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Waste Materials in Construction: Putting Theory into Practice

EVALUATION OF CONTAMINANT RELEASE MECHANISMS FOR STABILIZED/SOLIDIFIED WASTES

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ABSTRACT

Understanding mechanisms which control release of inorganic contaminants from stabilized/solidified wastes is important for predicting the long-term leaching of contaminants in the field. Frequently, release is assumed to be controlled either by solubility or diffusivity as limiting cases. These assumptions also dictate the interpretation of laboratory test methods. The consistency of testing and interpretation methods was evaluated using a synthetic solidified matrix spiked with lead chloride, using both batch extractions on size reduced materials, where equilibrium was anticipated, and tank leaching tests, where diffusion controlled release was anticipated. Differing approaches to determining availability, solubility and dynamic release are compared.

1. INTRODUCTION

The development of stabilization/solidification processes using hydraulic binders has resulted from increasingly stringent regulations in the field of environmental protection. The characterization of the leaching behavior of these materials is crucial in the environmental assessment of disposal or re-use scenarios. Numerous leaching tests with different purposes have been developed: simple tests used for regulatory compliance, and more "elaborate" tests used to better understanding of the physical-chemical phenomena that occur during and leaching and provide a basis for long-term prediction. One possible classification of leaching tests is to distinguish between equilibrium and mass transfer leaching tests. Equilibrium leaching tests, which typically are conducted on crushed materials, aim to determine an available release potential (e.g., Availability Test [1]), constituent solubilities (e.g., by varying pH [2, 3]), or matrix alkalinity. Mass transfer leaching tests carried out on

monolithic samples over a long period (from one week to 3 months or more) aim to determine release rates and account for chemical and physical properties of the matrix. Their interpretation is generally carried out by coupling results obtained with the equilibrium leaching tests.

A comparison of leaching tests applied to stabilized/solidified wastes and their interpretation was carried out within the framework of the international scientific collaboration between the Association RE.CO.R.D (Waste Research Cooperative Network, France) and the HSMRC (Hazardous Substance Management Research Center, US). Results discussed in this paper are the outcome from two parallel studies conducted by the research groups at the National Institute of Applied Sciences — LAEPSI/POLDEN — (INSA), Lyon, France [4] and Rutgers, The State University of New Jersey (RU), USA [5]. The specific objectives of the study were to compare testing results from varied conditions for measurement of (i) matrix alkalinity, (ii) constituent solubility as a function of pH and (iii) dynamic release. A synthetic solidified waste spiked with lead chloride was used for the initial phase of this study.

2. MATERIALS AND METHODS

2.1 Solidified Matrix Preparation

The synthetic solidified waste was prepared by mixing 34 wt% Ordinary Portland Cement, 11 wt% water, 44 wt% sand, 10 wt% lead chloride, and 1 wt% sodium chloride. Although the INSA and RU samples were prepared using the same mixture, they were prepared at two different times using different lots of cement. Samples for INSA were cast as blocks of 15x20x10 cm^3. Samples for RU were cast as 10 cm diameter by 10 cm height cylinders using polyethylene molds. Samples were vibrated in the molds for one minute before being stored at room temperature in sealed plastic bags. After 28 days of curing, samples for INSA were cored from the cast blocks as 4 cm diameter cylinders with 1, 2 or 8 cm heights. Fragments of the blocks were saved in sealed plastic bags as source material for tests on crushed materials. After 6 months of curing, samples for RU were removed from the molds and used for testing.

2.2. Measurement of Matrix Alkalinity and Lead Solubility

Determination of matrix alkalinity and lead solubility from the matrix as a function of pH was carried out using modifications of the Acid Neutralization Capacity leach test [6]. RU samples were crushed and jar milled until > 85% passed through a 300 μm sieve. In parallel, ten 5 g aliquots of the size-reduced matrix were extracted using nitric acid solutions

of varying acidity. Deionized water and nitric acid were added in varying proportion to each 5 g sample to achieve acid additions ranging from 0 to 10 meq/g solid and a final liquid addition of 30 ml (liquid to solid (LS) ratio of 6:1 ml/g). Each case was carried out in triplicate. After liquid addition, the mixtures were agitated for 24 hr. Subsequently, the solid and liquid phases were separated using vacuum filtration through 0.45 μm pore size polypropylene filters. The filtered extract was analyzed for pH and lead.

