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Cement and Concrete Research 36 (2006) 1628–1634
Cement and Concrete Research

Stabilization of electric arc furnace dust by the use of cementitious materials: Ionic competition and long-term leachability

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Received 21 July 2005; accepted 19 May 2006

Abstract

Ionic competition in stabilization of major heavy metals from electric arc furnace dust (EAFD) was investigated. The cementitious materials tested (ground granulated blast furnace slag (GGBFS) and ordinary Portland cement (OPC)) were put in contact with solutions made of various combinations of Cr(VI), Pb(II), Zn(II), Ni(II) and Mo(VI) ions. The presence of Ni, Zn or Mo did not influence the Cr fixation by GGBFS and OPC. The ionic competition phenomenon between Cr and Pb was observed for GGBFS in particular.

Long-term leaching tests with OPC-EAFD and GGBFS-EAFD showed that OPC is more effective in fixing Cr at 7 days (4.7 mg/L in solution) than GGBFS (79.4 mg/L in solution). GGBFS becomes effective with time and offers a better performance from 56 to 365 days (under 1 mg/L in solution) than OPC (until 11 mg/L in solution). GGBFS and OPC decreased Zn, Pb and Ni concentrations in leaching solutions under 2.5 mg/L.

Keywords: Heavy metals; Granulated blast furnace slag; Portland cement; Waste management

1. Introduction

Solidification/stabilization (S/S) techniques have been widely used to immobilize contaminated wastes before their disposal into the environment [1]. S/S techniques are a very effective tool and a standard process in liquid and hazardous waste treatment and disposal [2,3] and may be used to treat electric arc furnace dust (EAFD). EAFD is a toxic waste by-product of the stainless steel industry and is not reused or recycled to any extent [4]. This type of waste has particularly high contents of Cr and other toxic metals (Pb, Zn and Ni) [5–7]. The follow-up of Cr is of special interest due to its important content and high toxicity, particularly in the Cr(VI) state.

Solidification/stabilization techniques involve two principles. Solidification is the improvement of matrix engineering properties [3,8,9]. Stabilization is the chemical fixation of undesirable constituents in matrix [3,8]. Due to the high levels and variety of heavy metals in EAFD, this study will focus only on the stabilization process.

The investigation of the interaction of EAFD's other toxic metal ions on Cr fixation has not been reported in the literature. The influence of the presence of another ion on Cr fixation by ordinary Portland cement (OPC) and ground granulated blast furnace slag (GGBFS) was studied by Duchesne and Laforest [10] with synthetic solutions. Molybdenum ions were introduced to verify if another ion with similar behaviour to Cr would have a negative influence on Cr fixation. According to data obtained, no ionic competition was found between Cr and Mo (at initial concentrations of about 20 mg/L). However, Laforest and Duchesne [11] have reported high Cr concentrations (up to 331 mg/L) in EAFD leachate (water/EAFD: 3/1) after 84 days of reaction and about 225 mg/L after only 7 days. These concentrations are many times higher than the 20 mg/L previously reported [10]. Cohen and Petrie [12] have also shown the same range of concentrations (306.4 and 491.1 ppm) in a pore solution of EAFD S/S products. Thus, the high concentrations of leachable Cr and the presence of several heavy metal ions in EAFD justify the collection of supplementary data on chromium ionic competition.

The stabilization of EAFD by the use of OPC and GGBFS has been investigated by Laforest [13]. After 28 days of reaction at constant pH value of 13, samples of EAFD in contact with

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doi:10.1016/j.cemconres.2006.05.012

75% OPC or 75% GGBFS have shown Cr concentrations of 9 and 177 mg/L, respectively. As shown previously [11], an increase in Cr concentrations was observed in EAFD leachate between 7 and 84 days of reaction. Thus, the long-term fixation of metals by the use of these cementitious materials (OPC and GGBFS) needs further study.

