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Interactions between lead and different binders

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Abstract

The present study describes the interactions between lead nitrate, representative of soluble lead compounds, and seven cements based on either ordinary Portland cement (OPC) or ground-granulated blast furnace slag (GGBFS). Suspensions of cements contaminated by lead nitrate were investigated by means of electrical conductivity, powder X-ray diffraction, Fourier Transform-infrared spectrometry, scanning electron microscopy, inductively coupled plasma-optic emission spectroscopy, and ionic chromatography. Pastes cast with these contaminated cements were submitted to leaching tests. The presence of fly ash was not favourable toward lead fixation and the best retention of lead was obtained with GGBFS-based cements. The most efficient binder was SPINOR, an ultrafine GGBFS-based cement.

Keywords: Waste management; Heavy metals; Blended cement; Microstructure; Diffusion

1. Introduction

Lead, which is listed as a priority pollutant by the EPA (EPA code D0008) and other environmental agencies, has been shown to retard cement hydration; the effects with soluble PbO are more pronounced than with soluble $Pb(NO_3)_2$ [1]. Early work by Bishop [2] found that lead leached at a rate much slower than what was expected based on hydroxide solubility. Strong complexation of lead in the cement matrix was suggested as a possible explanation. Ortego and associates [3] performed thermal analysis and Fourier transform infrared studies based on lead nitrate-doped cement and concluded that the lead was present primarily on the surface. They suggested that lead nitrate retards cement hydration through the formation of lead sulfate or its various basic forms [3].

Despite such investigations, additional tests have to be carried out, especially when choosing a binder for solidification/stabilization (S/S) 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 series of test:

1. The study of interactions between the waste and the binder by means of suspension analysis. 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 inductively coupled plasma-optic emission spectroscopy (ICP-OES). The filtered solid particles of the suspension are also analyzed using X-ray diffraction (XRD), differential thermal analysis (DTA), and scanning electron microscopy (SEM).
2. The leaching test according to the specified regulations. Microstructural investigations are done on the paste that is used for the leaching test. This method was already used to analyze the interactions between different cements and sodium dichromate, and has been published in a previous paper [4]. In the present study, the interactions between seven binders and lead nitrate, $Pb(NO_3)_2$, are analyzed.

2. Methods

2.1. Materials

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

1. Three ordinary Portland-based cements: plain (OPC), or blended with fly ash (FA) or metakaolin (MK) formulated as: OPC: 100% OPC, FA: 80% OPC + 20% FA, MK: 80% OPC + 20% MK
2. Four ground-granulated blast furnace slag (GGBFS)-based cements, referred as: CLK: 80% GGBFS + 20% OPC, CLC: 25% GGBFS + 50% OPC + 25% FA, SPINOR: 75% GGBFS + 25% OPC, ultrafine cement (3 μm average diameter), CLC + SPINOR: 90% CLC + 10% SPINOR.

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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	.00	586

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

The pollutant used in this study was $Pb(NO_3)_2$, a soluble lead salt. It was introduced at two contents: 0.069 mole/Kg of binder (i.e., 14.3 g Pb/Kg of binder) in pastes used for leaching tests; and 0.024 mole/Kg of binder (i.e., 4.97 g Pb/Kg of binder) in suspensions.

2.2. Analyses

The suspensions were prepared at a water-to-binder ratio of 4 [5]. Each series of tests was repeated three times to check reproducibility. The electric conductivity of the suspension was measured for at least 12 h, using a multiplexor conductometer developed by Vernet [6]. The apparatus enables nine cells to be monitored simultaneously, each of them containing 70 mL of the model pollutant solution and 17.5 g of binder. The apparatus was already used with other experimental conditions (water over solid ratio of 2) [7]. At different times (ranging from 1 min to 48 h), the suspension was filtered. The liquid was analyzed by means of ICP-OES and ionic chromatography. The solid part of the suspension was dried in the presence of methanol, ground below 100 μm, and investigated by DTA, infrared spectrometry, and XRD. The test carried out on suspensions allow us to quantify the fixation of metal by the S/S system due to the hydration and sorption reactions while preventing physical encapsulation [8].

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. Those paste were then crushed below 100 μm. Crushed paste (100 g) was put in 1,000 g of deionized water and stirred. Leachates were collected at 16, 32, and 48 hours, and analyzed by ICP-OES and ionic chromatography.