INSA samples were size reduced by grinding to pass through either a 160 μm or 315 μm sieve [4]. Two different size reduction criteria were used to assess the impact of size reduction on observed results. In parallel, twenty 15 g aliquots of the size-reduced matrix were extracted using nitric acid solutions of varying acidity. Deionized water and nitric acid were added in varying proportion to each 15 g sample to achieve acid additions ranging from 0 to 10 meq/g solid and a final liquid addition of 150 ml (LS of 10:1 ml/g). After liquid addition, the mixtures were agitated for either 24 hr or 5 days to assess the time required to achieve equilibrium for both the < 160 and < 315 μm material. The case using < 315 μm material with agitation for 24 hr was carried out in triplicate, while the other cases were carried out without replication. Subsequently, the solid and liquid phases were separated using vacuum filtration through 0.45 μm pore size polypropylene filters. The filtered extract was analyzed for pH and lead.

The acid neutralization behavior of the materials was evaluated by plotting the pH of each extract as a function of milli-equivalents of acid added per gram of dry solid. Lead concentration in each extract is plotted as a function of extract final pH to provide solubility as a function of pH. The acid quantities required to reach pH 11.9 and pH 9 were observed because of the nature of the studied materials (cement matrix). Indeed, pH 11.9 is the pH theoretically reached after the complete neutralization of the portlandite (pH of transition between the predominance of CSH (i.e., gel formed of hydrated calcium silicates) and portlandite (i.e., $Ca(OH)_2$) and the predominance of rich calcium silicate CSH gels) [4]. pH 9 is the pH for which the solubility of most amphoteric metals is minimum and corresponds also to the stability limit of the main hydrate phases of a cement matrix. The concentration of calcium hydroxide produced during the hydration reactions of the cement, can also be estimated from the curve.

2.2. Mass Transfer Leaching Tests

Short duration monolith leaching tests were carried out on the RU samples in triplicate. Each monolithic sample (10 cm diameter by 10 cm height) was immersed in 4.6 l of deionized water, equivalent to 10 ml of leachant/cm^2 exposed surface area. The leachant

was refreshed with an equal volume of deionized water, following a 2^N progression of 3, 6, and 12 hours, 1, 2, 4 and 8 days. The leachant to surface area ratio and refreshing intervals were selected to minimize solubility limitations of lead in the leachant. After each extraction interval, the resulting extracts were filtered through 0.45 μm pore size polypropylene filters and preserved with nitric acid to pH < 2 for chemical analysis. The concentration of constituents in each leachate was measured with flame atomic absorption spectrometry.

The observed diffusivity for each replicate was calculated as the mean of the observed diffusivity for each leaching interval. The observed diffusivity for each leaching interval was calculated as

$$D_{obs,\,i} = \pi \left[\frac{B_i}{2 \rho A_o \left(t_{i+1}^{0.5} - t_i^{0.5} \right)} \right] \quad [m^2/s]$$

where

B_i = mass released per unit surface area [mg/m^2] during the leaching interval [t_i, t_{i+1}];

ρ = sample density [kg/m^3];

A_o = constituent availability determined by the Availability Leach Test [1] or initial concentration [mg/kg]; and

t_i = leaching time interval i [s].

Only intervals during which the slope of mass released as a function of the log of the time interval was between 0.35-0.65 were included in calculation of the mean diffusivity.

Long-term monolith leaching tests were carried out on INSA samples. Cylinders 4 cm diameter and 1, 2 or 8 cm height were used to provide variable external surface area and to observe the depletion of very soluble species (e.g., sodium and chloride) during the testing. Three replicates were carried out on the 2 and 8 cm height samples. Deionized water was employed as the leachant based on a liquid-to-solid ratio of 10 ml of leachant per gram of solid. The leachant was refreshed with an equal volume of deionized water at time intervals of 3, 5, 16, 24 hours, 2, 3, 4 days, 1, 2, 3, 4 weeks and thereafter every month up to a cumulative leaching period of 8 months. After each extraction interval, the resulting extracts were filtered through 0.45 μm pore size polypropylene filters and preserved with nitric acid to pH < 2 for chemical analysis. The pH of the filtered leachates was measured prior to preservation. Leachates also were analyzed for Na, Ca, Pb with ICP-AES and Cl with ion chromatography. Complete results have been reported elsewhere [4].

