Influence of Void Change, Cracking, and Bitumen Aging on Diffusional Leaching Behavior of Pavement Monoliths Constructed with MSW Combustion Bottom Ash

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The evaluation of leaching behavior of monolithic forms containing waste materials requires the use of leaching tests that evaluate diffusional release mechanisms. This can be coupled to procedures that accelerate the aging of the monolith to better predict long-term leaching behavior. As part of a comprehensive demonstration project examining the use of municipal solid waste combustion bottom ash as an aggregate substitute in bituminous binder course, the diffusional leaching behavior of ash constituents was assessed as a function of pavement voids level (5, 7.5, 10 percent), bitumen aging (none, medium, high), and monolith cracking (none, medium, high). Effective diffusion coefficients (D_e, m^2/sec) were determined for some ash constituents that leach; values ranged from 10^{-12} to 10^{-16} m^2/sec. These values are typical for ion diffusion in polymer systems. Species with small ionic radii (e.g., Na, Cl) were more diffusive than those with large ionic radii (e.g., Si, Zn). Monolith tortuosity values (τ) were found to be influenced only by monolith cracking. Diffusing specie chemical retention values (R) and effective diffusion coefficients were not influenced by voids, cracking, or aging. Bituminous pavement monoliths release ash constituents at very low fluxes. Modeled releases of chloride for 10 percent wetting ranged from 1.7 g Cl/m^2/1.5 years to 54.9 g Cl/m^2/100 years. These fluxes are considerably smaller than fluxes associated with road salting during a single snowfall. The tortuous, hydrophobic nature of the monolith system controls diffusion.

In the fields of waste stabilization and waste use, it is frequently desirable to produce monolithic forms from granular material. Examples include the solidification and stabilization of contaminated granular soils with portland cement and use of coal fly ash as filler in bituminous asphalt. In either case, the environmental performance of the product depends on the analysis of leaching behavior of monolithic instead of granular material.

In monolithic leaching scenarios (Figure 1), the very low specific surface area, permeability, and high structural integrity result in leaching regimes where diffusion is the dominant mass transfer mechanism. This differs dramatically from granular materials where solvent percolation and advection produce contaminant fluxes that are orders of magnitude larger than diffusional fluxes. Diffusional leaching involves diffusion into the monolith of solvents, such as H_2O, and diffusion out of the monolith of dissolving solutes such as cationic and anionic species. In the nuclear waste disposal field, the ANSI/ANS 16.1 monolith leach test (*1*) is used to assess diffusional release. In the hazardous waste disposal field, both the ANSI/ANS 16.1 and ASTM single batch extraction test (*2*) can be used to assess diffusional release. Finally, in the field of waste use, the Dutch NEN 7345 monolithic tank diffusion leaching test (*3*) is used to examine diffusional leaching behavior. To accelerate leaching, flow-through triaxial devices (*4*) or pressure vessels (Van der Sloot, personal communication) can be used to speed up the influx of solvent into the monolith. In virtually all these cases, an effective diffusion coefficient (D_e in m^2/sec) can be calculated and then used to model release as a function of geometry and application cycle of leachant.

The issue of monolith integrity plays an important role in evaluating diffusional leaching behavior. Improper solidified and stabilized (S/S) formulations, freeze or thaw, or wetting or drying phenomena can cause monolithic waste forms to spall, crack, or devitrify. When granular wastes or conventional materials are used in civil construction applications, the materials can undergo physical changes due to loading, temperature, and oxidation (O_2, UV) that can also result in loss of monolithic integrity. The ability to induce such loss of integrity in the laboratory for a monolithic leaching scenario allows the prediction and modeling of

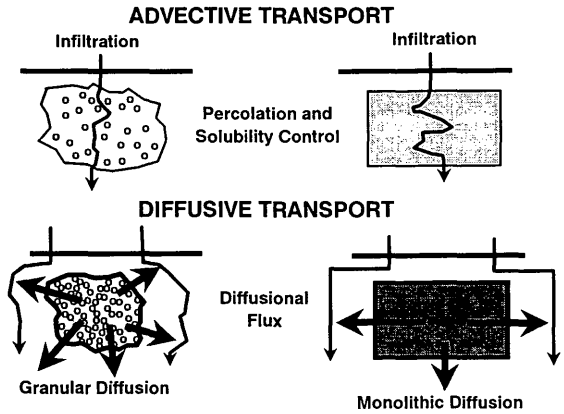

FIGURE 1 Advective and diffusive release (*19*).

