

Long-term behaviour of toxic metals in stabilized steel foundry dusts

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Abstract

A study of the long-term behaviour of stabilized steel foundry dust (SFD) wastes has been performed using a dynamic leaching test (DLT). Two stabilized/solidified forms were produced by solidifying the SFD (containing Pb, Cr, Cd and Zn) using either cement or cement and anhydrite (waste material) as binders.

The results of the dynamic leaching test were fitted to a semi-empirical mathematical model based on simple leaching rate mechanisms, which permitted the evaluation of an apparent diffusion coefficient and a leachability index, thus providing a measure of the contaminants' mobility in the solidified waste. In the case of Pb and Zn, the rate of leaching was controlled by either an initial resistance or an initial washoff, followed by diffusion of the metallic contaminants. The leaching indexes obtained in both cases were higher than 12, suggesting that both solidification/stabilization processes are acceptable.

1. Introduction

The most important factor determining whether a particular solidification/stabilization (S/S) process and its process parameters are effective in treating a particular kind of waste, is the reduction in the short- and long-term leachability of the waste.

Previous works [1, 2] have evaluated the toxicity of the solid exhausting from gas filtration units in steel factories, the so-called steel foundry dust (SFD), which contains a high content of Cr, Cd, Pb and Zn. This evaluation was based on a leaching test protocol, toxicity characteristic leaching procedure (TCLP) and chemical characterization of the aqueous solution, as well as determination of the SFDs biotoxicity (photoluminescence bioassay). From the obtained results, SFD was classified as a hazardous waste, requiring the application of detoxification technologies before landfill disposal.

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In the following works, the feasibility of the S/S process of SFD, using Portland cement and anhydrite as binders, was studied [3–5]. The toxicity of the final solids was evaluated according to the Spanish regulations, which are based on the biotoxicity of the leachate [6] and to the US EPA regulations, which are based on the concentration of the metals in the leachant [7].

It was verified that the S/S process satisfies the waste disposal rules, so the remaining question that needs to be addressed is the study of the long-term behaviour of the solidified waste.

This paper presents the results of a study which analyses the leachability of several waste forms using a dynamic leaching test. The test used in this work simulates leaching under well-managed disposal conditions. It has been developed for evaluation of solidified low-level-radioactive wastes [8] and has been proposed for use with hazardous wastes.

Regression analyses were conducted on the cumulative amounts of the contaminants leached from the solidified wastes, using a semi-empirical mathematical model based on simple leaching rate mechanisms. The resulting model parameter values give insights into the various leaching processes that occurred in the waste forms.

1.1. Background

The leaching mechanisms involved in solidified wastes are very complex. The rate of leaching from a waste can be controlled by diffusion and convection in the waste matrix, and surface phenomena at the interface, such as diffusion and leachant renewal in the aqueous solution.

Several authors [9–11] have determined that under mild leaching conditions, diffusion within the solid matrix usually controls the transfer of contaminants from the solid to the surrounding liquid phase.

A diffusion model is often used to study the kinetics of leaching in order to predict long-term leachability of the waste components. An expression based on Fick's diffusion theory is often used to describe the release of pollutants from the solidified waste, with the aid of the following assumptions [12, 13]:

(i) The mobility of a contaminant is limited by diffusion, (ii) the specimen behaves as a semi-infinite medium, provided that the cumulative fraction leached does not exceed 20% and (iii) the concentration of a contaminant at the specimen surface is approximately zero.

The diffusion model can be expressed as

$$\sum \left(\frac{A_n}{A_0} \right) \left(\frac{V}{S} \right) = 2 \left(\frac{D_e}{\pi} \right)^{0.5} t_n^{0.5}, \quad (1)$$

where A_n is the contaminant loss during the leaching period n in mg, A_0 is the initial amount of contaminant present in the specimen in mg, V is the volume of specimen in cm^3 , S is the surface area of specimen in cm^2 , t_n time (end of the leaching period) in seconds, and D_e is the effective diffusion coefficient in cm^2/s .