The initial leachable concentration, C_0, (i.e., availability) and the observed diffusivity of very soluble species (e.g., sodium or chloride) in the matrix were calculated using a 3-

dimensional diffusion model [7]. These parameters are simultaneously identified to account for depletion of the species in the solid core.

A model coupling dissolution and transport by diffusion was used for species whose solubilities exhibit a strong dependence on pore water pH [4]. This model was used because of large pH gradients which existed between the matrix interface with the leachant and within the solid matrix. This model can be divided into several stages:

- Release of portlandite using a shrinking front model;
- Calculation of the induced pH profile, assuming thermodynamic equilibrium occurs in the pore water;
- Determination of local lead solubility by calculation assuming the main equilibria or from experimental results (e.g., equilibrium leaching tests); and,
- Description and calculation of lead transport by diffusion in the pore water.

The coupled dissolution/diffusion model requires the knowledge of several parameters: matrix porosity, solid phases concentrations of constituents of interest (e.g., Ca, Na, Pb), constituent solubility as a function of pH, and observed diffusivity within the porous medium for each species of interest. The values of these parameters were initialized using experimental data and then adjusted by successive simulations until the simulated results coincided with the observed experimental results.

The equilibrium concentration of lead as a function of pH was determined using a simplified representation of the cement pore water solution assuming two principal components, calcium hydroxide and lead hydroxide. The resulting chemical equilibria considered were

$Ca(OH)_2 \Leftrightarrow Ca^{2+} + 2OH^-$ $10^{-5.2}=[Ca^{2+}]\,[OH^-]^2$ (If solid $Ca(OH)_2$ is present)

$Pb(OH)_2 \Leftrightarrow Pb^{2+} + 2OH^-$ $K_2= [Pb^{2+}]\,[OH^-]^2$ (If solid $Pb(OH)_2$ is present)

$Pb(OH)_3^- \Leftrightarrow Pb^{2+} + 3OH^-$ $K_3 = \dfrac{[Pb^{2+}]\,[OH^-]^3}{[Pb(OH)_3^-]}$

$2\,H_2O \Leftrightarrow H_3O^+ + OH^-$ $10^{-14} = [H_3O^+]\,[OH^-]$

The solubility product for calcium was provided by the literature [8]. The solubility constants for lead also could be found in the literature, but may be far removed from the experimental conditions. Thus, solubility constants for lead were estimated from the experimental solubility curve given by the equilibrium leaching tests and adjusted so that the simulated results were consistent with the results of the monolithic leaching test.

3. RESULTS

3.1 Acid Neutralization Capacity and Lead Solubility

Acid neutralization capacity curves for each experimental condition are provided in Figure 1. Contact time (24 hours or 5 days) did not significantly influence acid neutralization capacity for the INSA samples size reduced to < 160 μm and yielded the same results as the RU samples. RU samples size reduced to less than 300 μm appeared to exhibit similar behavior as the < 160 μm INSA samples because of the more aggressive size reduction procedure employed, which most likely resulted in a finer particle size distribution than 300 μm. Contact time did effect the acid neutralization capacity observed between pH 11 and 3 for INSA samples size reduced to < 315 μm. Initial neutralization of portlandite (to pH 11.9) appeared to be the same for all samples, but dissolution of the CSH gel at pH < 11.9 appeared to be incomplete when the larger particle size sample was contacted for only 24 hr. This is probably a consequence of less exposed surface area for the larger particles and thus slower dissolution. For a given particle size, the contact time required to reach 90% of constituent solubility increases with increasing fractional solubility and pD (Figure 2; Crank [9])[1,2]. Thus, the contact time required to reach 90% of solubility for a constituent with a pD_{obs} of 14.5 and fractional solubility much less than 10% (i.e., $M_\infty/M_o < 0.1$), is greater than 24 hr.

