

# Permeability and Leaching Characteristics of Fly Ash Liner Materials

DANIEL N. CREEK AND CHARLES D. SHACKELFORD

Rigid-wall column tests are performed at a hydraulic gradient of 100 on 21 compacted mixtures containing a Class F fly ash, sand, bentonite, and cement. Results indicate that combinations of the admixtures can lower the permeability of the fly ash by up to an order of magnitude. There appears to be an optimum percentage of fly ash that minimizes the permeability of fly ash-sand mixtures. Specimens from four mixtures, as well as 100 percent fly ash, are tested at four hydraulic gradients to establish different flow rates. Tap water is used as the permeant fluid, and the effluent is sampled every quarter pore volume of flow and tested for 13 metal concentrations, pH, and electrical conductivity. For most of the metals, most of the metal mass is leached within the first pore volume of flow. However, some metals (Ba, Ca, and Sr) are not leached in significant quantities until two or three pore volumes of flow have passed through the test specimens. These two leaching behaviors suggest that there is dissolution competition between metal ions. No simple trend in leaching rate as a function of flow rate was observed for all mixtures and metals. However, for specific elements between different mixtures at approximately the same flow rate, an increase in the fly ash content tended to increase the leaching rate of most metals. A 2.5 percent cement content was more effective than a 10 percent bentonite content in reducing the leaching rate of metals for mixtures containing the same amount (75 percent) of fly ash.

Fly ash is the finer portion of the noncombustible residue produced during the operation of coal-fired boiler units. It is composed primarily of fine-grained particles ( $< 0.075$  mm in diameter), most of which are glassy spheres, with some crystalline matter, carbon, and scoria. The glassy spheres are relatively inert and immune to dissolution due to their elemental composition and glasslike structure (1). However, on the surface of the spheres, there exist molecules of trace metals that can dissolve in the presence of liquid (1). The potential for leaching of these metals causes concern regarding the environmental consequences of using fly ash.

Despite the self-hardening characteristics that allow fly ash to be used as a soil stabilizer and as an additive in portland cement, only 25 percent of the fly ash produced in the United States currently is used for productive purposes (2), whereas the rest is being disposed of in landfills and surface impoundments. The safe use of fly ash has potential for great economic benefits.

Recently, several studies have investigated the application of fly ash in waste containment liner systems (3-6). Perme-

ability and leaching tests have been performed on mixtures containing fly ash. The studies have shown that permeabilities on the order of  $10^{-7}$  cm/sec can be obtained for stabilized fly ash, though no definite trends of metal leaching were found. None of the previous studies considered the effect of the leaching fluid flow rate on the leaching of metal species from mixtures containing fly ash. The flow rate, which is dependent on field conditions or laboratory procedures, may affect metal leaching. Investigation of the characteristics of metal leaching from fly ash is needed to prove its potential as a liner material for waste containment.

The objectives of this study are to (a) determine if flow rate affects the leaching of heavy metals from fly ash and stabilized fly ash; (b) investigate the effects of sand, bentonite, and cement admixtures on the permeability of fly ash; and (c) determine the effects of these admixtures on the leachability of metals associated with fly ash.

## MATERIALS AND METHODS

### Material Mixtures

For this study, 21 combinations of various percentages of fly ash, sand, bentonite, and cement were chosen. The mixes were grouped into Stage 1 (pure fly ash), Stage 2 (fly ash with sand), Stages 3A and 3B (fly ash with sand and bentonite), and Stage 4 (fly ash with sand and cement). The percentages, by dry weight, of each of the fly ash-soil-cement mixes are given in Table 1.

### Properties of Materials

#### Admixture Materials

Results of specific gravity (ASTM D854), grain size (ASTM D421, D422), and Atterberg limits (ASTM 4318) tests were performed on each of the materials and are presented in Table 2. Also given in Table 2 are results of tests to determine the cation exchange capacity (CEC) of the bentonite and cement. The measurable CEC for cement probably is due to the pozzolanic nature of cement.

The fly ash used in this study was a well-graded Class F pozzolan with the majority of its particles in the silt-size range (0.002 to 0.075 mm). About 28 percent of the fly ash is retained on the No. 325 (0.045-mm) sieve; hence, it is a fine-grained material.

D. N. Creek, Woodward-Clyde Consultants, Stanford Place 3, Suite 1000, 4582 S. Ulster St. Pkwy., Denver, Colo. 80237. C. D. Shackelford, Department of Civil Engineering, Colorado State University, Fort Collins, Colo. 80523.

TABLE 1 FLY ASH-SOIL-CEMENT MIXTURES USED IN TESTING PROGRAM

| Stage | Mix # | Percentage of Constituent (by dry weight) |      |           |        |
|-------|-------|---|------|-----------|--------|
|       |       | Fly Ash                                   | Sand | Bentonite | Cement |
| 1     | 1     | 100                                       | 0    | 0         | 0      |
| 2     | 2     | 75  | 25   | 0         | 0      |
| 2     | 3     | 50  | 50   | 0         | 0      |
| 2     | 4     | 25  | 75   | 0         | 0      |
| 2     | 5     | 15  | 85   | 0         | 0      |
| 2     | 6     | 5   | 95   | 0         | 0      |
| 3A    | 7     | 5   | 85.5 | 9.5       | 0      |
| 3A    | 8     | 15  | 76.5 | 8.5       | 0      |
| 3A    | 9     | 25  | 67.5 | 7.5       | 0      |
| 3A    | 10    | 50  | 45   | 5         | 0      |
| 3A    | 11    | 75  | 22.5 | 2.5       | 0      |
| 3B    | 12    | 5   | 85   | 10        | 0      |
| 3B    | 13    | 15  | 75   | 10        | 0      |
| 3B    | 14    | 25  | 65   | 10        | 0      |
| 3B    | 15    | 50  | 40   | 10        | 0      |
| 3B    | 16    | 75  | 15   | 10        | 0      |
| 4     | 17    | 5   | 85.5 | 0         | 9.5    |
| 4     | 18    | 15  | 76.5 | 0         | 8.5    |
| 4     | 19    | 25  | 67.5 | 0         | 7.5    |
| 4     | 20    | 50  | 45   | 0         | 5      |
| 4     | 21    | 75  | 22.5 | 0         | 2.5    |

TABLE 2 PROPERTIES OF MATERIALS USED IN TESTING PROGRAM

| Property                          | Fly Ash | Sand | Bentonite | Cement |
|-----------------------------------|---------|------|-----------|--------|
| G <sub>s</sub> (ASTM D854)        | 2.28    | 2.65 | 2.60      | 3.25   |
| USCS Classification (ASTM D2487)  | ML      | SP   | CH        | —      |
| Liquid Limit (%) (ASTM D4318)     | NP      | NP   | 465       | NP     |
| Plasticity Index (%) (ASTM D4318) | NP      | NP   | 425       | NP     |
| CEC (meq/100g)                    | —       | 0    | 76        | 6      |
| % Passing #200                    | 89.0    | 0    | 100       | 100    |

NP - non-plastic

| Property of Nixon Fly Ash *                        | Value |
|--|-------|
| Percent Retained on #325 (Fineness)(%)             | 28.2  |
| Silicon Dioxide, SiO <sub>2</sub> (%)              | 57.0  |
| Aluminum Oxide, Al <sub>2</sub> O <sub>3</sub> (%) | 27.1  |
| Iron Oxide, Fe <sub>2</sub> O <sub>3</sub> (%)     | 5.3   |
| Calcium Oxide, CaO (%)                             | 6.4   |
| Loss on Ignition (%)                               | 0.86  |
| <b>Pozzolanic Activity Index (see ASTM C311)</b>   |       |
| Portland Cement at 7 days % of Control             | 71    |
| Portland Cement at 28 days % of Control            | 93    |
| Lime at 7 days (psi)                               | 1004  |

\* Testing by Resource Materials Testing, Inc., of Lakewood, CO

A clean, medium sand identified as 20-40 silica sand was used. It is a poorly graded sand with approximately 95 percent of the particles between the No. 20 (0.085-mm) and the No. 40 (0.425-mm) sieve sizes.

