

## Modeling of zinc solubility in stabilized/solidified electric arc furnace dust

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### Abstract

Equilibrium models which attempt for the influence of pH on the solubility of metals can improve the dynamic leaching models developed to describe the long-term behavior of waste-derived forms. In addition, such models can be used to predict the concentration of metals in equilibrium leaching tests at a given pH. The aim of this work is to model the equilibrium concentration of Zn from untreated and stabilized/solidified (S/S) electric arc furnace dust (EAFD) using experimental data obtained from a pH-dependence leaching test (acid neutralization capacity, ANC). EAFD is a hazardous waste generated in electric arc furnace steel factories; it contains significant amounts of heavy metals such as Zn, Pb, Cr or Cd. EAFD from a local factory was characterized by X-ray fluorescence (XRF), acid digestion and X-ray diffraction (XRD). Zn and Fe were the main components while the XRD analysis revealed that zincite, zinc ferrite and hematite were the main crystalline phases. Different cement/EAFD formulations ranging from 7 to 20% dry weight of cement were prepared and subjected to the ANC leaching test. An amphoteric behavior of Zn was found from the pH dependence test. To model this behavior, the geochemical model Visual MINTEQ (VMINTEQ) was used. In addition to the geochemical model, an empirical model based on the dissolution of Zn in the acidic zone and the re-dissolution of zinc compounds in the alkaline zone was considered showing a similar prediction than that obtained with VMINTEQ. This empirical model seems to be more appropriate when the metal speciation is unknown, or when if known, the theoretical solid phases included in the database of VMINTEQ do not allow to describe the experimental data.

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### 1. Introduction

The importance of equilibrium chemistry on leaching models for waste materials has been demonstrated in former works [1,2]. Solubility and availability determine the distribution of a contaminant between the mobile and immobile forms, and therefore the fraction of contaminant to be released from the waste form. In addition, the equilibrium concentration of metals is usually pH dependent. Thus, equilibrium models which attempt for the influence of pH on the solubility of metals can improve the dynamic leaching models developed to describe the long-term behavior of waste-derived forms. In addition, such models can be used to estimate the concentration of metals at a given pH in compliance leaching tests performed under equilibrium conditions. The resulting pH depends on the acid neutralization capacity of

the untreated or treated waste and the acidity/alkalinity of the leachant used in the test.

The potential release of heavy metals must be considered when untreated or treated industrial wastes are disposed of in landfills. Together with compliance leaching tests, basic characterization tests such as the acid neutralization capacity (ANC) test should be carried out according to the Landfill Directive [3]. Electric arc furnace dust (EAFD) is one of the most common industrial waste generated in the north of Spain, mainly in the Basque Country and Cantabria Region. EAFD is a hazardous waste generated in filtration units of electric arc furnace steel factories; it contains significant amounts of heavy metals such as Zn, Pb, Cr or Cd. The Zn content can reach values of 30–40%. This waste has been classified according to the European Waste Catalogue as 10 02 07<sup>\*</sup> “solid wastes from gas treatment generated from an electric arc furnace containing hazardous materials”. EAFD is generated at a rate of 15–25 kg dust/tonne steel, resulting in huge amounts of EAFD to be managed. Despite the recovery of Zn is technically viable by

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### Nomenclature

$a_i$	coefficients of the polynomial equation (12)
$C_{\text{exp}}$	experimental concentration of zinc in ANC leaching test
$C_m$	concentration of metal at equilibrium conditions
$C_o$	concentration of zinc available for leaching
$C_{\text{Pb}}$	concentration of lead at equilibrium conditions
$C_{\text{sim}}$	simulated concentration of zinc
$C_{\text{Zn}}$	concentration of zinc at equilibrium conditions
$k_1$	empirical parameter for the acidic region in Eqs. (6) and (10)
$k_2$	empirical parameter for the alkaline region in Eqs. (8) and (10)
$K_a$	equilibrium constant of Eq. (3)
$K_s$	solubility product constant
$K_w$	water ionization constant
$K'$	lumped constant for the acidic region
$K''$	lumped constant for the alkaline region
$K'_e$	lumped empirical parameter in Eq. (7)
$K''_e$	lumped empirical parameter in Eq. (9)
$n$	number of experimental data
$n_1$	empirical slope for the acidic region in Eqs. (6) and (10)
$n_2$	empirical slope for the alkaline region in Eqs. (8) and (10)
$p_i$	empirical parameters in Eq. (13)
<i>Greek symbol</i>	
$\sigma$	standard deviation

pyrometallurgical and hydrometallurgical processes, EAFD is usually managed in Spain by stabilization/solidification (S/S) techniques and disposed of in landfills after treatment.

