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Durability of Double-Shell Slurry Feed Grouts: FY-90 Results

R. O. Lokken
P. F. C. Martin

December 1992

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Pacific Northwest Laboratory
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DURABILITY OF DOUBLE-SHELL SLURRY FEED GROUTS: FY-90 RESULTS

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Pacific Northwest Laboratory
Richland Washington

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SUMMARY

This report summarizes results from studies conducted during FY 1990 to assess the durability of grouted double-shell slurry feed (DSSF) waste. These studies were performed in support of Westinghouse Hanford Company's Grout Disposal Program to determine the physical and chemical properties of simulated DSSF grouts cured at elevated temperatures.

Previous studies (Lokken et al. 1989 and Lokken et al. 1992a) have indicated a strong impact from curing temperature and curing time on the strength and leach resistance of DSSF grouts. The current studies were expanded to determine whether these impacts could be attributed to other factors, such as the dry blend composition and the waste concentration. Some major conclusions from these studies include the following:

- Grouts prepared with dry blends containing 40 wt% limestone had lower strengths than grouts prepared without limestone.
- Compressive strengths decreased with increased curing temperature.
- Leach resistance decreased with increased curing temperature and curing time, with curing time having the greatest effect at the lower temperature.
- Waste concentration (dilution) had a major, positive effect on leachability, i.e., leach resistance increased for the grouts prepared with dilute DSSF.
- Nitrate leach resistance increased with high slag-to-cement ratios, dilute DSSF and low curing temperatures.
- The amount of drainable liquids for the grouts prepared with diluted DSSF was lowest when the slag content was high, suggesting that the slag reacts faster than the fly ash to produce a rigid structure within the grout that minimizes particle settling.
- The two most significant factors affecting the grout properties were the slag-to-cement ratio and waste dilution. Interactions between these two factors were also significant, indicating that reactions between the slag and the waste appear to dominate the properties of DSSF grouts.
- The effects of curing time and curing temperature were consistent with results from previous studies (Lokken et al. 1989).

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INTRODUCTION

Current plans for disposal of the low-level fraction of selected double-shell tank wastes at Hanford, Washington include grouting. Grout disposal in this application is the process of mixing low-level liquid waste with cementitious powders and pumping the resultant slurry to near-surface, underground concrete vaults. Once the slurry is in the vaults, the hydration reactions that occur result in the formation of a solid product that binds and/or encapsulates the radioactive and hazardous constituents.

Cementitious materials have been or will be used at many locations for the solidification and disposal of low-level radioactive wastes. Oak Ridge National Laboratory (ORNL) began disposing of low-level liquid wastes in 1966 using a process known as hydraulic fracturing (de Laguna 1966, Weeren 1976). This process involved mixing liquid wastes with a blend of cement, fly ash, pottery clay, and attapulgite clay, and pumping the slurry at 3000 to 5000 psi into shale formations underlying the ORNL site. The high pressures caused the shale to fracture, and the grout filled the resultant fissures. The Savannah River Plant (SRP) is planning to dispose of 400 million liters of a low-level salt solution using the "saltstone" process (Langton 1988, Wilhite et al. 1988). Saltstone is the name given to the product prepared by mixing the salt solution with a blend of fly ash, blast furnace slag, and cement. This process is very similar to the grouting process at Hanford (Guymon et al. 1988). Both processes use the same type of dry solids, and the major constituents in the waste solutions are the same (i.e., $NaNO_3$, $NaNO_2$, $NaAl(OH)_4$ and NaOH).

Westinghouse Hanford Company (WHC) operates the Grout Treatment Facility (GTF) for the U.S. Department of Energy (DOE). The GTF includes the Dry Materials Facility (DMF), the Grout Processing Facility (GPF), and the grout disposal vaults. The DMF receives, stores, batches, and blends the individual dry materials for use in the grouting operation. The blended solids are transported to the site of the GPF where they are mixed with the low-level waste in a continuous process at rates up to 70 gallons of grout per minute. The grout slurry is pumped to underground concrete vaults where it hardens and immobilizes the hazardous and radioactive constituents through chemical reactions and/or microencapsulation.

Pacific Northwest Laboratory[a] (PNL) provides support to the Grout Disposal Program at Hanford through laboratory support activities (Lokken et al. 1987), radioactive grout leach testing (Serne et al. 1987), performance assessments (Sewart et al. 1987), and pilot-scale tests (Fow et al. 1987). A major pilot-scale test was performed in 1986 with a simulated phosphate/sulfate waste (PSW) to assess the effectiveness of the grouting operations and to characterize the grout produced with pilot-scale equipment and cured in a large mass. The results of that test are presented in Fow et al. (1987), Lokken et al. (1988), and Lokken and Mitchell (1988). Characterization of grout samples taken from an actual disposal vault have also been reported by Martin and Lokken (1992).

Grout disposal will be used for double-shell slurry feed (DSSF) waste, one of the types of low-level wastes stored in double-shell tanks on the Hanford Site. The initial formulation for DSSF waste included a dry blend consisting of approximately 47 wt% ground blast furnace slag, 47 wt% fly ash, and 6 wt% Portland cement. The dry blend is mixed with liquid waste at a nominal ratio of 9 lb of solids per gallon of waste (1080 g/L). This formulation was developed to meet specified criteria for processing, leachability, and physical properties of the grouted waste form. Because of time constraints during formulation studies, tests using these grouts were conducted after relatively short curing times at temperatures that do not accurately simulate the temperatures that will occur under the expected disposal conditions. While the grouts prepared with the initial formulation met most formulation criteria, additional information was needed to verify that long-term reactions within the grout at elevated temperatures will not sacrifice the integrity of the disposal system and result in less favorable performance.

A second pilot-scale test using the formulation described above was conducted at PNL in 1988. The results of this test and of subsequent product characterization are presented by Lokken et al. (1992b). Results of the pilot-scale test indicated that the temperature rise during curing of DSSF grout produced with the initial dry blend formulation would exceed design criteria. Also, long-term, high-temperature curing studies on this

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formulation showed that leach resistance and compressive strengths decreased with increases in temperature and curing time (Lokken et al. 1989 and Lokken et al. 1992a). Subsequently, a modified formulation was tested to reduce the total heat generated during hydration. This formulation included 40 wt% ground limestone which was added to reduce the amount of heat-generating solids. The remainder of the formulation included 4 wt% Portland cement, 28 wt% fly ash, and 28 wt% blast furnace slag.

The objective of this study was to qualitatively determine which factors affect selected properties of DSSF grouts. The factors included dry blend composition, waste composition (by dilution), curing temperature, curing time, and the presence (or absence) of additional water during curing. Properties that were measured included critical flow rate, slurry density, bulk density, drainable liquids, compressive strength, evaporable water, and leach resistance.

CONCLUSIONS AND RECOMMENDATIONS

Two statistically designed experimental matrices were used to determine the effects of various factors on the properties of DSSF grouts. One matrix utilized a Plackett-Burman screening design to determine the effects of fly ash-to-cement ratio, slag-to-cement ratio, limestone content, curing temperature, curing time, and the effect of additional water available during hydration. The only significant effect indicated by the data from this matrix was the fly ash-to-cement ratio. This ratio had significant effects on the critical flow rate, bulk density, evaporable water content, and on the nitrate and sodium leachability. The effects of all the other factors on the selected grout properties were not significant at the 95% confidence levels. Because the experimental matrix was designed for screening studies, only the main effects of the variables could be determined; interactions between variables were not assessed. However, the data from this matrix indicate the following:

- Leach resistance decreases with increased curing temperature and curing time, with curing time having the greatest effect at the lower temperature.
- Grouts prepared with dry blends containing 40 wt% limestone had lower strengths than grouts prepared without limestone.
- Compressive strengths decreased with increased curing temperature.
- The presence of additional water during curing had no significant effects on the grout properties; however, the amount of water adsorbed by the grouts was less than 1 wt%.

The other matrix was based on a two-level, fourth-order factorial design, with three replicates at the center of the design space. This design allows for the determination of main effects of the variables and two-way interactions between variables. Based on the statistical analyses of these data, the following conclusions can be made:

- Waste concentration (dilution) had a major, positive effect on leachability, i.e., leach resistance increased for the grouts prepared with dilute DSSF. Grouts prepared with dilute DSSF had drainable liquids up to about 30 vol%.

- Nitrate leach resistance increased with high slag-to-cement ratios, dilute DSSF and low curing temperatures.

- A combination of high slag content, no fly ash, undiluted DSSF, and high temperature resulted in the worst leachability for nitrate.

- The amount of drainable liquids for the grouts prepared with diluted DSSF was lowest when the slag content was high, suggesting that the slag reacts faster than the fly ash to produce a rigid structure within the grout that minimizes particle settling.

- The two most significant factors affecting the grout properties were the slag-to-cement ratio and waste dilution. Interactions between these two factors were also significant, indicating that reactions between the slag and the waste appear to dominate the properties of DSSF grouts.

The effects of curing time and curing temperature were consistent with results from previous studies (Lokken et al. 1989 and Lokken et al. 1992a).

Pore size distribution measurements were conducted to determine whether leach resistance could be correlated with the pore size distribution. However, the high salt content in pore solutions apparently left precipitated salt crystals in the pores, which in turn did not allow an accurate representation of the pores available for ionic diffusion during leaching.

The results obtained from these studies will provide valuable guidance in defining future formulation enhancement activities aimed at improving grout properties.

MATERIALS AND METHODS

SIMULATED WASTE AND DRY BLEND

The simulated DSSF waste used in these studies was obtained from a large batch prepared for use in the November 1988 pilot-scale test of grout processing characteristics (Lokken 1992b). The nominal and analyzed composition of the simulated waste is listed in Table 1.

Dry blends were prepared with a combination of blast furnace slag, type I/II Portland cement and limestone flour obtained from Ash Grove Cement West, and class F fly ash from Centralia, Washington. The dry materials were mixed in a V-blender for 23 hours prior to grout preparation. The oxide composition of the major constituents in the slag, fly ash, and cement, as determined by inductively coupled plasma (ICP) spectroscopy, is listed in Table 2. Table 3 lists the concentrations of trace metals in these materials as determined by X-ray fluorescence (XRF) analysis.

EXPERIMENTAL MATRICES

Two experimental matrices were used in the studies. The first matrix, listed in Table 4, was used to determine the effects of various factors on the properties of cured DSSF grouts. The specific factors included dry blend composition, waste composition, and curing temperature. This matrix utilizes a two-level fourth-order factorial design with three replicates of the midpoint of the design.

The second experimental matrix, listed in Table 5, was used to determine the effects of various curing conditions and dry blend compositions on DSSF grout properties. This matrix follows a Plackett-Burman screening design and was used to determine main effects of the various factors.

