

# Solidification/stabilization of electric arc furnace dust using coal fly ash Analysis of the stabilization process

Constantino F. Pereira\*, Miguel Rodríguez-Piñero, José Vale

*Dpto. Ingeniería Química y Ambiental, E.S. Ingenieros Industriales,  
Universidad de Sevilla, E-41092 Sevilla, Spain*

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## Abstract

In this paper, the stabilization of electric arc furnace (EAF) dust containing hazardous metals such as Pb, Cd, Cr or Zn is described. The treatment involves a waste solidification/stabilization (S/S) process, using coal fly ash as the fundamental raw material and main binder. The article also contains a brief review of the most important recent publications related to the use of fly ash as S/S agents.

The efficacy of the process has been evaluated mainly through leaching tests on the solidified products and compliance with some imposed leachate limits. The concentration of metals leaching from the S/S products was strongly leachate pH dependent; thus, the final pH of the leachate is the most important variable in reaching the limits and, therefore, in meeting the stabilization goals.

In this study, the dependence relationship between the leachate pH and the concentrations of metals in the leachate are analyzed; in some cases, this allows us to estimate the speciation of contaminants in the S/S solids and to understand the mechanism responsible for reduced leachability of heavy metals from solidified wastes. © 2001 Elsevier Science B.V. All rights reserved.

*Keywords:* Solidification/stabilization; Heavy metals; Arc furnace dust; Coal fly ash; Leachability

## 1. Introduction

Stabilization/solidification (S/S) technologies are widely used for treatment of hazardous wastes that are mostly inorganic (aqueous wastes, sludges, slags, and ashes containing hazardous metals) and contaminated soils before final disposal [1–5].

Some recently published papers concerning the use of fly ash as S/S agents to immobilize hazardous wastes are discussed below. One of the most important publications in the last few years is no doubt the study by Conner and Hoeffner, “A Critical Review of

\* Corresponding author. Fax: +34-95-4461775.  
*E-mail address:* pereira@esi.us.es (C.F. Pereira).

Stabilization/Solidification (S/S) Technology” [2]. Conner, one of the leaders in this field, presents an overview of the technology, showing that fly ash is currently one of the most common binders in waste stabilization formulations, mainly in two kinds of mixtures: Portland cement + fly ash or lime + fly ash. Fly ash in Portland cement/fly ash processes acts as a bulking agent and as a pozzolan. Lime-based and lime/fly ash processes are useful in the solidification of oily wastes and other water insoluble organic materials, as well as the more common inorganic sludges.

Other interesting papers which are worth mentioning include the study by Hassett and Pflughoeft-Hassett [6], who studied the use of coal combustion by-products for the S/S of hazardous wastes. Some of the fly ash was selected because of ettringite formed in the solid in long-term leaching experiments and associated reduction in leachate concentration of trace elements. The stabilization experiments were designed to evaluate the removal of relatively high concentrations of boron and selenium from simulated wastewater.

The behavior of stabilized waste materials using Portland cement and fly ash (pulverized fuel ash) was studied by Chang et al. [7]. The binding mechanisms and chemical forms of metal contaminants (Zn) were determined using sequential chemical extraction (SCE) and TCLP tests. The properties of brown-coal fly ash combined with incinerator fly ash as binder systems for immobilizing hazardous waste containing Pb and Zn has also been investigated by Krug et al. [8]. Coal ashes have also been used as a binder for solidifying municipal waste incineration residues prior to their disposal in special landfills [9]. Fluidized bed combustion coal ash has proven effective for use as a binder for the solidification and stabilization of metal-bearing sludges from a hazardous waste treatment facility [10].

Two interesting articles [11,12] describe the technology of geopolymerization for the stabilization and solidification of waste materials. In one of the papers [11], fly ash is used as a reactant in creating a geopolymeric matrix for the immobilization of process water containing important amounts of Cu and Pb.

Another paper describes the stabilization/solidification of a steel industry waste using a common type-F fly ash from a pulverized coal power station as the main binder [13]. The waste, which contains hazardous levels of metals, may be stabilized by a conventional S/S to achieve permissible Pb, Cd, and Zn concentrations in the TCLP leachates of S/S solids. On the other hand, the stabilization of Cr(VI), also present in the waste, requires a reducing pretreatment stage with ferrous sulfate to attain TCLP leachates within limits.

