission electron microscopy (TEM) to explain those mechanisms (12–15). Laboratory experiments also deal with the effect of water on the leachability of ions confined in a cement matrix and assess the leaching mechanisms and increase in retention of soluble species.

Despite such investigations, additional tests have to be carried out, especially when choosing a binder for the stabilization of a given waste. In this study, a procedure is presented to evaluate a given S/S cementitious system. The proposed experimental procedure relies on two substrates:

1. The study of interactions between the waste and the binder by means of suspension analyses. This allows us to verify that the binder hydrates in the presence of the waste and characterize the mechanisms of waste fixation. This procedure utilizes electric

conductimetry, ion chromatography, and ICP. The filtered solid particles of the suspension are also analyzed, using XRD, DTA, and SEM.
2. The leaching tests according to the specified regulations. Microstructural investigations are done on the paste that is used for the leaching test.

In the following study, the interactions between six binders and sodium dichromate, $Na_2Cr_2O_7 \cdot H_2O$, are analyzed.

Experiment

Materials

Six binders were investigated. They belonged to two main families:

Three ordinary portland-based cements: plain (OPC) or blended either with fly ash (FA) or metakaolin (MK), formulated as:

OPC: 100% OPC,
FA: 80% OPC + 20% FA,
MK: 80% OPC + 20% MK,

Three ground granulated blast-furnace slag (GGBFS)-based cements, referred as:

CLK: 80% GGBFS + 20% OPC,
CLC: 50% OPC + 25 GGBFS + 25% FA,
SPINOR: ultra-fine cement (3 μm average diameter); 75% GGBFS + 25% OPC.

The chemical and physical properties of these cements are summarized in Table 1.

The pollutant used in the study was $Na_2Cr_2O_7$, 2 H_2O, a very soluble salt of chromium (VI). Chromium (III) forms an insoluble hydroxide on addition of basic S/S reagents such as cement, but chromium (VI) does not. It was introduced at two contents: 0.069 mole/kg of binder, i.e., 3.6 g Cr (VI)/kg of binder, in pastes used for leaching tests; and 0.024 mole/kg, i.e., 1.25 g Cr (VI)/kg of binder, in suspensions.

Analyses

The suspensions were prepared at a water to binder ratio of 4 (16). Each series of tests was repeated three times to check reproductibility. The electric conductivity of the suspension was measured for 12 hours, using a multiplexor conductometer developed by Vernet (17). Nine cells were simultaneously investigated, each of them containing 70 mL of the model pollutant solution and 17.5 g of binder. This type of experiment was already used with other experimental conditions (water to solid ratio of 2) (18). At different times (varying from 1 min to 12 hours), the suspension was filtered. The liquid was analyzed by means of inductively coupled plasma-optic emission spectroscopy (ICP-OES) and ion chromatography. ICP-OES measured total Cr, whereas Cr (VI) was determined by specific colorimetry (19). The solid was dried in the presence of methanol, ground to a width of less than 100 μm,

TABLE 1
Chemical composition (% by mass) and physical properties of the binders

	Composition of the binder and its physical properties					
Oxides	OPC	FA	MK	CLK	CLC	SPINOR
SiO_2	19.7	24.8	26.3	26.7	30.2	26.6
Al_2O_3	6.6	8.8	11.7	8.0	9.8	8.0
CaO	64.2	51.0	51.0	50.9	47.3	50.9
Fe_2O_3	3.2	4.5	2.6	2.9	3.1	2.9
SO_3	3.2	2.6	2.6	3.3	3.1	4.0
MgO	0.7	1.5	0.6	5.1	5.8	5.1
K_2O	0.6	0.5	1.6	0.4	1.4	0.4
TiO_2	0.3	0.2	0.7	0.5	0.5	0.5
Mn_2O_3	0.1	0.1	0.1	0.3	0.3	0.3
Na_2O	0.2	0.3	0.2	0.3	0.5	0.3
P_2O_5	0.3	0.2	0.2	0.1	0.1	0.1
LOI	0.3	0.2	0.2	1.5	1.5	1.5
Density	3.1	2.7	2.7	3.2	3.0	3.2
Blaine specific surface area (m^2/kg)	300	428	457	357	300	586

and investigated by DTA, I-R spectrometry, and XRD. The tests carried out on suspensions allow us to quantify the fixation of metals by the S/S systems due to hydration and sorption reactions while preventing physical encapsulation (4).