GROUT PREPARATION

Grouts were prepared using a Hobart mixer and a wire whip. The waste was preheated to approximately 45°C, and then poured into the mixer bowl. Room temperature dry blend was added to the waste at a mix ratio of 9 lb/gal

TABLE 1. Composition of Simulated DSSF Waste

	Composition, g/L	
Species	Analyzed[(a)]	Nominal[(b)]
Al	22.4	20.3
B	0.136	0.105
Ba	0.6	0.623
Ca	0.573	0.2
Cr	1.26	1.15
Fe	1.49	1.41
K	11.5	9.72
Mg	0.32	
Mn	3.01	2.75
Mo	0.068	0.049
Na	122	121.8
Si	0.502	0.56
Zn	2.93	1.63
Cl^-	5.36	3.86
NO_2^-	27.2	23.0
PO_4^{-3}	5.4	5.65
NO_3^-	186	154.4
SO_4^{-2}	5.1	5.05
TOC[(c)]	1.556	1.28

(a) Analyzed for this study
(b) Claghorn (1987)
(c) Total organic carbon as EDTA and citrate

(1.08 kg/L). After mixing, grout slurry samples were tested for density and for rheology using a Fann viscometer. The grouts were prepared in random order as listed by run number in Tables 4 and 5. The slurry was prepared for curing as discussed below.

TABLE 2. Oxide Composition of Blast Furnace Slag, Class F Fly Ash, and Cement

Oxide	Composition, wt%(a)		
	Slag	Fly Ash	Cement
Al_2O_3	13.4	23.5	3.3
B_2O_3		0.5	0.105
BaO	0.117	0.169	0.084
CaO	43.4	8.05	65.4
Fe_2O_3	0.377	5.73	4.08
K_2O	0.89	0.98	0.65
MgO	5.62	1.57	1.38
MnO_2	1.03	0.088	0.072
Na_2O	0.401	3.02	0.32
P_2O_5		0.94	
SiO_2	33.3	47.8	22.2
SrO	0.078	0.31	0.035
TiO_2	1.08	4.43	0.22
=====	=======	=======	=======
Total	99.693	97.087	97.846

(a) Determined by ICP analysis

TABLE 3. Concentration of Trace Metals in Ground Blast Furnace Slag and Class F Fly Ash

Trace Metal	Concentration, ppm(a)	
	Slag	Fly Ash
Ag	<4.6	<4.7
As	3.1	22.2
Cd	<5.2	<5.1
Hg	<4.3	<4.6
Pb	<3.9	22.9
Se	2.0	2.0

(a) Determined by XRF analysis

TABLE 4. Experimental Matrix for Determining the Effects of Dry Blend Composition, Waste Composition, and Curing Temperature on DSSF Grout Properties (All grouts were cured for 1 month)

Run No.	Trial No.	F/C[b]	S/C[c]	Waste Dil.	Temp., °C	Dry Blend Composition[a], wt% C	F	S	L
15	1	0	0	1X	55	60.0	0	0	40.0
8	2	8	0	1X	55	6.7	53.3	0	40.0
7	3	0	8	1X	55	6.7	0	53.3	40.0
4	4	8	8	1X	55	3.5	28.2	28.2	40.0
6	5	0	0	100X	55	60.0	0	0	40.0
3	6	8	0	100X	55	6.7	53.3	0	40.0
2	7	0	8	100X	55	6.7	0	53.3	40.0
10	8	8	8	100X	55	3.5	28.2	28.2	40.0
18	9	0	0	1X	95	60.0	0	0	40.0
19	10	8	0	1X	95	6.7	53.3	0	40.0
14	11	0	8	1X	95	6.7	0	53.3	40.0
12	12	8	8	1X	95	3.5	28.2	28.2	40.0
11	13	0	0	100X	95	60.0	0	0	40.0
16	14	8	0	100X	95	6.7	53.3	0	40.0
17	15	0	8	100X	95	6.7	0	53.3	40.0
13	16	8	8	100X	95	3.5	28.2	28.2	40.0
1	17	4	4	10X	75	6.7	26.7	26.7	40.0
5	18	4	4	10X	75	6.7	26.7	26.7	40.0
9	19	4	4	10X	75	6.7	26.7	26.7	40.0

(a) C = type I/II Portland cement
F = Class F fly ash
S = Ground blast furnace slag
L = Limestone flour
(b) Fly ash-to-cement ratio
(c) Slag-to-cement ratio

TABLE 5. Experimental Matrix for Determining the Effect of Curing Conditions and Dry Blend Composition on Grout Properties

Run No.	Trial No.	F/C[b]	S/C[c]	Dry Blend Composition, wt%[a] L	C	F	S	Curing Conditions Water[d]	°C	months
10	1	8	8	40	3.6	28.2	28.2	0	95	3
2	2	8	0	0	11.1	88.9	0	10	95	3
8	3	0	8	0	11.1	0	88.9	10	95	1
12	4	8	8	0	5.8	47.1	47.1	10	55	1
4	5	8	8	40	3.6	28.2	28.2	0	55	1
11	6	8	0	0	11.1	88.9	0	0	55	3
7	7	0	0	40	60	0	0	0	95	1
5	8	0	0	0	100	0	0	10	55	3
6	9	0	8	40	6.7	0	53.3	0	95	3
9	10	8	0	40	6.7	53.3	0	10	95	1
1	11	0	8	40	6.7	0	53.3	10	55	3
3	12	0	0	0	100	0	0	0	55	1

(a) C = type I/II Portland cement
F = Class F fly ash
S = Ground blast furnace slag
L = Limestone flour

(b) Fly Ash/Cement ratio

(c) Slag/Cement ratio

(d) 0 indicates no additional water present during curing
10 indicates that additional water equal to 10 wt% of the grout was added to the curing container during curing

CURING PROCEDURES

After mixing, grout slurry was poured into 250-mL plastic graduated cylinders. The cylinders were weighed and then placed into copper tubes. Copper end caps were placed onto the copper tube and the assembly was placed into a steel frame that was used to keep the caps tight. The assemblies were then placed in ovens initially operating at approximately 35°C. The temperature of the ovens was increased over a 3-day period to 55°C, 75°C, or 95°C.

After curing for the desired time periods, the samples were allowed to cool slowly to room temperature. The grout samples were removed from the graduated cylinders by cutting through the cylinder and then pushing the grout sample out. The grout specimens were weighed and placed into plastic bags until tested. Testing was normally conducted within 5 days after removal.

PHYSICAL PROPERTY TESTS

Compressive strength testing was conducted with an Instron(a) test machine at a constant crosshead speed of 0.05 in./min. The load-to-failure was determined from the maximum point of a load-deformation curve. Compressive strength values were calculated by dividing the maximum load by the cross-sectional surface area of the cylinders. The length-to-diameter ratio of these samples was one. Bulk density was determined by dividing the weight of the compressive strength samples by their bulk volume as determined by length and diameter measurements.

The moisture content, or amount of evaporable water, of the grouts was determined by drying the compressive strength samples (after testing) to a constant weight at 105°C ± 2°C.

PORE SIZE DISTRIBUTION

Pore size distribution was determined for selected samples using mercury intrusion porosimetry. These tests were conducted by Coors Analytical Laboratory, Golden, Colorado. Grout samples were dried to a constant weight at 105°C ± 2°C prior to testing. Testing was conducted at nominal intrusion pressures of 0.5 psia to 60,000 psia, corresponding to pore diameters from 375 μm to 0.003 μm.

AMERICAN NUCLEAR SOCIETY (ANS 16.1) LEACH TEST

The ANS 16.1 leach test (ANS 1986) was used to determine the effects of curing temperature and curing time on leachability. The test is an intermittent leachate exchange test designed to simulate a dynamic leaching situation. Cylindrical samples were suspended by nylon monofilament in deionized water within polyethylene containers. The leachant volume-to-sample surface area ratio used was 10 cm. The ANS leach tests were conducted for 28 to 35 days. After the elapsed time periods, the samples were removed from the

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(a) Instron Corporation, Canton, Massachusetts leachates and placed into containers containing fresh leachant. The pH of the leachates was measured immediately after removing the samples. Aliquots of leachate were filtered through a 0.45 µm filter and then submitted for cation analysis by ICP and for anion analysis by ion chromatography (IC).

The ANS 16.1 leach test was used in these studies to determine the relative leaching resistance for major cations and anions and to determine changes in leachability due to different curing conditions. Also, changes in the leaching behavior can provide some insight into physical and chemical changes that may be occurring in the grout. The ANS 16.1 leach test is designed to determine a "figure of merit" parameter called the leachability index (L). The leachability index for a given species is defined as the negative logarithm (base 10) of the effective diffusion coefficient (D) of that species. When less than 20% of a given species is leached, the effective diffusivity is given by (ANS 1986):

$$D = \pi \left[\frac{(a_n/A_0)}{(\Delta t)_n}\right]^2 \left[\frac{V}{S}\right]^2 T$$

where D = effective diffusivity, cm^2/s
a_n = concentration of ion released from the specimen during the leaching interval n
A_o = total amount of species in the specimen at the beginning of the leach test
$(\Delta t)_n$ = $t_n - t_{n-1}$, duration of the n-th leaching interval, s
V = volume of specimen, cm^3
S = geometric surface area of specimen, cm^2
T = $[1/2\ (\sqrt{t_n} + \sqrt{t_{n-1}})]^2$, representing the "mean time" of the leaching interval, s.

When greater than 20% of the total inventory of a species is leached, the effective diffusivity is calculated by:

$$D = \frac{Gd^2}{t}$$

where D = effective diffusivity, cm^2/s
G = dimensionless time factor for cylinder
d = cylinder diameter, cm
t = elapsed leaching time from beginning of test, s.

RESULTS AND DISCUSSION

This section presents the results of tests conducted to determine the effects of varying conditions and compositions on properties of DSSF grouts.

SLURRY PROPERTIES

Slurry density and viscometry measurements were conducted on all grout slurry samples to calculate the critical flow rate, i.e., the minimum flow rate required to attain turbulent flow in a nominal 2-in.-dia. pipe. The results of the density measurements and critical flow rate calculations are shown in Table 6 for the matrix-1 samples and in Table 7 for the matrix-2 samples. The data in Table 6 clearly show the dependence of waste dilution on the grout slurry densities, with the grouts prepared with diluted DSSF having the lowest densities. The critical flow rates also follow similar trends, with undiluted DSSF resulting in grouts with the highest values. The dry blend parameter having the greatest effect on both density and critical flow rate was the fly ash/cement ratio, i.e., high fly ash contents resulted in lower densities and lower critical flow rates. This is evidenced in both Table 6 and Table 7.

The amount of 28-day drainable liquids is also very much dependent on the waste dilution, as seen in Table 6. All grouts prepared with undiluted DSSF had no drainable liquids. The grouts prepared with waste diluted 100 times had drainable liquid contents up to about 31 vol%. The amount of drainable liquids for the grouts prepared with diluted DSSF was lowest when the slag content was high, suggesting that the slag reacts faster than the fly ash to produce a rigid structure within the grout that minimizes particle settling. Only two matrix-2 samples contained small amounts (less than 1 vol%) of drainable liquids.

Additional discussions of slurry density, critical flow rate, and drainable liquid data are presented in the "Statistical Evaluation" section.