Several studies report the leaching behavior of untreated and treated EAFD waste [4–8]. However, little information is available in the literature regarding the modeling of the solubility of heavy metals in EAFD matrices. Thus, Fernández-Pereira et al. [8] presented a simple model based on the solubilization of  $\text{Zn}(\text{OH})_2$  under acidic and alkaline conditions for S/S EAFD; however, despite the qualitative behavior was well described, the model superestimates the leaching of Zn. The authors also reported models for the solubilization of Pb, Cr and Cd. Standard softwares based on geochemical models have been widely used to describe the solubility of heavy metals in soils [9–11], residual matrices [12–14], and cement-based stabilized wastes [15–17]. Different geochemical models based on the equilibrium chemistry of metals are commercially available, some of these codes being freeware. The most common geochemical models used in the literature to describe the behavior of heavy metals in soils and wastes are MINTEQ A2, Visual MINTEQ (VMINTEQ), ORCHESTRA, PHREEQE and SOLTEQ.

The aim of this work is to model the equilibrium concentration of Zn from untreated and S/S electric arc furnace dust (EAFD) using experimental data obtained from a pH-

Table 1

Oxide composition of EAFD waste determined by XRF

Oxide	PA <sub>1</sub> (% dry wt.)	PA <sub>2</sub> (% dry wt.)
ZnO	48.2	40.2
Fe <sub>2</sub> O <sub>3</sub>	29.9	36.0
CaO	4.67	6.22
MgO	4.06	5.56
MnO	5.85	3.77
SiO <sub>2</sub>	1.74	1.96
SO <sub>3</sub>	–	1.66
PbO	1.29	1.30
Cl	0.48	0.90
K <sub>2</sub> O	0.62	0.80
Cr <sub>2</sub> O <sub>3</sub>	1.10	0.67
Al <sub>2</sub> O <sub>3</sub>	0.15	0.32
CuO	0.21	0.21
P <sub>2</sub> O <sub>3</sub>	–	0.14

dependence leaching test, ANC. For this purpose, two models were used: the geochemical mechanistic model VMINTEQ, and an empirical model developed by the authors for amphoteric metals.

## 2. Experimental methods

### 2.1. Experimental procedures

Three EAFD samples (PA1, PA2, PA3) were provided by a local factory and characterized by XRF, acid digestion and XRD; Table 1 shows the oxide composition of PA1 and PA2 samples determined by XRF; the metal composition was obtained by acid digestion based on APHA 3030 H using HNO<sub>3</sub> and HClO<sub>4</sub>, and it is shown in Table 2.

A multiple step single-extraction batch leaching test: acid neutralization capacity test (ANC) [18] was performed to determine the behavior of the leaching of Zn with pH in EAFD waste. Eleven series of ground and dried samples were in contact with a nitric acid solution of increasing acidity for 24 h at a liquid to solid ratio of 6. The pH of the different leachates are measured and the concentrations of heavy metals are determined by inductively coupled plasma emission spectroscopy (ICP-ES). The experimental pH range varies from 3–5 to 12–13.

Table 2

Chemical composition of EAFD waste determined by acid digestion

Metal	PA <sub>1</sub>	PA <sub>2</sub>
Zn (% dry wt.)	32.9	32.5
Fe (% dry wt.)	26.3	26.7
Ca (% dry wt.)	3.96	4.65
Mn (% dry wt.)	2.34	2.08
Si (% dry wt.)	0.38	0.03
Pb (% dry wt.)	1.15	1.38
Cr (% dry wt.)	0.46	0.41
Al (% dry wt.)	0.21	–
Cu (% dry wt.)	0.10	0.31
Mo (mg/kg, dry wt.)	264	1267
Ni (mg/kg, dry wt.)	350	268
Cd (mg/kg, dry wt.)	88.9	200

Table 3  
EAFD/cement formulations

Code	Cement (% dry wt.)
M0	7
M3	11
M6	20

Different cement/EAFD formulations ranging from 7 to 20% dry weight of cement were prepared and subjected to the ANC leaching test (see Table 3). The cement type was Portland cement CEM I 42.5 R according to EN-UNE 80301-96. The mixing was performed according to ASTM C305 mixing procedure for cement pastes at a water to total solids ratio of 0.30. The paste was poured in plastic moulds for curing at room temperature for 28 and 56 days. After curing, samples were dried at 60 °C, ground to less than 125 µm and subjected to the ANC leaching test.