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potential leaching behavior at the end of a design life of an application.

The authors are involved in a large demonstration project that examines the feasibility of using bottom ash from the combustion of municipal solid waste as an aggregate substitute in asphaltic binder course (*5–10*). A demonstration project involving the paving of a portion of Route 3 in Laconia, New Hampshire, has been underway since May 1993. To understand better the potential environmental behavior of monolithic pavement material over its 20- to 30-year design life, the Dutch monolith tank leaching procedure was used to evaluate the potential effects of void change, cracking, and bitumen aging on the diffusional leaching of bottom ash constituents from asphaltic pavement monoliths.

This research has shown that the bottom ash constituents that leach from the pavement monoliths are Si, Ca, SO_4, Mn, Cl, and Mg. The effective diffusion coefficients (D_e m^2/sec), reported as their negative logs (pD_e), are in the range of 12 to 16. These values are typical for ash constituent elements diffusing in polymer monoliths (*7,11*). The influence of monolith cracking was the only experimental variable to alter diffusional leaching behavior. Cracking reduces the diffusion path length from which diffusional release occurs. Void change and bitumen aging had no significant effects.

MONOLITH LEACHING FUNDAMENTALS

Figure 2 depicts the fundamental processes involved in leaching from monolithic specimens. The figure depicts the diffusive release of constituents via pathways of varying tortuosity, long-term dissolution of solid phase material at the monolith surface, and initial wash-off of material from the monolith surface. These phenomena exhibit different cumulative release behaviors (see Figure 3), as evidenced by plot slopes, on log-log cumulative release versus time plots.

Diffusion occurs by the random movement of individual molecules or ions. It is driven by the difference in chemical potential between the solid and the leachant (*12*). In a slightly porous solid, the ion flux of a soluble contaminant in the pore water system is defined by Fick's Second Law, which relates the concentration of a diffusing substance to both space and time:

$$\frac{dC}{dt} = L\frac{d^2C}{dx^2} \tag{1}$$

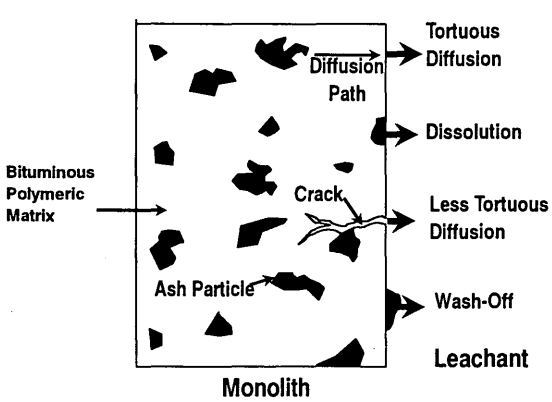

FIGURE 2 Monolithic leaching behavior.

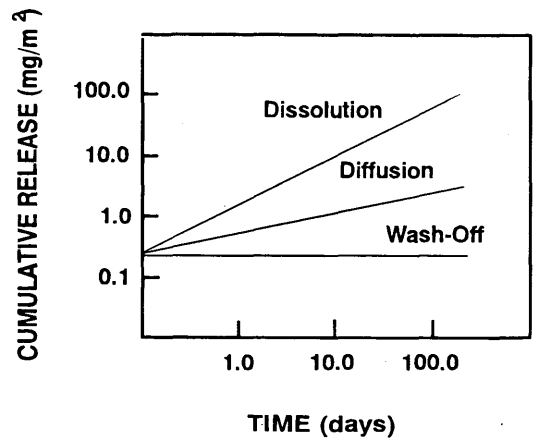

FIGURE 3 Hypothetical cumulative release plots.

where C is the concentration of the diffusing ion, and L is the leach constant, with units of a diffusion coefficient (m^2/sec). In the case of one-dimensional diffusion, the leach constant, L, is obtained by applying the following relationship:

$$L = \frac{f^2 \cdot D_o}{R \cdot \tau} \tag{2}$$

where

f = available leachable fraction of element in material,
D_o = mobility of element in water (m^2/sec),
R = chemical retention factor of element (unitless), and
τ = physical retardation or tortuosity factor (unitless).