TABLE 6. Critical Flow Rate, Density, and Drainable Liquid Data for Matrix-1 DSSF Grout Samples (All samples were cured for 1 month)

Run No.	Trial No.	F/C	S/C	Dil.	Temp., °C	Slurry Density, lb/gal	Critical Flow Rate, gpm	Drainable Liquids, Vol%
15	1	0	0	1X	55	14.37	29.94	0.0
8	2	8	0	1X	55	13.66	12.45	0.0
7	3	0	8	1X	55	14.14	25.37	0.0
4	4	8	8	1X	55	13.82	15.91	0.0
6	5	0	0	100X	55	12.84	9.64	19.6
3	6	8	0	100X	55	12.19	6.27	30.9
2	7	0	8	100X	55	12.57	18.82	4.8
10	8	8	8	100X	55	12.32	8.68	15.2
18	9	0	0	1X	95	14.41	32.31	0.0
19	10	8	0	1X	95	13.58	12.93	0.0
14	11	0	8	1X	95	14.12	25.04	0.0
12	12	8	8	1X	95	13.79	17.15	0.0
11	13	0	0	100X	95	12.86	9.62	20.2
16	14	8	0	100X	95	12.23	6.25	30.2
17	15	0	8	100X	95	12.59	19.04	3.5
13	16	8	8	100X	95	12.34	9.46	15.3
1	17	4	4	10X	75	12.48	7.24	8.4
5	18	4	4	10X	75	12.43	8.60	7.9
9	19	4	4	10X	75	12.44	9.45	8.6

TABLE 7. Critical Flow Rate and Density Data for Matrix-2 DSSF Grout Samples[a]

Run No.	Trial No.	Dry Blend Composition, Wt%				Curing Conditions[b]	Critical Flow Rate, gpm	Slurry Density g/cm^3
		C	F	S	L			
10	1	3.6	28.2	28.2	40	D/95/3	18.42	13.99
2	2	11.1	88.9	0	0	W/95/3	12.89	13.53
8	3	11.1	0	88.9	0	W/95/1	43.50	14.35
12	4	5.8	47.1	47.1	0	W/55/1	21.98	13.86
4	5	3.6	28.2	28.2	40	D/55/1	14.78	14.06
11	6	11.1	88.9	0	0	D/55/3	14.37	13.44
7	7	60.0	0	0	40	D/95/1	23.22	14.45
5	8	100.0	0	0	0	W/55/3	38.76	14.70
6	9	6.7	0	53.3	40	D/95/3	20.92	14.31
9	10	6.7	53.3	0	40	W/95/1	13.12	13.76
1	11	6.7	0	53.3	40	W/55/3	24.09	14.25
3	12	100.0	0	0	0	D/55/1	28.69	14.68

(a) All grouts were prepared with undiluted DSSF

(b) X/YY/Z
Curing time, months
Temperature, °C
W = additional water added during curing
D = no additional water added

COMPRESSIVE STRENGTH, DENSITY, AND MOISTURE CONTENT

Unconfined compressive strength, bulk density, and evaporable water content data for the matrix-1 and matrix-2 DSSF grout samples are given in Tables 8 and 9, respectively. There does not appear to be a large effect from any of the parameters on the density of the Matrix-1 samples. Although dilution had a large effect on the slurry density, dilution also played a significant role in the amount of drainable liquids. The net effect was densification of the grout due to particle settling prior to grout setting. The data in Table 9 show a relationship for bulk density with dry blend composition. In general, the density increases with decreases in fly ash content and increases in cement and slag content. Limestone content and curing conditions do not have an effect on the density.

Compressive strength values shown in Table 8 indicate an effect from temperature on strength. Increased temperature results in higher strengths for those grouts with fly ash and without slag (F/C = 8 and S/C = 0), and lower strengths for the others. Dilution also has an effect on the strengths, particularly for the blends without fly ash and slag. For example, comparing trial numbers 1 and 5, dilution of the DSSF resulted in a grout strength of 1022 psi compared with only 370 psi for the grout prepared with undiluted DSSF. The compressive strengths of the matrix-2 samples are influenced by the presence of limestone in the grouts, with lower strengths for those samples containing limestone.

Additional discussions of compressive strength, density, and evaporable water data are presented in the "Statistical Evaluation" section.

TABLE 8. Bulk Density, Compressive Strength, and Evaporable Water Content Data for Matrix-1 DSSF Grout Samples (All samples were cured for 1 month)

Run No.	Trial No.	F/C	S/C	Dil.	Temp °C	Bulk Density, g/cm^3	Compressive Strength, psi	Evaporable Water Content, Wt%
15	1	0	0	1X	55	1.723	370	24.10
8	2	8	0	1X	55	1.664	371	27.19
7	3	0	8	1X	55	1.707	973	24.22
4	4	8	8	1X	55	1.671	613	26.44
6	5	0	0	100X	55	1.713	1022	23.76
3	6	8	0	100X	55	1.699	85	24.43
2	7	0	8	100X	55	1.546	504	31.98
10	8	8	8	100X	55	1.592	501	30.09
18	9	0	0	1X	95	1.735	359	23.57
19	10	8	0	1X	95	1.671	254	26.75
14	11	0	8	1X	95	1.706	348	24.34
12	12	8	8	1X	95	1.680	367	26.31
11	13	0	0	100X	95	1.708	613	24.63
16	14	8	0	100X	95	1.717	236	24.19
17	15	0	8	100X	95	1.536	373	33.28
13	16	8	8	100X	95	1.576	358	30.64
1	17	4	4	10X	75	1.550	254	31.88
5	18	4	4	10X	75	1.558	260	32.33
9	19	4	4	10X	75	1.555	269	32.03

TABLE 9. Bulk Density, Compressive Strength, and Evaporable Water Content Data for Matrix 2 DSSF Grout Samples

Run No.	Trial No.	Dry Blend Composition, Wt% C	F	S	L	Curing Conditions(a)	Compressive Strength psi	Bulk Density g/cm3	Evaporable Water Content Wt%
10	1	3.6	28.2	28.2	40	D/95/3	212	1.695	33.9
2	2	11.1	88.9	0	0	W/95/3	1512	1.629	34.5
8	3	11.1	0	88.9	0	W/95/1	1330	1.719	29.3
12	4	5.8	47.1	47.1	0	W/55/1	1292	1.649	33.6
4	5	3.6	28.2	28.2	40	D/55/1	899	1.679	33.5
11	6	11.1	88.9	0	0	D/55/3	1449	1.619	34.2
7	7	60.0	0	0	40	D/95/1	361	1.741	31.3
5	8	100.0	0	0	0	W/55/3	878	1.754	28.9
6	9	6.7	0	53.3	40	D/95/3	307	1.709	31.8
9	10	6.7	53.3	0	40	W/95/1	244	1.665	35.2
1	11	6.7	0	53.3	40	W/55/3	653	1.709	31.4
3	12	100.0	0	0	0	D/55/1	750	1.760	29.9

(a) X/YY/Z
Curing time, months
Temperature, °C
W = additional water added during curing
D = no additional water added

LEACHABILITY

ANS 16.1 leach tests were conducted on DSSF grout samples cured for 1 or 3 months. These tests were conducted to determine relative changes in the leach behavior of DSSF grout as a function of various parameters, rather than to provide direct data for assessing the performance of the grout disposal system. Also, changes in the leaching behavior can provide insight into physical and chemical changes that may be occurring within the grout. The grout samples were leached for 28 to 35 days in deionized water. The leachates were analyzed by ICP for cations and IC for anions. Nitrate and sodium data were evaluated because they were expected to remain mostly within the liquid phase in the grout, where their release would be controlled by diffusion.

In the calculation of effective diffusion coefficients, the original inventory of species present in the grout samples (A_0) was calculated from analytical data for the starting waste and the dry blend constituents. The following discussion focuses primarily on NO_3 and Na release from the grout samples. A summary of the leachability indices for Na and NO_3 for the DSSF grout samples is shown in Tables 10 and 11 for the Matrix-1 and Matrix-2 samples, respectively. The lowest leachability indices for nitrate and sodium occur for the samples made with undiluted DSSF and cured at 95°C, as seen in Table 10. Temperature had the greatest effect on the leachability of those samples that contained equal amounts of slag and fly ash (F/C = 8 and S/C = 8).

The effects of temperature and waste dilution on the nitrate leachability are further illustrated in Figures 1 through 4, which show the cumulative fraction nitrate leached as a function of the square root of time. The labels in these figures (i.e., Slag, Cement, Fly Ash, and Blend) represent the solids most predominant in the dry blend. For example, "Slag" represents a dry blend of 6.7 wt% cement, 40 wt% limestone flour, and 53.3 wt% slag. "Blend" is close to the reference dry blend and consists of 3.6 wt% cement, 28.2 wt% slag, 28.2 wt% fly ash, and 40 wt% limestone flour. The curves in Figure 1 illustrate the poor leach resistance of grouts prepared with the various blends using full strength DSSF and curing at 95°C. In all cases, greater than 60% of the original NO_3 was leached out of the samples in less than 2 days. These results are consistent with previous data (Lokken et al. 1992a). The rate of NO_3 release from all these grouts decreased dramatically after about 3 days as the inventory in the larger accessible pores became depleted.

The effect of curing grouts prepared with undiluted DSSF at 55°C on the nitrate leachability is shown in Figure 2. The nitrate leachability is highest for the grouts made with with cement and high fly ash blends and was similar to those cured at 95°C. The effect of lower curing temperatures is most pronounced for the high slag blend and the blend with equal slag and fly ash contents. The amount of nitrate leached after 28 days decreased from about 70% for the 95°C-cured sample to about 20% for the 55°C-cured sample.

Figure 3 illustrates the effects of dry blend on the nitrate leachability for grouts prepared with diluted DSSF and cured at 95°C. The fraction of nitrate leached from these samples is less than for those prepared with undiluted DSSF (Figure 1), especially for the blend and high-slag grouts. The percentage of nitrate leached from the high-slag grout prepared with undiluted DSSF was nearly 90%, compared to only 10% from the grout prepared with diluted DSSF.

Figure 4 shows the cumulative fraction nitrate leached from grout samples prepared with diluted DSSF and cured at 55°C. The high fly ash grout released nitrate the fastest, with greater than 50% being leached after 1 day. The blend and high-slag grouts retained nitrate the best, with less than 10% being leached after 28 days of leaching. The lower curing temperature was beneficial in decreasing nitrate leachability for the blend and the high-cement grouts, but was detrimental for the high-fly ash grout.

Temperature also had a large influence on the nitrate and sodium leachability for the Matrix-2 samples cured for 1 month, as seen in Table 11 and Figure 5. The 55°C-cured samples are represented by trial numbers 4, 5, and 12. After 3 months of curing, the temperature effect was smaller (see Figure 6). The 55°C-cured samples in Figure 6 are trial numbers 6, 8, and 11.