The acid quantities per gram of solid dry required to reach pH 11.9 and pH 9 are presented in Table 1 according to the size reduction and contact time. The concentration of calcium hydroxide produced during the hydration reactions of the cement can be estimated at 230 kg/m^3 of porous medium (ca. 21% of the hydrated cement paste) based on the acid quantity required to reach the pH 11.9.

Lead solubility as a function of pH for each experimental condition is presented in Figure 3.

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[1] pD=-log(D) where D is in units of m^2/s.

[2] The diffusion of a constituent from a sphere into an infinite solution can be modeled as

$$\frac{M_t}{Mo} = \frac{6}{\pi^2}\sum_{n=1}^{\infty}\frac{1}{n^2}\exp\left(\frac{-D_{obs}\cdot n^2\cdot\pi^2\cdot t}{a^2}\right)$$

where,

M_t = constituent mass released from the particle size in time t [mg];
M_o = initial constituent mass in the particle (availability) [mg];
M_t/M_o = fractional release;
D_{obs} = observed constituent diffusivity in the particle matrix [m^2/s];
t = contact time or test duration [s];
a = particle radius [m]; and
M_∞ = constituent mass released from the particle size in an infinite time [mg].

Overall, test results obtained from all experimental conditions were in good agreement. Neither contact time nor extent of particle size reduction influenced the observed lead solubility. This is in contrast to the particle size effect observed for acid neutralization capacity but consistent with the dissolution of $Ca(OH)_2$ from the solid matrix. The impact of liquid-to-solid ratio appeared to be slight. For pH < 5, the solubility obtained with the ratio of 6:1 ml/g was slightly greater than the results obtained with the ratio of 10:1 ml/g; for pH > 5, the results of solubility obtained with the ratio of 6:1 ml/g were slightly less than those obtained with the ratio of 10:1 ml/g. The minimum solubility was observed between pH 8 and 10 which suggests that lead hydroxide is the solid phase controlling solubility [8].

3.2 Assessment of Dynamic Release

3.2.1 Leaching behavior of soluble species (sodium and chloride)

Cumulative release of sodium and chloride as a function of time for all experimental are presented in Figures 4 and 5. A comparison of the leachant to surface area and volume ratios, as well as sample surface area to volume ratios are useful for interpreting release data (Table 2). The release of sodium was rapid with > 80% of the total content released from the long-term monolithic leaching test samples but only 22% from the short-term test samples after one week of leaching. The release of chloride during one week of leaching was *ca.* 50% of the total chloride from the long-term monolithic leaching test samples and 4% from the short-term leaching samples. The smaller fraction of sodium and chloride released from the short-term test samples over this time period was attributed to the smaller sample surface area to sample volume ratio. Release over 8 months from the long-term samples was consistent with the sample surface area to volume ratio for each case. Depletion occurred most rapidly for the sample with the greatest surface area to volume ratio. Initial release flux for each element was similar for all cases. However, there was greater initial release of sodium from the RU samples which may be attributable to surface washoff as a consequence of sample molding.

Values of availability and diffusivity for sodium and chloride determined using the different interpretation techniques are compared in Table 3. Availability for the long-term release test cases was estimated using the 3-dimensional diffusion model, while availability for the short-term release test cases was measured using the Availability Leach Test. The available sodium content was different for the long and short-term test cases resulting from the different batches of the solidified matrix. However, the fraction of total sodium which was available was consistent between the two approaches (92 vs. 95%). Availability

determinations for chloride also provided consistent results for the two approaches (67 vs. 63%).

Observed diffusivity values for sodium were consistent for both approaches. However, the observed diffusivity for chloride using the short-term testing approach was sixfold less than that obtained from the long-term testing approach. A comparison of the long-term flux measured and predicted by the 3-dimensional diffusion model indicates that good agreement is obtained up to *ca.* 500 hr but measured fluxes are greater than predicted at longer intervals (Figure 6). The residual flux observed after 500 hr may be a result of dissolution of the constitutive phases of the solidified matrix.