When the leachable amount of the element equals the total amount of the element present in the material (i.e., $f = 1$), the leach constant L equals the effective diffusion coefficient, which is diffusion modified for retention and tortuosity (*13*).

If Equation 2 is transformed to logarithmic values and the relationship $pD_o = -\log D_o$ is used, then the new relationship is as follows:

$$pL = pD_o + 2pf - pR - p\tau \tag{3}$$

Because the available leachable fraction of the element may be obtained from analytical data and remains as a constant, Equation 3 may be simplified to:

$$pD_e = pD_o - pR - p\tau \tag{4}$$

where D_e is the effective diffusion coefficient (m^2/sec) (*13*). This equation allows for the determination of the relative contributions of D_o, R, and τ to the magnitude of D_e.

The mobility of an element within the monolithic pore space may be compared with its free mobility in water by determining the physical and chemical retardation factors of the product and the element, respectively. To calculate the physical retardation factor, it is necessary to choose an ion that does not chemically interact with the matrix (i.e., $R = 1$). In most studies, sodium is chosen and the physical retardation is calculated with the formula (*13*).

$$\tau = D_{Na}/D_{e,Na} \quad (5)$$

where D_{Na} is the diffusion coefficient of sodium in water (pD_{Na} = 8.88 m²/sec at 25°C) and $D_{e,Na}$ is the effective diffusion coefficient of sodium in the monolith (m²/sec). The chemical retardation factor for the element is determined using the following formula:

$$R = D_x/(D_{e,x}\tau) \quad (6)$$

where D_x is the diffusion coefficient for the element in water (m²/sec) and $D_{e,x}$ is the effective diffusion coefficient for the element in the monolith (m²/sec).

In monolith leach testing, the material is considered homogeneous and is immersed in a leachant that is renewed at regular intervals. The concentration of the element is uniformly distributed and the surface is maintained at a constant concentration. The solution to Equation 1 under such conditions is presented elsewhere (*14*).

$$\frac{C - C_1}{C_o - C_1} = \text{erf}\,\frac{x}{2\sqrt{(D \cdot t)}} \quad (7)$$

where

C = element's concentration in monolithic material as function of place and time,
C_1 = constant concentration at surface of monolith,
C_o = initial concentration of element in material,
D = diffusion coefficient, and
erf = standard error function.

Because the leachant is regularly renewed during the leaching experiment, the surface concentration, C_1, is assumed to be zero (*13*), and the solution to Equation 7 under this assumption is

$$D_e = \frac{\pi \cdot B_t^2}{4t(U_{max} \cdot d)^2} \quad (8)$$

where

B_t = cumulative release of element at time t (mg/m²),
U_{max} = maximum leachable quality of element from monolith (mg/kg), and
d = bulk density of monolithic material (kg/m³).

By plotting the cumulative release of an element leaching from the ash material as a function of time, it can be determined whether matrix diffusion or other leaching mechanisms, such as dissolution or surface wash-off, more common to granular materials, are occurring. The solubility of certain elements within the solid material can be significantly high, such that longer-term dissolution of the element at the surface proceeds faster than diffusion through the pores of the solid matrix. In addition, because of process conditions, a material may be covered with a soluble surface coating that is readily leached with initial leachant contact.

To determine which leaching mechanism is controlling release of the element from the monolith, Equation 8 is rearranged to yield the following (*13*):

$$B_t = U_{max}\, d\sqrt{(4D_e/\pi)}\sqrt{t} \quad (9)$$

After log transformation, Equation 9 becomes

$$\log(B_t) = {}^1/_2 \log(t) + \log\left[U_{max}\, d\sqrt{(4D_e/\pi)}\right] \quad (10)$$

From the monolith leach test results, the release of the element per time interval may be calculated using the formula (*13*)

$$B_i = \frac{c_i V_i}{1{,}000A} \quad (11)$$

where

B_i = release of element per unit area in period i (mg/m²),
c_i = concentration of element in ith period (μg/L),
V_i = volume of leachant (L), and
A = surface area of monolithic material (m²).

The cumulative release of the element for all N periods (N = 8) is calculated from

$$B_{t,i} = B_i \frac{\sqrt{t}}{\sqrt{t_i} - \sqrt{t_{i-1}}} \text{ for } i = 1 \text{ to } N \quad (12)$$

where

$B_{t,i}$ = cumulative release of element for all periods (mg/m²),
t_i = contact time after period i (sec), and
t_{i-1} = contact time after $i-1$ periods (sec) (*13*).