Finally, an interesting study was carried out by Stegemann et al. [14] to examine the response of various solidification systems to acid addition. The solidified products obtained using different cementing systems, one of them lime and coal fly ash, were tested as ground products at several ages using the acid neutralization capacity test. The authors also examined the effect of acid on the monolithic structure of the solidified products [15].

The research carried out included a previous step of laboratory tests intended to define the main operation parameters from which a pilot plant able to treat up to 600 kg of S/S solids per hour was designed and constructed [16]. The plant was conceived to allow the maximum operation flexibility by dosing and mixing different wastes and stabilizing agents (binders and additives). In the course of the project (called the IRIS project) different real industrial wastes were treated, selected from among those proposed by the waste management partner as the most problematic and difficult to stabilize. This partner operates an S/S plant using blast furnace slag as the main binder.

In this paper, the stabilization of arc furnace dust waste (s-WA) is described. s-WA is a dust that results from the collecting systems of particulate material in a carbon–steel electric arc furnace. Electric arc furnace (EAF) dust has been designated by the USEPA as a hazardous waste (K061) and must be treated for safe disposal in accordance with environmental regulations. The hazardous metals normally found in this kind of wastes are: lead; cadmium and chromium. In addition, the dust usually has a high zinc content which may reach up to 40% (though typical values are normally around 20%) resulting from the use of galvanized scrap [17]. Because of its heavy metal content, s-WA is classified as hazardous waste, according to Spanish regulations.

In a survey carried out in the US in 1997, there was an estimated annual production of 730,000 t of EAF dust, and this figure was expected to grow in the future [18]. Half of the dust generated in the USA was landfilled this year [19]. The main treatment options for this metallurgic dust are: (1) recovery of metals at high temperature, which requires treatment with a reductant such as coal or coke to obtain mainly Zn (also Pb and Cd) in the form of a metal or oxide; (2) direct recycling, for which the dust first has to be concentrated by means of some method such as pelletization or briquetting in order to reduce its volume; (3) stabilization/solidification and (4) other applications such as production of ceramic materials, mineral wool or cement, or the manufacturing of abrasive products for blasting and polishing applications or for use in hydrometallurgical processes.

Lead is frequently the most hazardous component of electric arc furnace dust. In the literature [20], the Super Detox process is described; this process uses fly ash, lime and other additives to stabilize electric arc furnace dust before it is sent to the dump. Pretreatment of dust with acid solutions to form a leachate in which the metals solubilize and can be separated from the solid waste is also mentioned. The solution with the metals can later be treated in different ways, as if it were wastewater, e.g. precipitating the metals like hydroxides [21]. EAF dust was chosen to evaluate the efficacy of an S/S process and the behavior of the solidified waste in the field. The waste was selected because of its high concentration of potentially hazardous components and the presence of a number of different high level contaminants, rather than a single major contaminant. Three cementing systems were tested: (1) type F coal fly ash + dolomitic lime; (2) type I Portland cement and (3) a combination of blast furnace slag + dolomitic lime + silica fume, which in the end, was the system chosen [22].

## 2. Materials and methods

Different coal combustion residues were used as solidification agents in this project. Low calcium fly ash (ASTM class F) from the combustion of high quality pulverized coals in the biggest coal power plant in the south of Spain, Los Barrios (550 MW), was used as the main silico-aluminous agent in the IRIS project. Other stabilization agents coming from different desulphurization processes were used in this work and are included in Table 1. The behavior of these agents was compared with that of standard solidification agents, like metallurgical slag, high alumina cement or Portland cement (OPC) which were used for reference purposes.

Table 1  
Chemical composition (major species) of S/S agents (% w/w, dry basis)

Agent	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO
Fly ash (Los Barrios)	50.0	8.8	20.8	4.0	1.3
Fly ash (fluidized bed)	40.0	9.4	10.1	38.7	0.9
Fly ash (spray dryer)	28.6	1.6	14.3	24.6	3.1
Aluminum cement	3.0	15.6	43.5	36.0	0.5
Portland cement (OPC)	23.0	3.8	2.5	64.7	1.3
Blast furnace slag (BFS)	39.0	0.6	8.1	41.4	7.1

Table 2  
Average hazardous metal content in waste (% w/w, dry basis)

Waste	s-WA
As	0.29
Cd	0.08
Cr	0.95
Ni	0.20
Pb	1.30
Zn	26.0

The heavy metal content of the waste (s-WA) is shown in Table 2. Average concentrations are shown since the metal contents varied throughout the project as different batches were used.

