EVALUATION OF CONTROL TECHNOLOGY FOR MODIFIED IN SITU OIL SHALE RETORTS

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ABSTRACT

Experiments were conducted to evaluate two technologies to control groundwater pollution due to leaching of abandoned modified in-situ (MIS) retorts, retort grouting and intentional leaching. Retort grouting to reduce permeability was evaluated by measuring the permeability of grouts containing only raw or refined waste materials (Lurgi spent shale, fly ash, gypsum tailings, and lignosulfonate fluidizers). The principal factor controlling grout formulation was the requirement for adequate fluidity without bleeding. This was achieved by inclusion of 0.25% lignosulfonate fluidizer in the grout. Permeability of the cured grouts decreased with increasing confining pressure; at 200 psi confining pressure, permeabilities as low as 5×10^{-7} cm/sec were measured. Electrical conductivity measurements on the permeate produced during permeability measurements suggest that grouting abandoned MIS retorts would increase the TDS of leachate by a factor of approximately 3; benefit of the proposed grouting operation would depend upon the flow rate through retorts being reduced by a greater factor to reduce the total mass (concentration x flow) of solute released. Comparison of the measured grout permeabilities to the permeability of surrounding rock suggest that this would be the case.

Costs for intentional leaching depend primarily upon the volume of leachate to be treated. In order to estimate the number of pore volumes which must be intentionally leached, an analytical model of the leaching process was applied. The required number of pore volumes of leaching to reduce leachate concentration to 10% of its initial value was found to be 2.1 at tract C-a and 3.4 at tract C-b; the difference is due primarily to the greater void volume used at tract C-a (40% compared to 23%).

Both technologies would require a large amount of water. Retort grouting requires water to prewet the MIS spent shale and to prepare the grout. These requirements were estimated at 140 to 210 gal/bbl of oil, considering only oil recovered by in-situ retorting. Intentional leaching requires water to saturate the MIS spent shale and to replace blowdown or rejected brine from the leachate treatment process. These requirements were estimated at approximately 120 gal/bbl of oil.

INTRODUCTION

Modified in-situ (MIS) retorting has been proposed as a means to develop the oil shale resource of the Piceance Creek Basin. Advantages of MIS retorting include a reduced amount of raw shale to be mined and a reduced amount of spent shale to be disposed of on the surface, compared to surface retorting. However, MIS retorting introduces the problem of MIS spent shale leaching. Proposed MIS retorts at tracts C-a and C-b intersect aquifers, which are dewatered to permit mining and retorting. Following site abandonment, groundwater would reinvade the dewatered region, leaching the spent shale in abandoned MIS retorts and transporting leached material into aquifers and surface streams. Reinvasion would be slow because of the large amount of water to be replaced and the low transmissivity of the aquifers to deliver water to the site; recovery of the piezometric surface at tract C-b might take over 200 years (Mehran, Narasimhan, and Fox, 1980, 1981). Following recovery of the piezometric surface, transport of the leachate plume to surface streams also would be slow (4 to 160 ft/yr, depending upon local conditions) (Fox, 1980). Laboratory leaching of simulated and actual MIS spent shale has shown that, compared to native groundwater, leachate would be elevated in total dissolved solids (TDS), organic N, organic C, phenols, Se, Pb, and V (Amy, 1978; Fox, 1980; Peterson, et al., 1982; Persoff and Fox, 1983).

In 1978, a program to evaluate control technology for groundwater protection was initiated at Lawrence Berkeley Laboratory. Several candidate control technologies were identified as potentially effective at reasonable cost (Persoff and Fox 1979a,b). Two of these, retort grouting and intentional leaching, were selected for laboratory investigation. This paper

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summarizes the results of these investigations; a complete report is presented in Persoff and Fox (1983).