TABLE 10. Average NO_3 and Na Leachability Indices for Matrix-1 DSSF Grout Samples Leached by the ANS 16.1 Procedure (All samples were cured for 1 month)

Run No.	Trial No.	F/C	S/C	Dil.	Temp °C	Leachability Index NO_3	SD[(a)]	Na	SD
15	1	0	0	1X	55	6.43	0.14	6.29	0.10
8	2	8	0	1X	55	6.57	0.23	6.99	0.22
7	3	0	8	1X	55	7.17	0.03	7.08	0.05
4	4	8	8	1X	55	8.11	0.10	8.48	0.82
6	5	0	0	100X	55	7.22	0.21	6.72	0.35
3	6	8	0	100X	55	6.42	0.49	7.75	0.38
2	7	0	8	100X	55	9.04	0.69	9.25	0.41
10	8	8	8	100X	55	9.55	0.66	10.08	0.35
18	9	0	0	1X	95	6.30	0.32	6.01	0.29
19	10	8	0	1X	95	6.25	0.51	6.96	0.41
14	11	0	8	1X	95	6.14	0.34	6.08	0.31
12	12	8	8	1X	95	6.32	0.40	6.69	0.37
11	13	0	0	100X	95	7.01	0.23	6.53	0.40
16	14	8	0	100X	95	6.65	0.49	7.68	0.33
17	15	0	8	100X	95	8.95	0.43	8.48	0.26
13	16	8	8	100X	95	8.30	0.24	8.92	0.19
1	17	4	4	10X	75	8.78	0.47	8.78	0.39
5	18	4	4	10X	75	9.03	0.37	8.92	0.28
9	19	4	4	10X	75	9.07	0.27	8.88	0.25

(a) SD = one standard deviation of 7 leaching intervals

TABLE 11. Average NO_3 and Na Leachability Indices for Matrix-2 DSSF Grout Samples Leached by the ANS 16.1 Procedure

Run No.	Trial No.	Dry Blend Composition, Wt%				Curing Conditions[a]	Leachability Index			
		C	F	S	L		NO_3	SD[b]	Na	SD
10	1	3.6	28.2	28.2	40	D/95/3	6.35	0.46	6.61	0.41
2	2	11.1	88.9	0	0	W/95/3	6.89	0.16	7.46	0.17
8	3	11.1	0	88.9	0	W/95/1	6.32	0.28	6.36	0.27
12	4	5.8	47.1	47.1	0	W/55/1	8.39	0.11	8.44	0.11
4	5	3.6	28.2	28.2	40	D/55/1	7.95	0.06	7.95	0.09
11	6	11.1	88.9	0	0	D/55/3	7.07	0.12	7.49	0.14
7	7	60.0	0	0	40	D/95/1	6.24	0.37	6.00	0.33
5	8	100.0	0	0	0	W/55/3	7.09	0.04	7.01	0.04
6	9	6.7	0	53.3	40	D/95/3	6.12	0.39	6.12	0.35
9	10	6.7	53.3	0	40	W/95/1	6.41	0.49	6.76	0.45
1	11	6.7	0	53.3	40	W/55/3	6.63	0.09	6.67	0.08
3	12	100.0	0	0	0	D/55/1	7.30	0.02	6.95	0.02

(a) X/YY/Z
Curing time, months
Temperature, °C
W = additional water added during curing
D = no additional water added

(b) SD = one standard deviation of 7 leaching intervals

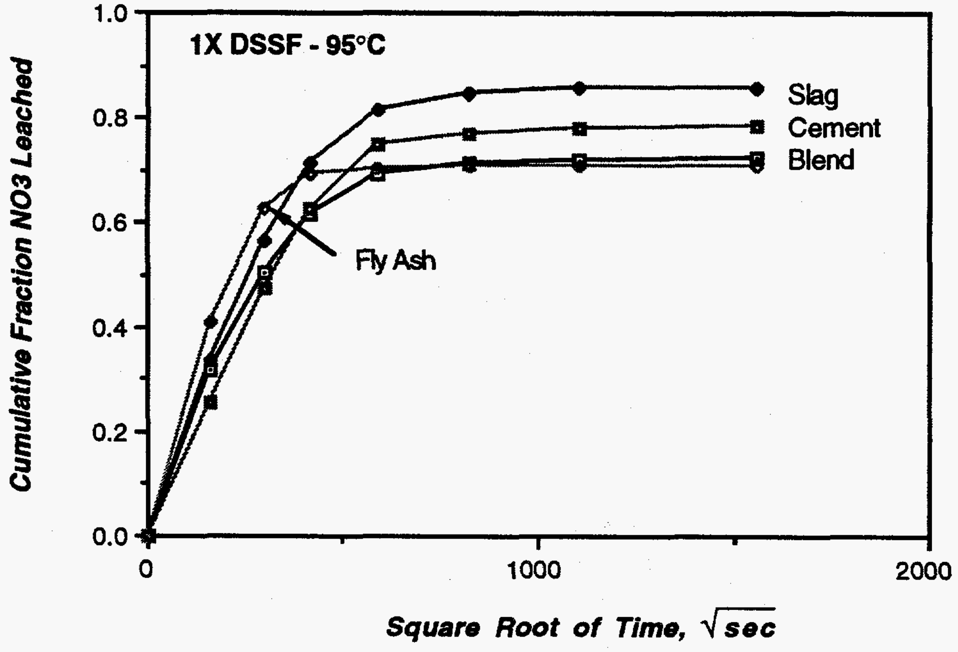

FIGURE 1. Cumulative Fraction of Nitrate Leached from DSSF Grout Samples Prepared with Full-Strength DSSF and Cured at 95°C

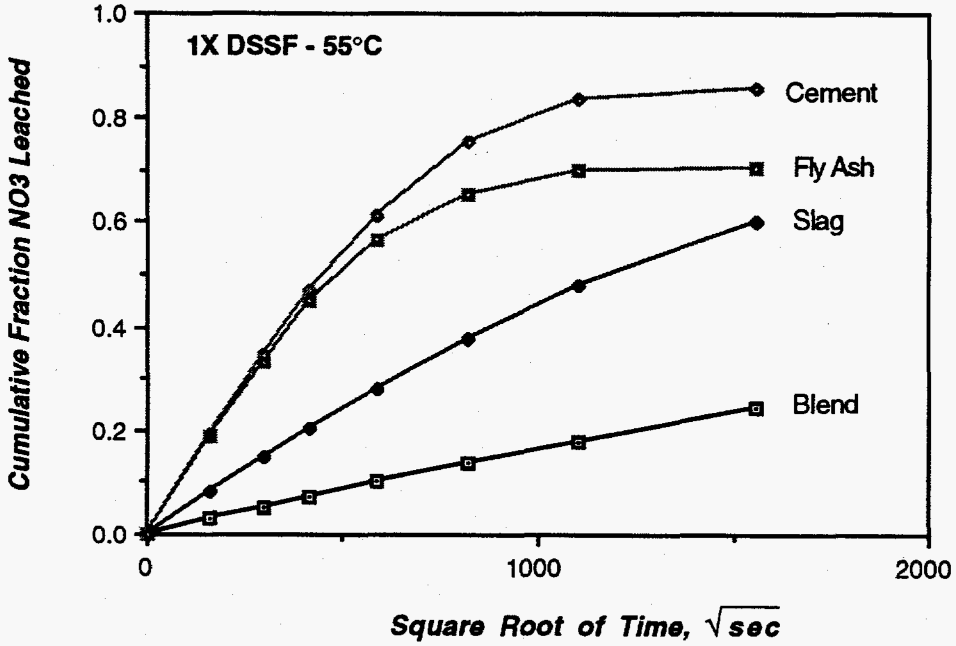

FIGURE 2. Cumulative Fraction of Nitrate Leached from DSSF Grout Samples Prepared with Full-Strength DSSF and Cured at 55°C

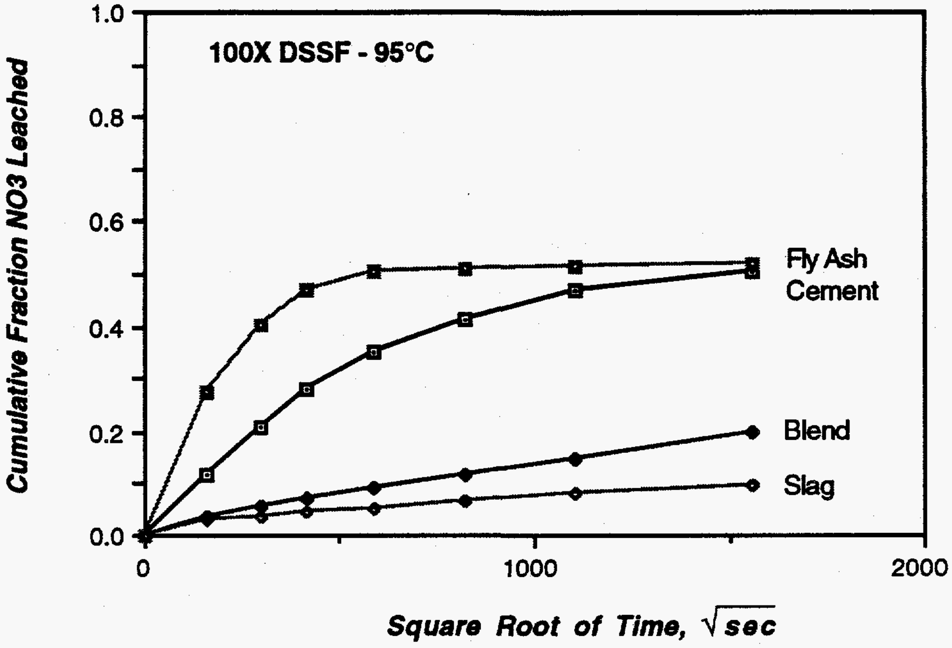

FIGURE 3. Cumulative Fraction of Nitrate Leached from DSSF Grout Samples Prepared with Dilute DSSF and Cured at 95°C

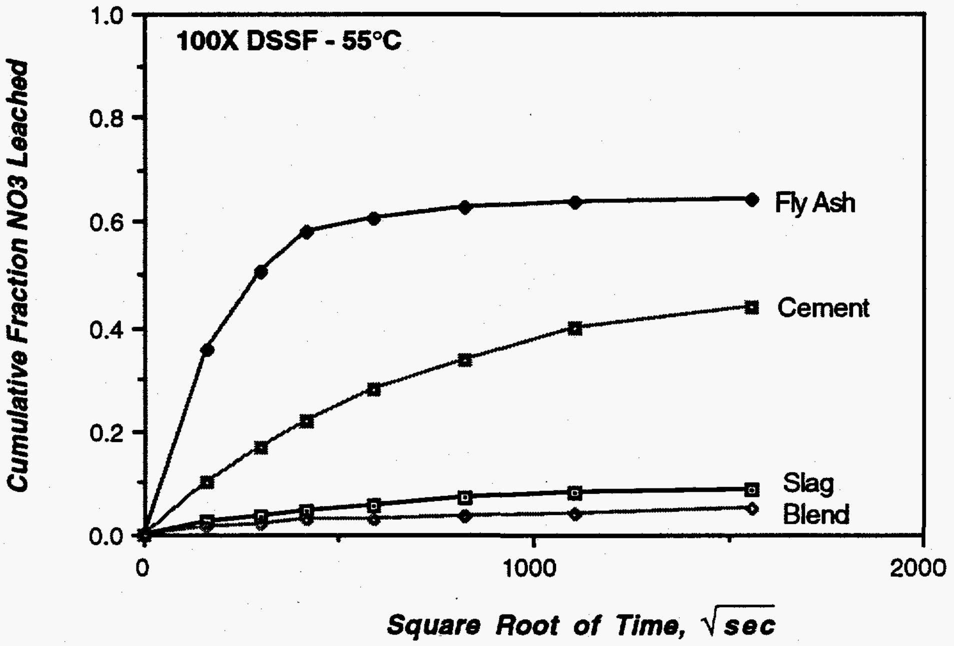

FIGURE 4. Cumulative Fraction of Nitrate Leached from DSSF Grout Samples Prepared with Dilute DSSF and Cured at 55°C