3.2.2 Leaching behavior of lead

The coupled dissolution/diffusion model was used to simulate results obtained from both long-term and short-term monolith leaching tests. Parameter estimates used in the model and a comparison of model results with long-term leaching of lead and calcium are presented in Table 4 and Figure 7, respectively. A comparison of model results with short-term leaching for lead are presented in Figure 8. Overall, the parameters estimated from the short-term testing are consistent with those from the long-term testing. The equilibrium constants derived from the model fitting to the experimental data also permit simulation of lead solubility in the pore water as a function of pH. This simulated lead solubility is compared with the experimental results from equilibrium testing described earlier (Figure 9). The lower predicted solubility as compared to the measured solubility at pH > 8 may from differences in the aqueous ionic strength and chloride complexation between the experimental conditions. Within the pore matrix during long-term testing, ionic strength and chloride complexation with lead may be less than in batch experiments because of the rapid removal of sodium and chloride from the matrix.

An observed diffusivity for lead also can be calculated from the short-term testing results using the interval method. The resulting D_{obs}, equal to 10^{-16} m^2/s, also can be related to a retardation factor and effective diffusivity according to

$$D_{obs} = \frac{D_e}{R}.$$

If we consider as the effective diffusion coefficient the identified coefficient obtained by using the coupled dissolution/diffusion model (i.e., $D_e = 10^{-9.1}$ m^2/s) the retardation factor R is then equal to 8×10^6. However, this does not clearly correlate with the equilibrium constants according to the relation

$$\text{Solubility} = \left[\frac{AVLT * \rho}{1000 * \eta}\right] * \frac{1}{R-1}$$

where

AVLT = constituent availability or initial concentration [mg/kg];

ρ = sample density [kg/m^3];

η = porosity; and

R = retention factor.

The coupled dissolution/diffusion model also indicates that no depletion of lead in the solid phase occurs at the solid-liquid interface during the time scale of the long-term laboratory testing. Thus, lead release is controlled by solubilization phenomenon at the solid-liquid interface. In this case, the coupled dissolution/diffusion model can be simplified. The diffusional transport of lead within the matrix can be neglected and the flux of lead can be expressed in terms of a mass transfer coefficient and the difference between the lead saturation concentration and the concentration in the leaching solution. Consequently a shrinking core model can be used to describe the release of calcium and pH changes at the solid-liquid interface. Lead release then can be described using a constant mass transfer coefficient and changes in lead solubility as a function of pH [4].

3. CONCLUSIONS

Based on the experimental results, modeling and previous observations, the following conclusions can be made:

- Measurement of acid neutralization capacity is sensitive to the extent of particle size reduction and extraction period. Use of particle size reduction to < 165 μm in conjuction with a 24 hr agitated extraction has been demonstrated to achieve equilibrium. However, determination of lead solubility in conjunction with acid neutralization capacity was not sensitive to extent of particle size reduction or extraction period over the range of conditions examined. These relatively simple tests provide a rapid method to measure the buffering capacity of solidified materials and the solubility of specific metals as a function of equilibrium pH. Results also provide information on the chemical speciation of the pollutants considered and allow prediction of the amount of acid required to neutralize a certain alkalinity and to decrease the pH to a certain point. This information can be used to estimate how long the buffering capacity will last [10] and consequently when the solubility of amphoteric metals may increase dramatically.

- Evaluation of leaching parameters did not appear to be sensitive to curing intervals in excess of 28 days.

- Short-term (8 days) and long-term (8 months) dynamic leach tests on monolithic samples provided consistent estimates of leaching parameters (observed diffusivity, leachable concentration or availability) for very soluble species. Thus, short-term testing may be used to evaluate long-term release when the principal mechanisms of release are well understood. Evaluation long-term leaching should consider the effect of the depletion of very soluble species from the solid matrix on leaching parameters (e.g., porosity).

- Determination of constituent availability based on direct experimental measurement and two parameter modeling of dynamic release provided consistent results.

- Lead appeared to be less soluble in the pore water of the solidified matrix than observed during determination of solubility using a batch extraction on size reduced material. This effect may have resulted from rapid initial release and depletion of very soluble species (sodium and chloride) during monolith leach testing.

Table 1. Acid neutralization capacity from experimental conditions evaluated.

mEq/g dry	INSA samples				RU samples
	< 160 μm		< 315 μm		< 300 μm
Contact time	24 hr	5 days	24 hr	5 days	24 hr
pH 11.9	2.2	2.3	2.6±0.005[a]	2.8	2.4±0.6[a]
pH 9	5.9	5.8	3.2±0.08[a]	5.7	6.5±0.2[a]

[a] Standard deviation of data points from their mean (three replicates).