Assuming that the leaching rate is linear over a leachant renewal period, the following solution can also be derived [14]:

$$\left(\frac{A_n}{A_0}\right)\left(\frac{V}{S}\right)\left(\frac{1}{\Delta t_n}\right) = \left(\frac{D_e}{\pi}\right)^{0.5} \frac{1}{T_n^{0.5}}, \quad (2)$$

where Δt_n is the duration of the leaching period in seconds, and T_n is time (middle of the leachant period) in seconds.

Eqs. (1) and (2) provide two ways of calculating the effective diffusion coefficient, D_e . With Eq. (1), D_e can be calculated from the slope of a plot of the cumulative fraction leached, (A_n/A_0) versus the square root of time, $t_n^{1/2}$. Using Eq. (2), an average value of D_e can be derived from the incremental fraction, A_n/A_0 , leached at each period, n .

The American Nuclear Society [8] has proposed that the results can be a function of a leachability index, LX, that can be interpreted as the negative logarithm of the effective diffusivity, provided that long-term leaching data support the hypothesis that diffusivity controls leachability. The value of LX is given by

$$LX = \frac{1}{m} \sum_{n=1}^m \log \left(\frac{\beta}{D_e}_n \right), \quad (3)$$

where β is constant, $1 \text{ cm}^2/\text{s}$, n is the leaching period, and m is the number of leaching periods [12]. This index can be used to compare the relative mobility of different contaminants on a uniform scale that varies from 5 (D_e : $10^{-5} \text{ cm}^2/\text{s}$, very mobile) to 15 (D_e : $10^{-15} \text{ cm}^2/\text{s}$, immobile) [15].

If the calculated coefficient " D_e " remains constant over the entire leaching period, it is possible to conclude that diffusion is the primary transport mechanism responsible for leaching. On the other hand, Bishop [16] has observed that the value of D_e changes with time during a dynamic leaching test for some metals. Other authors [10, 17] have analysed the leachability of solidified metallic wastes, under carefully controlled laboratory conditions with most boundary conditions of the diffusion model properly addressed. The results obtained from the leaching test showed that mechanisms other than diffusion were also affecting the leaching process.

The development of a model that takes into account all the leaching phenomena is mathematically very complex and the resultant expression is not likely to have practical application. To overcome this problem, Côté et al. [18] have adopted a semi-empirical method to obtain a model, that describes the long-term leaching characteristics of a waste component. In this model, the cumulative amount of contaminant leached is expressed by:

$$\sum(A_n/A_0) = K_1 + K_2 t^{1/2} + K_3 t, \quad (4)$$

where K_1 is a constant representing the immediate dissolution, K_2 is a constant representing the diffusion controlled transport mechanism, and K_3 is a constant representing the long-term kinetically controlled dissolution.

2. Materials and methods

2.1. Production of stabilized/solidified (S/S) wastes

Steel foundry dust (SFD) with a high metallic content, considered as hazardous waste, was analysed in this study. The results of X-ray fluorescence characterization are reported in Table 1. The S/S forms were produced by mixing the following binders: Portland cement (commercial quality) and anhydrite (waste material) with the SFD. The anhydrite was obtained as a residual product in the hydrofluoric acid (HF) manufacturing process, which is obtained by the reaction between dry fluorspar (CaF_2) and sulphuric acid (H_2SO_4).

The initial amount of contaminants present in the specimen in each solidified waste matrix, as well as the formulations selected for each process, which have been determined and optimized in previous works [1, 5], are shown in Table 2.

The “A” matrix was obtained using a S/S process with a ratio of binder (Portland cement) to waste, 1/3, while the “B” matrix used Portland cement together with anhydrite, in a ratio 1/1, and a binder/waste ratio of 1/1.