Ecco Gel, the bentonite, purchased from Eisenman Chemical Co., Greeley, Colorado, was warranted to contain at least 81.5 percent sodium bentonite and 4.5 percent free silica.

The cement was a low alkali portland cement purchased from Ideal Cement Co., Denver, Colorado. The cement was a fine-grained material with 100 percent of its particles finer than 0.075 mm.

### Permeant Fluid

Tap water was used as the permeant fluid. Background concentrations of metals in the tap water were determined using inductively coupled plasma optical emissions spectrometry (ICP-OES) (7). This method also was used, in conjunction with extraction procedures, to determine background metal concentrations in the soils, cement, and fly ash. Average metal concentrations of nine water samples were below detection limits or drinking water standards (DWSs) (8) for all metals except Pb. The average background concentration of Pb was 0.07 mg/L, whereas the DWS is 0.05 mg/L. The electrical conductivity of the tap water was 27.9  $\mu$ mhos/cm at 25°C, and the pH was 6.3 at 25°C.

### Selection of Metal Species for Tracing During Column Tests

On the basis of values of leaching potential for 21 metal species, 13 were chosen to be traced during the column tests either because of a high background concentration in the fly ash or DWS (9) consideration. The traced metals are Al, B, Ba, Ca, Cd, Cr, Cu, Fe, Mn, Mo, Pb, Sr, and Zn.

Results from two extraction procedures on each of the materials, using tap water and ammonium bicarbonate diethylenetriaminepentaacetic acid (AB-DTPA), were analyzed to determine the approximate metal concentrations for mass balance analyses.

Metal quantities available for leaching were calculated for the mass balance analyses used during the flow-rate testing phase. Available metal masses were calculated for each admixture constituent. Because neither AB-DTPA extracts nor tap water extracts consistently resulted in the highest concentrations, the maximum concentration value for each metal from either of the two procedures was used in the mass balance analyses. Since tap water was used as the permeant fluid, the percentage of the maximum available mass of a given metal species leached in three pore volumes of flow may be somewhat low.

### Methods

The following is a summary of the methods and equipment used in the testing program. Details are given elsewhere (10).

### Proctor Tests

Five-point Proctor curves were developed using a Soiltest Mechanical Compactor (Model CN-4230). The constituents were mixed by hand until the blend was homogeneous. Tap water was added and mixed before compaction. Virgin fly

ash, cement, and soil were used for each separate Proctor point to ensure that particle deformation due to compaction did not affect densities.

### Permeability and Flow-Rate Tests

The test equipment (Figure 1) uses a pair of plexiglass accumulators on either side of a soil column. The influent liquid is pressurized in the inflow accumulator by means of compressed air acting on an air-water interface. The water flows through the soil column to the outflow accumulator, which is vented to atmospheric pressure. The outflow head stays constant because of the elevated exit tube contained within the outflow accumulator. The inflow head drops as the liquid infiltrates into the soil cell. The test procedure actually models a falling-head situation. However, considering the maximum drop in tap water height and the air pressure applied to the water surface, the gradient changed by less than 2.5 percent during the preliminary permeability tests and by a maximum of 4.2 percent during the lowest gradient flow-rate tests. This was the basis for the constant gradient assumption.

The coefficient of permeability was calculated for each measurement interval and for the cumulative outflow by using Darcy's law:

$$k = \frac{Q}{iA} \quad (1)$$

where  $k$  is the hydraulic conductivity or coefficient of permeability,  $Q$  is the volumetric flow rate,  $i$  is the hydraulic gradient, and  $A$  is the cross-sectional area of the sample. Stainless steel, standard Proctor compaction molds were used as the soil columns. For the flow-rate tests, dry unit weights were

allowed to vary by 1.5 percent within each stage, and the water contents were required to be within 1 percent water content of optimum. The compacted samples were cured at room temperature in double ziploc plastic bags. Time for curing was 1 day for the preliminary tests, but 7 days for the flow-rate tests. Laboratory temperature varied between 20°C and 27°C. All permeability tests used the same tap water for the permeant fluid as was used in the compaction of the samples. Leachate samples were collected every quarter pore volume of flow during the flow-rate tests. For the flow-rate tests, three pore volumes of effluent, based on the total, initial porosity of the mixture, were collected before the test was stopped. All of the preliminary tests were conducted at a gradient of about 100, whereas the flow-rate tests were performed at gradients of approximately 25, 50, 100, and 150.

## RESULTS AND DISCUSSION

### Proctor Tests

The moisture-density curves resulting from Proctor tests on the mixtures containing fly ash are similar to those for typical soils. The maximum dry unit weights ranged from 86.5 pcf (100 percent fly ash) to 111.0 pcf (25 percent fly ash, 7.5 percent cement, and 67.5 percent sand). Optimum water content of the mixtures ranged from 9.0 to 19.3 percent.

### Preliminary Column Tests

The preliminary column tests on the 21 mixtures resulted in steady-state permeabilities ranging from  $1.4 \times 10^{-4}$  to  $1.0 \times 10^{-7}$  cm/sec. Water contents, degrees of saturation, mixture

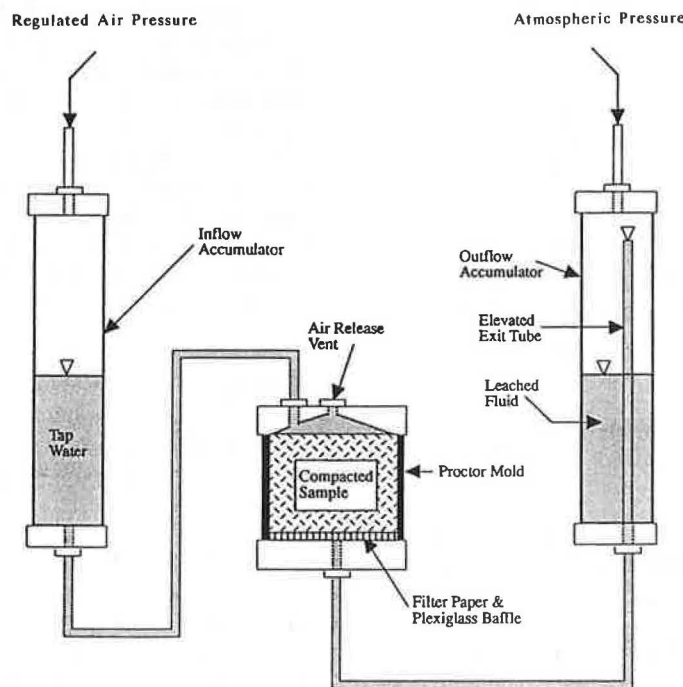


FIGURE 1 Schematic of column testing unit.

porosities, dry unit weights, and steady-state permeabilities are summarized in Table 3. The permeabilities obtained correspond well with those reported in the literature (3,5).