### 2.1. Quality criteria

To evaluate the degree to which the S/S objectives were met, some specific criteria were defined, in part taken from the Spanish regulations on hazardous waste characterization, which are summarized in Table 3. The compressive strength refers to S/S solids cured

Table 3  
Quality criteria for S/S solids

Physical properties	
Setting time (h)	5–72
Compressive strength (MPa)	≥0.35
Chemical properties	
pH	2–12.5
Metal concentrations in TCLP leachates (mg/l)	
Cd	0.5
Cr	5
Pb	5
Zn	300
Ecotoxicity (TCLP leachate)	
Ecotoxicity to <i>Daphnia Magna</i>	EC50 ≤ 750 mg/l

Table 4  
Complementary criteria<sup>a</sup>

Cd	0.5
Cd (III)	4.0
Cd (VI)	0.5
Pb	0.5
Zn	20.0

<sup>a</sup> Metal concentrations in DIN leachates (mg/l).

under ambient conditions. The pH values are measured after mixing the crushed solid with distilled water in a water/waste ratio of 10 (w/w).

Ecotoxicity tests were performed according to the ISO standard protocol [23] with a few modifications. The *Daphnia magna* inhibition test is used to determine the acute toxicity of a substance using this organism. The toxicity is expressed by the initial concentration that inhibits the mobility of 50% of the daphnids in the batch being tested during a 24-h period of exposure (EC50, 24 h). Ten neonates (daphnids with less than 24 h of age) were introduced into containers that had different concentrations of the test substance (in our case, the TCLP leachate). Immobilization percentages were determined visually after 24 h. The EC50 (24 h) value was obtained using the regression line, which was determined by plotting the concentration against the immobilization percentage. The concentrations were presented on a logarithmic scale and the immobilization percentages, on a probit scale.

To complete the information on the behavior of the wastes treated at their final destination, it was decided to carry out a leaching test with water, in addition to the toxicity characteristic leaching test (TCLP). The German standard DIN-38414 S4 is an extraction method similar to the TCLP, that uses distilled water as leachant in a L/S proportion of 10, instead of the TCLP acetic acid solution (Table 4).

All metal analyses were performed in accordance with the procedures described in standard methods for the examination of water and wastewater [24].

## 2.2. Preparation of S/S solids

A methodology for obtaining solids was established that includes the following steps:

1. Enough water is added to the waste-fly ash mixture (generally 1:1 by weight) to make the paste castable; then Portland cement (or aluminum cement) is sometimes incorporated. Finally, if necessary, some lime is added so that the paste will have a pH near 12. The resulting mixture, with a water/solid ratio of around 0.25, is agitated in a planetary mixer. The pH of the paste is measured in the solution obtained by mixing 5 g of paste with 100 ml of distilled water after agitating the mixture. In some cases, the mixing process can be improved by adding the water of the formulation as part of a waste or fly ash slurry.
2. With the mass obtained, cylindrical plastic molds (28 mm of diameter and 30 mm high) are filled and compacted. During setting, the samples obtained are placed in a controlled-moisture chamber (90% RH).
3. After 28 days, the samples are crushed and sieved to between 0.25 and 4 mm, and are then extracted using leaching tests.

### 3. Results and discussion

#### 3.1. Compliance with quality criteria

About 80 different S/S mixtures were tested. Solidification objectives were easily achieved with setting times and compressive strengths within the framework of the quality criteria mentioned earlier. The same occurs with the ecotoxicological limit for waste TCLP leachates which never were surpassed. The most decisive criterion to evaluate the efficiency of the process was in compliance with the limits for metal concentrations in leachates.

Figs. 1–4 show the concentrations of the metals studied in leachates obtained from many S/S stabilization mixtures as a function of the leachate pH, using the two leaching tests previously mentioned. As can be seen, the lowest Cd, Pb, Cr or Zn concentrations in leachates appear within an optimum pH range (pH window).

This behavior, which is cited many times in the bibliography [1] for zinc bearing wastes that are stabilized with different agents and formulations seems to show that the metal is found as an oxide or hydroxide and that, as such, displays an amphoteric behavior, redissolving in a basic medium as zincate.