## 2.2. Equilibrium models

Two equilibrium models were used to describe the solubility behavior of zinc in untreated and treated EAFD. First, the geochemical model VMINTEQ ver. 2.31 was used [19]. This is a mechanistic model based on MINTEQA2, which was developed first by the US-EPA. It is a chemical equilibrium model for the calculation of metal speciation and solubility equilibria for natural waters and soils, but it can also be applied to the leaching of solid wastes. It includes an extensive database containing several metal species in aqueous and solid forms. The main zinc species considered in the MINTEQA2 database in aqueous forms and solid phases are shown in Table 4. The latest versions of VMINTEQ software also allow to manage a liquid to solid ratio parameter in order to represent the leaching of a solid form.

Considering the amphoteric behavior of Zn in untreated and treated EAFD, an empirical model based on the dissolution of Zn in the acidic zone and the re-dissolution of zinc in the alkaline zone has been considered. The model was based on the theoretical solubility behavior of Zn(OH)<sub>2</sub>, which is governed by the equilibrium:



Table 4  
Zinc species considered in MINTEQA2 database (NIST 46.7)

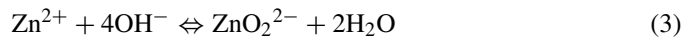
Species name	Phase	Log K
ZnO (zincite)	Solid	11.23
Zn(OH) <sub>2</sub> (am)	Solid	12.474
Zn(OH) <sub>2</sub> (beta)	Solid	11.754
Zn(OH) <sub>2</sub> (delta)	Solid	11.844
Zn(OH) <sub>2</sub> (epsilon)	Solid	11.534
Zn(OH) <sub>2</sub> (gamma)	Solid	11.734
Zn(OH) <sub>2</sub> (aq)	Aqueous	-16.894
Zn(OH) <sub>3</sub> <sup>-</sup>	Aqueous	-28.391
Zn(OH) <sub>4</sub> <sup>2-</sup>	Aqueous	-41.188
Zn <sub>2</sub> (OH) <sup>3+</sup>	Aqueous	-8.997
Zn(OH) <sup>+</sup>	Aqueous	-8.997
Zn <sup>2+</sup>	Aqueous	-

In the acidic region, the solubility of Zn<sup>2+</sup> is calculated by the following equation:

$$C_{\text{Zn}^{2+}} = \frac{K_s}{(\text{OH}^-)^2} = \frac{K_s}{K_w^2} (\text{H}^+)^2 = K' (\text{H}^+)^2 \quad (2)$$

where  $K_s$  is the solubility product constant of zinc hydroxide,  $K_w$  the water ionization constant, and  $K'$  a lumped constant for the acidic region.

In the alkaline region it was assumed that aqueous ZnO<sub>2</sub><sup>2-</sup> is in equilibrium with Zn<sup>2+</sup>:



The concentration of ZnO<sub>2</sub><sup>2-</sup> in the alkaline region is calculated by the following equation:

$$C_{\text{ZnO}_2^{2-}} = K_a (\text{Zn}^{2+}) (\text{OH}^-)^4 = K_a K_s K_w^2 \frac{1}{(\text{H}^+)^2} = K'' \frac{1}{(\text{H}^+)^2} \quad (4)$$

where  $K_a$  is the equilibrium constant of Eq. (3) and  $K''$  a lumped constant for the alkaline region. Assuming that Zn<sup>2+</sup> and ZnO<sub>2</sub><sup>2-</sup> are the main aqueous species, the solubility of zinc is calculated by the sum of Eqs. (2) and (4):

$$C_{\text{Zn}} = K' (\text{H}^+)^2 + K'' \frac{1}{(\text{H}^+)^2} \quad (5)$$

To describe the zinc solubility in the acidic region the following empirical equation is proposed:

$$C_{\text{Zn}} = C_o \left[ \frac{1}{(1 + k_1(1/(\text{H}^+)))^{n_1}} \right] \quad (6)$$

where  $C_o$  is the concentration of Zn available for leaching which can be determined experimentally by the ANC test at pH < 4. In the case of Zn this value is close to the total Zn content calculated from XRF and acid digestion;  $k_1$  and  $n_1$  are empirical parameters.

When  $1 \ll k_1(1/(\text{H}^+))$ , Eq. (6) becomes

$$C_{\text{Zn}} = \frac{C_o}{k_1^{n_1}} (\text{H}^+)^{n_1} = K'_e (\text{H}^+)^{n_1} \quad (7)$$

which is similar to Eq. (2);  $K'_e$  is a lumped empirical parameter and  $n_1$  represents the slope of the concentration versus pH plot in logarithmic scale.