For all but one of the mixtures, the permeability increased slightly before leveling off at a steady-state value. In the case of 100 percent fly ash (Mix 1), the permeability increased slightly, then dropped significantly before reaching a steady-state value of  $1.0 \times 10^{-7}$  cm/sec. This permeability value is contradicted by results of the 100 percent fly ash flow-rate tests (reported later), in which permeabilities ranged from  $1.2 \times 10^{-6}$  to  $2.4 \times 10^{-6}$  cm/sec.

For all stages except Stage 3B, there appeared to be an optimum percentage of fines that minimized the permeability and maximized the dry unit weight of a given mixture of materials. An increase in fines over the optimum percentage resulted in a higher permeability and a lower maximum dry unit weight. For the Stage 3B specimens, the permeability continued to decrease with increasing percentage of fines.

### Flow-Rate Column Tests

The criteria for choice of mixtures used in the flow-rate testing phase were permeability and percent fly ash. Mixes 3, 10, and 16, from Stages 2, 3A, and 3B, respectively, were chosen because their permeabilities were substantially lower than the permeabilities of the other mixtures in the respective stages (see Table 3). Mix 21, from Stage 4, was chosen because it consisted of a higher percentage of fly ash than Mix 20, which had approximately the same permeability. Mix 1, the 100 percent fly ash mixture, also was chosen.

Column tests were performed on test specimens prepared from the five chosen mixtures to investigate the effect of flow rate on the leachability of metals from mixtures containing fly ash. Each of the five mixtures was tested at four different hydraulic gradients, producing four flow rates (i.e., fluid flow flux). Although the applied gradient was kept constant, the permeability varied during each test, resulting in slight variations in the flow rate.

### Mixture Permeabilities

As indicated in Table 4, the steady-state permeability values of the mixtures varied from  $1.5 \times 10^{-7}$  to  $2.4 \times 10^{-6}$  cm/sec. Like previous studies (3,5), this study indicated that bentonite is the most effective of the three admixture materials in reducing the permeability value for the fly ash mixtures. The permeability values between preliminary and flow-rate tests varied by no more than a factor of about two for all of the mixtures except 100 percent fly ash.

### DWSs

The average concentrations for the metal species in the DWSs (9) based on three pore volumes of flow are plotted versus mean flow rate in Figure 2. For the 100 percent fly ash specimens (Figure 2a) and the 50 percent fly ash/50 percent sand specimens, the average concentrations are greater than the DWS for B, Ba, Cd, Cr, and Pb, regardless of the flow rate. Except for Ba, the same trend is evident for the two fly ash/sand/bentonite mixtures (Figures 2b and 2c). Ba apparently is preferentially adsorbed by the bentonite.

For the mixture containing cement (Figure 2d), Ba was the only metal species that exceeded its DWS for all four flow rates. Five other metals (B, Cr, Fe, Mn, and Pb) were leached in sufficient quantities to exceed the DWS level for at least one flow rate, typically the lowest or the highest flow rate, or both.

### Leaching Behavior

In the flow-rate tests, two distinct shapes for the elution curves, or leaching behaviors, are apparent for the majority of the metal species traced. "Early" leaching behavior is characterized by concentrations that initially are high, peak before one pore volume of flow, then decrease, sometimes leveling off, for the remainder of the three pore volumes of flow. "De-

TABLE 3 DATA FROM PRELIMINARY COLUMN TESTS

| Stage | Mix # | Fly Ash (%) | Fines <sup>a</sup> | G <sub>s</sub> | γ <sub>unit</sub> (pcf) | n     | w <sub>unit</sub> (%) | w <sub>water</sub> (%) | S <sub>sat</sub> (%) | S <sub>unit</sub> (%) | θ <sub>unit</sub> (%) | θ <sub>sat</sub> (%) | Permeability (cm/sec) |
|-------|-------|-------------|--------------------|----------------|-------------------------|-------|-----------------------|------------------------|----------------------|-----------------------|-----------------------|----------------------|-----------------------|
| 1     | 1     | 100         | 100                | 2.28           | 87.6                    | 0.384 | 14.1                  | 19.2                   | 52                   | 70                    | 20                    | 27                   | $1.0 \times 10^{-7}$  |
| 2     | 2     | 75          | 75                 | 2.36           | 95.4                    | 0.352 | 11.4                  | 15.6                   | 50                   | 68                    | 18                    | 24                   | $2.0 \times 10^{-4}$  |
|       | 3     | 50          | 50                 | 2.45           | 102.9                   | 0.327 | 7.6                   | 13.3                   | 38                   | 67                    | 12                    | 22                   | $6.4 \times 10^{-7}$  |
|       | 4     | 25          | 25                 | 2.55           | 104.1                   | 0.346 | 12.1                  | 13.6                   | 58                   | 66                    | 20                    | 23                   | $7.5 \times 10^{-4}$  |
|       | 5     | 15          | 15                 | 2.59           | 97.1                    | 0.400 | 15.0                  | 16.4                   | 58                   | 64                    | 23                    | 26                   | $7.5 \times 10^{-5}$  |
|       | 6     | 5           | 5                  | 2.63           | 88.0                    | 0.464 | 19.5                  | 21.6                   | 59                   | 65                    | 27                    | 30                   | $6.4 \times 10^{-4}$  |
| 3A    | 7     | 5           | 14.5               | 2.63           | 93.6                    | 0.430 | 16.3                  | 20.7                   | 57                   | 72                    | 25                    | 31                   | $2.1 \times 10^{-4}$  |
|       | 8     | 15          | 23.5               | 2.59           | 99.6                    | 0.384 | 13.4                  | 16.6                   | 56                   | 69                    | 22                    | 26                   | $2.0 \times 10^{-4}$  |
|       | 9     | 25          | 32.5               | 2.55           | 103.4                   | 0.350 | 13.2                  | 14.5                   | 63                   | 69                    | 22                    | 24                   | $2.7 \times 10^{-5}$  |
|       | 10    | 50          | 55                 | 2.45           | 102.4                   | 0.330 | 11.1                  | 14.2                   | 55                   | 70                    | 18                    | 23                   | $4.5 \times 10^{-7}$  |
|       | 11    | 75          | 77.5               | 2.36           | 93.3                    | 0.366 | 12.8                  | 16.8                   | 52                   | 69                    | 19                    | 25                   | $1.1 \times 10^{-4}$  |
| 3B    | 12    | 5           | 15                 | 2.63           | 97.4                    | 0.406 | 15.7                  | 18.9                   | 60                   | 73                    | 24                    | 30                   | $7.5 \times 10^{-5}$  |
|       | 13    | 15          | 25                 | 2.59           | 96.7                    | 0.402 | 15.1                  | 17.7                   | 58                   | 68                    | 23                    | 27                   | $1.4 \times 10^{-4}$  |
|       | 14    | 25          | 35                 | 2.55           | 98.4                    | 0.382 | 15.8                  | 16.7                   | 65                   | 69                    | 25                    | 26                   | $8.2 \times 10^{-4}$  |
|       | 15    | 50          | 60                 | 2.46           | 97.2                    | 0.367 | 12.7                  | NA                     | 54                   | NA                    | 20                    | NA                   | $1.7 \times 10^{-4}$  |
|       | 16    | 75          | 85                 | 2.37           | 90.0                    | 0.391 | 15.9                  | NA                     | 59                   | NA                    | 23                    | NA                   | $5.2 \times 10^{-7}$  |
| 4     | 17    | 5           | 14.5               | 2.67           | 100.7                   | 0.396 | 14.0                  | 13.8                   | 57                   | 56                    | 26                    | 22                   | $4.8 \times 10^{-5}$  |
|       | 18    | 15          | 23.5               | 2.62           | 107.8                   | 0.341 | 10.8                  | NA                     | 55                   | NA                    | 19                    | NA                   | $9.2 \times 10^{-4}$  |
|       | 19    | 25          | 32.5               | 2.58           | 109.0                   | 0.323 | 8.9                   | 9.8                    | 48                   | 53                    | 16                    | 17                   | $4.0 \times 10^{-4}$  |
|       | 20    | 50          | 55                 | 2.47           | 103.2                   | 0.330 | 10.3                  | 11.8                   | 52                   | 59                    | 17                    | 19                   | $4.4 \times 10^{-7}$  |
|       | 21    | 75          | 77.5               | 2.36           | 95.2                    | 0.354 | 13.7                  | 13.9                   | 59                   | 60                    | 21                    | 21                   | $4.5 \times 10^{-7}$  |