Table 2. Comparison between sample surface area, volume and leachant volume for experiment conditions evaluated.

	INSA samples			RU samples
Ratios	d 4 cm, h 1 cm	d 4 cm, h 2 cm	d 4 cm, h 8 cm	d 10 cm, h 10 cm
$\left(\frac{\text{Sample surface area}}{\text{Sample volume}}\right)$ (cm^{-1})	3	2	1.25	0.6
$\left(\frac{\text{Leachate volume}}{\text{Sample surface area}}\right)$ (cm)	8.4	12.5	20	10
$\left(\frac{\text{Leachate volume}}{\text{Sample mass}}\right)$ (ml/g)	10	10	10	2.4

d = diameter, h = height.

Table 3. Comparison of the leaching parameters for monolith leaching tests.

	Long term monolithic leaching tests (8 months of leaching)					Diffusivity test method (One week of leaching)		
	3-D diffusional model			*Interval diffusion coefficient method*		*Availability test - Interval diffusion coefficient method*		
Elements	C_0 (kg/m^3)	C_0/C_i (%)	- log D_{obs} (m^2/s)	C_0 (kg/m^3)	- log D_{obs} (m^2/s)	C_0 (kg/m^3)	C_0/C_i (%)	- log D_{obs} (m^2/s)
Sodium	9.0	92	10.2	9.0[b]	10.5	17±0.8[a]	95	10.3±0.02[a]
Chloride	52.7	67	10.5	52.7[b]	10.9	58.7±0.3[a]	63	11.3±0.01[a]

[a] Standard deviation of data points from their mean (three replicates).

[b] The availability test was not carried out on these samples. Therefore, the values used for the available potential release are the identified values obtained by using the 3-D diffusional model.

Table 4. Parameters values from the coupled dissolution/diffusion model.

Porosity		22%[a]	
Solid phases concentrations (kg/m³ of porous medium)	$Ca(OH)_2$	230[b]	
	$Pb(OH)_2$	217[c]	
		INSA results	RU results
Equilibrium constants	Product of solubility: $[Pb^{2+}][OH^-]^2$	$10^{-16.5}$	$10^{-16.7}$
	Constant of complexation: $\frac{[Pb^{2+}][OH^-]^3}{[Pb(OH)_3^-]}$	$10^{-13.98}$	$10^{-13.98}$
Effective diffusion coefficients: -log D_e (m^2/s)	Ca^{2+}	9.15	9.15
	Pb^{2+}	9.1	9.3
	$Pb(OH)_3^-$	9.1	9.3

[a] The experimental porosity measured by mercury intrusion was 11% on unleached samples. However, the identified porosity was 22%. This difference may be due to the variation of the porosity during the leaching according to the release of soluble species and precipitation of phases (i.e., pursuit of the hydration reactions, carbonation). As the structural evolution of the matrix during the leaching is not included in the model, the porosity is adjusted by successive simulations.

[b] Estimated from the acid neutralization results.

[c] Calculated from the initial quantity incorporated.

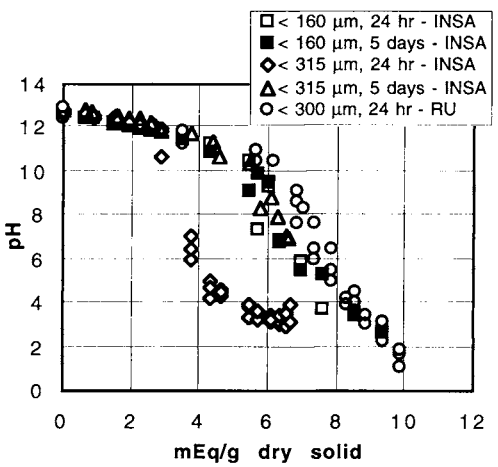

Figure 1. Acid neutralization capacity curves from experimental conditions evaluated.