The proposed empirical equation for the alkaline region is:

$$C_{\text{Zn}} = C_o \left[ \frac{1}{(1 + k_2(\text{H}^+))^{n_2}} \right] \quad (8)$$

where  $k_2$  and  $n_2$  are empirical parameters. When  $1 \ll k_2(\text{H}^+)$ , Eq. (8) becomes

$$C_{\text{Zn}} = \frac{C_o}{k_2^{n_2}} \frac{1}{(\text{H}^+)^{n_2}} = K''_e \frac{1}{(\text{H}^+)^{n_2}} \quad (9)$$

which is similar to Eq. (4);  $K''_e$  is a lumped empirical parameter and  $n_2$  represents the slope of the concentration versus pH plot in logarithmic scale in the alkaline region.

Thus, the solubility of zinc in the whole pH range can be calculated by the sum of Eqs. (6) and (8) leading to the empirical model shown in the following equation:

$$C_{Zn} = C_o \left[ \frac{1}{(1 + k_1(1/(H^+)))^{n_1}} + \frac{1}{(1 + k_2(H^+))^{n_2}} \right] \quad (10)$$

The model has four parameters,  $k_1$  and  $n_1$  for the acidic region and  $k_2$  and  $n_2$  for the alkaline region. These parameters were obtained after minimization of the error between experimental and simulated data using an optimization software (Gams).

### 3. Results and discussion

The characterization of PA1 and PA2 EAFD samples allows to identify zinc and iron oxides as the main components, while calcium and magnesium oxides were minor components, leading to an alkaline pH in water. Other pollutants were found at low levels, being Pb, Cr, Cu and Cd the most important contaminants from the environmental point of view. The XRD analysis revealed that zincite, zinc ferrite and hematite were the main crystalline phases.

The concentration of Zn is shown against leachate pH in Fig. 1 for untreated EAFD samples (PA1, PA2 and PA3). Each point shown in Fig. 1 has been determined at equilibrium conditions. The equilibrium models for Zn in these matrices must attempt for the amphoteric behavior shown in the figure. VMINTEQ was used to simulate the solubility curve of Zn in EAFD. Taking into account the XRD analysis, zincite was considered as the main Zn solid species. Four simulations were performed and compared with the experimental curve:

- Considering zincite (ZnO) as the only compound present in the solid phase. The measured concentration of ZnO in EAFD was introduced in mol/kg.
- Considering the main four oxides in the solid phase: zincite (ZnO), hematite (Fe<sub>2</sub>O<sub>3</sub>), lime (CaO) and periclase (MgO).
- Considering zincite (ZnO) as the only compound present in the solid phase and the targeted nitric concentration in leachate; increasing amounts of nitric acid are required to obtain low leachate pH values; then, the ionic strength increases as pH decreases.

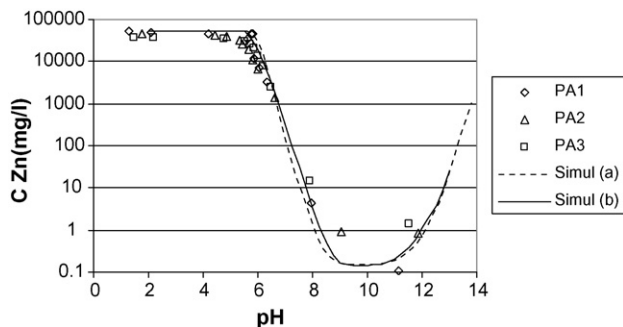


Fig. 1. Solubility of zinc as a function of leachate pH for untreated EAFD waste: experimental vs. simulated curves (using VMINTEQ ver. 2.31).

Table 5

Relative standard deviations ( $\sigma$ ) of VMINTEQ based zinc solubility models for untreated and treated EAFD waste

	Experimental code	(a)	(b)	(c)	(d)
Untreated	PA2	1.66	1.00	2.08	1.44
Treated	M0, 28 days	1.63	1.68	–	–
	M0, 56 days	1.51	0.89	–	–
	M3, 28 days	3.15	1.64	–	–
	M3, 56 days	2.10	1.32	–	–
	M6, 28 days	1.67	0.89	–	–
	M6, 56 days	1.87	0.89	–	–

- Considering the main four oxides in the solid phase: zincite (ZnO), hematite (Fe<sub>2</sub>O<sub>3</sub>), lime (CaO) and periclase (MgO) and the targeted nitric concentration in leachate.

The standard deviation of each simulation was calculated according to the following equation.

$$\sigma = \sqrt{\frac{\sum ((c_{exp} - c_{sim})/c_{exp})^2}{n - 1}} \quad (11)$$

The standard deviation of the simulations is shown in Table 5. Small differences between the models are found; the error diminishes when other solid phases are introduced together with zincite. Then, the composition of the solid phase affects the solubility of Zn, as it can be observed in Fig. 1, where the simulated (a) and (b) curves are plotted together with the experimental curves.