<sup>a</sup>Fines include fly ash, bentonite and/or cement. NA - data not available. Symbols: γ-unit weight; w-water content; S-degree of saturation; θ - volumetric water content; n - porosity; G<sub>s</sub> - specific gravity of solids of mixture.

TABLE 4 FLOW-RATE TEST DATA

| Mixture                               | Hydraulic Gradient | $\gamma_{sat}$ (pcf) | n     | $W_{sat}$ (%) | $W_{final}$ (%) | $Sr_{sat}$ (%) | $Sr_{final}$ (%) | $\theta_{sat}$ (%) | $\theta_{final}$ (%) | Permeability (cm/sec) |
|---------------------------------------|--------------------|----------------------|-------|---------------|-----------------|----------------|------------------|--------------------|----------------------|-----------------------|
| Mix #1<br>100% FA                     | 24                 | 84.5                 | 0.406 | 14.8          | 19.9            | 49.4           | 66.4             | 20.1               | 27.0                 | $1.2 \times 10^{-4}$  |
|                                       | 51                 | 85.3                 | 0.400 | 14.9          | NA              | 51.0           | NA               | 20.4               | NA                   | $1.8 \times 10^{-4}$  |
|                                       | 103                | 85.3                 | 0.400 | 14.7          | 17.0            | 50.3           | 58.1             | 20.1               | 23.2                 | $2.4 \times 10^{-4}$  |
|                                       | 154                | 85.6                 | 0.392 | 15.2          | 18.3            | 53.8           | 64.1             | 21.1               | 25.4                 | $2.1 \times 10^{-4}$  |
| Mix #3<br>50% FA, 50% S               | 24                 | 105.0                | 0.313 | 10.1          | 10.9            | 54.3           | 58.6             | 17.0               | 18.3                 | $6.5 \times 10^{-7}$  |
|                                       | 51                 | 104.6                | 0.316 | 10.0          | 11.2            | 53.3           | 59.7             | 16.8               | 18.9                 | $7.7 \times 10^{-7}$  |
|                                       | 103                | 104.1                | 0.318 | 10.1          | 11.2            | 53.1           | 58.9             | 16.8               | 18.7                 | $1.8 \times 10^{-6}$  |
|                                       | 154                | 104.3                | 0.318 | 9.9           | 11.8            | 52.0           | 62.0             | 16.5               | 19.7                 | $1.6 \times 10^{-6}$  |
| Mix #10<br>50% FA, 45% S,<br>5% B     | 24                 | 102.0                | 0.333 | 11.3          | 13.0            | 55.5           | 63.8             | 18.5               | 21.2                 | $1.5 \times 10^{-7}$  |
|                                       | 51                 | 102.1                | 0.332 | 11.2          | 13.5            | 55.2           | 66.6             | 18.3               | 22.1                 | $2.3 \times 10^{-7}$  |
|                                       | 103                | 102.2                | 0.332 | 11.2          | 13.1            | 55.2           | 64.6             | 18.3               | 21.4                 | $2.7 \times 10^{-7}$  |
|                                       | 154                | 101.3                | 0.338 | 11.7          | 14.0            | 56.1           | 67.2             | 19.0               | 22.7                 | $3.4 \times 10^{-7}$  |
| Mix #16<br>75% FA, 15% S,<br>10% B    | 24                 | 99.4                 | 0.328 | 16.0          | 19.1            | 77.7           | 92.7             | 25.5               | 30.4                 | $2.3 \times 10^{-7}$  |
|                                       | 52                 | 99.2                 | 0.329 | 16.5          | 19.3            | 79.8           | 93.3             | 26.3               | 30.7                 | $3.1 \times 10^{-7}$  |
|                                       | 103                | 99.8                 | 0.325 | 16.2          | 19.0            | 79.7           | 93.5             | 25.9               | 30.4                 | $3.3 \times 10^{-7}$  |
|                                       | 155                | 99.8                 | 0.325 | 16.2          | 17.3            | 79.7           | 85.2             | 25.9               | 27.7                 | $3.1 \times 10^{-7}$  |
| Mix #21<br>75% FA, 22.5% S,<br>2.5% C | 24                 | 103.9                | 0.295 | 13.3          | 15.2            | 75.0           | 85.7             | 22.1               | 25.3                 | $4.4 \times 10^{-7}$  |
|                                       | 52                 | 104.5                | 0.291 | 13.3          | 14.7            | 76.5           | 84.5             | 22.3               | 24.6                 | $6.2 \times 10^{-7}$  |
|                                       | 103                | 104.8                | 0.289 | 13.3          | 14.4            | 77.2           | 83.6             | 22.3               | 24.2                 | $9.3 \times 10^{-7}$  |
|                                       | 155                | 104.8                | 0.288 | 13.3          | 15.5            | 77.6           | 90.7             | 22.3               | 26.1                 | $8.9 \times 10^{-7}$  |

NA - data not available. Symbols:  $\gamma$  - unit weight; n - porosity; w - water content; Sr - degree of saturation;  $\theta$  - volumetric water content; FA - fly ash; S - sand; B - bentonite; C - cement.