As can be seen in the diagram presented in Fig. 1, the solubility of Zn seems to follow the typical tendency of zinc hydroxide. When characterizing the interactions responsible for the stabilization mechanism of the metals in different S/S formulations, given the complexity of

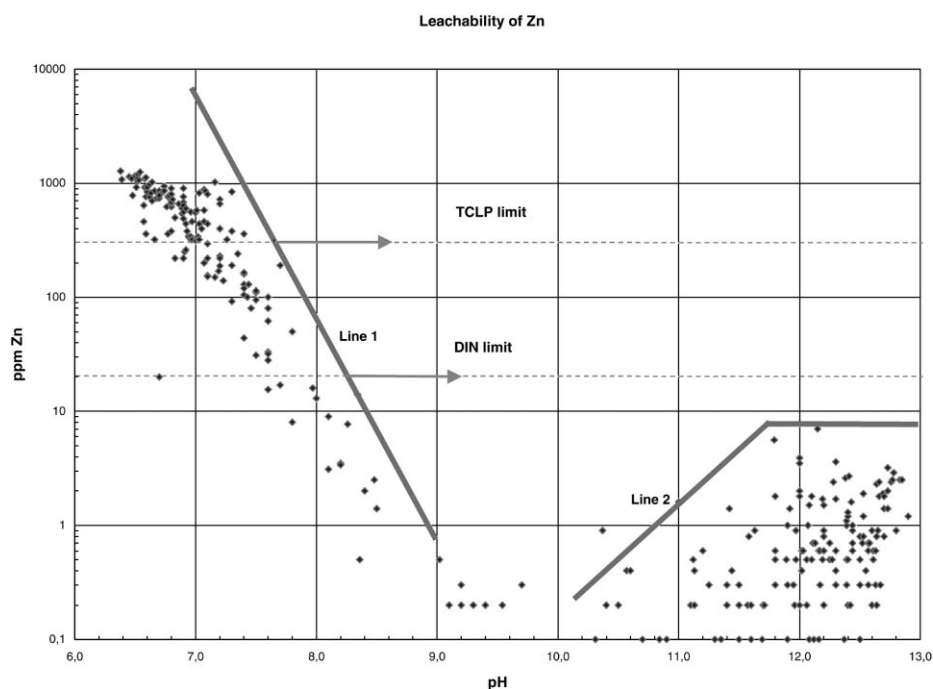


Fig. 1. Leachability of zinc as a function of pH.

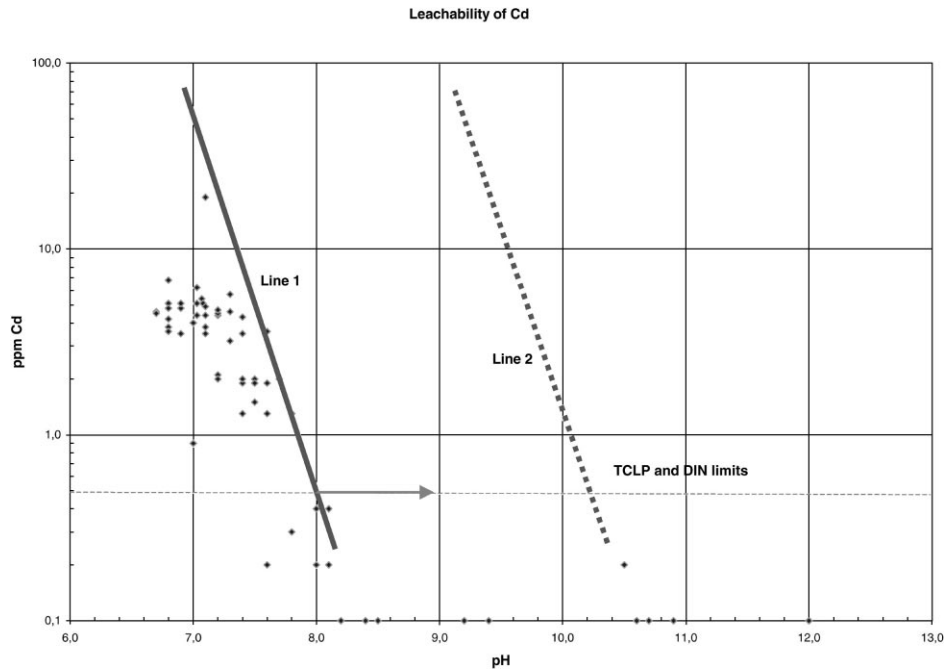
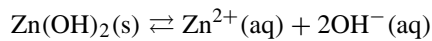