BACKFILLING ABANDONED RETORTS WITH SPENT SHALE GROUT

The object of retort grouting is to reduce the permeability of abandoned retorts to a value lower than that of the surrounding rock. This would reduce the rate of flow of groundwater through retorts, although the quality of leachate would not be improved. Golder Associates (1977) suggested that a post-grouting permeability of 10^{-6} cm/sec, or two orders of magnitude lower than that of the host rock, would be necessary to prevent increased salt loading to aquifers and surface streams. The large volume of voids to be filled dictated that the grout material be spent shale (on-site waste material). It was assumed that surface retorting of the mined raw shale would accompany MIS development.

Experiments reported elsewhere (Mehta and Persoff, 1980; Mehta, Persoff, and Fox, 1980) showed that spent shale could be converted to a true hydraulic cement similar to portland cement by heating with added $CaCO_3$. Cost considerations, however, impelled evaluation of spent shale as received, with no heat treatment and a minimum of added material. Lurgi spent shale was selected for this investigation because it has a low organic C content as well as the finest particle size distribution (57% finer than 4 µm) of any surface spent shale. Fine particle size contributes to low permeability.

Fluidity Criterion

For low permeability of the grouted retort, the spent shale grout must completely penetrate all the macrovoids between particles of spent shale (but not microvoids within particles) in an abandoned retort. This requirement, coupled with the economic requirement that injection holes be drilled far apart, imposes a fluidity criterion on the grout. The fluidity criterion can be approached either theoretically or empirically.

Particulate grouts (as distinct from chemical or solution grouts) are non-Newtonian fluids characterized by the Bingham or Casson model; they have a yield stress, τ_y greater than zero, which is the minimum stress needed to cause flow. Raffle and Greenwood (1961) showed, by analysis of forces on a plug of fluid in a cylindrical pore, that the maximum yield stress which would permit grout to penetrate to a specified distance through a cylindrical pore is given by

$$\tau_y = (R\rho gh)/(2d)$$

where τ_y = maximum allowable yield stress
R = pore radius
ρ = density of grout
g = acceleration of gravity
h = injection head
d = required penetration distance (no safety factor)

To apply this model to an MIS retort, the pore radius R must be replaced by an equivalent "effective" pore radius. Substituting values typical of an MIS retort (h = 150 m, d = 25 m, R = 0.1 cm, ρ =1.6 g/cm^3), the maximum τ_y is 470 dyne/cm^2. In practice, a safety factor must be used, and in particular it is difficult to characterize an MIS retort by an effective pore radius. Therefore, an empirical fluidity criterion was used.

Retort grouting, in which spent shale grout would be injected into MIS spent shale rubble, is similar to intrusion grouting of preplaced aggregate concrete, in which mortar is injected into gap-graded coarse aggregate preplaced in a form. Mortar is considered adequately fluid for the latter process if it flows through a standard US Army Corps of Engineers flow cone in 20±2 sec (American Concrete Institute, 1969; Crosby, 1971; du Plessis, 1970). All grouts tested were designed to meet this empirical fluidity criterion; rheometry showed that the yield stress of these grouts was about 60 dyne/cm^2. This suggests that the empirical fluidity criterion is quite conservative.

Fluidity of grouts can be improved by increasing the water-solids ratio (WSR), but this can also result in bleeding (separation of a clear supernatant by sedimentation). Bleeding is undesirable because it results in ungrouted voids and a more permeable grouted mass. Using only Lurgi spent shale and water, bleeding occurred if the WSR was greater than 0.8, but grout with this WSR was too thick to pass through the flow cone. Therefore an additive was needed to meet both the fluidity and non-bleeding requirements. In a preliminary study we found that replacing one-third of the spent shale by -30 +50 mesh sand enabled the grout to meet both criteria. However, because inclusion of sand in the grout could cause blocking of small pores, a chemical fluidizer was sought for the present series of grouts. A variety of such materials are available, of which the least costly are lignosulfonate fluidizers refined from waste liquor from sulfite pulping.

Materials

Spent shale was Green River oil shale which had been retorted in a Lurgi retort in Germany for Amoco Research, Inc. This sample was collected from the electrostatic precipitator during run 9, 1976. The chemical, mineralogical, and particle size analysis of this material are reported by Mehta and Persoff (1980), Mehta, Persoff, and Fox (1980), and Persoff and Fox (1983).