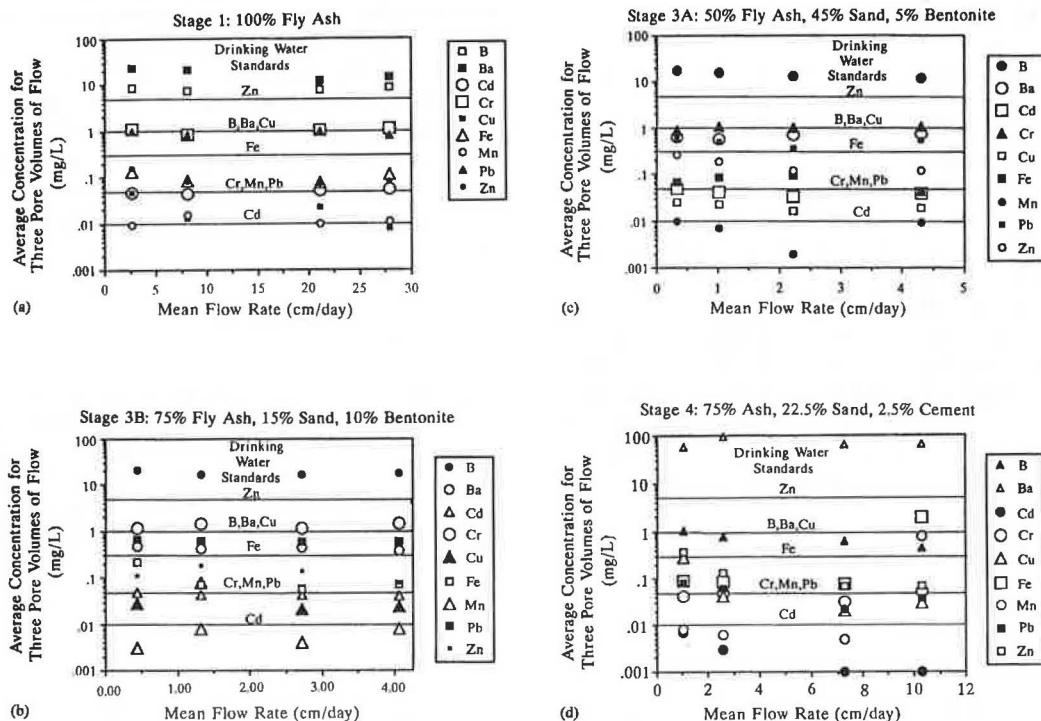


FIGURE 2 Average metal concentrations versus mean flow rate.

layed" leaching behavior is characterized by concentrations that start at relatively low values, increase, then either level off or peak before decreasing. In some cases, the metal concentrations were still increasing at three pore volumes of flow. Figure 3 shows typical early and delayed leaching elution curves found in this study.

Of the 13 metals traced, only Ba, Ca, and Sr show delayed leaching behavior. These three metals appear in the second column of the periodic table and are divalent ions in their most stable oxidation states. Comparisons of the relative charge densities of the metal ions monitored in this study indicate that  $Ba^{2+}$ ,  $Ca^{2+}$ , and  $Sr^{2+}$  possess the three smallest charge

densities. The consistent relationship between the two types of leaching behaviors with respect to the charge densities of the metal ions suggests that the equilibrium chemistry of the leaching fluid may play a significant role in the rate at which metal ions dissolve into solution (i.e., it appears that the ions with higher charge densities dissolve into solution first). After the more competitive ions are leached away from the particle surfaces, the ions with lower charge densities dissolve into solution.

Although early leaching behavior has previously been recognized (3), no mention has been made of delayed leaching behavior for similar tests, although similar behavior is seen



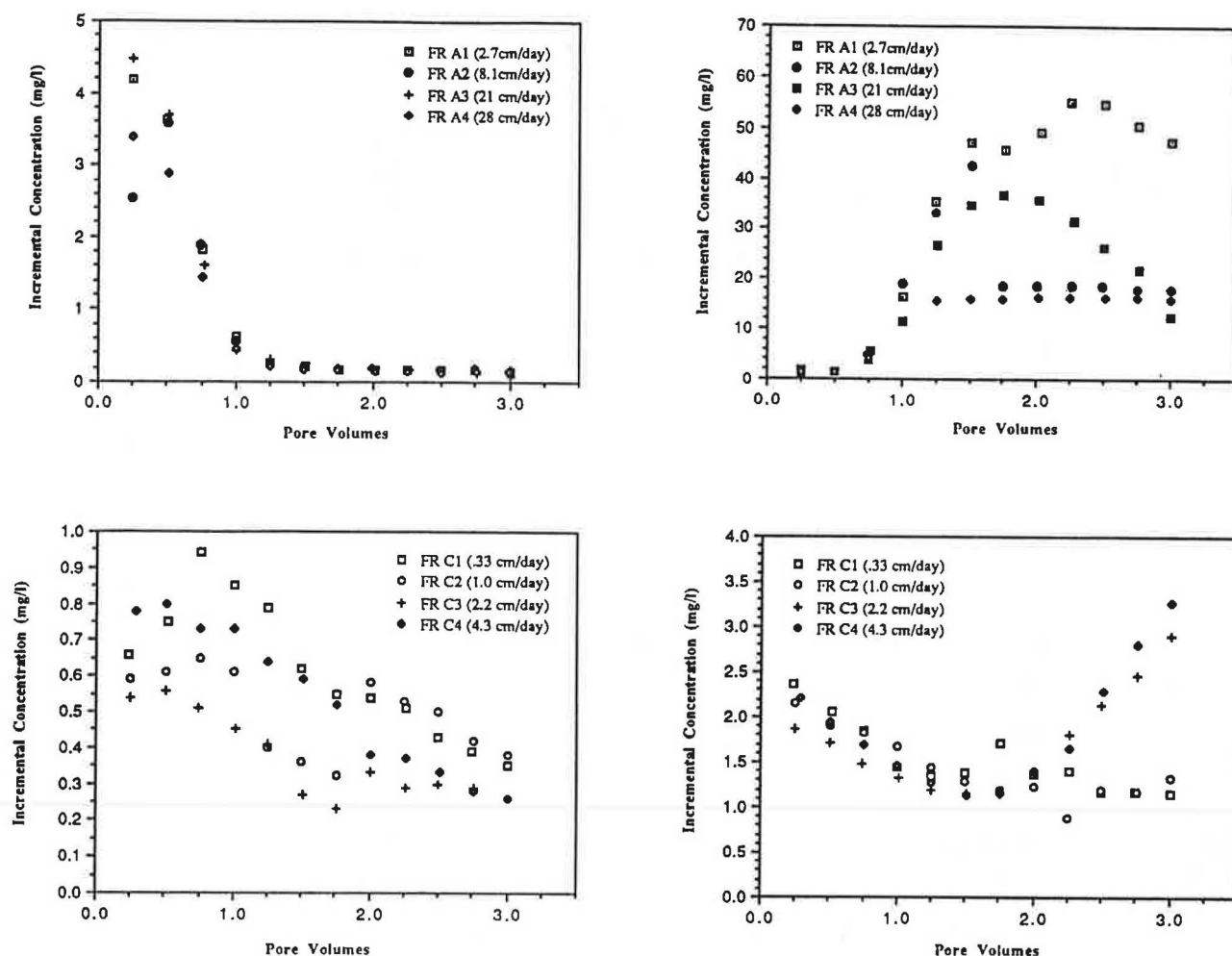


FIGURE 3 Typical early and delayed leaching curves.

for Ca in the leaching curves of Edil et al. (5). Nonetheless, it appears from the results of this study that the equilibrium chemistry of the leaching fluid may play a significant role in the leaching of metals from fly ash particle surfaces. However, additional study is needed to verify this trend.

#### Percent Mass Leached

The percentages of the mass of each metal leached per mass available for five test specimens (one from each of the five different sample mixtures) are provided in Table 5. The test specimens were chosen such that the flow rates for the test specimens are approximately the same (i.e., relative to the range of flow rates in the flow-rate tests). Overall,  $\leq 0.1$  percent of Fe and Mn was leached within three pore volumes of flow regardless of the mixture constituency. The 317 percent reported for Ba in Test FR E2 and the values exceeding 100 percent for Mo in Tests FR C3 and FR D3 are probably due to insufficient extractions procedures.