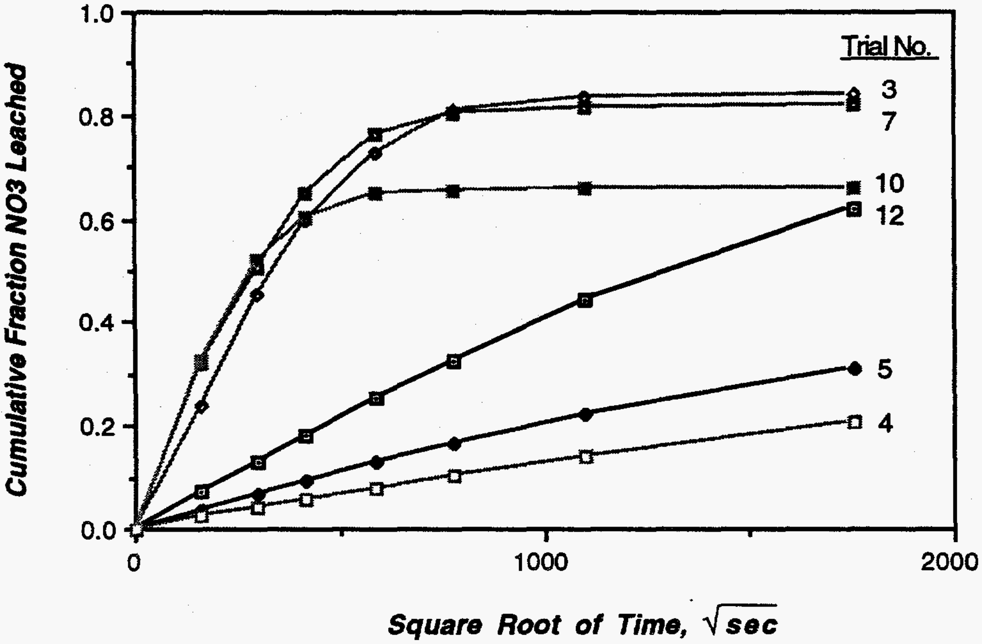

FIGURE 5. Cumulative Fraction of Nitrate Leached from Matrix-2 DSSF Grout Samples Cured for 1 Month

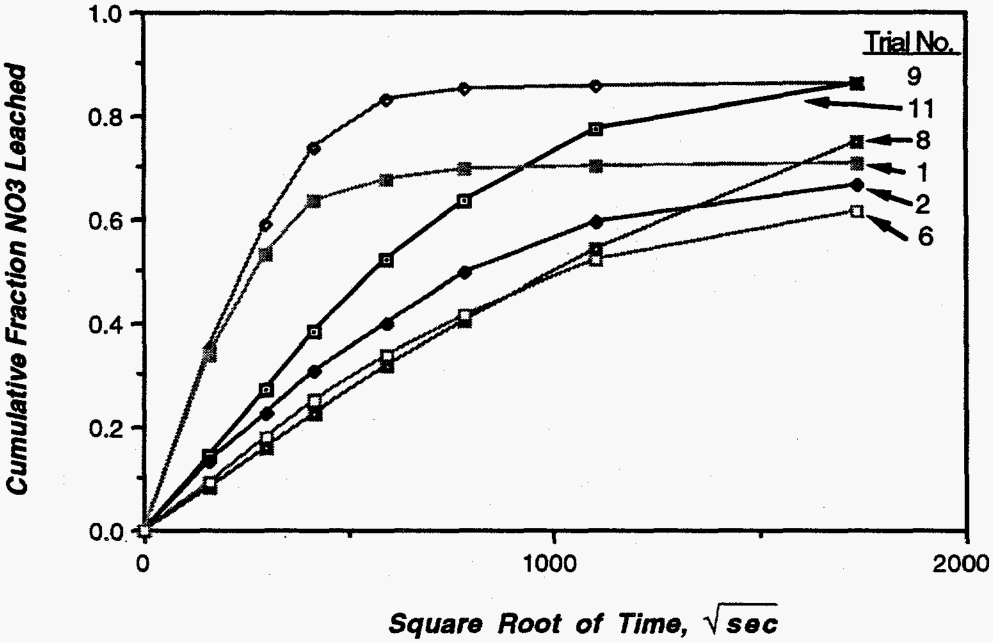

FIGURE 6. Cumulative Fraction of Nitrate Leached from Matrix-2 DSSF Grout Samples Cured for 3 Months

PORE SIZE DISTRIBUTION

Mercury intrusion porosimetry was conducted on selected grout samples from the matrix-1 group to determine whether the large changes in leach resistance could be correlated with changes in the distribution of pores within the grout. Figures 7 and 8 show the cumulative volume intruded vs. pore diameter for grout samples prepared with undiluted DSSF and cured at 55°C and 95°C, respectively. There do not appear to be any large, clear correlations between the nitrate leachability (Figures 1 and 2) and the pore size distributions. For example, comparing the curves of the cement and the fly ash grouts in Figure 2 shows a relatively small difference in the amount of nitrate leached. This would suggest similar pore size distributions for these two grouts; however, as seen in Figure 7, the fly ash grout contains a greater number of larger pores and more total intruded volume than the cement grout. Except for the fly ash grouts, increased curing temperature did not appear to alter the pore size distributions (see Figure 8). The pore size distribution of the fly ash grout shifted toward larger pores as curing temperature increased, i.e., the curve shifted to the left.

Figure 9 shows the pore size distribution of DSSF grout samples prepared with 3.5 wt% cement, 28.2 wt% slag, 28.2 wt% fly ash, and 40 wt% limestone flour. Waste dilution has a large influence on the resultant pore size distributions of these grouts. For grouts prepared with diluted DSSF, increased curing temperature resulted in a shift in the pore size distribution toward slightly larger pores and increased total intruded volume. Based on these data, it is suspected that the measured pore sizes of the grouts prepared with undiluted DSSF is not representative of the actual pores that would contain dissolved species during the leach tests. The samples for porosimetry were dried prior to testing, resulting in the precipitation of salts which may have filled some of the larger pores. One possible method of obtaining more representative pore size distributions in the future would be to leach the salts from the grouts prior to testing. A potential problem with this method, however, is that the structure of the grout matrix could change, depending on the solubility of the reaction products.

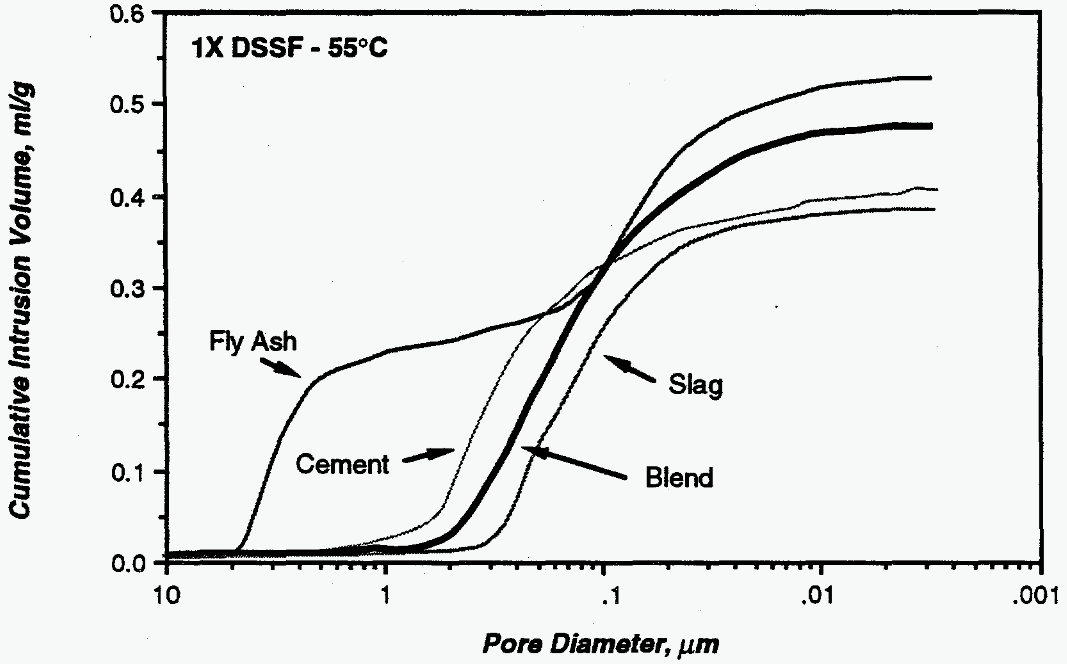

FIGURE 7. Pore Size Distribution of DSSF Grout Samples Prepared with Undiluted DSSF and Cured at 55°C

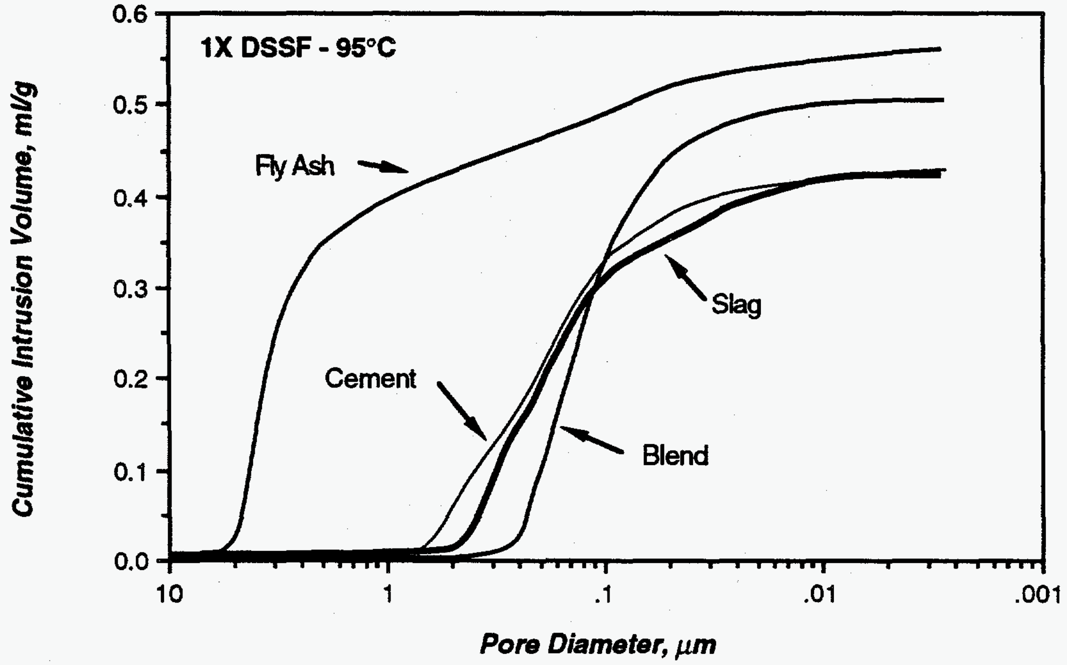

FIGURE 8. Pore Size Distribution of DSSF Grout Samples Prepared with Undiluted DSSF and Cured at 95°C