Two leaching procedures were used in the experimental program. The first one evaluated the maximum quantity of pollutant released and was performed according to the French standard NF X31-210. Pastes, cast at the same workability (Table 2), were hydrated for 28 days in sealed bags at 20°C, then crushed to less than 4 mm. We placed 100 g of crushed paste in 1000 g of de-ionized water and stirred. Leachates were collected at 16, 32, and 48 hours, and analyzed by ICP-OES and ion chromatography.

The second test was a diffusion test (DLT adapted from ANS 16.1). It was carried out on cylinders of polluted paste hydrated for 360 days ($\varnothing = 30$ mm; $H = 43$ mm). Cylinders were placed in de-ionized water (273.3 mL), which means a volume to surface ratio of 50 mm. At different times (0, 1, 2, 7, 24 hours; 2, 3, 5, 7, 14, 28 and 45 days), the solution was analyzed by ICP-OES, colorimetry and the pH measured. At the end of the test, the porosity of the paste was investigated by mercury intrusion and the microstructure observed by SEM-EDXA.

TABLE 2
Water to solid ratios (W/S) of pastes

Type of binder	OPC	FA	MK	CLK	CLC	SPINOR
W/S	0.325	0.350	0.375	0.325	0.325	0.450

TABLE 3
Influence of sodium dichromate on the conductivity of binder suspensions

Type of binder	Polluted (P) or not (N)	Initial conductivity (mS/cm)	Maximum conductivity (mS/cm)	Length of the dormant period (min)
OPC	N	10.5	14.4	150
	P	11.3	15.4	180
FA	N	8.8	14.1	255
	P	9.3	15.3	285
MK	N	9.2	14.2	225
	P	9.4	15.7	240
CLK	N	8.2	14.5	270
	P	8.2	15.0	285
CLC	N	9.6	14.4	210
	P	9.6	15.9	225
SPINOR	N	10.0	17.0	210
	P	9.5	17.5	225

Results and Discussion

Suspensions

The results obtained from the conductimetric tests are summarized in Table 3.

Sodium dichromate leads to an increase of the dormant period length: from 15 to 30 min, according to the binder used. The maximum value of conductivity is generally increased by 1 mS/cm, which is the conductivity of a sodium dichromate solution.

In order to understand the mechanisms involved in the reactions between the binder and the pollutant, three binders were more deeply investigated: OPC, CLK, and SPINOR. The liquid phase of the suspensions was analyzed as well as the microstructure of the filtration cake. The ion analysis of the liquid phases is presented in Table 4. For each binder, the initial Cr (VI) content was 6 mmol/L.

The reactions between sodium dichromate and OPC can be summarized as follows. A strong decrease in Cr (VI) concentration is recorded during the first minute (0.7 mmol/L), then, there is a slight decrease until 360 min, followed by a high consumption of Cr (VI) at 6 hours (2.5 mmol/L). The solution of the polluted binder is deficient in Ca ions for 6 hrs, due to the precipitation of calcium-chromate, $Ca_3(CrO_4)_2$, as shown by XRD investigation of the cake. The sulphate ion concentration of the suspension with Cr (VI) is always higher than that of plain OPC (+ 1 mmol/L), which proves that the sodium dichromate does not modify gypsum dissolution. The OH^- concentration in the presence of dichromate is less than that found in the reference solution, due to the precipitation of hydrates.