Class F fly ash was from the Craig, CO power plant; this is the nearest power plant to the Piceance Creek Basin. Class F fly ash is pozzolanic, but not cementitious. Class C fly ash was from the Wyodak power plant, Gillette, WY. Class C fly ash is not only pozzolanic but also cementitious. X-ray diffraction analysis showed that it contained some tricalcium aluminate (C_3A), which can react with gypsum to form ettringite, enhancing cementing properties. Therefore reagent gypsum was used along with this fly ash. Separate experiments (Persoff and Fox, 1983) showed that this reagent gypsum could be replaced by waste gypsum tailings.

Two lignosulfonate fluidizers, CZ-503 and CZ-512, were supplied by the Crown Zellerbach Corp. These are sodium salts of lignosulfonic acid, which are refined from sulfite pulping waste liquor.

Table 1. Spent Shale Grouts Containing Lignosulfonate Fluidizers.

	R-1	R-2	R-3	R-4	R-5
Lurgi spent shale, g	100	100	100	100	100
Class F fly ash, g	0	0	0	0	10
Class C fly ash, g	0	0	0	9.5	0
Reagent gypsum,g	0	0	0	0.5	0
Lignosulfonate fluidizer CZ-503, g	0.5	0.25	0	0	0
Lignosulfonate fluidizer CZ-512, g	0	0	0.25	0.25	0.25
Distilled water, mL	69.4	74.6	71.8	68.9	63.3
Water-to-solids ratio (WSR)	0.69	0.74	0.72	0.69	0.63
Flow cone time, sec	17	16	18	22	22

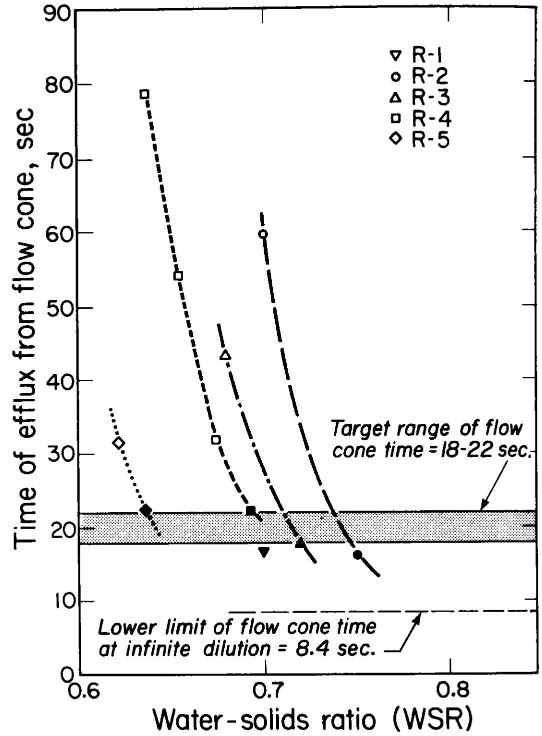

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Figure 1. Relationship between water-solids ratio (WSR) and flow-cone time. Solid points represent the final composition of the grouts (see Table 1).

Methods

The grout formulae tested are shown in Table 1. Grouts were prepared by dry blending the solid ingredients and mixing with the least amount of water needed to produce a grout fluid enough to pass through the flow cone. Water was then added in increments until the flow cone time was reduced to the target range of 20±2 sec. The grout was mixed for 3 min at 1300 rpm initially and after each incremental addition of water, using a Jiffy^{TM} mixer (Jiffy Co., Irvine, CA). The decrease of flow cone time with incremental addition of water is shown in Figure 1. The flow cone time did not change with additional mixing or after standing undisturbed for 10 min. Grout samples for permeability measurement contained no coarse aggregate. They were prepared by pouring the grout into waxed cardboard cylinder molds and curing in 100% relative humidity at 73°F for 5 to 7 months. Additional samples for measurement of triaxial compressive strength were prepared with coarse aggregate to simulate grouted MIS spent shale; the test methods and results are reported elsewhere (Persoff and Fox, 1983).