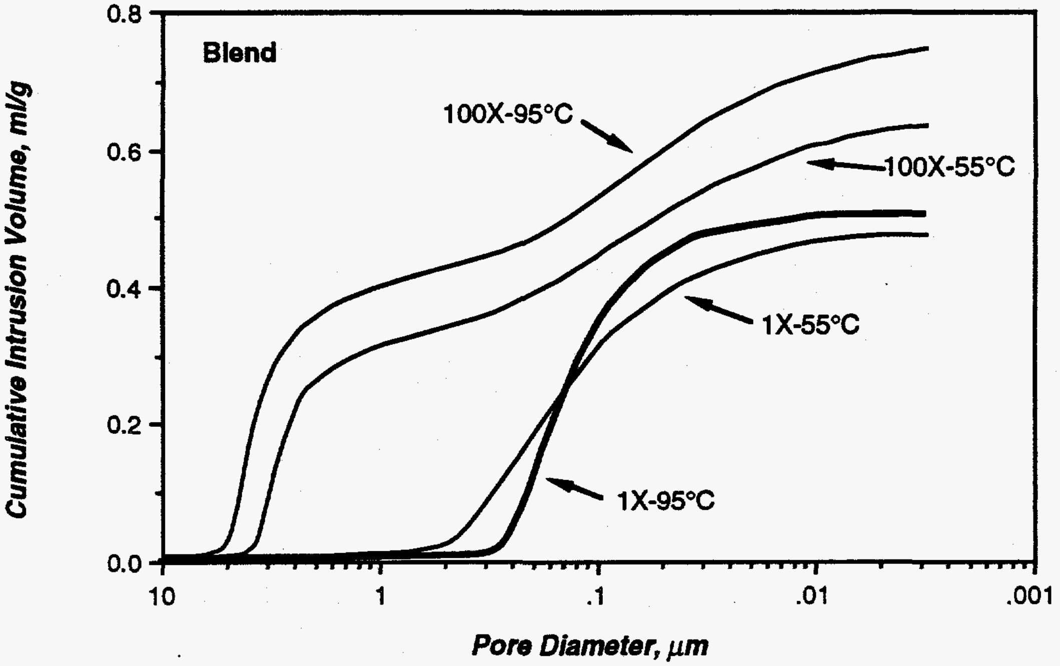

FIGURE 9. Pore Size Distribution of DSSF Grout Samples Prepared with 3.6 wt% Cement, 28.2 wt% Fly Ash, 28.2 wt% Slag, and 40 wt% Limestone Flour

STATISTICAL EVALUATION

The experimental matrices used in these studies were designed to allow statistical evaluation of the data using multiple regression analysis. Matrix 1 was a two-level, fourth-order factorial design for determining the main effects of fly ash-to-cement ratio, slag-to-cement ratio, DSSF waste dilution, and curing temperature on selected properties of the grouts. This design also allows the determination of interactions between factors and, because three replicates of the center of the factor space were done, an estimate of curvature (i.e., deviation from linearity) can be made. Matrix 2 was a Plackett-Burman screening design, which allows only main effects to be estimated.

The results of the statistical evaluation of the matrix-1 data are listed in Table 12. The effects listed are significant at a 95% confidence level. Unassigned factor effects of the three-way and four-way interactions were used to determine the confidence intervals. The main effects of F/C, S/C, dilution, and temperature are listed in the first four columns and the two-way interactions between these factors are listed in the last six columns. The effects are listed in order of magnitude, with a "1" having the largest effect, and whether the effect was positive or negative. Dilution had the largest effect on the slurry properties, i.e., critical flow rate, slurry density, and drainable liquids. The fly ash/cement ratio also had significant effects on the slurry properties. There were also two 2-way interactions affecting the slurry density and drainable liquids, the most significant including dilution. There were no significant effects (at the 95% confidence level) of any of the factors on the compressive strength values, primarily because the error values were large. Bulk density and evaporable water content were affected by the same factors and two-way interactions, but in opposite directions. The slag/cement ratio and dilution had the largest effect on the sodium and nitrate leachability. These properties were the only ones in which curing temperature had a significant effect. There were also two-way interactions between S/C and dilution and between dilution and temperature. These effects and the interactions were also evident in Figures 1 to 4, as discussed above. Based on the above discussions, the two most significant factors affecting the grout properties were the slag/cement ratio and waste dilution. Interactions between these two factors were also significant, indicating that reactions between the slag and the waste appear to dominate the properties of DSSF grouts. A summary of the multiple regression analyses is listed in the Appendix.

An analysis was also conducted on the matrix-2 data; however, very few significant effects were observed. A summary of the multiple regression analyses is listed in the Appendix.

TABLE 12. Results from the Statistical Evaluation of Main Effects and Two-Way Interactions for Matrix-1 Data(a)

	F/C	S/C	Dil	Temp	Interactions					
	X1	X2	X3	X4	X1X2	X1X3	X1X4	X2X3	X2X4	X3X4
Critical Flow Rate	2(b) -(c)		1 -							
Slurry Density	2 -	4 -	1 -		3 +	5 +				
Drainable Liquid	3 +	2 -	1 +			3 +		2 -		
Compressive Strength										
Bulk Density	6 -	1 -	3 -		5 +	4 +		2 -		
Evaporable Water	6 +	2 +	3 +		5 -	4 -		1 +		
NO_3 Leach Index		1 +	2 +	4 -				3 +	5 -	
Na Leach Index	3 +	2 +	1 +	5 -				4 +	6 -	

(a) Effects are significant at a 95% confidence level. Effects were calculated using a 2-level, 4th order factorial design. Unassigned factor effects using three-way and four-way interactions were used to determine confidence intervals.

(b) Numbers represent the order of the effect, i.e., 1 represents the largest effect

(c) + represents a positive effect; - represents a negative effect

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APPENDIX

SUMMARIES OF THE
MULTIPLE REGRESSION ANALYSES

Multiple Regression Y_1:CFR 4 X variables

Count:	R:	R-squared:	Adj. R-squared:	RMS Residual:
16	.914	.836	.777	3.93

Analysis of Variance Table

Source	DF:	Sum Squares:	Mean Square:	F-test:
REGRESSION	4	867.26	216.815	14.04
RESIDUAL	11	169.875	15.443	p = .0003
TOTAL	15	1037.135		

Residual Information Table

SS[e(i)-e(i-1)]:	e ≥ 0:	e < 0:	DW test:
296.54	8	8	1.746

Multiple Regression Y_1:CFR 4 X variables

Beta Coefficient Table

Variable:	Coefficient:	Std. Err.:	Std. Coeff.:	t-Value:	Probability:
INTERCEPT	24.175				
F/C	-1.261	.246	-.626	5.133	.0003
S/C	.313	.246	.156	1.276	.2282
Dil	-.105	.02	-.647	5.301	.0003
Temp	.015	.049	.037	.3	.7696

Multiple Regression Y_1:CFR 4 X variables

Confidence Intervals and Partial F Table

Variable:	95% Lower:	95% Upper:	90% Lower:	90% Upper:	Partial F:
INTERCEPT					
F/C	-1.801	-.72	-1.702	-.819	26.344
S/C	-.227	.854	-.128	.755	1.629
Dil	-.149	-.062	-.141	-.07	28.096
Temp	-.093	.123	-.073	.103	.09

TABLE A1. Multiple Regression Analysis for Critical Flow Rate for the Matrix-1 Grout Samples

Multiple Regression Y_2:Slurry Density 4 X variables

Count:	R:	R-squared:	Adj. R-squared:	RMS Residual:
16	.99	.981	.974	.132

Analysis of Variance Table

Source	DF:	Sum Squares:	Mean Square:	F-test:
REGRESSION	4	9.923	2.481	141.374
RESIDUAL	11	.193	.018	p = .0001
TOTAL	15	10.116		

Residual Information Table

SS[e(i)-e(i-1)]:	$e \geq 0$:	$e < 0$:	DW test:
.404	8	8	2.091

Multiple Regression Y_2:Slurry Density 4 X variables

Beta Coefficient Table

Variable:	Coefficient:	Std. Err.:	Std. Coeff.:	t-Value:	Probability:
INTERCEPT	14.275				
F/C	-.062	.008	-.312	7.493	.0001
S/C	-.007	.008	-.035	.849	.4138
Dil	-.015	.001	-.939	22.553	.0001
Temp	3.125E-5	.002	.001	.019	.9853

Multiple Regression Y_2:Slurry Density 4 X variables

Confidence Intervals and Partial F Table

Variable:	95% Lower:	95% Upper:	90% Lower:	90% Upper:	Partial F:
INTERCEPT					
F/C	-.08	-.044	-.077	-.047	56.138
S/C	-.025	.011	-.022	.008	.721
Dil	-.017	-.014	-.016	-.014	508.638
Temp	-.004	.004	-.003	.003	3.562E-4

TABLE A2. Multiple Regression Analysis for Slurry Density for the Matrix-1 Grout Samples

Multiple Regression Y_3:Shrinkage 4 X variables

Count:	R:	R-squared:	Adj. R-squared:	RMS Residual:
16	.993	.987	.982	.579

Analysis of Variance Table

Source	DF:	Sum Squares:	Mean Square:	F-test:
REGRESSION	4	276.75	69.188	206.39
RESIDUAL	11	3.687	.335	p = .0001
TOTAL	15	280.438		

Residual Information Table

SS[e(i)-e(i-1)]:	e ≥ 0:	e < 0:	DW test:
1.906	7	9	.517

Multiple Regression Y_3:Shrinkage 4 X variables

Beta Coefficient Table

Variable:	Coefficient:	Std. Err.:	Std. Coeff.:	t-Value:	Probability:
INTERCEPT	-8.987				
F/C	.109	.036	.105	3.023	.0116
S/C	.203	.036	.194	5.613	.0002
Dil	.034	.003	.403	11.658	.0001
Temp	.184	.007	.881	25.475	.0001