Stabilization aims at solubility control of the EAFD heavy metals. Reardon et al. [14] recommended conducting leaching tests on a particular waste material with at least two different water/solid ratios. They point out that, if an element's concentration does not double when the water/solid ratio is halved, there must be a solid phase control of its concentration in solution.

2. Objectives

The first objective of this study is to evaluate Cr fixation in the presence of one or several other ions (Zn, Ni, Pb and Mo). The ionic competition will be evaluated for OPC and GGBFS with synthetic contaminated solutions.

The second objective of this study is to evaluate the long-term effect of various cementitious materials on the concentration of heavy metals in EAFD leachates. The long-term behaviour will be evaluated on mixtures of EAFD with GGBFS and/or OPC by a static leaching test with a 365-day reaction time.

Reactions are conducted at two different water/solid ratios (3/1 and 6/1) as a means of distinguishing elements in the leachates that have solubility controls versus those that do not. The performance of GGBFS at fixing undesirable elements from EAFD is compared to that of OPC.

3. Materials

The cementitious materials used were GGBFS and OPC. The GGBFS studied here comes from Algoma Steel (Sault Ste-Marie, Canada). The chemical composition of the cementitious materials is presented in Table 1.

The EAFD studied comes from Atlas Stainless Steels (Sorel, Canada) and was obtained in the summer of 2000. EAFD comes from dust collected from arc furnaces where coal and scrap are mixed together at an approximate temperature of 1500 °C. The chemical composition of this dust varies according to the type of steel produced and the variations of compositions are considerable. For example, the proportion of Fe, which is the major element, can vary from 15% to 62% and represents around 43% in EAFD from the stainless steel industry [6]. Table 1 presents the chemical composition of the major oxides of the EAFD tested. The EAFD studied contained 10.9%, 4.1%, 1.4% and 5.2% mass of Cr, Ni, Pb and Zn, respectively. EAFD shows two fractions: a very fine-grained portion and a coarser part. Particle sizes range from less than 2.8 μm to more than 176 μm. The majority (94%) of the particles are smaller than 5.5 μm in diameter.

The EAFD particles appear as elongated grains, or spherical, fine-grained and irregular particles. The predominance of spinel group minerals was identified. Magnetite (Fe_3O_4) is the main phase present in EAFD along with chromite ($FeCr_2O_4$). Most grains are magnetite (FeO_4)–franklinite ((Zn,Fe,Mn)(Fe,Mn)$_2$O$_4$)–jacobsite ($MnFe_2O_4$) solid solutions. A more detailed characterisation of EAFD was presented by Laforest and Duchesne [11].

Table 1
Chemical composition of materials

Oxides (mass%)	GGBFS	OPC	EAFD
CaO	37.31	62.49	6.59
SiO_2	36.77	19.75	5.76
MgO	13.91	2.62	4.25
Al_2O_3	7.77	4.41	0.74
MnO	1.02	0.05	5.88
Fe_2O_3	0.85	2.92	39.56
K_2O	0.43	0.89	0.48
TiO_2	0.36	0.17	0.16
Na_2O	0.31	0.28	1.01
P_2O_5	0.00	0.23	0.04
PbO	–	–	1.51
Cr_2O_3	–	–	15.93
NiO	–	–	5.22
ZnO	–	–	6.47
LOI	0.00	1.90	3.67
Total	98.73 [a]	95.71 [a]	97.25 [a]

(–) Not analysed constituents.

[a] The sum does not equal 100, because the sample contains constituents that were not analysed for (carbonates, for example).

4. Methods

4.1. Ionic competition test

This test was run to determine the effect of the presence of several ions on metal ion fixation by cementitious materials. In fact, some ions are often linked in a preferential way, compared to others. This effect, ionic competition, can be very important when the residues studied, such as EAFD, contain several contaminants.