Eight leaching periods are usually used (0.25, 1, 2, 4, 8, 16, 32, and 64 days). This conforms to a time series relationship (renewal time of the nth period is equal to the square of the period sequence number times the renewal time of the first period) that is based on both leaching behavior and on data distribution in log ($B_{t,i}$) − log(t_i) plots.

If the logarithm of the cumulative release, $B_{t,i}$, is plotted versus the logarithm of time, t_i, for the eight periods, the slope of the resulting graph indicates the mechanism controlling the release of the element. Because the slope may change over different time intervals, the slope is examined over the following ranges (*13*):

- Total range—i = 1 to 8,
- Initial range—i = 1 to 3,
- Middle range—i = 3 to 6, and
- Final range—i = 5 to 8.

The relevance of the change in slope at the different intervals is summarized as follows:

	Slope		
Range	*<0.35*	*0.35–0.65*	*>0.65*
Initial	Surface washoff	Diffusion	Lag time/dissolution
Middle	Depletion	Diffusion	Dissolution
Final	Depletion	Diffusion	Dissolution

The effective diffusion coefficient for the element is then calculated from each period for the release per period (B_i) using the data points where the slope is 0.50 ± 0.15 with a deviation of less than 50 percent, and the slope of the final range is smaller than 0.65 by

$$D_{e,i,x} = \frac{\pi B_i^2}{4(U_{max}d)^2 \cdot (\sqrt{t_i} - \sqrt{t_{i-1}})^2} \quad (13)$$

where $D_{e,i,x}$ is the effective diffusion coefficient of element x calculated from the release in the ith period (m^2/sec), and the other terms are as previously described.

MATERIALS AND METHODS

The purpose of the monolith leach test was to evaluate the effects of void content, aging, and cracking on monolith specimens generated from the job mix formula using the NEN 7345 monolith leach test procedure. The monolith leach test examines diffusive leaching mechanisms from monolith specimens. Estimates of monolith tortuosity (τ), and chemical retention (R), via sorption or precipitation of diffusing solutes, were used with estimates of the effective diffusion coefficient (D_e) to model leaching.

A partial $3^3 + 6$ factorial design experiment was conducted on 14 gyratory compacted specimens. The material used to make the specimens came from the actual production run for the full-scale demonstration (50 percent bottom ash, 7 percent asphalt content). Three degrees of voids (low, medium, high), aging (none, medium, high), and cracking (none, medium, high) were integrated into the experimental design. Voids (5, 7.5, 10 percent) were created during the compaction of the specimens using the gyratory testing method (GTM). Voids of 10, 7.5, and 5 percent were produced using 60, 110, and 300 cycles, respectively, on the GTM. Aging was simulated by heat treatment in the presence of forced hot air (107°C for 5 days for moderate aging and 107°C for 10 days for severe aging). The regimen was based on methods compiled by von Quintus et al. (*15*). An Instron machine was used to subject some of the samples to cyclical loading parallel to the direction of compaction to produce micro and macro cracking (i.e., moderate and high cracking levels). A load of 8.22 MPa at 2 cycles/sec was used. Moderate cracking was produced after 1,250 ± 250 cycles. High cracking was produced after 1,750 ± 250 cycles.

Each specimen was placed in a 3-L container filled with contact solution (5 times the specimen volume). Contact solution consisted of Nanopure ASTM Type II (double-deionized) water reduced to pH 4 using Baker Analyzed Ultrex II Ultrapure nitric acid. The leachate was filtered and analyzed periodically (0.25, 1, 2, 4, 8, 16, 32, and 64 days) to determine specified element concentrations leached per indicated time frame. After each filtration, new contact solution was added to the sample. Samples were analyzed using ion chromatography, graphite furnace atomic absorption spectrophotometry, and inductively coupled argon plasma atomic emission spectrometry. Monolith specimens were also ground to less than 300 μm and subjected to the Dutch total availability leaching procedure (NEN 7341) to determine U_{max}, the fraction available for leaching.