Fig. 2. Leachability of cadmium as a function of pH.

the problem, a certain speciation for the metal is usually assumed and then that assumption is contrasted with the experimental results. Thus, if to explain the behavior of the set of points shown in Fig. 1 for a pH less than 9, it is assumed that the solubility of the Zn in that pH range is governed by the equilibrium



whose equilibrium constant is the solubility product constant ( $K_{\text{sp}}$ ) of the zinc hydroxide; as a result, the Zn concentration (mol/l) would be limited by

$$[\text{Zn}^{2+}] \leq \frac{K_{\text{sp}}}{[\text{OH}^{-}]^2}$$

or, if this concentration is expressed in parts per million (ppm) and as a function of  $[\text{H}^{+}]$  at 25°C, the following should be true:

$$\text{Zn (ppm)} \leq 65,390 K_{\text{sp}} \left( \frac{[\text{H}^{+}]}{10^{-14}} \right)^2$$

That is, if the hypothesis of a solubilization of Zn limited by the equilibrium of its hydroxide is true, the concentrations measured in the leachates of the different mixtures made should be equal to or less than the concentrations that correspond to the solubilization equilibrium of the hydroxide.

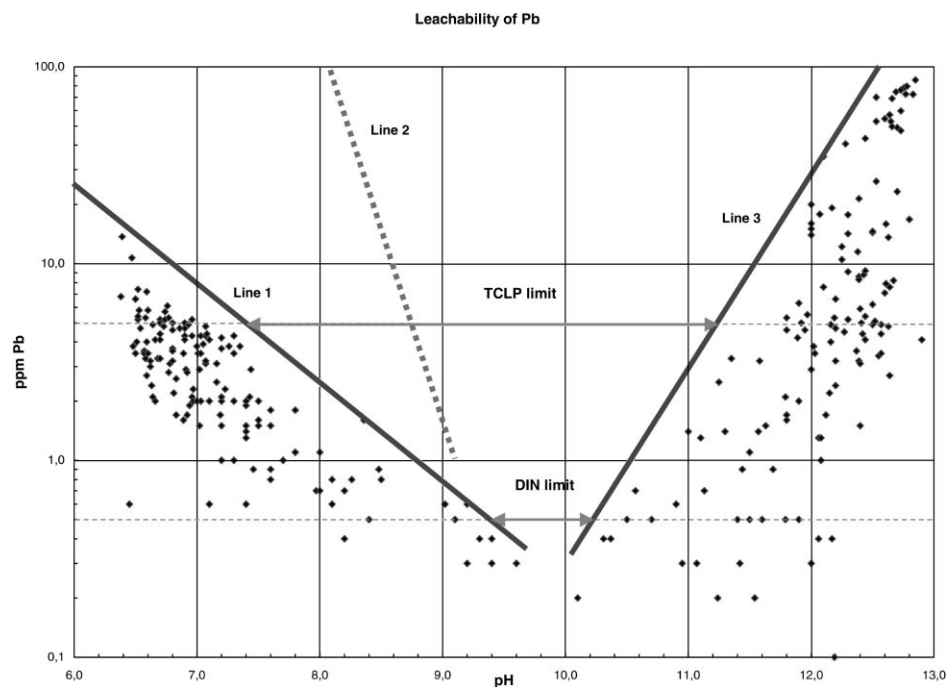


Fig. 3. Leachability of lead as a function of pH.

If the “limit” line (line 1 in Fig. 1) for the solubilization of the hydroxide is drawn (a straight line whose slope is two units, in accordance with the presumed relationship between the Zn concentration and the pH), a line beneath which all the points that correspond to the values of the  $Zn^{2+}$  concentrations in the leachates are found, we obtain a straight line with the following equation:

$$Zn \text{ (ppm)} = 6.539 \times 10^{(32 - pK_{sp} - 2pH)}$$

This line has a  $K_{sp}$  value of  $1 \times 10^{-15}$ , which coincides with the solubility product for zinc hydroxide mentioned in the literature [25,26]. The hypothesis that we started from, then, seems to be in agreement with the experimental results.

According to this value for the equilibrium constant, the  $Zn^{2+}$  concentration (ppm) in the leachates will be limited by

$$Zn \text{ (ppm)} \leq 6.539 \times 10^{(17 - 2pH)}$$

For any leachate of this waste, the corresponding quality criteria will be met (in regard to the maximum concentration of  $Zn^{2+}$ ) as long as its pH is greater than 7.7 for TCLP leachates or greater than 8.3 for DIN leachates.