The samples studied were unmolded after 24 h from geometrically defined containers, and cured for 28 days at $20 \pm 3^\circ\text{C}$ constant temperature to obtain a

Table 1
Chemical analysis of steel foundry dust (SFD) (% weight)

Composition	SFD
Zn	24.37
Pb	5.70
Cd	0.07
Cr	0.30
Ni	0.06
Cu	0.50
Fe	28.9
Si	1.60
Ca	4.00
Mg	1.10
Al	0.60
Mn	4.00
Sn	0.08
K	2.00
Cl	4.60
C	1.00
S	1.50
F	0.50
Ba	< 0.01
As	< 0.01
Na	2.50
O	16.60

Table 2

Initial amount of contaminants ($\text{mg} \times 10^3$) in matrix *A* and matrix *B* wastes, and solidification/stabilization process variables

	Matrix <i>A</i>	Matrix <i>B</i>
Pb ₀	8.55	5.70
Zn ₀	36.55	24.37
Cd ₀	0.105	0.07
Cr ₀	0.45	0.30
Binder type	Portland cement	Portland cement + Anhydrite
Waste/binder ratio (w/w)	3/1	1/1
	Mixing time (min)	10
	Water/solid ratio (w/w)	4.88
	Curing temperature (°C)	20
	Curing time (days)	28

Table 3

Results of the toxicity evaluation of the steel foundry dust and solidified/stabilized forms

Solid	Chemical characterization (mg/l)					Toxicity EC ₅₀ (mg/l)
	pH	Pb	Cr	Cd	Zn	
SFD	6.24	328	0.02	28	4500	270
Matrix <i>A</i>	11	0.82	0.23	0.07	0.17	60,000
Matrix <i>B</i>	11	0.89	0.23	0.12	0.20	75,000
Limit	12 ^{a,b}	< 5 ^a	< 5 ^a	< 1 ^a	—	3000 ^b

^a US EPA regulations.

^b Spanish regulation.

monolithic cylindrical specimen of 4.5 cm diameter and 7.4 cm length. Triplicate leaching tests were made with each sample.

Results of the evaluation of the toxicity of the steel foundry dust waste and the solidified/stabilized waste forms (Matrix *A* and Matrix *B*) are reported in Table 3. Taking into account the US EPA [6] and Spanish regulations [7], the initial waste is toxic, but not the solidified/stabilized forms.

2.2. Dynamic leaching test [19]

The dynamic leaching tests were carried out in closed 2 l plastic bottles. A monolithic cylindrical specimen was immersed in distilled water at a specified ratio of leachant volume to sample surface area. The leachant was renewed at frequent intervals and the concentrations of the species leached during each interval were

determined. The test bottles were not agitated, because it was assumed that the migration rate of the contaminants in the leachant was several orders of magnitude larger than in the solid matrix, thus eliminating concentration gradients in the leachant.

The dynamic leach test is adapted from the American Society Test ANS 16.1 [8]. The leachant was renewed according to schedules proposed by Côté and Isabel [14]. The leaching intervals were calculated using a diffusion model such that the amount leached in each interval is equal. Therefore, the selected of these schedules ensure that the contaminant can be detected and that none equilibrium leaching conditions prevail.

Two schedules (A and B) are available for contaminants of greater or lesser mobility. Schedule A involved nine leaching intervals over a total period of 5 days, while schedule B involved seven leaching intervals over a total period of 9 days.

The pH value of each leachate sample was measured and aliquots were analysed for Cd, Cr, Pb and Zn concentration using a Perkin Elmer 3100B atomic absorption spectrometer.

It was checked that the set of data corresponding to the leachant renewal schedule A was above the analytical detection limit, so that the data obtained from schedule B were discarded.

For each contaminant the effective diffusion coefficient for each leaching interval was calculated from Eq. (2) and a leachability index from Eq. (3).

3. Results and discussion

In both schedules A and B, the concentration values of the Cd and Cr in the leachant were of the same order as that of the detection limit of the absorption spectrophotometer (0.001 mg/l). Therefore, these metals have not been considered in the following study.