The cementitious materials tested (GGBFS and OPC) were immersed in different solutions made up of various combinations of heavy metals. The immersion solutions were made up of various combinations of Cr(VI), Pb(II), Zn(II), Ni(II) and Mo (VI) ions. The choice of the first four metals was based on their presence in EAFD. Mo was selected because of its similar behaviour to that of Cr. First, the metals were studied individually. Then, Cr(VI) was studied in the presence of the other ions (Cr+Ni, Cr+Pb, Cr+Zn, Cr+Mo). Finally, more complex solutions were evaluated (Cr+Ni+Pb+Zn and Cr+Ni+Pb+Zn+Mo). The initial concentration of each element in solution was 1000 mg/L. The reagent solids used were CrO_3, $NiCl_2{\cdot}6H_2O$, $PbCl_2$, $ZnCl_2$ and MoO_3. The immersion solutions were made up with distilled water. An amount of cementitious material (1× = 7.5 g or 2× = 15 g) was put in contact with 45 mL of immersion solution (containing 45 mg of each metals). These mixtures were placed in 60 mL high density polyethylene bottles mounted horizontally on a Plexiglas carousel. The carousel was suspended in a temperature bath maintained to within 0.5 °C at 25 °C and was rotated between

Table 2
Maximum concentration of contaminants for toxicity characteristic and safe drinking water, U.S. Environmental Protection Agency [16,17]

Contaminant	Toxicity Characteristic Regulatory Level	Safe drinking water	
	TCRL (mg/L)	MCLs [a] (mg/L)	NSDWRs [b] (mg/L)
Cr	5.0	0.1	
Pb	5.0	0.015	
Zn	n.a.		0.3

n.a.: Not available.

[a] Maximum concentration level.

[b] National Secondary Drinking Water Regulations.

10 and 20 rpm for 28 days. After 28 days of reaction, cation (Cr, Ni, Pb, Zn and Mo) concentrations were analysed by Atomic Absorption Spectrometer (AAS). The 28-day limit required to reach equilibrium was retained in accordance with Delagrave et al. [15]. In that study, the data have clearly shown that equilibrium was achieved with powdered cement paste samples and a chloride solution after a 3-week exposure period. This period was confirmed by Duchesne and Laforest [10]; equilibrium was attained after approximately 7 days for a solution rich in Cr and Mo in contact with different binders.

Reacted solid samples (dried at 60 °C) were observed under a JEOL JSM-840A scanning electron microscope (SEM) equipped with an energy dispersive X-ray analysis system (EDXA).

The percentage relative error was evaluated for concentration measurements. Experimental error was calculated as the smallest division of the instrument. Analytical error was calculated in accordance with the AAS detection limit for each metal.

4.2. Long-term static leaching test

The static leaching test was carried out to quantify available metals over the long term (365 days) from mixtures of EAFD with GGBFS and/or OPC. The presence or not of a solubility control was also investigated.

A series of batch leaching experiments was performed by reacting samples of EAFD and cementitious materials at 3/1 and 6/1 water/solid ratios. The samples were placed in 60 mL high density polyethylene containers. The containers were mounted horizontally on a Plexiglas carousel that was suspended in a temperature bath maintained to within 0.5 °C at 25 °C. The carousel was rotated between 10 and 20 rpm. A sufficient number of replicate samples for each water/solid mix were reacted to provide all the solution samples required for each sampling period for chemical analyses. Solution samples were obtained after 7, 56, 84 and 365 days of reaction. Samples were filtered through 0.22 μm membrane filters and were acidified with HCl for cation analyses (Cr, Zn, Ni and Pb) by AAS.

The three cementitious formulations tested were GGBFS, OPC and a 50:50 mixture (by mass) of GGBFS and OPC. Each of these cementitious materials was mixed with EAFD at a ratio by mass of 75:25. The leaching solution was distilled water.