The addition of bentonite (5 or 10 percent) resulted in significant reductions in the leached masses of Ba, Ca, and Sr relative to the 100 percent fly ash or the 50 percent fly ash/

50 percent sand specimens. For the same comparison, reductions in the leached amounts of Al, Cd, Cu, Pb, and Zn were less significant, and a slightly adverse effect was noticed for the leached masses of B, Cr, and Mo.

The reductions in the leached masses of Ba, Ca, and Sr apparently represent a retardation effect wherein there is a delay in the leaching of these three metals due to a greater adsorption affinity for the exchange sites of the bentonite. A greater adsorption affinity of Ba, Ca, and Sr would indicate that the charge densities of these three metals are greater than the charge densities of the other competing ions, contradicting the hypothesis that the delayed leaching behavior of these three metals in all mixtures is due to smaller charge densities. The contradiction may be related to the difference between the charge densities of unhydrated versus hydrated chemical species (i.e., a smaller charge density for an unhydrated chemical species may result in a greater charge density for the same chemical species when hydrated, and vice versa). Therefore, smaller charge densities for unhydrated  $\text{Ba}^{2+}$ ,  $\text{Ca}^{2+}$ , and  $\text{Sr}^{2+}$  may result in greater charge densities for hydrated  $\text{Ba}^{2+}$ ,  $\text{Ca}^{2+}$ , and  $\text{Sr}^{2+}$ . Once dissolved, greater hydrated charge densities for  $\text{Ba}^{2+}$ ,  $\text{Ca}^{2+}$ , and  $\text{Sr}^{2+}$  would result in relatively more competitive adsorption reactions for these three species rel-

TABLE 5 PERCENTAGE MASS LEACHED AND CENTROIDAL PORE VOLUME VERSUS MIXTURE

| STAGE                 | STAGE 1                       |                              | STAGE 2                       |                              | STAGE 3A                      |                              | STAGE 3B                      |                              | STAGE 4                       |                              |
|-----------------------|-------------------------------|------------------------------|-------------------------------|------------------------------|-------------------------------|------------------------------|-------------------------------|------------------------------|-------------------------------|------------------------------|
| MIXTURE               | 100% FA                       |                              | 50% FA,<br>50% S              |                              | 50% FA,<br>45% S,<br>5% B     |                              | 75% FA,<br>15% S,<br>10% B    |                              | 75% FA,<br>22.5% S,<br>2.5% C |                              |
| Flow rate<br>(cm/day) | FR A1<br>(2.73)               |                              | FR B2<br>(3.41)               |                              | FR C3<br>(2.22)               |                              | FR D3<br>(2.73)               |                              | FR E2<br>(2.59)               |                              |
|                       | Percentage<br>Mass<br>Leached | Centroidal<br>Pore<br>Volume | Percentage<br>Mass<br>Leached | Centroidal<br>Pore<br>Volume | Percentage<br>Mass<br>Leached | Centroidal<br>Pore<br>Volume | Percentage<br>Mass<br>Leached | Centroidal<br>Pore<br>Volume | Percentage<br>Mass<br>Leached | Centroidal<br>Pore<br>Volume |
| <b>Metal:</b>         |                               |                              |                               |                              |                               |                              |                               |                              |                               |                              |
| Al                    | 87.2                          | 1.00                         | 86.7                          | 1.20                         | 76.5                          | 2.11                         | 53.3                          | 1.60                         | 2.1                           | 1.24                         |
| B                     | 9.9                           | 1.17                         | 11.9                          | 1.62                         | 21.1                          | 2.19                         | 16.5                          | 1.62                         | 0.7                           | 0.92                         |
| Ba                    | 99.2                          | 3.60                         | 61.5                          | 3.76                         | 4.0                           | 3.09                         | 1.6                           | 1.50                         | 317.0                         | 2.16                         |
| Ca                    | 12.5                          | 3.41                         | 11.8                          | 3.79                         | 1.7                           | 3.11                         | 0.6                           | 1.44                         | 12.8                          | 2.55                         |
| Cd                    | 11.5                          | 0.77                         | 12.2                          | 1.26                         | 10.4                          | 2.13                         | 8.8                           | 1.55                         | 0.7                           | 0.77                         |
| Cr                    | 44.4                          | 0.57                         | 33.0                          | 0.85                         | 50.7                          | 1.32                         | 46.7                          | 1.20                         | 1.4                           | 1.43                         |
| Cu                    | 0.6                           | 0.91                         | 0.7                           | 1.15                         | 0.3                           | 1.77                         | 0.2                           | 0.97                         | 1.4                           | 1.13                         |
| Fe                    | < 0.1                         | 2.94                         | < 0.1                         | 3.12                         | < 0.1                         | 1.92                         | < 0.1                         | 1.09                         | < 0.1                         | 2.12                         |
| Mn                    | 0.1                           | 1.01                         | 0.1                           | 2.28                         | < 0.1                         | 0.80                         | < 0.1                         | 0.72                         | < 0.1                         | 2.57                         |
| Mo                    | 96.7                          | 0.52                         | 99.0                          | 0.69                         | 111.0                         | 0.97                         | 108.3                         | 0.70                         | 7.6                           | 0.78                         |
| Pb                    | 32.5                          | 0.93                         | 17.5                          | 1.12                         | 13.0                          | 2.12                         | 13.4                          | 1.57                         | 1.5                           | 0.77                         |
| Sr                    | 24.5                          | 3.40                         | 17.1                          | 3.79                         | 1.6                           | 2.77                         | 0.7                           | 1.36                         | 25.9                          | 2.32                         |
| Zn                    | 3.2                           | 1.24                         | 4.2                           | 1.19                         | 2.7                           | 1.70                         | 1.9                           | 0.46                         | 0.7                           | 1.10                         |

\* Percentage Mass Leached = (mass leached/mass available) X 100%  
NOTE: FA = Fly Ash; S = Sand; B = Bentonite; C = Cement

ative to the other species in solution. Whereas this explanation may appear feasible, it cannot be substantiated by the data from the present study.

Except for Ba, Ca, Cu, and Sr, the addition of only 2.5 percent cement (Stage 4) to a mixture including 75 percent fly ash and 22.5 percent sand resulted in a drastic reduction in the mass of metal species leached in three pore volumes of flow. These reductions may be due to a pozzolanic reaction between the cement and the migrating metal species.

#### Leaching Rate

To evaluate the effect of flow rate on the leaching rate of the fly ash mixtures, a centroidal coordinate system was used. In this case, leaching rate refers to the rate of migration of the eluted center of mass of the metal species relative to the average flow rate. Since each effluent sample represents one-quarter pore volume of flow, the measured concentrations of the metals in the effluent samples represent only average values of concentration for a one-quarter pore volume of effluent. As a result, the elution curve represents a histogram of concentration, or mass, of metal leached versus pore volumes of flow. For any given histogram, the center of mass can be described by the centroidal coordinates  $\bar{m}$  and  $\bar{PV}$ , where  $\bar{m}$  is given by

$$\bar{m} = \frac{\sum_i A_i \cdot m_{ci}}{\sum_i A_i} \quad (2)$$

and  $\bar{PV}$  is given by

$$\bar{PV} = \frac{\sum_i A_i \cdot PV_{ci}}{\sum_i A_i} \quad (3)$$

where

$$A_i = m_i \cdot PV_i \quad (4)$$

$$m_{ci} = \frac{m_i}{2} \quad (5)$$

and

$$PV_{ci} = \frac{PV_i}{2} \quad (6)$$

where  $m_i$  is the mass collected in a sample volume (i.e., in  $PV_i$ ) of effluent.