The results obtained from this kind of models are discrete data of pH-metal concentration. However, when chemical equilibrium models are needed to introduce into dynamic leaching models (coupled chemical/transport model), a mathematical equation is required. To solve this problem, some authors consider simple empirical models: the simplest case is to consider a polynomial equation, such as that used by Tiruta-Barna et al. [20] for lead solubilization in a synthetic waste stabilized with cement:

$$\log C_{Pb} = a_0 + a_1pH + a_2pH^2 + a_3pH^3 \quad (12)$$

Kim and Batchelor used the same polynomial equation to describe the concentration of Cr and Cu as a function of pH [21]. However, the authors used a different empirical equation for Cd, Pb and Zn:

$$C_m = 10^{p_1 + p_2pH + p_3(1/pH)} + 10^{p_4} + 10^{p_5 + p_6pH} \quad (13)$$

where  $C_m$  is the equilibrium concentration of metal; this model has six fitting coefficients to be determined by regression.

Considering the amphoteric behavior of Zn in untreated and treated EAFD, the empirical model shown in Eq. (10) has been proposed; the model has four parameters to be estimated and the available concentration of Zn must be known. The simulated curve obtained from this model is plotted in Fig. 2 together with the best VMINTEQ simulation for EAFD waste. The values of the parameters of the model were:  $k_1 = 1.0 \times 10^{-6}$  mol/L;  $k_2 = 1.0 \times 10^{17}$  L/mol;  $n_1 = 2.18$ ,  $n_2 = 0.99$ . The parameters  $n_1$

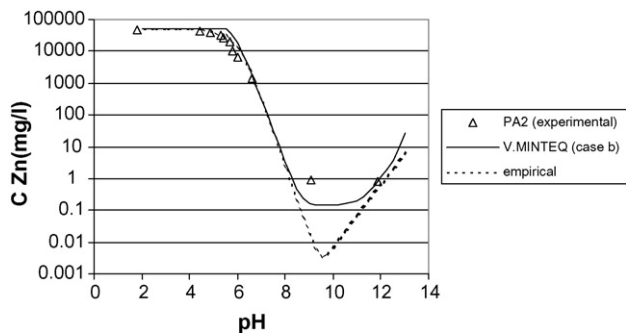


Fig. 2. Solubility of zinc as a function of leachate pH for untreated EAFD waste: experimental vs. simulated curves (using VMINTEQ ver. 2.31 – case (b) – and the proposed empirical model).

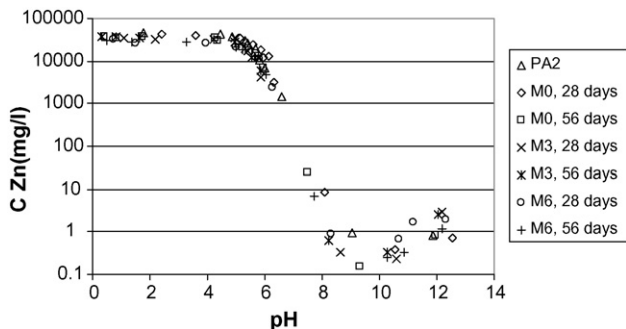


Fig. 3. Solubility of zinc as a function of leachate pH for stabilized EAFD waste cured at 28 and 56 days.

and  $n_2$  have similar meaning than that of a mechanistic model based on the solubilization of  $Zn(OH)_2$  in acidic media and its re-dissolution in alkaline media, where  $n_1$  and  $n_2$  represent the slopes of  $\log C_{Zn}$  versus pH plots in the acidic and alkaline regions.

The simulation of the Zn concentration–pH profiles for the stabilized EAFD samples using the VMINTEQ software has also been performed, and the standard deviation of each simulation is shown in Table 5. Since the experimental zinc solubility curves for the stabilized EAFD were similar to that of the untreated EAFD (see Fig. 3), the simulation results agree well with that obtained for the untreated waste.

In summary, both simulation models describe well the solubility behavior of zinc in EAFD matrices; however, the empirical model shows some advantages with respect to the geochemical model: first, the metal speciation in waste-derived forms is usually not well understood; the users of geochemical models need to introduce the main metal solid phases in the software in order to perform the calculations. In addition, the experimental solubility data are not always properly described by geochemical models; in such situations, the empirical model seems to be more appropriate. Furthermore, geochemical models generate series of metal concentration–pH data which cannot be introduced into dynamic leaching models; solubility-

pH mathematical equations are required for such dynamic models.

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