For measurement of permeability, the waxed cardboard molds were stripped from the sample, a porous stone was placed on each end, and the sample was encased in a flexible rubber jacket and submerged in deaired water in a vacuum chamber for 10 days. This was done to assure complete saturation of the pore space within the sample. After saturation, the sample was placed in a triaxial load cell as shown in

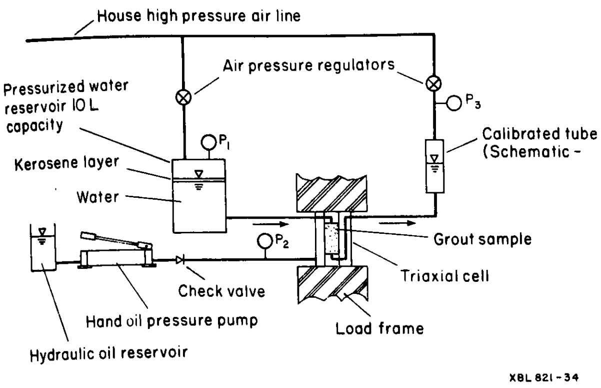

Figure 2. Permeability measuring system.
P_1 = upstream pressure, P_2 = confining pressure,
P_3 = downstream pressure.
Hydraulic gradient = $(P_1 - P_3)/(\text{sample length})$.

Figure 2. Confining pressure was applied to the sample in the triaxial cell and the load frame was adjusted to provide a uniform state of stress (confining pressure equal in all directions). A hydraulic gradient was applied across the sample, and flow through the sample was measured by the motion of the fluid interface in a calibrated tube (Chan and Duncan, 1966). This "volume change device" allowed measurement of small flow rates into a pressurized reservoir by allowing the operator to reverse the direction of flow through the calibrated tube without changing the direction of flow through the sample and into the reservoir.

The typical permeability measurement test lasted one week, with permeability being measured at confining pressures of 60, 120, and 240 psi, and at hydraulic gradients of 250, 340, and 420 ft/ft. At intervals during the test, the permeate was drained from the collection reservoir for electrical conductivity measurements.

Results and Discussion

The results of permeability measurements are shown in Figure 3. The consolidation of grout samples under confining pressure caused permeability to decrease at higher confining pressures. Comparison of the results for the various grouts shows that permeability was lower for R-1 and R-2, which included fluidizer CZ-503, than for the other grouts, which contained CZ-512. This suggests that the fluidizer itself was the cause of low permeability. According to information supplied by the manufacturer, both 503 and 512 are sodium salts of lignosulfonic acid, the only difference being that residual sugars are present in 503 but have been removed from 512 (residual sugars would retard the setting of portland cement). Whether this is an adequate explanation of the observed effect is not known, and it should be tested with other desugared and nondesugared fluidizer pairs.

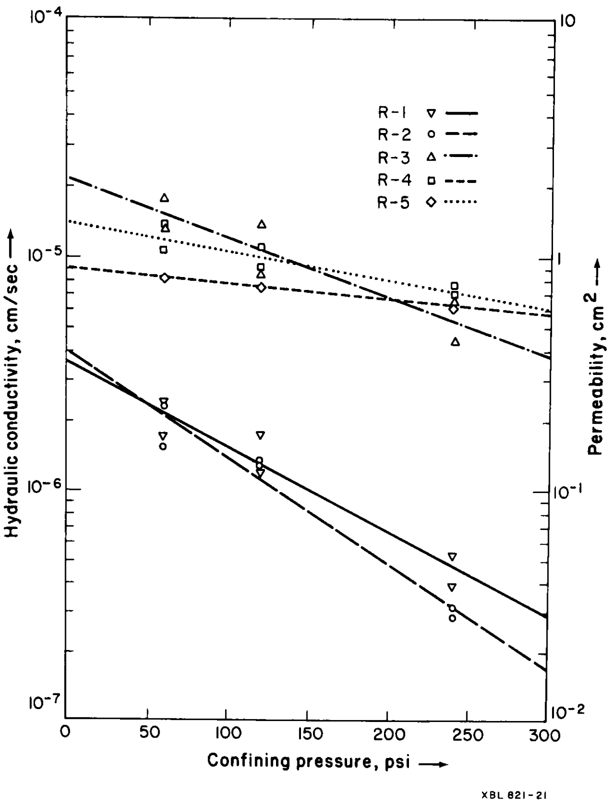

Figure 3. Variation of grout permeability with confining pressure.