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

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

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 (∅= 30 mm, height = 43 mm). Cylinders were placed in deionized water (273.3 mL), which provides needed volume-to-surface ratio of 50 mm. At different times (0, 1, 2, 7, 24 h, 2, 3, 5, 7, 14, 28 days), the solution was analyzed by ICP-OES, colorimetry, and pH measurement. At the end of the test, the porosity of the paste was investigated by mercury intrusion and the microstructure observed by SEM.

3. Results and discussion

3.1. Suspensions

The results derived from the conductometric test are summarized in Table 3. The binders investigated behave in a similar way; differences lie in the intensity of events. Suspensions with lead nitrate show a lower initial conductivity and a longer dormant period than those without the pollutant. The period increase ranges from 3 h for OPC binder up to 16 h for CLK. The dormant period is always characterized by a linear increase of conductivity up to a maximum value. However, the slopes recorded on nonpolluted suspensions are much more pronounced than those recorded on polluted ones (between −0.3 and −1.2 mS/cm/mn). This shows a delay in the precipitation of calcium hydroxide that starts once the maximum of conductivity is reached.

To understand the mechanisms involved in the reactions between the binder and the pollutant, three binders were more fully 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 phase is presented in Table 4. For each binder the initial concentration of lead was 6 mmol/l.

The reaction between lead nitrate and OPC may be summarized as follows. A strong decrease in Pb concentration is recorded within the first minute (4.9 mmol/L) (Table 4). Then a slow linear decrease (0.1 mmol/h) occurs for the next 10 h. From the first minutes, the solution is strongly deficient in hydroxyl ions (between −20 to −50 mmol/L) due to the precipitation of oxides and hydroxides of lead as shown by XRD and DTA investigations of the filtration cake. The calcium ion concentration of the suspension with lead nitrate is lower than that of plain OPC, whereas that of sulfate remains similar. DTA confirms that gypsum dissolution is not modified by the presence of lead nitrate in the suspension. Because calcium is no longer in the form of

Table 3
Influence of lead nitrate on the conductivity of binder suspensions

Type of binder	Polluted (P) or not (N)	Initial conductivity (mS/cm)	Maximal conductivity (mS/cm)	Slope of the dormant period (mS/cm/min)	Length of the dormant period
OPC	N	10.5	14.4	1.15	2 h, 30 min
	P	10.5	14.4	0.80	6 h, 00 min
FA	N	8.8	14.1	1.00	4 h, 15 min
	P	8.5	15.5	0.20	15 h, 45 min
MK	N	9.2	14.2	1.20	3 h, 45 min
	P	9.0	13.9	1.40	7 h, 45 min
CLK	N	8.2	14.5	0.40	4 h, 30 min
	P	6.4	15.3	0.20	20 h, 45 min
CLC	N	9.6	14.4	1.20	3 h, 30 min
	P	6.6	13.4	0.40	16 h, 45 min
SPINOR	N	10	17.0	1.80	3 h, 30 min
	P	9.5	17.0	1.20	5 h, 30 min
CLC-SPINOR	N	9.0	14.8	1.60	3 h, 30 min
	P	6.5	13.8	0.40	5 h, 20 min

gypsum and indeed absent from the liquid phase, it must be involved in the formation of new solid compounds or gels.

Addition of Pb to CLK leads to a strong reduction of the conductivity slope over the dormant period. The conductivity curve in the presence of Pb is almost flat. The Pb suspension analysis reveals a strong lack of hydroxyl ions. Moreover a severe drop in the lead concentration is recorded initially (only 0.96 Pb mmol/L after 1 min out of the 6 mmol/L initially added). At that time, the quantity of the sulfate and lead missing from solution are equal. Afterwards, lead immobilization continues rather slowly (0.03 mmol/h) between 1 h, 30 min and 20 h. Precipitation of lead nitrate is not responsible for this, since the nitrate added remains in solution over the test. Calcium ion concentration is