RESULTS AND DISCUSSION

Results from preliminary evaluations (*16*) of the leaching behavior of the bottom ash before this study indicated the following general environmental behavior. Neutron activation analyses detected the presence of approximately 45 elements in the bottom ash. Although 45 elements are present in the bottom ash, only 16 consistently leached from bottom ash ground to <300 μm when subjected to the total availability leach test: Cl, Ca, Zn, Cd, Mg, Cu, Mn, Pb, Sr, Si, Fe, Al, Na, K, Ba, and Cr. Lysimeter leaching data for the granular (<1.9 cm) bottom ash lysimeter showed that the bottom ash leached Cl, SO_4^{2-}, Ca, K, Mg, Na, Fe, Mn, Si, and Sr. Similar constituents were leached from a lysimeter containing pavement rubble containing bottom ash, but at much lower levels. Only seven constituents (Na, Cl, SO_4^{2-}, Ca, Si, Mg, and Zn) were routinely leached from the bottom ash test specimens during the monolith leach test.

In this study, monoliths that were ground to less than 300 μm were subjected to the Dutch availability leaching test (NEN 7434). The data, shown in Table 1, indicate that Ca, SO_4, K, Cl, Si, Na, Mg, and Al are the species with the largest potential to be released if the integrity of the monolith were to be severely compromised. Their availabilities range from 7860 to 200 mg/kg. These values are considered extremely low and reflect the role of the bitumen in coating ash particles and reducing surface area available for leaching (*7*). A number of additional species are also available for leaching but at low levels (<200 mg/kg).

In monolithic form, however, leaching behavior is markedly different. The elements Ca, Pb, Mn, Si, Na, Zn, SO_4, Cu, Fe, Mg, and Cl were released from the monoliths in the factorial designed experiment. Only the elements Si, Ca, SO_4, Mn, Cl, Na, and Mg were routinely leached from the monoliths in accordance with diffusional

TABLE 1 Availability of Potentially Diffusing Species in Pavement Monoliths

Element/Specie	Availability mg/kg
Al	221.8
As	<2.02
Ba	20.2
B	<100.8
Cd	<1.01
Ca	7,860
Cr	<2.02
Cu	102.8
Fe	137.1
Pb	98.8
Mg	362.9
Mn	98.8
Hg	<0.60
Mo	<20.2
K	1,008
Se	<2.02
Si	766.2
Na	403.3
Sr	<40.3
Zn	221.8
Cl	806.5
SO_4	3,630
Br	<201

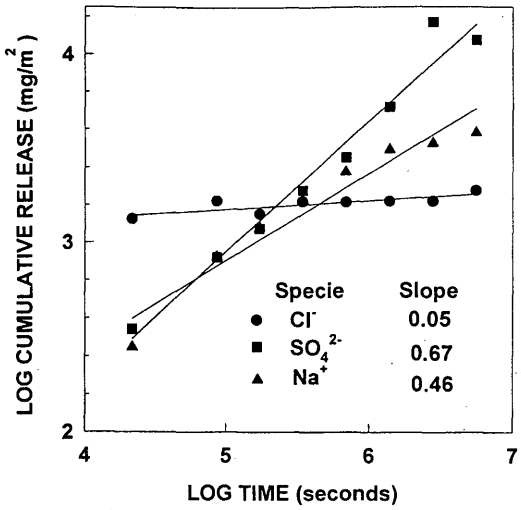

FIGURE 4 Cumulative release plots for Cl, SO_4^{2-}, and Na.

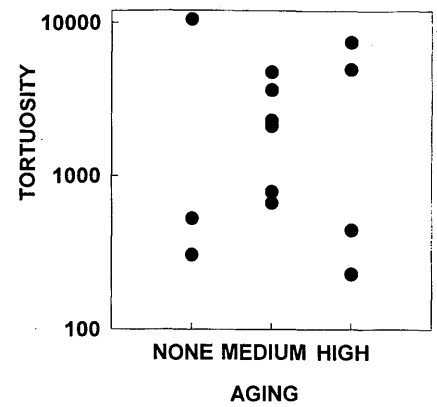

FIGURE 6 Influence of monolith bitumen aging on tortuosity.

processes. The elements Pb, Zn, Cu, and Fe were leached sporadically. Some of the data could be modeled using diffusional modeling. Cr, Al, K, and Sr were seen in only a few of the 112 leachate samples that were analyzed during the experiment. As, Ba, B, Cd, Hg, Mo, and Se were never detected. Some of the elements that exhibited high relative availabilities, such as K, Na, Mg, and Al, were not readily leachable from the monolith. Other elements that exhibited low relative availabilities, such as Mn, were readily leached. These behaviors can be attributed to pH effects and monolith integrity.