To evaluate the ion fixation, concentrations of free ions in solution were compared to regulatory limits and to Toxicity Characteristic Regulatory Level [16,17] (TCRL, Table 2). It is important to keep in mind that the TCRL was used only for comparison because the test carried out was not related to the Toxicity Characteristic Leaching Procedure (TCLP).

5. Results and discussion

5.1. Ionic competition test results

5.1.1. Influence of ionic competition on Cr ion fixation

The influence of the presence of Ni, Pb, Zn or/and Mo on Cr fixation by GGBFS and OPC is presented in Figs. 1 and 2, respectively. The final Cr concentrations in leaching solutions are presented for the solutions where Cr(VI) ions were present alone, with Ni or Pb or Zn or Mo, with Ni, Pb and Zn and with Ni, Pb, Zn and Mo.

GGBFS generally fixed more Cr ions than OPC. Fig. 1 shows that the presence of Ni, Zn or Mo has no influence on Cr fixation by GGBFS. The Cr concentrations were similar for these three cases (between 0 to 2.0 mg/L). However, the presence of Pb did affect Cr fixation. The Cr concentrations were increased from 1.9 mg/L (Cr ions alone) to 16.8 and 13.8 mg/L (2× GGBFS and 1× GGBFS, respectively) in the presence of Pb ions. The presence of several ions (Cr+Ni+Pb+Zn and Cr+Ni+Pb+Zn+Mo) has a significant influence on Cr fixation. The Cr concentrations in leachate were lower when Cr ions were alone (1.9 mg/L) than when Cr was in the presence of Ni, Pb and Zn (42.3 and 47.2 mg/L) or in the presence of Ni, Pb, Zn and Mo (43.1 and 47.0 mg/L). The

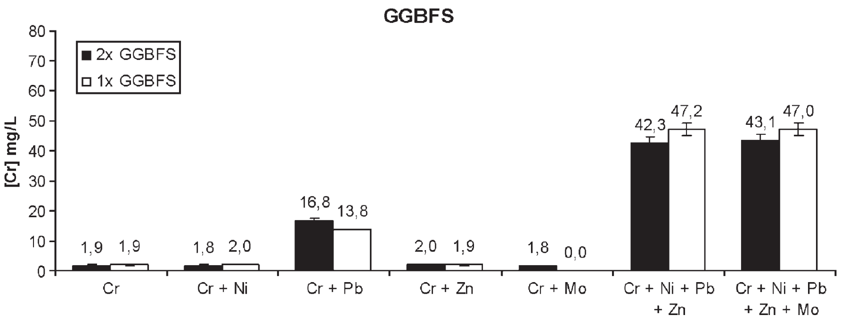

Fig. 1. Influence of the presence of Ni, Zn, Pb or/and Mo on Cr fixation by GGBFS.

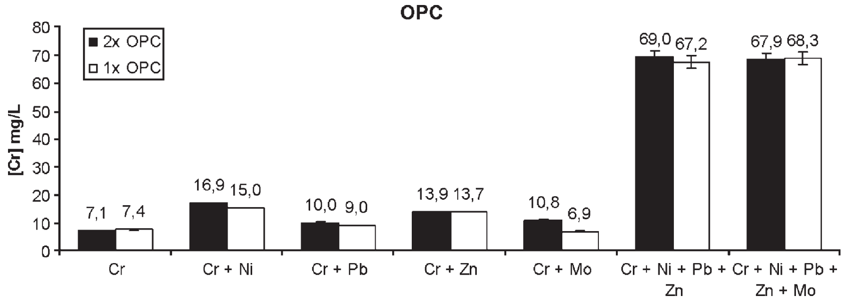

Fig. 2. Influence of the presence of Ni, Zn, Pb or/and Mo on Cr fixation by OPC.

fixation of Cr ions by GGBFS was around 4.3% less efficient in the presence of other ions (average fixation with Cr only (99.8%) − average fixation with Cr + Ni + Pb + Zn + Mo (95.5%)). The amount of GGBFS did not significantly affect Cr fixation, at this initial concentration.