The results of the cumulative fraction of Pb and Zn are shown in Figs. 1 and 2, where they are presented as a function of time for each type of S/S process. The pH history for the leaching test is presented in Fig. 3. The leachate pH values were initially 9.7, increasing approximately one unit during the test in both S/S processes.

The theoretical models described above considered the behaviour of a single species, not taking into account the transport and reactions of different species present in the solid, which could change the chemical environment. In the context of this study, the most important environmental change would result from a decrease in pH which would change the solubility of the metal [20]. The pH history (Fig. 3), however, shows that the leachate pH throughout the experiments remained in a range where the metal precipitates were stable (9.7–11.25).

The derivation of the models was also based on simple boundary conditions, therefore an analysis of the data was conducted first to verify the validity of the following assumptions:

(a) *The specimens behave as a semi-infinite media provided that the cumulative fraction leached does not exceed 20%*. Examination of the cumulative fraction leached after the last interval shows that the majority did not exceed 20%.

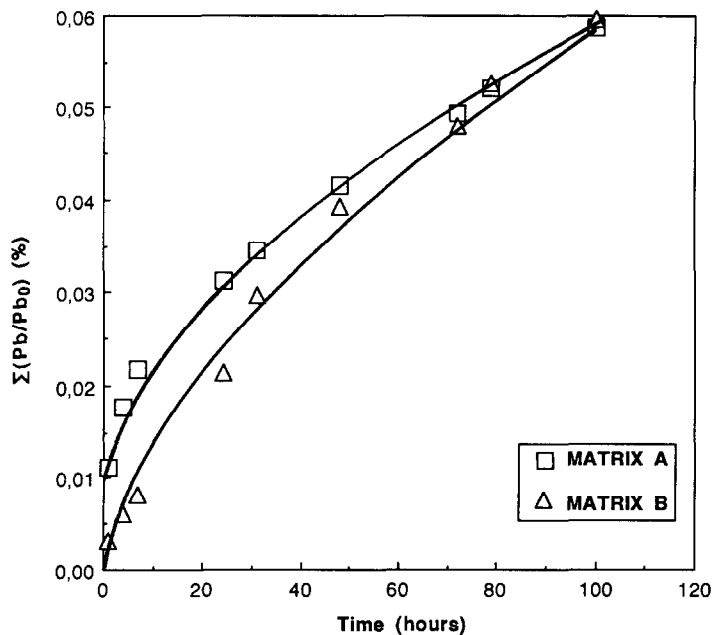


Fig. 1. Cumulative fraction of leached lead during the leaching time. \square Matrix A, \triangle Matrix B.

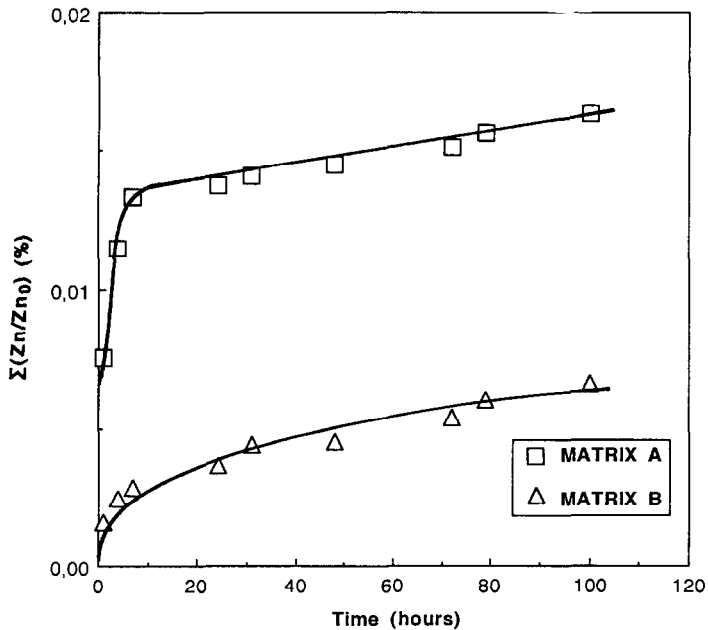


Fig. 2. Cumulative fraction of leached zinc during the leaching time. \square Matrix A, \triangle Matrix B.