Results of triaxial tests of simulated grouted cores are presented in Persoff and Fox (1983). The addition of fly ash increased the strength of the grouts, but it also increased their resistance to consolidation under confining pressure. As a result, the decrease in permeability with increasing confining pressure, which was observed for all grouts, was less for grouts R-4 and R-5 than for grouts containing no fly ash. Figure 3 shows this effect.

A typical confining pressure in an in-situ retort was estimated to be 200 psi. At this confining pressure, the permeability of grout R-2 would be 5×10^{-7} cm/sec. This permeability would also be representative of the grouted retort as a whole (assuming that it is completely grouted), because the MIS spent shale particles would be less permeable than the grout, and the grout, not the rubble, would be the continuous phase. Table 2 compares this value to the permeability of surrounding aquifers at tracts

Table 2. Estimated Flow Reduction Caused by Retort Grouting

	Tract C-a	Tract C-b
Permeability of upper aquifer, cm/sec[a]	5.3×10^{-4}	1.8×10^{-4} to 2.8×10^{-4}
Permeability of lower aquifer, cm/sec[a]	1.52×10^{-3}	0.4×10^{-4} to 1.1×10^{-4}
Permeability of grouted retort, cm/sec	5.0×10^{-7}	5.0×10^{-7}
Factor of flow reduction[b]	1060	80 to 220

[a] Permeabilities of aquifers reported by Fox (1980), p. 203.

[b] This factor is the ratio of the permeability of the upper or lower aquifer (whichever is less) to that of the grouted retort.

C-a and C-b, and shows that the permeability reduction would be sufficient to reduce flow through grouted retorts by a factor of 1060 or 80 to 220 at the two sites, respectively.

The total pollutant load to aquifers is the product of the concentration of leachate and the rate of flow through abandoned MIS retorts. Figure 4 shows that the conductivity of permeates, collected during permeability measurements, decreased with cumulative flow through the samples. Also plotted in this figure (+) are conductivity data from column leaching of spent shale recovered from Occidental Oil Shale retort 3E (core 1, section 1) (US DOE, 1980). This MIS spent shale is less leachable than Lurgi spent shale because it was exposed to more severe retorting conditions. Comparison of the two data sets suggests that the conductivity of leachate from a grouted retort would be approximately three times as great as that of leachate from an ungrouted retort. This deleterious effect would be offset by flow reduction by a factor greater than 3 (as calculated in Table 2). Thus the potential benefits of retort grouting are site-specific: it should only be considered for use at sites where rock surrounding the retort is relatively permeable and where the flow reduction accomplished by retort grouting would be large.

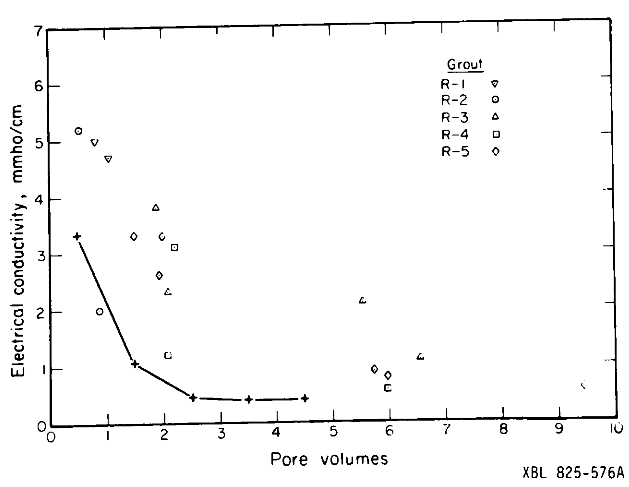

Figure 4. Decrease in electrical conductivity of permeates with cumulative flow through grout samples during permeability tests. Data are also shown (+) for section 1 of MIS core R3E1 (data from US DOE, 1980).