Some typical cumulative release plots are shown in Figure 4. The specie Cl shows wash-off behavior; the specie SO_4^{2-} shows dissolution behavior; and the specie Na shows diffusion behavior. These cumulative releases or fluxes are very low and close to that of control specimens containing bitumen and natural aggregates (*7,16,17*).

As shown in Figures 5 and 6, the influence of monolith void content and bitumen aging had little influence on monolith tortuosity and diffusion path length. However, as shown in Figure 7, the presence of micro and macro cracks dramatically reduced tortuosity (τ) values by reducing diffusion path length.

The effective diffusion coefficients that were calculated from the experiment are shown in Figure 8 (*top*). Diffusing species with small ionic radii usually were the most diffusive (D_e values of 10^{-12} m^2/sec or pD_e values of 12). Diffusing species or anions with larger ionic radii were less diffusive (pD_e values of 12 to 16). These values are similar to those reported by de Groot et al. (*11*) for asphalt paving blocks where MSW electrostatic precipitator ash was used as mineral filler and for earlier studies by the authors' group using the same bottom ash but at various substitution levels of 25, 50, and 75 percent (*16,17*). There were no strong relationships between pD_e and the variables tested (voids, aging, and cracking). Some weak relationships may exist given the interdependence of pD_e and tortuosity.

Reactivity (R) values are shown in Figure 8 (*bottom*). The values shown are typical (*11,16,17*) for bituminous asphalt systems governed by hydrophobic surfaces where reaction is less likely. Diffusing species with a small likelihood of sorption or coprecipitation in the matrix exhibit reactivities close to Na. However, species with high sorption or coprecipitation potential, such as Pb, Mn, Zn, and Cu, show high values. There were no strong relationships between R and the experimental variables that were tested.

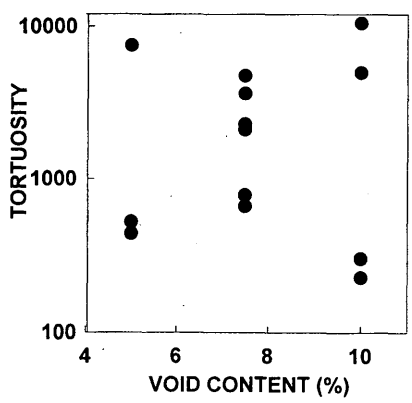

FIGURE 5 Influence of monolith void content on tortuosity.

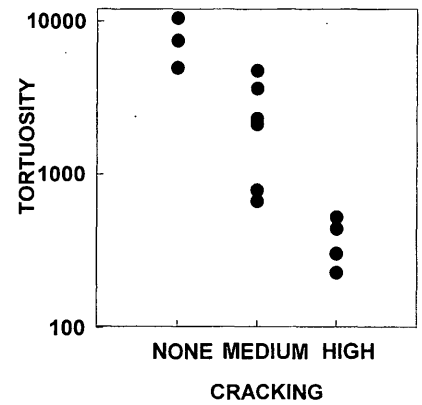

FIGURE 7 Influence of monolith cracking on tortuosity.

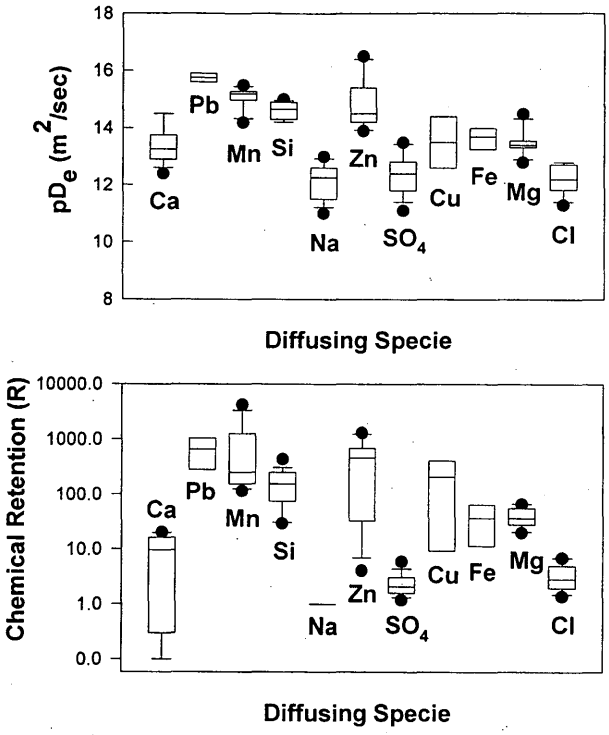

FIGURE 8 Diffusing species for all monoliths versus (*top*) pD_e and (*bottom*) chemical retention.