Chromium immobilization in cements was investigated by Cocke et al (20,21); Cr appears to nucleate the precipitation of hydrates. Ivey et al. (22) observed that, in the Cr-containing samples, ettringite was absent. McPhee (6) suggested that the immobilization of chromium may be associated with the formation of Ca, Cr aluminate hydrates. The uptake of ettringite for many species has been recognized for some time and recently McCarthy et al. (23)

TABLE 4
Evolution of the ion concentration of solutions (mmol/L)

Ion	Time (min)	Binder					
		OPC		CLK		SPINOR	
		N*	P†	N	P	N	P
Cr^{6+}	1	—	5.3	—	5.1	—	5.9
	150	—	5.0	—	5.1	—	5.9
	360	—	4.7	—	4.3	—	3.5
	720	—	2.2	—	3.0	—	0.8
Ca^{2+}	1	25.5	23.8	27.5	27.0	28.0	28.0
	150	31.0	28.0	31.5	28.0	33.0	35.0
	360	31.5	30.5	35.0	30.0	23.0	31.0
	720	26.5	26.5	30.0	30.0	7.0	7.0
SO_4^{2-}	1	15.0	16.0	19.0	20.0	35.0	35.0
	150	14.0	15.2	13.5	14.0	17.0	17.0
	360	13.0	14.0	12.5	13.0	5.0	15.0
	720	16.0	16.5	11.5	10.5	0.0	0.0
OH^-	1	52.0	52.0	56.0	65.0	5.0	5.0
	150	40.0	38.0	56.0	65.0	57.0	57.0
	210	37.0	28.0	42.0	45.0	62.0	60.0
	360	23.0	10.0	—	—	60.0	40.0

*N, plain binder; †P, binder doped with Cr (VI).

summarized work specifically relating to the uptake of oxyanion species of Se, Cr, and B. Cr (III) can substitute for Al in the columns $[Ca_6(Al(OH)_6)_2\ 24H_2O]^{6+}$, whereas chromate replaces sulphate in the channels $[(oxyanion)_{2-4}\ (OH)_{0-4}\ (H_2O_{0-6}]^{6-}$. Some authors have synthetized such phases from aqueous solutions (24–28).

The mineralogical XRD analysis using the radiation Cu-K_α was carried out; typical phases in the sulphate-cement system such as ettringite and monosulpho-aluminate were found, but some diffraction lines did not correspond to those hydrates. Cr-containing hydrates certainly

TABLE 5
Inter-reticular distances of different sulpho-aluminates (Å) (JCPDS-ICDD, 1994)

Sulpho-aluminate	d (Å)				
Ettringite 3CaO, Al_2O_3, $3CaSO_4$, $32H_2O$	9.72	5.61	3.87	3.48	3.24
Cr-Ettringite 3CaO, Al_2O_3, $3CaCrO_4$, $32H_2O$	9.87	5.70	4.71	3.91	3.52
Bentorite 3CaO, Cr_2O_3, $3CaSO_4$, $32H_2O$	9.66	5.59	3.89	3.60	3.23
Na-Ettringite 3CaO, Al_2O_3, $CaSO_4$, 0.5 Na_2SO_4, $15H_2O$	10.03	5.01	4.45	3.53	2.87
Experimental	10.14	5.05	4.48	3.55	2.88
	9.80	5.63	3.89	3.48	3.25