Water requirements for retort grouting were calculated by considering the water required to prewet the MIS spent shale plus the water required for the grout itself. Details of the calculation are presented in Persoff and Fox (1983). Taking typical values (porosity of spent shale = 0.3, density of raw shale = 140 lb/ft^3, density of grout = 100 lb/ft^3, WSR of grout = 0.7, recovery by MIS retorting = 65% of Fischer Assay, Fischer Assay = 24 gal/ton), the water requirements, neglecting any oil recovered by surface retorting, would be 140 gal/bbl of oil for a retort with 23% voids, or 210 gal/bbl for a retort with 40% voids.

INTENTIONAL LEACHING

Retort grouting is intended to reduce the flow rate of water through abandoned retorts, but not to improve the quality of leachate (indeed, it would become worse). Conversely, intentional leaching is intended to improve the quality of leachate entering aquifers, but not to reduce the flow rate. Many column leaching experiments have shown the effect noted in Figure 4: leachate concentrations are initially high but decrease to a "tail" after a few pore volumes. (Although the tail concentration is low, the total mass of solute contained in the tail is much greater than the mass contained in the initial pulse). For groundwater quality protection, the first few pore volumes would be recovered and treated (no treatment process has been demonstrated yet) until the concentration of subsequent leachate was acceptably low. Treatment costs for many processes depend mainly on the volume of water to be treated. Therefore, a procedure was needed to estimate the number of pore volumes of leachate that would require treatment.

Leaching behavior such as shown in Figure 4 suggests that concentration of the solute is controlled by the rate of mass transfer from the interior of spent shale particles. Hall (1982) developed an analytical model of the leaching process for mass-transfer-limited solutes, and verified this model with column leaching experiments in which the concentration of TOC in leachate from LETC S-55 simulated in-situ spent shale was monitored. The concentration of leachate exiting a column (or retort) is expressed in terms of dimensionless parameters ALPHAX (dimensionless column length) and

TIME (dimensionless time).

$$ALPHAX = (4D_m z)/(mb^2U_p)$$

where all symbols are given in Table 3

$$TIME = (\theta D_m/b^2)$$

where t = time

$\theta = t - (z/U_p)$ = time after arrival of the first pore volume at a given point in the column.

TIME/ALPHAX is a dimensionless inverse flow velocity and is nearly proportional to the number of pore volumes.

$$TIME/ALPHAX = (\theta mU_p)/(4z) = (n_{pv} - 1)/(4z)$$

Figure 5 plots the modeling results for various values of ALPHAX. Substituting appropriate values for the indicated quantities as shown in Table 3, ALPHAX values of 1.06 and 0.97 were calculated for tracts C-a and C-b, respectively. Interpolation in Figure 5 led to the results that the leachate concentration would be reduced to 10% of the initial concentration after 2.1 or 3.4 pore volumes of intentional leaching at tract C-a or C-b, respectively. Figure 5 shows that placing a more stringent requirement on the leachate concentration (e.g., reduction to 2% of the initial concentration) would sharply increase the required number of pore volumes.

This model assumes that solute concentrations in leachate are mass-transfer-limited, and it cannot be applied to solvents which are solubility-limited. It has been validated experimentally only for total organic carbon, and it should be validated for other solutes of concern.