The ionic competition phenomenon between Cr and Pb was less evident for OPC. The Cr concentrations were increased slightly from 7.1 and 7.4 mg/L (Cr ions alone) to 10.0 and 9.0 mg/L (2× GGBFS and 1× GGBFS, respectively) in the presence of Pb ions. Cr ionic competition with other metals was not evident and more tests at higher metal concentrations need to be done. High metal concentrations will allow for identification of metal-bearing phases in solids. Ni and Zn seemed to have more influence on Cr fixation. The presence of several ions (Cr + Ni + Pb + Zn and Cr + Ni + Pb + Zn + Mo) showed a major influence on Cr fixation. The Cr concentrations in leachate were lower when Cr ions were alone (7.1 and 7.4 mg/L) than when Cr was in the presence of Ni, Pb and Zn (69.0 and 67.2 mg/L) or in the presence of Ni, Pb, Zn and Mo (67.9 and 68.3 mg/L). The fixation of Cr by OPC was around 6.1% less efficient in the presence of many ions (average fixation with Cr only (99.3%) − average fixation with Cr + Ni + Pb + Zn + Mo (93.2%)). As for GGBFS, the amount of OPC did not significantly affect Cr fixation, at this initial concentration.

Results show that GGBFS was less affected than OPC by the simultaneous presence of many ions (Cr, Ni, Pb, Zn and Mo). Both cementitious materials were efficient at fixing Cr ions at 95.5% for GGBFS and 93.2% for OPC when Ni, Pb, Zn and Mo were present with Cr in solution. However, the Cr concentrations reach 47.0 and 68.3 mg/L in multi-element solutions (Cr + Ni + Pb + Zn + Mo) for GGBFS and OPC compared to 1.9 and 7.4 mg/L for solutions with only Cr ions.

Molybdenum was introduced to verify the Cr ionic competition with an element which has similar behaviour. Mo is just below Cr in the periodic table of the elements and its behaviour is amphoteric, like Cr. As mentioned in Section 1 (Introduction), Mo(VI) did not show any influence on Cr(VI) fixation. Fig. 1 shows that Cr concentrations for GGBFS samples were equal or inferior in the Cr + Mo case compared to the Cr alone case. The absence of competition between these two ions was confirmed for OPC samples (Fig. 2). This fact was also confirmed by comparison between the samples where the Cr ions were in presence of all heavy metals with the exception of Mo (Cr + Ni + Pb + Zn) and the samples with all of them (Cr + Ni + Pb + Zn + Mo). These results confirm some earlier observations presented by Duchesne and Laforest [10].

5.1.2. *Influence of ionic competition on Pb, Ni, Zn and Mo ion fixation*

The concentration of other metals (Pb, Ni, Zn and Mo) was also measured by AAS. The Ni and Zn concentrations were null or below the detection limit (0.14 and 0.018 mg/L, respectively). The Mo concentrations were not available. Pb ions were still present in the leaching solutions for GGBFS and OPC samples and their concentrations are presented in Figs. 3 and 4, respectively. The Pb concentrations were similar when the Pb ions were alone in an immersion solution and when they were in the presence of Cr ions for GGBFS and OPC samples. Thus, the ionic competition between Cr and Pb previously mentioned in Section 5.1.1 did not affect Pb fixation. SEM observations were undertaken to identify metal-bearing phases from GGBFS and OPC solid samples recovered from an immersion solution with Cr or Pb or Cr and Pb ions. No phase containing Cr or Pb was