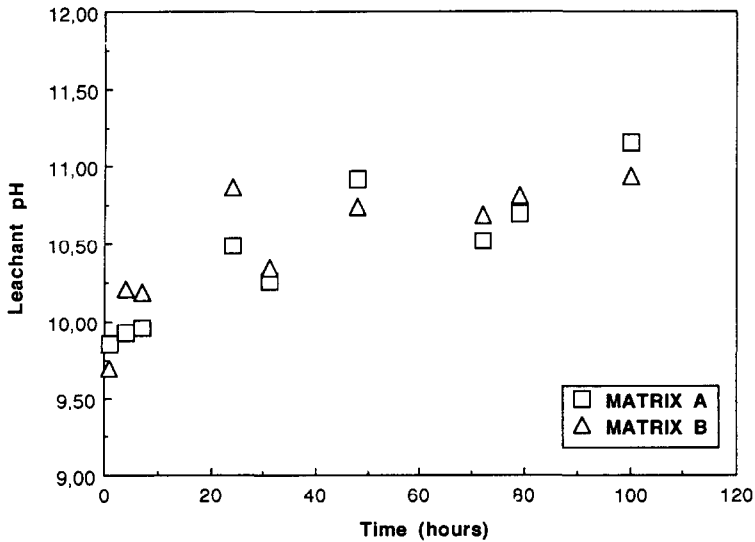


Fig. 3. Leachate pH during the dynamic leaching tests. □ Matrix A, △ Matrix B.

(b) *The model assumes zero surface concentration corresponding to the maximum driving force; this driving force becomes zero when the equilibrium concentration of the contaminant is attained in the leachant. Therefore, it is important to keep the system far from equilibrium. The schedules for leachant renewal were chosen as a compromise between this need and the requirement of a contaminant concentration higher than the detection limit for chemical analysis [21].*

The concentrations of Cd and Cr obtained in the dynamic leaching test were close to or below the detection limits for both tests. The concentrations of lead and zinc measured were small compared to the equilibrium soluble concentration. Therefore, for all metals a boundary condition of zero surface concentration was approximated.

(c) *The mobility of a contaminant is limited by diffusion. The cumulative fraction leached was represented against the square root of time for each contaminant from the developed S/S processes in order to verify the validity of the assumption of diffusion control (Figs. 4 and 5).*

The results fitted well to the semi-empirical model proposed by Côté et al. [18] (Eq. (4)). A linear regression fitting of the experimental results leads to a negligible influence of the variable t , which parameter (K_3) is not significant in the linear regression analysis. Results are shown in Table 4. They can be classified into two general categories of leaching mechanisms: (i) initial resistance to leaching followed by diffusion control; (ii) initial washoff followed by diffusion control.

A linear relationship between the cumulative fraction leached and time (Figs. 1 and 2), was not observed. It was therefore concluded that the dissolution rate was not a controlling factor in any of the leaching intervals considered.