In intentional leaching, leachate would be treated with the loss of a brine stream (assumed 10%); the remainder would be reused for further leaching. Treating the number of pore volumes calculated in Table 3 would result in consumption of 40 or 29 gal/bbl of oil at tracts C-a or C-b, respectively. More water would be consumed, however, in saturating the MIS spent shale; water in micropores could not be recovered for treatment. Assuming the porosity of MIS spent shale particles to be 30%, as suggested by the data of Hall (1982) (the porosity could be as high as 50%), this would consume 86 gal/bbl of oil, for a total water consumption for intentional leaching of 126 or 115 gal/bbl of oil, considering only oil recovered by MIS retorting. For comparison, Fox (1980) estimated the total water requirements for MIS retorting, excluding abandonment procedures, at 61 to 128 gal/bbl of oil.

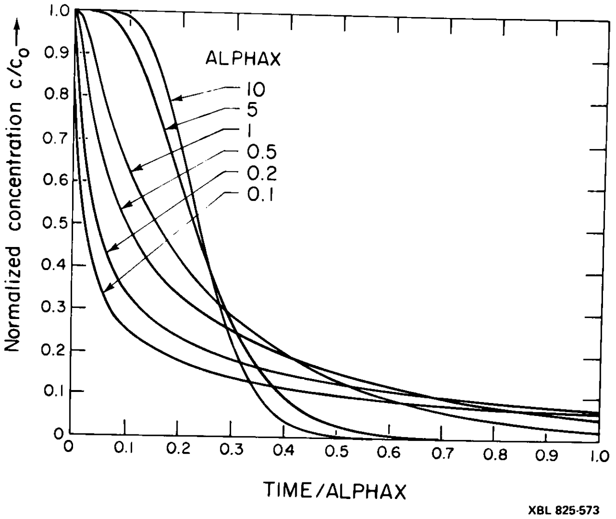

Figure 5. Plot of dimensionless breakthrough curves for ALPHAX values from 0.1 to 10 (Hall, 1982)

Table 3. Estimation of Number of Pore Volumes Required to Reduce Leachate Concentration to c/c_o = 0.1 by Intentional Leaching at Tracts C-a and C-b.

Parameter	Tract C-a	Tract C-b
D_m, solute diffusivity, assumed, cm^2/sec	1.0×10^{-5}	1.0×10^{-5}
macroporosity[a]	0.40	0.23
microporosity[b]	0.18	0.23
m, ratio of macro- to microporosity	2.22	1.00
U_p, vertical velocity of flow through retort, assumed,	1.55×10^{-2}	1.55×10^{-2}
b, particle radius, cm	5	5
z, retort height, m	229	94
ALPHAX	1.06	0.97
value of TIME/ALPHAX at which c/c_o = 0.1[c]	0.6	0.6
n_{pv}, number of pore volumes	2.1	3.4

[a] Based upon values reported for experimental MIS retorts.

[b] Microporosity = (0.3)(1 - macroporosity). That is, individual particles of spent shale have porosity of 0.3.

[c] By interpolation in Figure 5.

CONCLUSIONS

A grout that meets both fluidity and non-bleeding criteria cannot be made using only Lurgi spent shale and water; if 0.25% lignosulfonate fluidizer is added, both criteria can be met.

Permeability of these spent shale grouts decreases with increasing confining pressure; this effect is greater for grouts that contain no fly ash.

Electrical conductivity of permeates from grout permeability measurements decreases with increasing cumulative flow through the sample, and is approximately 3 times as great as conductivity of laboratory-produced MIS spent shale leachate.

Flow reduction through grouted MIS retorts depends upon the confining pressure in the retort and upon the permeability of surrounding rock; calculations for tracts C-a and C-b suggest that flow would be reduced by a factor of 1060 and 80-220, respectively.

A model of the leaching process for mass-transfer controlled solutes was applied to MIS retorts to determine the number of pore volumes of leachate that would be needed to reduce the concentration of subsequent leachate to 10% of its initial value. At tracts C-a and C-b, 2.1 and 3.4 pore volumes were required, respectively.

The water requirements are sensitive to the porosity of the MIS spent shale. Intentional leaching would require 115 to 126 gallons of water per barrel of oil; retort grouting would require 140 to 210 gallons of water per barrel of oil. These figures are for 23% to 40% voids in the retort, with 30% porosity of the MIS spent shale.

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