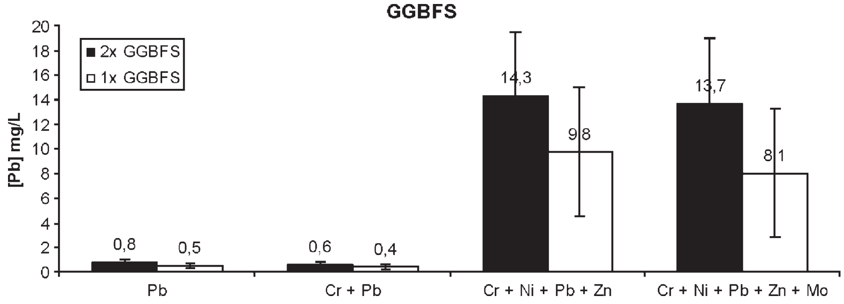

Fig. 3. Influence of the presence of Cr, Ni, Zn or/and Mo on Pb fixation by GGBFS.

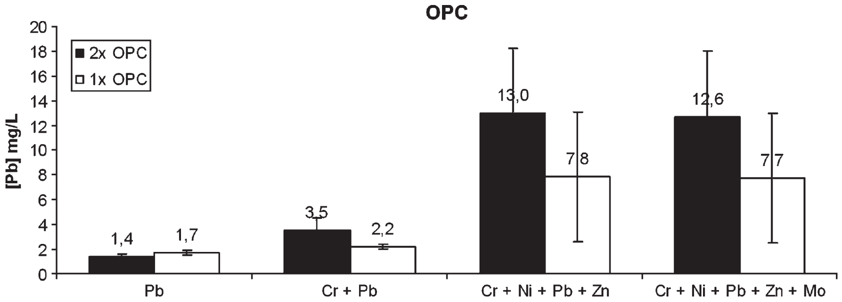

Fig. 4. Influence of the presence of Cr, Ni, Zn or/and Mo on Pb fixation by OPC.

observed. This should be due to metal levels too low to be detected by EDXA.

5.2. Long-term static leaching test results

Solubility control by EAFD on heavy metal concentrations has been studied by Laforest and Duchesne [11]. The results of static leaching tests conducted on EAFD (15 g and 7.5 g without binder materials in 45 mL of distilled water) have shown that Cr, Zn, Ni and Pb were controlled by mineral solubility for pH values varying from 9.8 to 11.5. Solubility control was established based on the fact that Cr, Zn, Ni and Pb concentrations did not double when the water/solid ratio was halved. In fact, Zn, Ni and Pb have shown concentrations in leaching solution under 1.3 mg/L. Fig. 5 presents the results of Cr concentration in solution according to Laforest and Duchesne [11]. The results show that the Cr has been leached from EAFD in large amounts (117 to 331 mg/L). However, the solubility control established by EAFD was not sufficient to decrease Cr concentrations below TCRL limits (Table 2). The addition of cementitious material was proposed as a way of reducing the quantities of Cr ions leached by EAFD. In this way, long-term static leaching tests were conducted on cementitious material–EAFD mixtures.

Fig. 6 shows static leaching test results for Cr, Pb and Zn concentrations recovered from samples of EAFD (25% in mass) mixed with OPC and/or GGBFS (75% in mass). Nickel concentrations were under AAS detection limit (0.14 mg/L) and are not represented. Fig. 6 shows high Cr short-term concentrations for GGBFS samples. This behaviour may be linked to the longer hydration time needed for GGBFS compared to OPC because C–S–H is an important Cr-bearing phase for GGBFS (confirmed by Laforest and Duchesne [18] and by Omotoso et al. [19]). After 56 days of reaction, this mixture became the best Cr-stabilizing material. Indeed, after

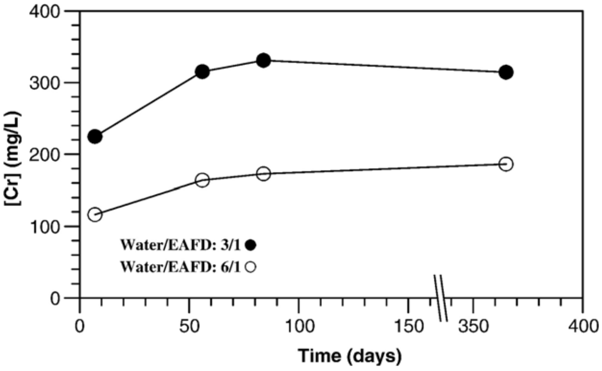

Fig. 5. Concentration of Cr in water/EAFD leachate over 365 days (after Laforest and Duchesne [11]).