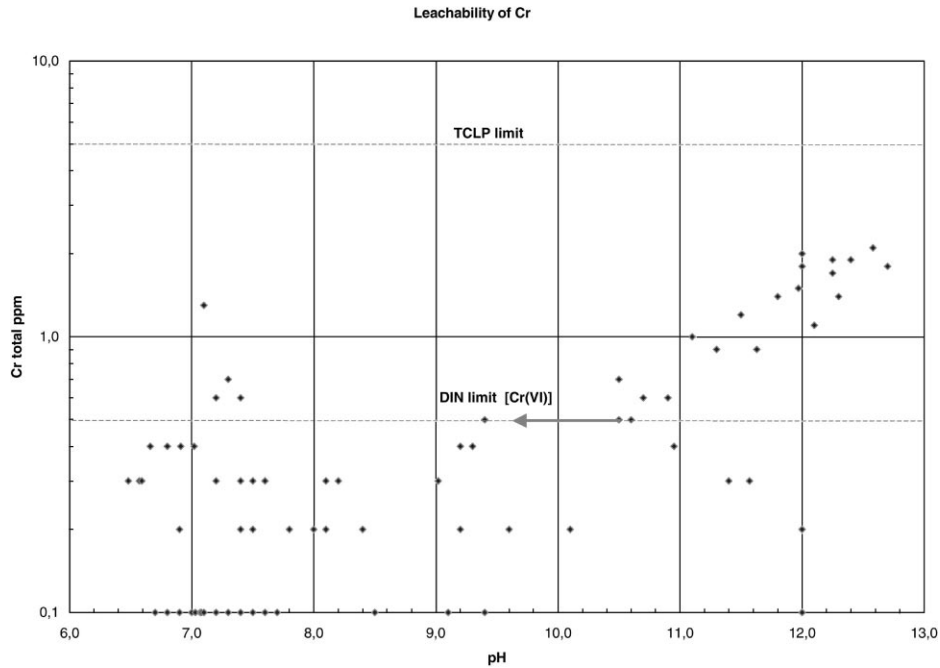
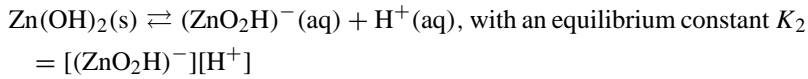


Fig. 4. Leachability of chromium as a function of pH.

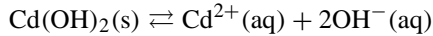
In the same way, for basic pH (between 10.5 and 11.5), taking into account the amphoteric nature of Zn, the set of points might be in accordance with a solubility of the Zn determined by the equilibrium (see line 2)



Finally, for very basic pH (>11.5) there is a limitation in the solubility, which some authors [27] attribute to a greater solubilization of  $\text{Ca}^{2+}$  ions and the subsequent precipitation of calcium and zincate compounds.

Fig. 2 shows the existing relationship between the Cd leachate concentration and the corresponding leachate pH.

If we apply the same procedure that was used with Zn, assuming that the equilibrium that determines the solubility of Cd for a pH less than 8.5 was of the type:



the following should be true for the concentration of Cd (ppm) in the leachates

$$\text{Cd (ppm)} \leq 112,410 \frac{K_{sp}}{[\text{OH}^-]^2}$$

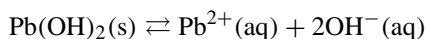
and as a result, from the “limit” line 1, a value of  $K_{sp} = 5.1 \times 10^{-18}$  is obtained, which corresponds to the equation

$$\text{Cd (ppm)} = 5.7329 \times 10^{(15-2\text{pH})}$$

The value obtained experimentally for the solubility product constant ( $K_{sp} = 5.1 \times 10^{-18}$ ) is considerably lower than the values mentioned in the literature (in the order of  $10^{-14}$ ) [25,26] that correspond to line 2 in Fig. 2. This seems to indicate that a different solid phase, other than  $\text{Cd(OH)}_2$ , must be responsible for the dissolution process or that the immobilization mechanism is different. Thus, for some authors, the S/S of Cd salts using Portland cement implies a physical encapsulation mechanism, according to which the Cd, in the form of the insoluble hydroxide, is protected by an impervious coating; consequently, Cd cannot be readily leached because it is neither very accessible to water nor in a very soluble chemical form [28,29].