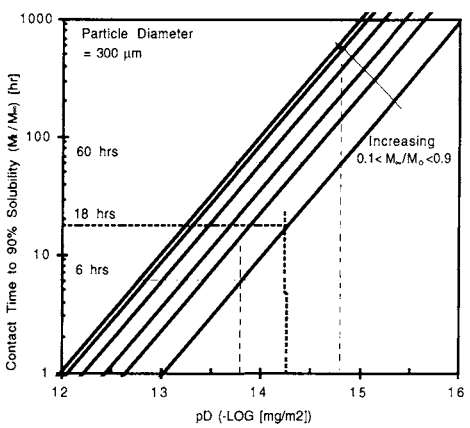

Figure 2. Required test contact time to achieve 90% solubility as a function of constituent solubility as a function of constituent diffusion coefficient with a 300 µm diameter particle.

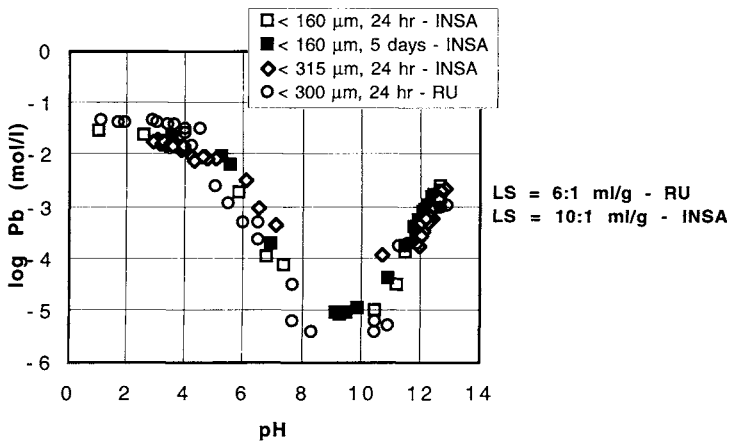

Figure 3. Lead solubility as a function of pH measured using batch extractions on size reduced material.

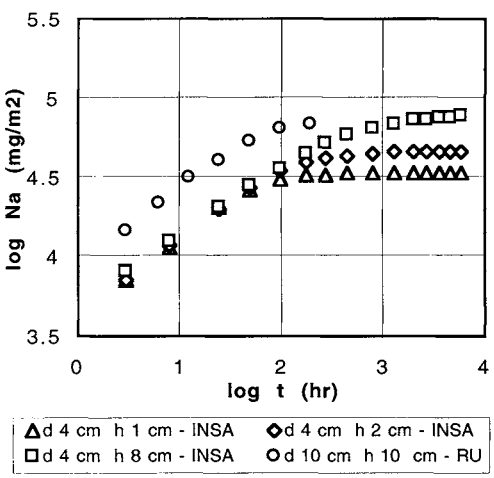

Figure 4. Cumulative release of sodium as a function of time.

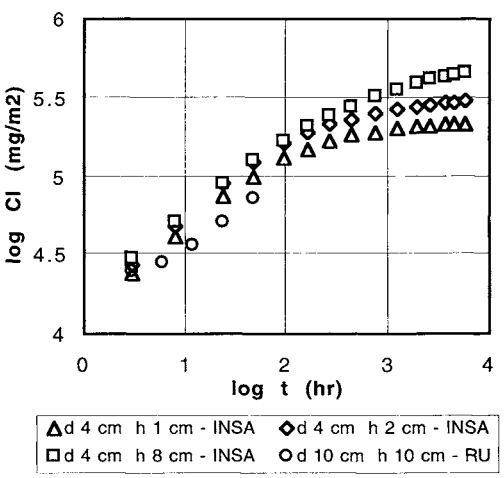

Figure 5. Cumulative release of chloride as a function of time.