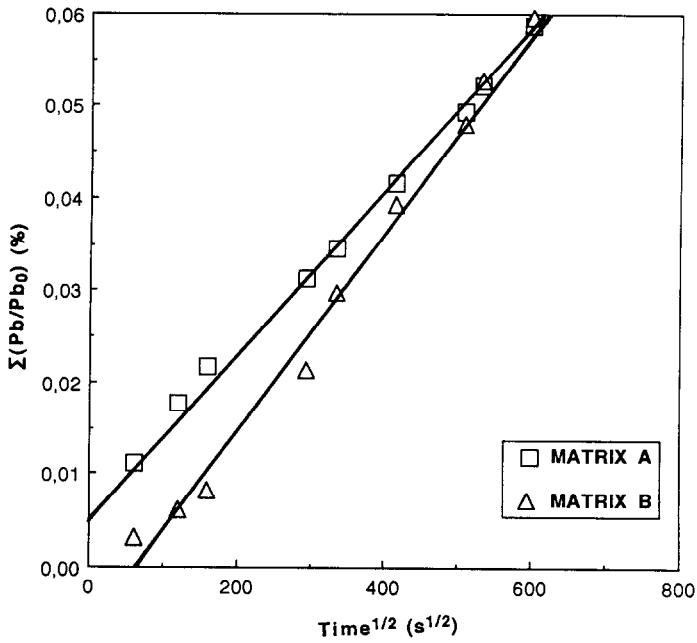


Fig. 4. Cumulative fraction of leached lead versus the square root of time (diffusion model, Eq. (4)).
 □ Matrix A, △ Matrix B.

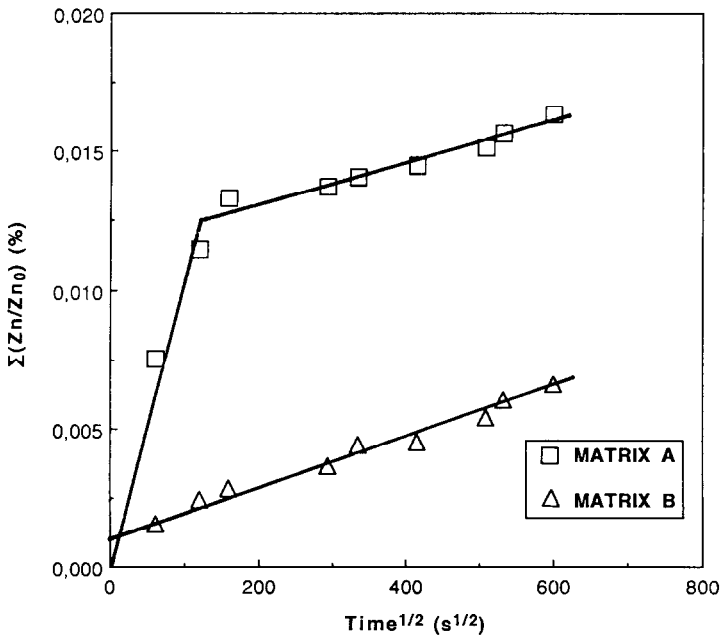


Fig. 5. Cumulative fraction of leached zinc versus the square root of time (diffusion model, Eq. (4)).
 □ Matrix A, △ Matrix B.

Table 4
Diffusivities (pD_e) and leaching indexes (LX) values of lead and zinc

Process	Metal	Controlling mechanisms			
		Surface phenomena		Diffusion pD_e^b	LX ^c
		K_1^a	K_2^a		
Matrix A	Pb	0.46×10^{-4}	8.99×10^{-7}	12.33	12.20
	Zn	1.13×10^{-4}	8.00×10^{-8}	14.43	13.78
Matrix B	Pb	-0.47×10^{-4}	1.04×10^{-6}	12.20	12.24
	Zn	8.52×10^{-6}	9.60×10^{-8}	14.27	14.14

$$pD_e = -\log D_e$$

^a Obtained from Eq. (4).

^b Calculated from Eq. (1).

^c Calculated from Eqs. (2) and (3).

The first term of Eq. (4) is related to surface phenomena. In this study, the initial surface exchanges were fast relative to the time of leaching, so these exchanges can be considered to have taken place instantaneously (time zero). They are represented by the term K_1 . A positive value of K_1 was assumed to denominate initial wash off. A negative value of K_1 was assumed to represent a delay or a resistance to leaching [20].

After an initial period, where the mentioned surface phenomena take place, diffusion appeared to be the controlling mechanism in the leaching of all the metals.