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

		Binder					
		OPC		CLK		SPINOR	
Ion	Time (min)	N[a]	P[b]	N[a]	P[b]	N[a]	P[b]
Pb^{2+}	1	–	1.2	–	0.9	–	0.02
	150	–	0.7	–	0.2	–	ND
	600	–	0.3	–	0.2	–	0.01
	C_{max}	–	(360 mn) 0.5	–	(1245) 0.1	–	(330 mn) ND
OH^-	1	33.1	10.8	22.8	0.6	5.9	0.3
	150	51.9	14.4	34.8	0.3	5.8	0.2
	240	61.9	11.4	61.9	0.4	5.8	5.3
	600	52.2	28.1	38.0	0.8	3.5	14.1
	C_{max}	(155) 54.5	(360) 12.9	(270) 55.70	(1245) 26.10	(210) 6.2	(330) 18.2
NO_3^{2-}	1	–	11.0	–	11.9	–	10.4
	150	–	10.7	–	11.8	–	10.4
	240	–	9.9	–	11.7	–	10.1
	600	–	9.6	–	11.6	–	9.8
	C_{max}		(360) 9.8		(1245) 11.5		(330) 5.7
Ca^{2+}	1	20.8	21.1	25.2	21.0	19.5	28.2
	150	24.9	23.2	27.4	24.0	31.2	29.8
	240	25.7	23.0	28.0	25.0	32.3	30.5
	600	26.9	26.8	32.2	27.0	7.6	5.8
	C_{max}	(155) 24.5	(360) 20.8	(270) 28.6	(1245) 32.7	(210) 31.0	(330) 31.5
SO_4^{2-}	1	16.5	16.1	18.6	13.0	12.1	16.3
	150	16.0	16.3	14.5	15.0	7.3	10.9
	240	16.0	15.4	14.1	15.0	8.7	10.7
	600	15.4	16.3	12.7	14.0	0	0.0
	C_{max}	(155) 15.7	(360) 16.4	(270) 13.2	(1245) 12.5	(210) 8.4	(330) 8.2

ND = not detected.
[a] Plain binder.
[b] Binder spiked with lead nitrate.

Table 5
Results of suspension study: modification due to $Pb(NO_3)_2 \cdot 4H_2O$

	Binder		
Kinetic of Fixation	OPC (two phases)	CLK (two phases)	SPINOR (one phase)
Phase I (time)	1 min	1 min	1 min
Pb% precipitated	79%	96%	100%
Modified concentration	$-OH/-NO_3$	$-OH/-Ca/-SO_4$	$-OH/-NO_3$ $+Ca/+SO_4$
Modified hydrates	Ettringite, CSH	Ettringite, CSH	Ettringite, CSH, $Pb_7(NO_3)_4OH_{10}$
New hydrates	$Pb_7(SiO_3) \cdot H_2O$ colloidal gel	$Pb_7(SiO_3) \cdot H_2O$ colloidal gel	$Pb_7(SiO_3) \cdot H_2O$ colloidal gel
Phase II (time)	1 min/10 h	1 min/10 h	1 min/10 h
Pb% precipitated	94%	96%	100%
Modified concentration	$-OH/-Ca/-Na$	$-OH/-Ca/-Na/-K+SO$	$-OH/-NO_3$ $+Ca/+SO$
Modified hydrates	Ettringite, CSH, gels	Ettringite, CSH, gels	Ettringite, CSH, gels
New hydrates	solid solution Pb-CSH	solid solution Pb-CSH	solid solution Pb-CSH

3 mmol/L lower than the reference one in the presence of Pb, regardless the testing time.

When Pb is added to SPINOR, a strong precipitation reaction occurs. Concentration of lead in solutions drops from the initial 6 mmol/L to traces. At 1 min, several differences exist between the spiked suspension and the reference ones: hydroxyl ions are missing. Calcium ion is in excess, and this excess is equal to the quantity of lead precipitated. This suggests that calcium may be replaced by lead.

The main findings from the study of suspensions are summarized in Table 5. The modification of C-S-H was observed by IR spectrometry, which showed a displacement of the Si-O band at 970 cm^{-1} toward higher wave numbers. The immobilization of lead by hydraulic binders (OPC, CLK, SPINOR) probably occurs by one or a combination of the following mechanisms:

Addition: C-S-H + Pb $\rightarrow$ Pb-C-S-H
Substitution: C-S-H + Pb $\rightarrow$ Pb-S-H + calcium
Precipitation of new compound: Pb + OH + Ca + SO_4 $\rightarrow$ mixed salts