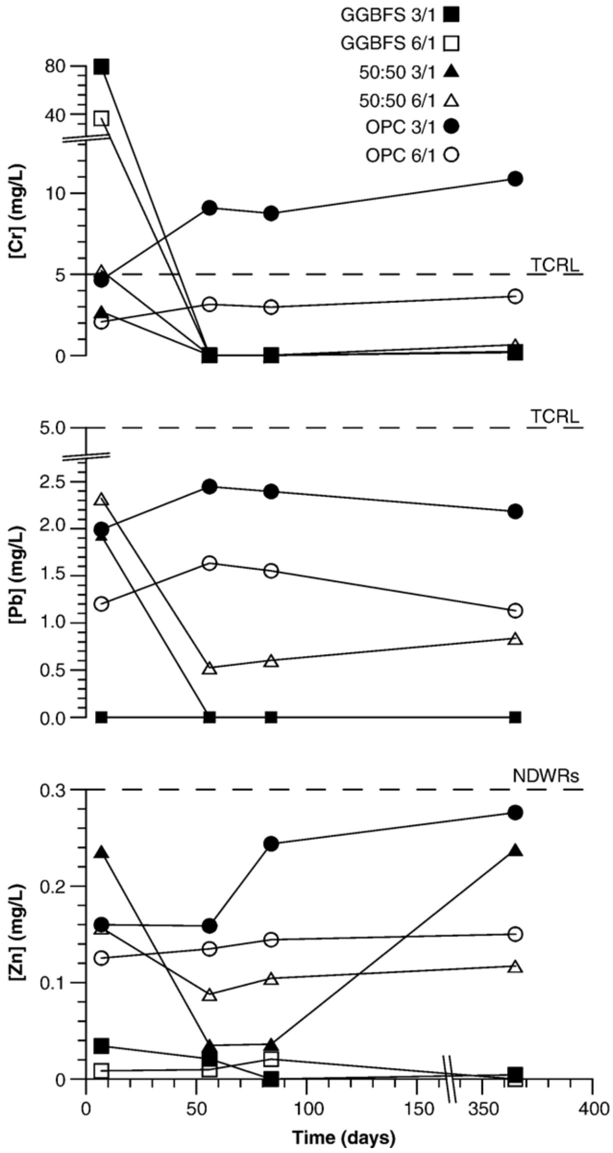

Fig. 6. Long-term effect of the binder mixtures on Cr, Pb and Zn concentrations in leachates from 25% EAFD (water/solid ratios = 3/1 and 6/1).

Table 3
Amount (in percentage) of Cr extracted from EAFD and cementitious materials–EAFD formulations

Water/solid	Time (days)	Amount of Cr extracted (%)			
		EAFD[a]	GGBFS	OPC	50:50
3/1	7	0.6185	0.0087	0.0005	0.0003
3/1	56	0.8679	0.0000	0.0010	0.0000
3/1	84	0.9115	0.0000	0.0010	0.0000
3/1	365	0.8674	0.0000	0.0012	0.0000
6/1	7	0.6414	0.0081	0.0005	0.0012
6/1	56	0.9010	0.0000	0.0007	0.0000
6/1	84	0.9508	0.0000	0.0007	0.0000
6/1	365	1.0246	0.0000	0.0008	0.0001

[a] After Laforest and Duchesne [11].

56 days, GGBFS had almost entirely fixed Cr ions from solution, probably in the C–S–H phases.