Here,  $PV_i = 1/4$ , so Equation 3 reduces to

$$\bar{PV} = \frac{1}{8} \frac{\sum_{i=1}^{12} m_i(2i-1)}{\sum_{i=1}^{12} m_i} \quad (7)$$

Since the total mass leached, or area under the histogram, is a more pertinent parameter than is the centroidal mass coordinate,  $\bar{m}$ , Equation 2 is not used in this study.

The centroidal pore volume coordinate,  $\overline{PV}$ , given by Equation 7 is a measure of the rate of leaching of the center of mass of a given element; the higher the value of  $\overline{PV}$ , the slower the rate of leaching of a given metal. If the metal species is uniformly and homogeneously mixed in the column sample before leaching, the center of mass is represented by the center of volume of the column. If the metal is not constrained from leaching by some time-dependent process and is not degraded, then the center of mass of the metal should appear at one-half a pore volume of leachate. A  $\overline{PV}$  value greater than one-half indicates a delay process (e.g., ion exchange) in the elution of the metal species. A  $\overline{PV}$  value less than one-half could indicate several possibilities, including nonuniform sample mixture, an error in the determination of chemical concentrations, or preferential flow paths in the sample. Since the value of  $\overline{PV}_i$  of  $\frac{1}{4}$  used in Equation 7 is based on a pore volume assuming a completely saturated sample, the centroidal pore volume coordinate for unsaturated samples must be corrected as follows:

$$\overline{PV}(\theta) = \left( \frac{n}{\theta} \right) \overline{PV}(n) = \frac{\overline{PV}(n)}{S} \quad (8)$$

where

- $n$  = total porosity of the sample,
- $\theta$  = volumetric moisture content of the sample,
- $S$  = degree of saturation (decimal),

$\overline{PV}(n)$  = centroidal pore volume based on  $n$  (i.e., Equation 7), and

$\overline{PV}(\theta)$  = centroidal pore volume based on  $\theta$ .

In this study, all of the samples were unsaturated, so Equation 8 was used in lieu of Equation 7. However, since  $\theta$  was not constant, an average value of  $\theta$ , or  $\theta_{AVE}$ , was used in Equation 8, where

$$\theta_{AVE} = \frac{\theta_i + \theta_f}{2} \quad (9)$$

and  $\theta_i$  and  $\theta_f$  are the initial and final volumetric moisture contents, respectively. The use of  $\theta$  or  $\theta_{AVE}$ , which is less than  $n$ , resulted in effluent volumes that represent more than the three pore volumes of flow based on a saturated flow system.

On the basis of the centroidal pore volume approach described, the centroidal pore volumes for each metal species and each mixture in the flow-rate tests are given in Table 5. Again, the test specimens were chosen such that the mean flow rates for the test specimens are approximately the same (i.e., relative to the range of flow rates in the flow-rate tests).

Except for Zn, the leaching rates of the metals in the 50 percent fly ash/50 percent sand mixture were lower (higher  $\overline{PV}$  values) relative to the 100 percent fly ash specimen. The substitution of 5 percent bentonite for 5 percent sand in the 50 percent fly ash/50 percent sand mixture resulted in the further reduction in the leaching rates of all metals except Ba, Ca, Fe, Mn, and Sr. The further reduction apparently is attributable to the ion exchange capacity of the sand/bentonite admixture. Ba, Ca, and Sr are the metal species that exhibited delayed leaching behavior, and Fe and Mn are the metal

species that essentially did not leach from the mixtures within three pore volumes of flow.

An increase in bentonite content from 5 to 10 percent did not lower the leaching rates as expected. On the contrary, the leaching rates of all 13 metals increased. However, the fly ash contents of the 10 percent mixtures were also greater by 25 percent relative to the 5 percent mixtures. As a result, an increase in fly ash content apparently controls over an increase in bentonite content. Therefore, on the basis of the results of the test specimens from Stages 1, 2, 3A, and 3B, it appears that an increase in fly ash content tends to increase the leaching rate of the center of mass of the metal species.

A comparison of the two test specimens containing 75 percent fly ash (Stages 3B and 4) indicates that only four (Al, B, Cd, and Pb) of the 13 metals were leached faster for the Stage 4 mixture containing 2.5 percent cement. Therefore, on the basis of this limited comparison, it appears that cement is more effective than bentonite at reducing the leaching rate of metals for mixtures containing the same amount of fly ash. However, when the Stage 4 results are compared with the results for the Stage 2 and Stage 3A tests with lower (50 percent) fly ash contents, 9 of the 13 metal species in Stage 4 were leached at faster rates relative to the Stage 2 mixture, and 10 of the 13 metal species in Stage 4 were leached at faster rates relative to the Stage 3A mixtures. This follows the same trend, already mentioned, of a faster leaching rate (lower  $\overline{PV}$  value) with a greater fly ash content, regardless of the admixture constituents.

No simple trend in the leaching rate of a given metal species as a function of the flow rate for any of the mixtures was found. In addition, within a given mixture, the leaching rates of different metal species may increase or decrease with increasing flow rate or show no apparent trend. It is obvious that from this study that either there are no general trends in leaching rate versus flow rate or the test procedures and setup used in this study do not exhibit enough control to detect such trends.

#### pH and Electrical Conductivity

In general, there was a reduction in pH and electrical conductivity (EC) with an increase in pore volumes of flow for all test specimens. Both the pH and the EC of the effluent tended to be greater than that of the influent, especially within the first pore volume of flow. Also, there was no significant effect of flow rate on the pH and EC of the effluent. The pH values of all effluent solutions fall within the relatively narrow range of 11.8 (Stage 2, FR B4) to 13.3 (Stage 4, FR E4). This range is significantly more basic than the pH of 6.3 measured for the tap water.

Typical plots of EC versus pore volumes of flow are shown in Figure 4. The elution of ions from the test specimens is reflected by the initially high EC values for the effluent solutions. The initially high EC values gradually decrease within three pore volumes of flow towards the background EC value of 29.7  $\mu\text{mhos/cm}$  for the tap water. In the case of the fly ash and fly ash/sand test specimens (Stages 1 and 2), the EC of the effluent approaches background more completely and rapidly, within the first one to two pore volumes of flow. The