Multiple Regression Y_3:Shrinkage 4 X variables

Confidence Intervals and Partial F Table

Variable:	95% Lower:	95% Upper:	90% Lower:	90% Upper:	Partial F:
INTERCEPT					
F/C	.03	.189	.044	.174	9.136
S/C	.123	.283	.138	.268	31.508
Dil	.028	.041	.029	.039	135.915
Temp	.168	.2	.171	.197	649

TABLE A3. Multiple Regression Analysis for Shrinkage for the Matrix-1 Grout Samples

Multiple Regression Y_4:Drainables 4 X variables

Count:	R:	R-squared:	Adj. R-squared:	RMS Residual:
16	.902	.814	.747	5.725

Analysis of Variance Table

Source	DF:	Sum Squares:	Mean Square:	F-test:
REGRESSION	4	1579.152	394.788	12.044
RESIDUAL	11	360.562	32.778	p = .0005
TOTAL	15	1939.714		

Residual Information Table

SS[e(i)-e(i-1)]:	e ≥ 0:	e < 0:	DW test:
935.914	8	8	2.596

Multiple Regression Y_4:Drainables 4 X variables

Beta Coefficient Table

Variable:	Coefficient:	Std. Err.:	Std. Coeff.:	t-Value:	Probability:
INTERCEPT	1.291				
F/C	.68	.358	.247	1.899	.084
S/C	-.97	.358	-.353	2.712	.0202
Dil	.176	.029	.793	6.1	.0001
Temp	-.004	.072	-.007	.057	.9557

Multiple Regression Y_4:Drainables 4 X variables

Confidence Intervals and Partial F Table

Variable:	95% Lower:	95% Upper:	90% Lower:	90% Upper:	Partial F:
INTERCEPT					
F/C	-.108	1.467	.037	1.322	3.608
S/C	-1.758	-.183	-1.613	-.328	7.353
Dil	.113	.24	.124	.228	37.212
Temp	-.162	.153	-.133	.124	.003

TABLE A4. Multiple Regression Analysis for Drainable Liquids for the Matrix-1 Grout Samples

Multiple Regression Y_5:CS 4 X variables

Count:	R:	R-squared:	Adj. R-squared:	RMS Residual:
16	.637	.406	.19	223.828

Analysis of Variance Table

Source	DF:	Sum Squares:	Mean Square:	F-test:
REGRESSION	4	376974.25	94243.562	1.881
RESIDUAL	11	551088.188	50098.926	p = .184
TOTAL	15	928062.438		

Residual Information Table

SS[e(i)-e(i-1)]:	$e \geq 0$:	$e < 0$:	DW test:
1032989.219	9	7	1.874

Multiple Regression Y_5:CS 4 X variables

Beta Coefficient Table

Variable:	Coefficient:	Std. Err.:	Std. Coeff.:	t-Value:	Probability:
INTERCEPT	881.281				
F/C	-27.766	13.989	-.461	1.985	.0727
S/C	11.359	13.989	.189	.812	.434
Dil	.047	1.13	.01	.041	.9678
Temp	-4.784	2.798	-.397	1.71	.1153

Multiple Regression Y_5:CS 4 X variables

Confidence Intervals and Partial F Table

Variable:	95% Lower:	95% Upper:	90% Lower:	90% Upper:	Partial F:
INTERCEPT					
F/C	-58.559	3.028	-52.891	-2.64	3.939
S/C	-19.434	42.153	-13.766	36.485	.659
Dil	-2.442	2.535	-1.984	2.077	.002
Temp	-10.943	1.374	-9.81	.241	2.924

TABLE A5. Multiple Regression Analysis for Compressive Strength for the Matrix-1 Grout Samples

Multiple Regression Y_6:Density 4 X variables

Count:	R:	R-squared:	Adj. R-squared:	RMS Residual:
16	.772	.596	.45	.048

Analysis of Variance Table

Source	DF:	Sum Squares:	Mean Square:	F-test:
REGRESSION	4	.038	.01	4.063
RESIDUAL	11	.026	.002	p = .0291
TOTAL	15	.064		

Residual Information Table

SS[e(i)-e(i-1)]:	$e \geq 0$:	$e < 0$:	DW test:
.055	9	7	2.136

Multiple Regression Y_6:Density 4 X variables

Beta Coefficient Table

Variable:	Coefficient:	Std. Err.:	Std. Coeff.:	t-Value:	Probability:
INTERCEPT	1.737				
F/C	-.002	.003	-.103	.536	.6025
S/C	-.01	.003	-.608	3.176	.0088
Dil	-.001	2.449E-4	-.464	2.423	.0338
Temp	4.375E-5	.001	.014	.072	.9438

Multiple Regression Y_6:Density 4 X variables

Confidence Intervals and Partial F Table

Variable:	95% Lower:	95% Upper:	90% Lower:	90% Upper:	Partial F:
INTERCEPT					
F/C	-.008	.005	-.007	.004	.288
S/C	-.016	-.003	-.015	-.004	10.088
Dil	-.001	-5.439E-5	-.001	-1.536E-4	5.873
Temp	-.001	.001	-.001	.001	.005

TABLE A6. Multiple Regression Analysis for Bulk Density for the Matrix-1 Grout Samples

Multiple Regression Y_7:Evaporable Water 4 X variables

Count:	R:	R-squared:	Adj. R-squared:	RMS Residual:
16	.723	.523	.35	2.56

Analysis of Variance Table

Source	DF:	Sum Squares:	Mean Square:	F-test:
REGRESSION	4	79.122	19.78	3.018
RESIDUAL	11	72.087	6.553	p = .0659
TOTAL	15	151.209		

Residual Information Table

SS[e(i)-e(i-1)]:	$e \geq 0$:	$e < 0$:	DW test:
158.223	8	8	2.195

Multiple Regression Y_7:Evaporable Water 4 X variables

Beta Coefficient Table

Variable:	Coefficient:	Std. Err.:	Std. Coeff.:	t-Value:	Probability:
INTERCEPT	22.811				
F/C	.096	.16	.125	.602	.5597
S/C	.448	.16	.583	2.801	.0172
Dil	.025	.013	.408	1.961	.0757
Temp	.005	.032	.03	.146	.8862

Multiple Regression Y_7:Evaporable Water 4 X variables

Confidence Intervals and Partial F Table

Variable:	95% Lower:	95% Upper:	90% Lower:	90% Upper:	Partial F:
INTERCEPT					
F/C	-.256	.448	-.191	.384	.362
S/C	.096	.8	.161	.735	7.845
Dil	-.003	.054	.002	.049	3.845
Temp	-.066	.075	-.053	.062	.021

TABLE A7. Multiple Regression Analysis for Evaporable Water Content for the Matrix-1 Grout Samples

Multiple Regression Y_8:NO3 Li 4 X variables

Count:	R:	R-squared:	Adj. R-squared:	RMS Residual:
16	.865	.749	.658	.666

Analysis of Variance Table

Source	DF:	Sum Squares:	Mean Square:	F-test:
REGRESSION	4	14.577	3.644	8.205
RESIDUAL	11	4.886	.444	p = .0026
TOTAL	15	19.463		

Residual Information Table

SS[e(i)-e(i-1)]:	e ≥ 0:	e < 0:	DW test:
8.86	10	6	1.813

Multiple Regression Y_8:NO3 Li 4 X variables

Beta Coefficient Table

Variable:	Coefficient:	Std. Err.:	Std. Coeff.:	t-Value:	Probability:
INTERCEPT	7.06				
F/C	-.001	.042	-.005	.034	.9737
S/C	.168	.042	.608	4.025	.002
Dil	.012	.003	.558	3.695	.0035
Temp	-.014	.008	-.26	1.722	.1131

Multiple Regression Y_8:NO3 Li 4 X variables

Confidence Intervals and Partial F Table

Variable:	95% Lower:	95% Upper:	90% Lower:	90% Upper:	Partial F:
INTERCEPT					
F/C	-.093	.09	-.076	.073	.001
S/C	.076	.259	.093	.242	16.201
Dil	.005	.02	.006	.018	13.653
Temp	-.033	.004	-.029	.001	2.965

TABLE A8. Multiple Regression Analysis for Nitrate Leachability Index for the Matrix-1 Grout Samples

Multiple Regression Yg:Na Li 4 X variables

Count:	R:	R-squared:	Adj. R-squared:	RMS Residual:
16	.913	.833	.773	.583

Analysis of Variance Table

Source	DF:	Sum Squares:	Mean Square:	F-test:
REGRESSION	4	18.653	4.663	13.743
RESIDUAL	11	3.732	.339	p = .0003
TOTAL	15	22.385		

Residual Information Table

SS[e(i)-e(i-1)]:	e ≥ 0:	e < 0:	DW test:
5.114	9	7	1.37

Multiple Regression Yg:Na Li 4 X variables

Beta Coefficient Table

Variable:	Coefficient:	Std. Err.:	Std. Coeff.:	t-Value:	Probability:
INTERCEPT	6.971				
F/C	.111	.036	.376	3.051	.011
S/C	.158	.036	.535	4.348	.0012
Dil	.014	.003	.572	4.648	.0007
Temp	-.017	.007	-.28	2.27	.0443

Multiple Regression Yg:Na Li 4 X variables

Confidence Intervals and Partial F Table

Variable:	95% Lower:	95% Upper:	90% Lower:	90% Upper:	Partial F:
INTERCEPT					
F/C	.031	.191	.046	.176	9.311
S/C	.078	.238	.093	.224	18.901
Dil	.007	.02	.008	.019	21.604
Temp	-.033	-.001	-.03	-.003	5.155

TABLE A9. Multiple Regression Analysis for Sodium Leachability Index for the Matrix-1 Grout Samples

Multiple Regression Y_1:CFR 6 X variables

Count:	R:	R-squared:	Adj. R-squared:	RMS Residual:
12	.904	.818	.599	6.243

Analysis of Variance Table

Source	DF:	Sum Squares:	Mean Square:	F-test:
REGRESSION	6	875.455	145.909	3.744
RESIDUAL	5	194.868	38.974	p = .0845
TOTAL	11	1070.323		

Residual Information Table

SS[e(i)-e(i-1)]:	e ≥ 0:	e < 0:	DW test:
557.453	5	7	2.861

Multiple Regression Y_1:CFR 6 X variables

Beta Coefficient Table

Variable:	Coefficient:	Std. Err.:	Std. Coeff.:	t-Value:	Probability:
INTERCEPT	30.84				
Fly Ash/Cement	-1.742	.451	-.738	3.867	.0118
Slag/Cement	.628	.487	.266	1.29	.2533
Limestone	-.219	.11	-.463	1.983	.1042
Water	.274	.389	.145	.704	.5131
Temperature	.029	.097	.061	.296	.7795
Time	-1.32	1.802	-.14	.732	.4968

Multiple Regression Y_1:CFR 6 X variables

Confidence Intervals and Partial F Table

Variable:	95% Lower:	95% Upper:	90% Lower:	90% Upper:	Partial F:
INTERCEPT					
Fly Ash/Cement	-2.9	-.584	-2.65	-.834	14.951
Slag/Cement	-.623	1.879	-.353	1.609	1.665
Limestone	-.503	.065	-.441	.004	3.93
Water	-.727	1.275	-.511	1.059	.495
Temperature	-.221	.279	-.167	.225	.087
Time	-5.953	3.313	-4.952	2.312	.536