The reported effective diffusion coefficients are 10^4 to 10^5 times smaller than diffusion coefficients of these ions in water. The dramatic reduction is due to the difficulties associated with ion diffusion in a bituminous polymer matrix where pathways are tortuous and reaction sites on ash particles can interrupt the diffusion process.

The pD_e values depend on both τ and R. Given the large τ values and the relatively low R values, it is apparent that the high degree of tortuosity provided by asphalt accounts for the very low fluxes occurring from such specimens. The maintenance of monolithic integrity is therefore one consideration for keeping fluxes of ash constituents at background levels.

The use of the diffusional modeling approach can be illustrative in estimating the release of constituents from monoliths (*14,18*). The cumulative release of a diffusing constituent from a monolith can be described by (*18*)

$$M_t = 2\rho U_{max}\left(\frac{D_e t}{\pi}\right)^{0.5} \tag{14}$$

where

M_t = cumulative mass of constituent released per unit surface area,
ρ = monolith density,
U_{max} = availability,
D_e = effective diffusion coefficient, and
t = some time period of interest.

This equation assumes that the concentration of the diffusing constituent remains dilute at the surface boundary and that the concentration does not deplete in the monolith. The use of this equation can be further enhanced by correcting D_e for temperature and correcting the cumulative release for the degree of wetting the monolith (*14,19*).

Table 2 illustrates the use of Equation 14 in estimating cumulative release data for Cl for a hypothetical binder course asphalt pavement scenario where the pavement changes its voids content, bitumen polymer aging, and degree of cracking over time. A 10 percent wetting time is assumed to mimic the potential release from the lower surface of a binder course. The 10 percent wetting time is based on an annual average best estimate of percentage of wetting from condensation of moisture vapor from the vadose zone on the lower binder surface; its use is illustrative and would require field verification.

Three relative time frames are used to illustrate the approach: 1.5, 20, and 100 years. The fluxes are shown in Table 2. The largest modeled cumulative flux of 54.9 g Cl/m^2 over a 100-year time frame is similar to the flux associated with road salting during a single snow storm in New Hampshire (20 g Cl/m^2) and less than that applied over a single season (500 g Cl/m^2). These calculations are to be used for simple order of magnitude comparisons only. In fact, the estimated flux from the lower road surface interface is very conservative. Chloride, a nonreactive and mobile anion, would still have to move via diffusive processes through capillary water and

TABLE 2 Modeled Flux of Chloride from Pavement Monoliths as Function of Monolith Integrity and Time

Scenario	Flux, g Cl/m^2		
	Years[a]		
	1.5	20	100
New Pavement[b] (Voids = 10%, Cracking = L, Aging = L)	1.7	6.4	14.3
Moderately Aged Pavement[b] (Voids = 7.5%, Cracking = M, Aging = M)	4.1	14.9	33.5
Aged Pavement[b] (Voids = 5%, Cracking = H, Aging = H)	6.7	24.5	54.9

[a] Based on an estimated 10% wetting time from condensation of vadose zone moisture vapor on the lower surface of the binder course.
[b] Based on conditions used in the monolith leach test.

condensate ephemerally present on soil particle surfaces a very large distance to the groundwater table via a tortuous, noncontiguous pathway. This diffusive process would be driven by a weak driving gradient. Given the preexisting chloride levels in RAP, roadside soils, and groundwater, this gradient could also be nonexistent. Nevertheless, the approach can allow order of magnitude comparisons of waste product leaching to background signals.

CONCLUSIONS

Monolith cracking was the only experimental variable influencing diffusional leaching behavior. Increased cracking decreased diffusional path lengths whereby release occurs. Void content and asphalt aging had little influence. The observed effective diffusion coefficients for those species exhibiting diffusional release are typical for diffusion in polymer systems. The results suggest that MSW bottom ash constituent leaching behavior is controlled by the tortuous hydrophobic nature of the bituminous polymer system in the pavement.

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