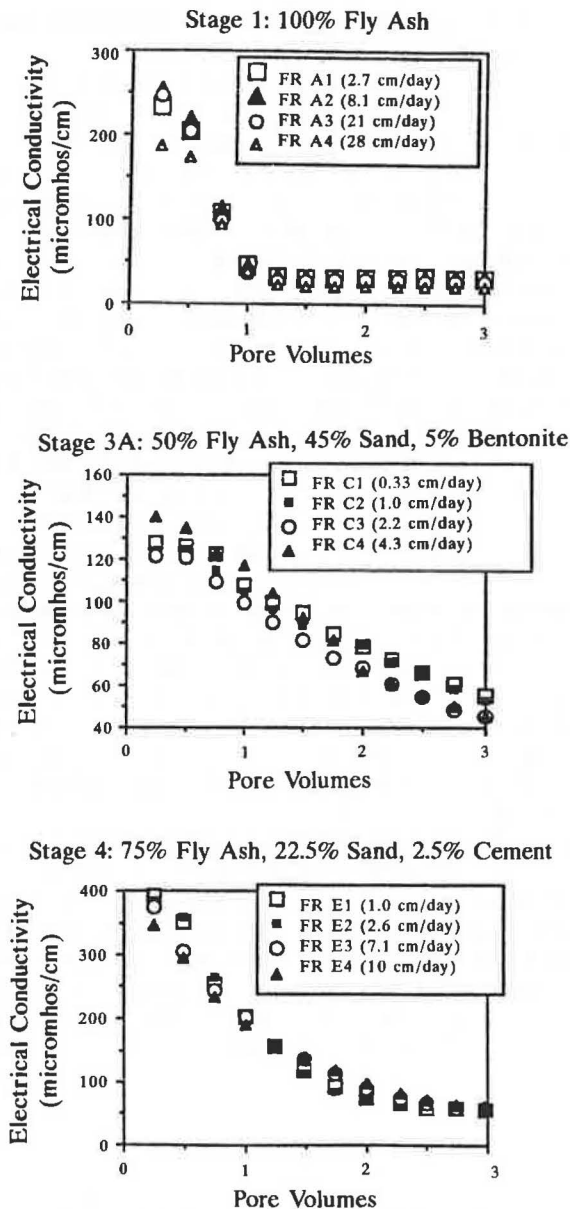


FIGURE 4 Typical curves of electrical conductivity versus pore volumes of flow.

trend is slightly more gradual and less complete for the fly ash/sand/cement specimens (Stage 4), and much more gradual for the specimens containing bentonite (Stages 3A and 3B). The more gradual reduction in EC with increase in pore volumes for the Stage 3A and 3B test specimens may be attributed to the retardation effect of the bentonite on the migrating metal ions.

An increase in fly ash content appears to result in an increase in the maximum EC value measured in the effluent. However, the maximum EC values for the Stage 4 test specimens containing 75 percent fly ash are significantly higher than the test specimen containing 100 percent fly ash (Stage 1), the difference being attributed to the cement content in the Stage 4 mixture.

For mixtures containing the same amount of fly ash, there is a contradiction in the maximum EC values measured in the effluent. In the case of the test specimen containing 50 percent fly ash, there appears to be little effect for the test specimens with bentonite (Stage 3A) relative to the test specimens without bentonite (Stage 2). However, for the test specimens containing 75 percent fly ash, there is a pronounced difference in maximum EC values, with significantly lower EC values for the test specimens containing bentonite (Stage 3B) relative to those containing cement (Stage 4). The differences apparently are due to the bentonite admixture of the Stage 3B test specimens or the incorporation of cement into the Stage 4 test specimens, or both.

In general, the EC data tend to reflect the previously drawn conclusions with respect to leaching rate of specific metal ions with some notable exceptions. First, the measured EC values of the effluent samples are not metal specific; the EC value represents the combined conductivity of all ions in solution. Second, anions (e.g.,  $\text{Cl}^-$ ) as well as cations (metals) contribute to the electrical conductivity of the effluent. Finally, whereas only ions contribute to the electrical conductivity of a solution, the measured metal concentrations probably include neutral metal species (e.g., metal oxides), which do not contribute to the measured EC value.

#### Sidewall Leakage

Although rigid-wall permeability tests are susceptible to sidewall leakage, certain factors suggest that sidewall leakage was not significant in this study. Sample preparation and curing conditions were nearly identical for every sample tested. No disturbance of the samples was noticed during the curing time and the testing period. Though final degrees of saturation varied slightly within the stages, no trend was evident indicating a correlation with leached metal quantities. The percentage of leached metal masses should be an inverse function of the degree of sidewall leakage. Less surface area within the porous media is exposed to the leaching fluid if there are preferential flow paths along the sidewalls. Less exposed surface area allows less dissolution of metals into the leaching fluid, resulting in lower quantities of metals leached. On the basis of an examination of the final values of the degree of saturation and the data for the percentage of metal mass leached within each stage, it is evident that the quantities of metals leached are not a function of the final saturation levels. No trends are evident indicating a particular test to have a low final degree of saturation and to have leached a lower percentage of metal mass relative to the other tests within the same stage. This type of trend would be expected if sidewall leakage were significant. In addition, permeability values measured in this testing program correlate well with those reported in recent literature (3,5,11).

#### SUMMARY AND CONCLUSIONS

##### Metal Leaching Versus Flow Rate

Metal leaching behavior from mixtures containing fly ash showed no dependence on flow rate for the range of flow

rates used in this study. Column tests at hydraulic gradients equal to 25, 50, 100, and 150 resulted in no consistent differences of metal leaching characteristics between tests. For all mixtures, average concentrations of four to six metals exceeded the DWSs (9) for at least one flow rate. However, no trends were apparent that linked magnitude of flow rate to relative metal concentrations in the leachate. On the basis of the centroidal pore volume approach, the leaching rate does not appear to be a function of flow rate. The leaching rate varied from metal to metal in each mixture tested, independent of flow rate. In summary, no evidence was found of a dependence of metal leaching on flow rate.

### Permeability Versus Admixture Constituents

This research indicates that compacted Class F fly ash, when stabilized with bentonite or cement, can have a permeability on the order of  $10^{-7}$  cm/sec, the current regulatory standard for earthen liners. Bentonite appears to be slightly more effective than cement at minimizing the permeability of mixtures containing fly ash and sand. There appears to be an optimum percentage of sand that minimizes the permeability of the fly ash-sand mixtures. For the three types of admixtures considered in this study, the permeability decreased in the following order: fly ash/sand > fly ash/sand/cement > fly ash/sand/bentonite.

### Leaching Behavior

Of the 13 metals traced, Ba, Ca, and Sr showed delayed leaching behavior. Fe and Mn showed no trend, and Al, B, Cd, Cr, Cu, Mo, Pb, and Zn showed early leaching behavior. The delayed leaching behavior contrasts with most other studies, which have indicated only early leaching behavior. These two leaching behaviors suggest that there is competition between metal ions for dissolution into the pore fluid.

### Metal Leaching Versus Admixture Constituents

Addition of sand resulted in little change in the percentage of mass leached of the metals traced. In contrast, the addition of bentonite resulted in a significant reduction in the mass of Ba, Ca, and Sr leached in three pore volumes of flow relative to the 100 percent fly ash mixture, whereas reductions in the amount of Al, Cd, Cu, Pb, and Zn were less significant. However, an adverse effect was noticed for the mass of B, Cr, and Mo leached in three pore volumes of flow. The reduction in the mass of Ba, Ca, and Sr is believed to be due to an increase in the delayed leaching behavior for these three chemical species resulting from the bentonite addition. Except for Ba, Ca, Cu, and Sr, the addition of only 2.5 percent cement to a mixture of 75 percent fly ash and 22.5 percent sand resulted in a drastic reduction in the mass of nine other metal species leached in three pore volumes of flow.

The centroidal pore volume approach was also used to compare the effects of different admixture constituents. For spe-

cific elements between different mixtures at approximately the same flow rate, increasing the fly ash content in fly ash/sand/bentonite mixtures tended to increase the leaching rate of most metals. The 2.5 percent cement content was more effective than the 10 percent bentonite at reducing the leaching rate of metals for mixtures containing the same amount of fly ash.

Both bentonite and cement admixtures reduce the leaching of metals in the first three pore volumes of flow. Overall, cement reduces the leaching of more metals to a greater degree than bentonite.

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