TABLE A10. Multiple Regression Analysis for Critical Flow Rate for the Matrix-2 Grout Samples

Multiple Regression Y_2:Density 6 X variables

Count:	R:	R-squared:	Adj. R-squared:	RMS Residual:
12	.901	.811	.584	.267

Analysis of Variance Table

Source	DF:	Sum Squares:	Mean Square:	F-test:
REGRESSION	6	1.535	.256	3.576
RESIDUAL	5	.358	.072	p = .0917
TOTAL	11	1.893		

Residual Information Table

SS[e(i)-e(i-1)]:	e ≥ 0:	e < 0:	DW test:
.774	4	8	2.165

Multiple Regression Y_2:Density 6 X variables

Beta Coefficient Table

Variable:	Coefficient:	Std. Err.:	Std. Coeff.:	t-Value:	Probability:
INTERCEPT	14.826				
Fly Ash/Cement	-.085	.019	-.86	4.425	.0069
Slag/Cement	.003	.021	.032	.153	.8842
Limestone	.001	.005	.067	.282	.7893
Water	-.006	.017	-.078	.373	.7244
Temperature	-.003	.004	-.148	.706	.5117
Time	-.078	.077	-.197	1.015	.3569

Multiple Regression Y_2:Density 6 X variables

Confidence Intervals and Partial F Table

Variable:	95% Lower:	95% Upper:	90% Lower:	90% Upper:	Partial F:
INTERCEPT					
Fly Ash/Cement	-.135	-.036	-.124	-.047	19.581
Slag/Cement	-.05	.057	-.039	.045	.023
Limestone	-.011	.013	-.008	.011	.08
Water	-.049	.037	-.04	.027	.139
Temperature	-.014	.008	-.011	.005	.499
Time	-.277	.12	-.234	.077	1.029

TABLE A11. Multiple Regression Analysis for Slurry Density for the Matrix-2 Grout Samples

Multiple Regression Y_3:Compressive Strength 6 X variables

Count:	R:	R-squared:	Adj. R-squared:	RMS Residual:
12	.877	.769	.491	345.217

Analysis of Variance Table

Source	DF:	Sum Squares:	Mean Square:	F-test:
REGRESSION	6	1979014.472	329835.745	2.768
RESIDUAL	5	595874.444	119174.889	p = .1418
TOTAL	11	2574888.917		

Residual Information Table

SS[e(i)-e(i-1)]:	e ≥ 0:	e < 0:	DW test:
1246874.015	6	6	2.093

Multiple Regression Y_3:Compressive Strength 6 X variables

Beta Coefficient Table

Variable:	Coefficient:	Std. Err.:	Std. Coeff.:	t-Value:	Probability:
INTERCEPT	1086.417				
Fly Ash/Cement	27.688	24.914	.239	1.111	.317
Slag/Cement	21.569	26.91	.186	.802	.4592
Limestone	-19.204	6.103	-.829	3.147	.0255
Water	6.578	21.528	.071	.306	.7723
Temperature	-1.744	5.382	-.075	.324	.759
Time	11.25	99.656	.024	.113	.9145

Multiple Regression Y_3:Compressive Strength 6 X variables

Confidence Intervals and Partial F Table

Variable:	95% Lower:	95% Upper:	90% Lower:	90% Upper:	Partial F:
INTERCEPT					
Fly Ash/Cement	-36.365	91.74	-22.521	77.896	1.235
Slag/Cement	-47.615	90.754	-32.662	75.801	.642
Limestone	-34.894	-3.515	-31.503	-6.906	9.903
Water	-48.77	61.925	-36.808	49.963	.093
Temperature	-15.581	12.092	-12.591	9.102	.105
Time	-244.96	267.46	-189.585	212.085	.013

TABLE A12. Multiple Regression Analysis for Compressive Strength for the Matrix-2 Grout Samples

Multiple Regression Y_4:Bulk Density 6 X variables

Count:	R:	R-squared:	Adj. R-squared:	RMS Residual:
12	.885	.784	.524	.032

Analysis of Variance Table

Source	DF:	Sum Squares:	Mean Square:	F-test:
REGRESSION	6	.019	.003	3.017
RESIDUAL	5	.005	.001	p = .123
TOTAL	11	.024		

Residual Information Table

SS[e(i)-e(i-1)]:	e ≥ 0:	e < 0:	DW test:
.011	4	8	2.137

Multiple Regression Y_4:Bulk Density 6 X variables

Beta Coefficient Table

Variable:	Coefficient:	Std. Err.:	Std. Coeff.:	t-Value:	Probability:
INTERCEPT	1.761				
Fly Ash/Cement	-.009	.002	-.847	4.069	.0096
Slag/Cement	-.001	.003	-.06	.267	.8001
Limestone	3.042E-4	.001	.136	.532	.6176
Water	-.001	.002	-.1	.443	.676
Temperature	-1.514E-4	.001	-.067	.3	.7761
Time	-.008	.009	-.182	.874	.4219

Multiple Regression Y_4:Bulk Density 6 X variables

Confidence Intervals and Partial F Table

Variable:	95% Lower:	95% Upper:	90% Lower:	90% Upper:	Partial F:
INTERCEPT					
Fly Ash/Cement	-.016	-.003	-.014	-.005	16.556
Slag/Cement	-.007	.006	-.006	.004	.071
Limestone	-.001	.002	-.001	.001	.283
Water	-.006	.004	-.005	.003	.197
Temperature	-.001	.001	-.001	.001	.09
Time	-.032	.016	-.027	.011	.765

TABLE A13. Multiple Regression Analysis for Bulk Density for the Matrix-2 Grout Samples

Multiple Regression Y_5:Evap H2O 6 X variables

Count:	R:	R-squared:	Adj. R-squared:	RMS Residual:
12	.956	.914	.812	.934

Analysis of Variance Table

Source	DF:	Sum Squares:	Mean Square:	F-test:
REGRESSION	6	46.571	7.762	8.905
RESIDUAL	5	4.358	.872	p = .0149
TOTAL	11	50.929		

Residual Information Table

SS[e(i)-e(i-1)]:	e ≥ 0:	e < 0:	DW test:
11.682	8	4	2.68

Multiple Regression Y_5:Evap H2O 6 X variables

Beta Coefficient Table

Variable:	Coefficient:	Std. Err.:	Std. Coeff.:	t-Value:	Probability:
INTERCEPT	29.044				
Fly Ash/Cement	.465	.067	.902	6.895	.001
Slag/Cement	-.06	.073	-.117	.83	.4443
Limestone	.03	.017	.291	1.818	.1288
Water	.012	.058	.028	.2	.8491
Temperature	.009	.015	.085	.601	.574
Time	.158	.27	.077	.587	.5824

Multiple Regression Y_5:Evap H2O 6 X variables

Confidence Intervals and Partial F Table

Variable:	95% Lower:	95% Upper:	90% Lower:	90% Upper:	Partial F:
INTERCEPT					
Fly Ash/Cement	.291	.638	.329	.6	47.542
Slag/Cement	-.248	.127	-.207	.086	.689
Limestone	-.012	.072	-.003	.063	3.304
Water	-.138	.161	-.106	.129	.04
Temperature	-.029	.046	-.021	.038	.361
Time	-.535	.851	-.385	.701	.345

TABLE A14. Multiple Regression Analysis for Evaporable Water Content for the Matrix-2 Grout Samples

Multiple Regression Y_6:NO3 LI 6 X variables

Count:	R:	R-squared:	Adj. R-squared:	RMS Residual:
12	.936	.877	.729	.369

Analysis of Variance Table

Source	DF:	Sum Squares:	Mean Square:	F-test:
REGRESSION	6	4.862	.81	5.941
RESIDUAL	5	.682	.136	p = .0348
TOTAL	11	5.543		

Residual Information Table

SS[e(i)-e(i-1)]:	e ≥ 0:	e < 0:	DW test:
1.87	6	6	2.742

Multiple Regression Y_6:NO3 LI 6 X variables

Beta Coefficient Table

Variable:	Coefficient:	Std. Err.:	Std. Coeff.:	t-Value:	Probability:
INTERCEPT	8.769				
Fly Ash/Cement	.07	.027	.412	2.627	.0467
Slag/Cement	.03	.029	.176	1.037	.3471
Limestone	-.008	.007	-.248	1.289	.2537
Water	4.444E-4	.023	.003	.019	.9853
Temperature	-.023	.006	-.665	3.927	.0111
Time	-.205	.107	-.302	1.923	.1125

Multiple Regression Y_6:NO3 LI 6 X variables

Confidence Intervals and Partial F Table

Variable:	95% Lower:	95% Upper:	90% Lower:	90% Upper:	Partial F:
INTERCEPT					
Fly Ash/Cement	.001	.139	.016	.124	6.899
Slag/Cement	-.044	.104	-.028	.088	1.076
Limestone	-.025	.008	-.022	.005	1.662
Water	-.059	.06	-.046	.047	3.725E-4
Temperature	-.037	-.008	-.034	-.011	15.424
Time	-.479	.069	-.42	.01	3.698

TABLE A15. Multiple Regression Analysis for Nitrate Leachability Index for the Matrix-2 Grout Samples

Multiple Regression Y_7:Na LI 6 X variables

Count:	R:	R-squared:	Adj. R-squared:	RMS Residual:
12	.961	.923	.831	.301

Analysis of Variance Table

Source	DF:	Sum Squares:	Mean Square:	F-test:
REGRESSION	6	5.476	.913	10.044
RESIDUAL	5	.454	.091	p = .0114
TOTAL	11	5.93		

Residual Information Table

SS[e(i)-e(i-1)]:	e ≥ 0:	e < 0:	DW test:
1.316	6	6	2.896

Multiple Regression Y_7:Na LI 6 X variables

Beta Coefficient Table

Variable:	Coefficient:	Std. Err.:	Std. Coeff.:	t-Value:	Probability:
INTERCEPT	8.108				
Fly Ash/Cement	.117	.022	.664	5.363	.003
Slag/Cement	.026	.023	.146	1.091	.3252
Limestone	-.009	.005	-.267	1.759	.1388
Water	.014	.019	.098	.736	.4948
Temperature	-.019	.005	-.528	3.945	.0109
Time	-.092	.087	-.13	1.053	.3404

Multiple Regression Y_7:Na LI 6 X variables

Confidence Intervals and Partial F Table

Variable:	95% Lower:	95% Upper:	90% Lower:	90% Upper:	Partial F:
INTERCEPT					
Fly Ash/Cement	.061	.173	.073	.161	28.761
Slag/Cement	-.035	.086	-.022	.073	1.189
Limestone	-.023	.004	-.02	.001	3.095
Water	-.034	.062	-.024	.052	.542
Temperature	-.031	-.006	-.028	-.009	15.567
Time	-.315	.132	-.267	.084	1.11

TABLE A16. Multiple Regression Analysis for Sodium Leachability Index for the Matrix-2 Grout Samples

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