TABLE 6

Results of suspension study: modifications due to $Na_2Cr_2O_7$, $2H_2O$

Events	Binder		
	OPC	CLK	SPINOR
Kinetics of hydration	2 phases	2 phases	1 delayed phase
Phase I	1 min	1 min	
· modified concentrations	Ca^{2+}, SO_4^{2-}	Ca^{2+}, SO_4^{2-}, OH^-	
· modified hydrates	ettringite, CSH	ettringite, CSH	—
· new hydrates		$Ca(CrO_2)_2$ $Ca_4Al_6O_{12}CrO_4$	
Phase II	6–24 hrs	6–24 hrs	2.5–12 hrs
· modified concentrations	Ca^{2+}, SO_4^{2-}, OH^-	Ca^{2+}, SO_4^-	Ca^{2+}, SO_4^{2-}, OH^-
· modified hydrates	ettringite, CSH	ettringite, CSH	ettringite, CSH
· new hydrates	calcium-chromate	—	—
	Na-Cr-AF_t	—	—
	mono-chromoaluminate	mono-chromoaluminate	mono-chromoaluminate

precipitated, as shown in Table 5. Table 5 summarizes the inter-reticular distances of the peaks of ettringite, Cr-modified ettringites, Na-modified ettringite, and experimental data.

One of the phases observed by XRD analysis is Cr-modified ettringite, and the other is close to 3CaO, Al_2O_3, $CaSO_4$, 0.5 Na_2SO_4, $15H_2O$, and was identified by Serclerat (29) to be: 3CaO, Al_2O_3, $CaCrO_4$, 0.5 Na_2CrO_4, $15H_2O$, a Na-Cr modified AF_t phase.

When CLK is used, the initial concentration of Cr (VI) decreases to 5.1 mmol/L, then remains constant and slightly decreases to 4.3 mmol/L after 6 hours. From 6 to 12 hours, the rate of Cr (VI) consumption is 0.21 mmol/L per hour, which is less than with OPC (−0,42 mmol/L per hour). Ca^{2+}, SO_4^{2-} and OH^- concentrations are higher in the polluted solution than in the reference one. The analysis of the filtration cake showed that the quantity of hydrates formed was lower until 150 min, whereas Cr (VI) was consumed to precipitate calcium-chromium oxide and calcium-monochromoaluminate (3CaO, Al_2O_3, CrO_4, $12H_2O$).

With SPINOR, the Cr (VI) concentration does not decrease instantaneously as with OPC and CLK. After 150 min, it decreases at a rate of 0.67 mmol/hour. At the end of the test, there is only 0.8 mmol/L of Cr (VI) remaining. In comparison with the reference, this solution exhibits larger concentrations in calcium and sulphate ions and lower OH^- concentrations. These differences are limited with time and they disappear after 12 hours. The analysis of the solid part of the suspension showed that monochromoaluminate and Cr-ettringite precipitated. The calcium-chromate formed in OPC polluted by sodium dichromate did not appear with SPINOR.

The main findings issued from the study of suspensions are summarized in Table 6.

The modification of C-S-H was observed by IR spectrometry which showed a displacement of the Si-O band at 970 cm^{-1} towards higher wave numbers.

The immobilization of Cr (VI) by hydraulic binders (OPC, CLK, SPINOR) probably occurs by one or a combination of the following mechanisms:

TABLE 7
Results of the leaching tests (NF X 31-210) (mg/kg)

Binder	Cr (VI) concentration			Efficiency of the binder (%)
	16 hrs	32 hrs	48 hrs	
OPC	3	6	9	99.67
FA	34	49	62	97.66
MK	13	21	30	98.85
CLK	5	10	15	99.44
CLC	16	26	37	98.62
SPINOR	7	14	21	99.15

Addition: C-S-H + chromate → Cr-C-S-H
Substitution: ettringite → Cr-ettringite
monosulphoaluminate → monochromoaluminate
Precipitation of calcium + chromate → calciumchromate
new compounds calcium + chromate + sodium → Na-Cr-AF_t

Leaching Tests

The results of the leaching tests carried out according to the French standard NF X 31-210 are given in Table 7.

From this table, two types of behaviour are distinguished: the rate of leaching is constant with time for OPC, CLK, SPINOR, and it can be concluded that Cr (VI) is immobilized in hydrates or new precipitated compounds; and the rate of leaching is variable with time for FA, MK, CLC.