During the experiments, the following modes of behaviour were observed;

– Initial washoff or immediate dissolution at $t = 0$, $\sum(A_n/A_0) > 0$, this was observed, for Pb (Matrix A) in Fig. 4 and Zn (Matrix B) in Fig. 5; the values of the leached fraction at time zero (K_1) are shown in Table 4. The linear regression of the experimental data gives the following parameters:

$$\sum(\text{Pb}/\text{Pb}_0)_A = 4.6 \times 10^{-3} + 8.99 \times 10^{-5} \times t^{1/2}, \quad r^2 = 0.99, \quad (5)$$

$$\sum(\text{Zn}/\text{Zn}_0)_B = 8.5 \times 10^{-4} + 9.6 \times 10^{-6} \times t^{1/2}, \quad r^2 = 0.98. \quad (6)$$

– Initial washing period of about 4 h ($t^{1/2} = 120 \text{ s}^{1/2}$) is the mechanism representing leaching of Zn from the matrix A (Fig. 5).

– A delay or resistance to leaching at $t = 0$, $\sum(A_n/A_0) < 0$, is observed for Pb from matrix B (Fig. 4), in this case K_1 has a negative value (Table 4). The cumulative fraction of leached lead in matrix B increases linearly with $t^{1/2}$ giving the following linear expression:

$$\sum(\text{Pb}/\text{Pb}_0)_B = -4.7 \times 10^{-3} + 1.0 \times 10^{-4} \times t^{1/2}, \quad r^2 = 0.99. \quad (7)$$

The diffusion coefficients calculated using Eqn (1) and expressed as $pD_e = -\log D_e$, are reported in Table 4.

The same data were also used to calculate the leachability index (LX) following the approach proposed by the ANS [Eq. (3)]. The value of LX was calculated from the

incremental leach rate corresponding to each period using Eq. (2). The arithmetic average of the leaching index obtained with all the leaching periods (including those in the initial phase) were then calculated using Eq. (3) to generate the values presented in the Table 4.

Examination of the values shown in Table 4 leads to the conclusion that Pb has a similar behaviour for both S/S processes, since similar values of pD_e and LX were obtained. However, the initial behaviour was different, depending on the matrix type; specifically, the obtained (K_1) were of the same order of magnitude but with different signs.

On the other hand, Zn had a different behaviour in each matrix, the diffusion phase was analogous, obtaining very similar values of pD_e , but the initial periods showed different leaching mechanisms.

Comparison of both metals, Pb and Zn, taking into account that the leaching index of Pb is approximately two units lower than the index of Zn, reflects that Pb has greater mobility than Zn, independently of the S/S process (Matrix A or Matrix B).

Considering the scale proposed by Nathwan and Phillips [17], LX = 5 ($D_e = 10^{-5}$ cm²/s very mobile or rapid diffusion, and LX = 15 ($D_e = 10^{-15}$ cm²/s) immobile or very slow diffusion, and taking into account that the leaching index obtained with the stabilized waste samples is greater than 12, suggests that both S/S processes are acceptable.

4. Conclusions

First, the study of the long-term behaviour of S/S process of SFD waste using the dynamic leaching test allowed the dynamic leaching models of Pb and Zn to be established from a representation of the cumulative fraction leached against the square root of time. This verifies the assumption of diffusion control inherent to this test, resulting in the identification of two different leaching mechanisms: (i) initial resistance to leaching following by diffusion control and (ii) initial washoff following by diffusion control of contaminants.

Second, the leachability index and diffusion coefficients for Zn and Pb were calculated from the amounts leached in each interval of the dynamic leach test, provide a measure of the mobility of the contaminants in the solidified waste matrix.

Lead has a similar behaviour in both S/S processes, while Zn presents greater mobility with matrix A than with matrix B, because a significant initial washoff period (≈ 4 h) takes place in the first case, whereas immediate dissolution occurs with matrix B. Pb presents greater mobility for this type of S/S process than Zn (LX_{Pb} is 1.5 units lower than LX_{Zn}).

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