Tashiro and others [9–11] showed that in the presence of lead the binder hydration occurred at a much slower rate and mortar mechanical strength was reduced. It is thought that coating of C_3S by metal ions or complex is responsible for the retardation. Moreover, a correlation seems to exist between the coating effect and the ionic potential of metal ion (ionic potential $Ca^{2+} = 0.50$, ionic potential $Pb^{2+} = 0.62$). Lead immobilization in cement was also investigated by Bhatty [8]. His conclusion is that Pb is included by an addition reaction in calcium silicate hydrates. The exact stochiometry of this calcium silicate is still unknown. Alford and Rahman [12] and Balzamo et al. [13] brought evidence that on lead-spiked samples mixed salts are precipitated in a colloidal gelatinous form. Those salts are sensitive to the pH of the pore solutions and undergo fluctuation and reprecipitation. The amount of salt formed is proportional to the amount of lead added. At some point the system might be overwhelmed by the expansive lead gel formation. This will induce microcracking effects and reduce mechanical and stabilization properties. Cocke et al. [14,15] determined by X-ray photoelectron spectroscopy (XPS) detection that lead has a tendency to form a selective coating on the outer portion of cement components. In this area, no trace of nitrate or sulfate was detected. This means that either lead is in the form of carbonate or failure occurs in detection. Grutzeck [16] studied lime silicate-spiked system. Nuclear magnetic resonance results demonstrated that lead nitrate enhances the degree of silicate polymerization and retards setting reaction. Chatelet et al. [17] confirmed that Pb immobilization in calcium silicate hydrate (C-S-H) occurs as soon as C_3S begins to hydrolyze. The "Pb-C-S-H" is referred as a C-S-H of regular stochiometry where lead replaces calcium. Other publications state that lead fixation is due to a double phenomenon: first the precipitation of a metallic hydroxide and then encapsulation of this compound in the C-S-H phase. Bishop [18] followed a lead-polluted cement sample submitted to some sequential extraction, and found that lead is bound together with Ca, Si, and to a lesser extent Fe and Al.

3.2. Leaching tests

The results of the leaching tests carried out according to the French standard NF X 31-210 are given in Fig. 1. OPC, FA, and CLK exceed the threshold reject value specified by this regulation, although the fraction of lead extracted from the binders is quite low (0.05 to 0.71% of the initial Pb extracted). SPINOR, MK, CLC, and CLC/SPINOR give extraction values lower than the defined threshold.

Fig. 1 illustrates two types of release behaviour: (1) after an initial wash off, the second and third lixivations release the same amount of lead (SPINOR, MK), and (2) the quantity leached decreases with time (OPC, FA, CLC, CLC/SPINOR, CLK). This can either mean that the fraction of lead released is stabilized in the form of various mixed salts of different solubility, or that the Pb precipitation salts undergo cycles of dissolution/reprecipitation during the test. The plot of total release against pH (Fig. 2) confirms that the extraction mecha-

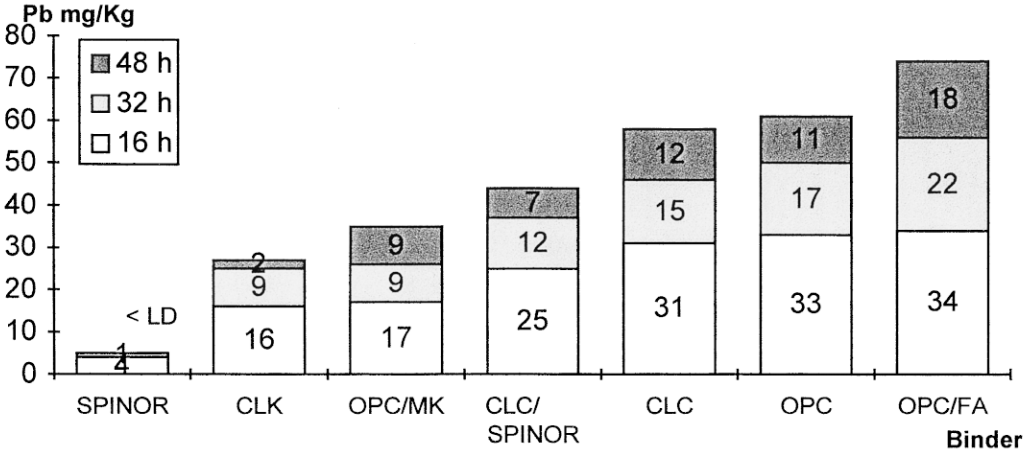

Fig. 1. Results of the leaching tests (NF X 31-210) (mg/Kg).

nism is function of pH. Moreover, analysis of the leachate shows that lead and nitrate releases are parallel.

The results obtained from the diffusion test are illustrated by two kinds of mechanisms depending upon the binder used (Table 6). Release of lead plotted vs. square root of time for SPINOR and CLK result in a straight line. Lead rejects are therefore modeled by a single phenomenon. Whereas the curves drawn for the five other binders (OPC, MK, FA, CLC, CLC/SPINOR) include an inflection point and therefore define two lines, the kinetics of release of lead from cylinders has to be modeled by a double phenomenon [19].

1. Initial release of the "lightly" fixed lead fraction governed by diffusion within 4 days or less depending upon the binder. Two parameters enable the model: initial leaching coefficient and initial apparent diffusion coefficient (D_{ai}).
2. Progressive release of fixed lead characterized by two new coefficients: fixed leaching coefficient and apparent diffusion coefficient (D_{af}).