The results obtained from the diffusion test (DLT) allowed the kinetics of Cr (VI) leaching from cylinders to be modelled by two phenomena (30):

TABLE 8
Double period of diffusion: coefficients derived from the modelling

Binder	First period		Second period	
	L_i	D_{ai} (cm^2/s)	L_f	D_f (cm^2/s)
OPC	2.11×10^{-4}	3.4×10^{-10}	11.40×10^{-4}	3.9×10^{-11}
FA	0.66×10^{-4}	6.6×10^{-10}	18.69×10^{-4}	12.5×10^{-11}
MK	0.44×10^{-4}	4.3×10^{-10}	11.02×10^{-4}	7.0×10^{-11}
CLK	7.07×10^{-4}	30.0×10^{-10}	23.36×10^{-4}	12.6×10^{-11}
CLC	4.05×10^{-4}	23.0×10^{-10}	26.66×10^{-4}	14.3×10^{-11}
SPINOR	11.56×10^{-4}	58.0×10^{-10}	28.07×10^{-4}	8.9×10^{-11}

TABLE 9
Porosity of samples after the DLT test

Binder	Total porosity (%)	Pore size distribution (%)		
		Micropores 0.006 to 0.1 μm	Mesopores 0.1 to 0.6 μm	Macropores >0.6 μm
OPC	15.57	80.17	16.95	2.88
FA	21.20	95.81	3.47	0.73
MK	21.56	93.92	1.33	4.75
CLK	19.97	82.78	12.48	4.75
CLC	21.31	92.58	3.42	3.99
SPINOR	28.98	92.92	1.61	5.46

1. the initital release of the nonimmobilized Cr (VI) fraction, governed by diffusion, within less than 24 hours. This is characterized by two parameters: initial leaching coefficient L_i, and initial apparent diffusion coefficient D_{ai}, which is high; and
2. the progressive leaching of fixed Cr (VI), characterized by two other parameters: a fixed leaching coefficient L_f; and a fixed apparent diffusion coefficient D_{af}, which is low.

The D_a values were calculated using the formula proposed by Cote (31):

$$D_a = \pi\left[\frac{C_i}{C_o} \times \frac{V}{S} \times \frac{1}{\Delta t_i}\right]^2 x t_i$$

where: D_a is the apparent diffusion coefficient (cm^2/s), C_i is the concentration of the solution within sequence i, C_o is the initial concentration of the solid, V is the volume of the solvant (cm^3), S is the surface of the sample (cm^2), Δt_i is the length of the sequence i (s), and $T_i$is the average value of this period (s).

The D_a values for each phenomenon are given in Table 8.

From the DLT test, it appears that OPC-based cements are less leachable than GGBFS-based cements. The presence of fly ash or metakaolin modifies the initial quantity of leached Cr (VI), which lowered. In the second period, these admixtures are less effective.

CLK and SPINOR are less efficient than FA and MK in the leaching test carried out on monolithic samples (DLT). This can be explained by the porosity of the pastes, as shown in Table 9. The porosity was measured by mercury intrusion using a Micromeritics Autopore III 9410 porosimeter.

SPINOR presents the highest total porosity due to a higher water demand for its placing. CLK does not contain a high amount of micropores compared to FA and MK.

Conclusion

A new procedure allowing the selection of an adequate binder for waste immobilization was described in this study. The regulations are based on leaching tests, variable from one country to another. It was shown that such tests are not sufficient to accept a binder for the S/S of wastes. Two types of experiments were proposed: the study of interactions between the waste and the binder through analyses of suspensions; and the standard leaching tests.

This procedure was applied to one waste (sodium dichromate) and six binders. The results obtained show that the most efficient binder is OPC. The presence of fly ash or metakaolin is not favourable towards chromate fixation. The slag-based cements (CLK or SPINOR) in general perform well, except in the diffusion test due to their total porosity and pore distribution.

The fixation of other wastes was studied according to this procedure and will be presented in another paper.

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