In the case of cadmium, the redissolution phenomenon at high pH showed by Zn was not observed. Instead, it can be seen that, above a pH of 8.0, Cd concentrations are always less than 0.5 ppm, the leachate limit. According to Mollah et al., in the course of the hydration of Portland cement, Cd may form hydroxycadmates, that after interact with calcium ions could produce the insoluble  $\text{CaCd(OH)}_2$  [30].

The behavior of Pb is similar to that of Zn, with higher metal concentrations in solution at a pH above 11. Nevertheless, in regard to the leachability of Pb (Fig. 3), unlike what happened with the Cd and Zn cations, for pH values less than 9, the solubility does not seem to be determined by the equilibrium of the hydroxide

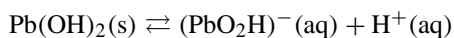


Indeed, the slope of the limit line based on the experimental data (line 1), approximately 0.5 units, does not coincide with that of the line that would be drawn based on the hydroxide solubility equilibrium (line 2), which would have a slope of two units. Based on the experimental values, the concentration of Pb (ppm) in the leachates can be expressed using the following equation

$$\text{Pb (ppm)} \leq 207,200 \times \frac{1.2 \times 10^{-8}}{[\text{OH}^{-}]^2} = 2.4864 \times 10^{(4-(1/2)\text{pH})}$$

Côté [31] mentions some differences between the experimental solubility/pH curves (in the neutral–acid area) and those calculated, assuming a speciation in the form of hydroxides (Cd and Pb), in S/S systems that use the Portland cement + fly ash mixture as a binder. Van der Sloot et al. [32] also show some diagrams concerning the leaching behavior of Cd and Pb as a function of pH for wastes stabilized using different hydraulic binders in which the slope observed for the experimental data in the aforementioned pH range is clearly less than two units. Without going into any more detail in regard to the speciation of Pb in leachates, the above-mentioned dependence relationship could be explained by the complex chemistry of Pb in solution, which implies the formation of several  $\text{Pb}^{2+}/\text{OH}^{-}$  polynuclear complexes [28,33].

At a pH less than 10, on the other hand, the solubility could be governed by an equilibrium of the following type



and the concentration in leachates (ppm) could be limited by

$$\text{Pb (ppm)} \leq 207,200 \times \frac{1.3 \times 10^{-16}}{[\text{H}^+]} = 2.6936 \times 10^{(\text{pH}-11)}$$

Consequently, the quality criteria (maximum concentration in DIN and TCLP leachates of 0.5 and 5 ppm, respectively) are met in the pH range 7.4–11.3 for the TCLP test, and 9.4–10.3 for the DIN test.

In contrast with the previous cases, the concentrations of chromium (see Fig. 4) do not show a clear relationship to the leachate pH. It can be seen in this graph that the leaching of Cr is very low. The concentration values in all of the cases were above 3 ppm. This behavior seems to indicate a major speciation of the chromium as Cr(VI), a species which as it is well known, is soluble in a wide pH range. A slight increase in the leachability of chromium as pH increases (above a pH of 11) is also observed. This could be partly explained by the redissolution of trivalent chromium as  $\text{CrO}_2^-$ , but in any case it seems to be of little importance.

In summary, to assure that the Pb, Zn, Cr and Cd concentration limits are not exceeded in leachates, the final pH of the leachate must be within a range of values corresponding to the minimum solubility of the metals in the leaching medium. This is normally achieved in the pH 8.0–11.3 interval for TCLP leachates and in the pH 9.4–10.3 interval for DIN leachates.

#### 4. Conclusion

In the solidification/stabilization processes of industrial wastes that contain hazardous metals, such as those described in this paper, the final pH of the leachate is the main variable that determines the effectiveness of the process. The leachate pH is usually responsible for the concentrations of metals in the leachate complying with certain limits, limits that guarantee the immobilization of the contaminants in the resulting S/S solids. In this paper, the S/S of EAF dust stabilized using coal fly ash as the main binder has been carried out. In the course of the study, we have analyzed the dependence relationship between the concentrations of the metals Zn, Cd, Pb, and Cr in the waste leachates and the leachate pH, to better understand the mechanism responsible for the stabilization and the degree of stabilization reached. Moreover, this study also better defines the pH limits of the leachate that guarantee the stabilization of the waste.

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