The apparent diffusion coefficient values were calculated using the formula proposed by Cote [20]. The D_a values for each phenomenon are given in Table 6.

Using Portland cement alone or substituting part of a binder by class F fly ash is detrimental to lead fixation. Tamas et al. [21] confirms that fly ash is a preferential substrate for metal fixation. Moreover, their high specific surface increases the contact binder/leachant and the resulting release.

Substitution by MK or SPINOR causes a lowering of the four coefficients defined. On the contrary, class F FA addition increases the diffusion coefficient of the fixed lead fraction. Wang and Vipulanandan [22] bring new arguments on the subject. Using a class C fly ash with hydraulic and pozzolanic activity enhances the fixation of lead nitrate and lowers the deleterious effect of lead nitrate on hydration.

The binders rank from the most to the least able to solidify/stabilize lead nitrate: SPINOR, CLK, MK, CLC/SPINOR, CLC, OPC, FA; this is the same according to DLT and NF tests. Macroporosity and diffusion towards the

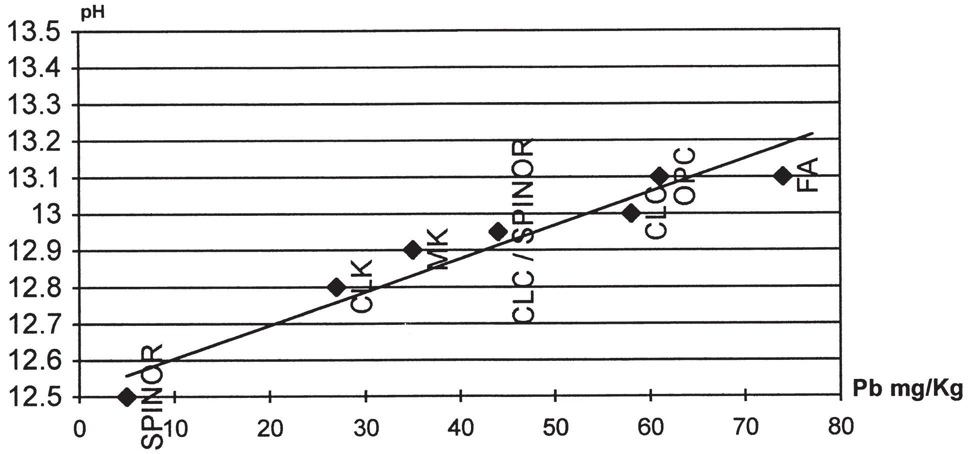

Fig. 2. Amount of lead extracted vs. pH of lixiviat (NF X31.210).

Table 6
Double period of diffusion: coefficients derived from modelling

	First period		Second period	
Type of binder	L_i	D_{ai} (cm^2/s)	L_f	D_{af} (cm^2/s)
OPC	$4.00\ 10^{-5}$	$60.0\ 10^{-13}$	$13.00\ 10^{-5}$	$4.65\ 10^{-13}$
FA	$-0.25\ 10^{-5}$	$7.7\ 10^{-13}$	$6.00\ 10^{-5}$	$0.42\ 10^{-13}$
MK	$-2.00\ 10^{-5}$	$20.0\ 10^{-13}$	$15.00\ 10^{-5}$	$8.49\ 10^{-13}$
CLK	–	–	$0.35\ 10^{-5}$	$0.84\ 10^{-13}$
CLC	–	–	$0.50\ 10^{-5}$	$0.81\ 10^{-13}$
SPINOR	$-1.15\ 10^{-5}$	$13.0\ 10^{-13}$	$12\ 10^{-5}$	$5.25\ 10^{-13}$
CLC-SPINOR	$-0.75\ 10^{-5}$	$1.2\ 10^{-13}$	$4\ 10^{-5}$	$0.90\ 10^{-13}$

cylinder sample are not the governing leaching factors. NF test shows that the seven binders used are able to fix between 99.3 and 99.95% of the lead added, whereas the DLT test shows a fixation ability of 99.65 up to 99.995%.

4. Conclusions

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 and vary 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 suspension and the standard leaching tests.

This procedure was applied to one waste (lead nitrate) and seven binders. The results obtained show that the most efficient binder is SPINOR. The presence of fly ash is not favourable toward lead fixation. In general, GGBFS cements perform better than OPC cements. Addition of metakaolin modifies the porosity and the solubility of the mixed lead salts that are formed.

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