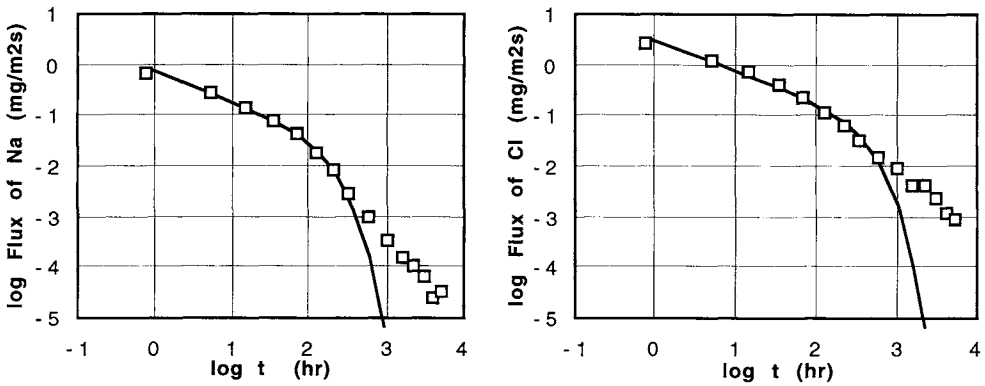

Figure 6. Comparison of diffusional model prediction and experimental data. The flux of sodium and chloride are shown.

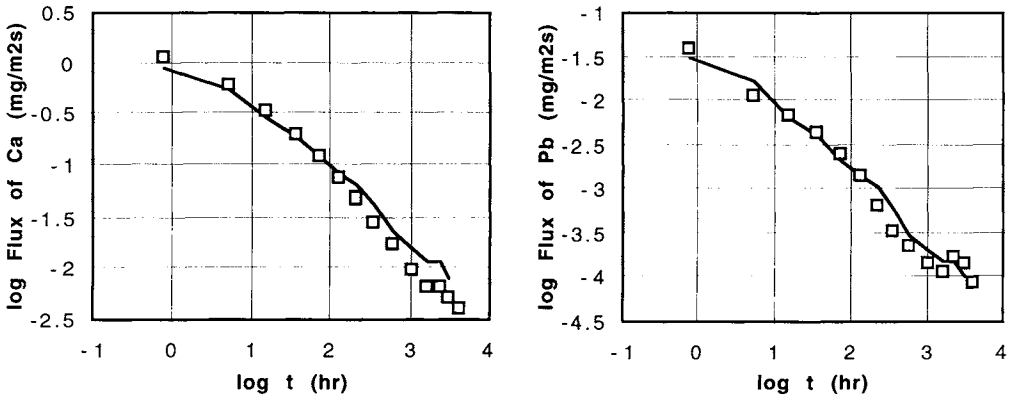

Figure 7. Comparison of coupled dissolution/diffusion model prediction and experimental data. The flux of calcium and lead during the long-term monolith leaching tests are shown.

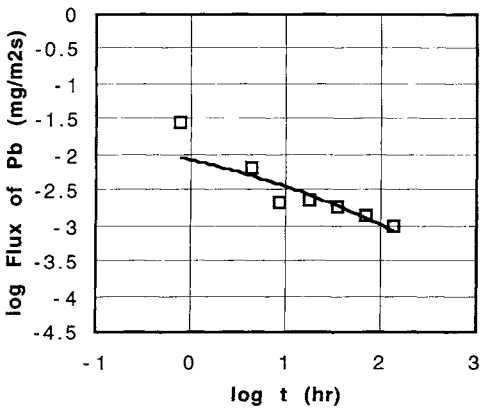

Figure 8. Comparison of coupled dissolution/diffusion model prediction and experimental data. The lead flux during the diffusivity test (i.e., one week of leaching) is shown.

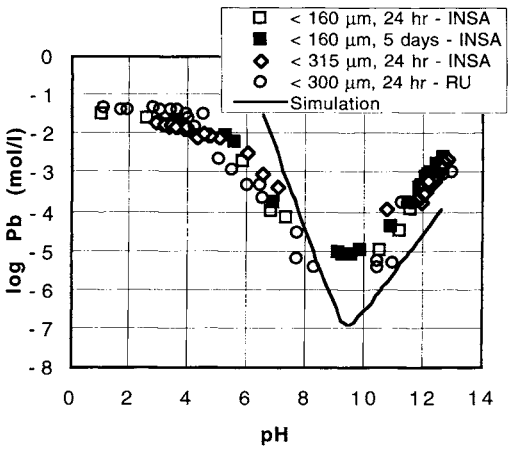

Figure 9. Comparison of simulated and experimental data of lead solubility.

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