All samples show values under TCRL (Table 2) with the exception of the sample with OPC at water/solid ratio of 3/1. The best combination for fixing Cr from EAFD (25%), for all time intervals, was the mixture of OPC and GGBFS (50:50). GGBFS offered the best performance at fixing Pb for all time intervals. It is nevertheless important to note that OPC was also very effective because all the Pb concentrations were very low (lower than 2.5 mg/L) and under TCRL (Table 2). Almost all the Zn concentrations were null (lower than 0.3 mg/L). These values were under the National Secondary Drinking Water Regulations (NSDWRs) (Table 2). GGBFS was the most effective cementitious material at fixing Zn ions. Overall, GGBFS was generally, irrespective of time, the most effective cementitious material at incorporating metals leached by EAFD.

Fig. 6 shows that all mixture (GGBFS, OPC, 50:50) have exerted a solubility control on Pb, Ni and Zn. In fact, metal (Pb, Ni and Zn) concentrations are under the AAS limits of detection (0.19 mg/L, 0.14 and 0.018 mg/L, respectively) or do not double when the solution is halved. For chromium, the situation is the same, with the exception of GGBFS samples. For these samples, the solubility control at an early point in time (7 days) was uncertain because the ratio between the 3/1 and 6/1 samples was near two (2.18) and the concentrations were higher (79.4 and 36.5 mg/L) than the other observed values with GGBFS samples (under 4 mg/L). However, Table 3 shows that the amount of Cr extracted from EAFD alone is higher (0.6185% and 0.6414%) than the amount of Cr extracted from GGBFS samples at 7 days (0.0087% and 0.0081%). Thus, a certain control was exerted at 7 days because the GGBFS samples have had less Cr leached than the EAFD alone.

Chromium was the only metal whose concentrations exceeded the TCRL limit. OPC and GGBFS had an inverse behaviour over time. At first, OPC was more effective. With time, GGBFS became more effective and offered the best or comparable performance because of its late hydration, which slows down the development of C–S–H.

6. Conclusions

The presence of Ni, Zn or Mo has not affected Cr fixation by GGBFS. However, the presence of Pb has affected Cr fixation, particularly for GGBFS. The influence of several ions (Cr, Ni, Pb, Zn and Mo) had a significant influence on Cr ion fixation. The fixation of Cr ions by GGBFS was around 4.3% less efficient in the presence of other ions (average fixation with Cr only (99.8%) − average fixation with Cr, Ni, Pb, Zn and Mo (95.5%)). The fixation of Cr by OPC was around 6.1% less efficient in the presence of many ions (average fixation with Cr only (99.3%) − average fixation with Cr, Ni, Pb, Zn and Mo (93.2%)). The ionic competition phenomenon between Cr and Pb was less evident for OPC. The amount of GGBFS or OPC has not affected Cr fixation, at this initial concentration.

Long-term leaching tests showed that OPC was more effective at fixing Cr in the short term. GGBFS became effective with time and offered the best performance over the long term. Thus, GGBFS is a good option in S/S formulations. Results have shown that all Cr concentrations were under the TCRL limit with the exception of a sample with OPC at water/solid ratio of 3/1. The best combination at fixing Cr from EAFD (25%), for all time intervals, was the mixture of OPC and GGBFS (50:50). GGBFS offered the best performance at fixing Pb for all time intervals. It is nevertheless important to note that OPC was also very effective because all the Pb concentrations were very low (lower than 2.5 mg/L) and under the TCRL limit. Almost all the Zn concentrations were null (lower than 0.3 mg/L). These values were under the NSDWRs. GGBFS was the most effective cementitious material at fixing Zn ions. Overall, GGBFS was generally, irrespective of time, the most effective cementitious material at incorporating metals leached by EAFD.

Acknowledgments

This study was supported by the National Science and Engineering Research Council of Canada (NSERC) and by the Fonds de recherche sur la nature et les technologies of the Province of Québec (FQRNT). The authors would like to thank also Atlas Stainless Steels in Sorel, Canada.

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