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Initial Formulation Results for In Situ Grouting of a Waste Trench at ORNL Site No. 6

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Chemical Technology Division

NUCLEAR AND CHEMICAL WASTE PROGRAMS
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INITIAL FORMULATION RESULTS FOR IN SITU GROUTING OF A WASTE TRENCH AT ORNL SITE NO. 6

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O. K. Tallent, E. W. McDaniel, R. D. Spence, and T. T. Godsey

ABSTRACT

An investigation is being conducted by the Chemical Technology Division to assist the Environmental Sciences Division in developing a grout formulation for use in testing in situ grouting in a waste trench at ORNL Site 6. This final report satifies the milestone of Subtask 12 entitled, "Low Level Waste (LLW) Trench Grouting Assessment," which was initially issued as RAP-86-7, December 31, 1985. Grouts prepared from dry-solid blends containing Type I Portland cement, ASTM Class C or Class F fly ash, and bentonite, mixed with water at ratios of 10 to 15 lb/gal, were evaluated. The grouts prepared with ASTM Class C fly ash exhibited significantly better properties than those prepared with ASTM Class F fly ash. The grouts containing ASTM Class C fly ash satisfy tentative performance criteria for the project.

1. INTRODUCTION

One of the major problems with shallow landfill trenches has been subsidence. In situ grouting would prevent subsidence by filling the large accessible voids in the trench with a cheap, coarse grout. This remedial action would also help to minimize water intrusion and to reduce the overall hydraulic conductivity of the trench. In turn, radionuclude migration would be retarded, hopefully to a negligible level. The objective of Subtask 1 for the "ORNL Site Correctives Measures Technology Development" program (ONLWN02) is to test an economical field-scale grouting that will prevent subsidence.

The Chemical Technology Division is assisting the Environmental Sciences Division in conducting tests of in situ grouting in a waste trench at ORNL Site 6. The Chemical Technology Division's responsibilities are (1) to develop a grout formula to be used in the trench, (2) to develop a grout implementation plan, (3) to work with Martin Marietta Purchasing Department in selecting a grouting subcontractor, and (4) to provide limited field support for the grout injection. This final report satifies the milestone of Subtask 12 entitled, "Low Level Waste (LLW) Trench Grouting Assessment," which was initially issued as RAP-86-7, December 31, 1985.

Dry-solid blends containing Type I Portland cement, ASTM fly ash (Classes C and F), and bentonite were mixed with water at ratios ranging from 10 to 15 lb/gal. Phase separation, compressive strength, and various rheological tests were then conducted on the resulting grout mixes. The methods for calculating Reynolds number, critical velocity, and frictional pressure drop will be described. It is recognized that these variables apply to grout pumping, which may not be necessary for this project. Values of these variables have been calculated since data to calculate them were acquired for other purposes and since knowledge of the values may serve to more completely characterize the grout. Brief explanations will be presented (Sect. 4) for the grout performance criteria used in the investigation.

2. TEST METHODS AND DATA USES

2.1 BLEND COMPOSITION AND PREPARATION

Two dry-solid blends were prepared: (1) a type containing ASTM Class C fly ash and (2) a type containing ASTM Class F fly ash. The compositions of three test blends containing ASTM Class C fly ash are shown in Table 1. These blends vary in composition from 30 to 41 wt % Type I Portland cement, from 54 to 70 wt % ASTM Class C fly ash, and from 0 to 6 wt % bentonite. The compositions of the blends prepared containing ASTM Class F fly ash are shown in Table 2. Those blends contained from 42 to 58 wt % Type I Portland cement, from 33 to 50 wt % ASTM Class F fly ash, and 8 or 9 wt % bentonite. Class C fly ash exhibits more cementitious properties than does Class F fly ash; however, both are pozzolanic materials. Bentonite was included in the blends to increase the water absorption properties of the grouts.[1,2] The blends were tumbled in a V-blender for 2.0 h before being mixed with water (see Sect. 3). The blends listed in Table 1, which contain ASTM Class C fly ash (along with other blend materials), were mixed with water at ratios of 12, 13, 14, and 15 lb/gal (i.e., lb dry solids/gal water). The blends listed in Table 2, which contain ASTM Class F Fly ash (along with other blend materials), were mixed with water at ratios ranging from 10 to 13 lb/gal.

Table 1. Blends of solids containing ASTM Class C fly ash[a]

Material	Blend 1 (wt %)	Blend 2 (wt %)	Blend 3 (wt %)
Type I Portland cement	36	41	30
ASTM Class C fly ash[b]	58	54	70
Bentonite	6	5	0.0
CFR-1[c]	0.02	0.02	0.02

[a]Blends mixed with water at ratios of 12, 13, 14, and 15 lb/gal.
[b]Obtained from American Fly Ash Co.; source, power plant at Owensboro, Ky.
[c]CFR-1, a sugar type set retarder was added as wt % of the blends and was dissolved in the water mixed with the blends.

Table 2. Blends of solids containing ASTM Class F fly ash

Material	Blend 4 (wt %)	Blend 5 (wt %)	Blend 6 (wt %)
Type I Portland Cement	42.0	49.5	58.0
ASTM class F fly ash[a]	50.0	41.5	33.0
Bentonite	8.0	9.0	9.0
CFR-1[b]	0.02	0.02	0.02

[a]Fly ash obtained from TVA power plant at Kingston, Tenn.
[b]CFR-1, a sugar type set retarder, was added as wt % of the blends and was dissolved in the water mixed with the blends.

2.2 PHASE SEPARATION TESTS

Phase separation refers to the liquid or water layer that collects at the top of freshly mixed grout. The volume of liquid is usually found to increase for a short period after the grout has been mixed and then gradually decrease to zero with additional cure time. The volume of the liquid layer is determined by a settling test in a 1-L plastic bottle. In the test, a known volume of freshly mixed grout (usually 500 mL) is poured into the bottle; then the bottle is capped and allowed to stand for intervals up to 28 d. The phase separation, in vol %, is calculated as the volume of clear, drainable surface liquid divided by the total initial grout volume times 100.

The procedure[3,4] for preparing grouts for the phase separation test was as follows: The appropriate weight of a given dry blend was added to 500 mL of the waste solution while stirring at low speed (setting 1) in a Hobart Model N-50 mixer over a period of 15 s. The setting was left at low speed for an additional 15 s and then increased to high speed (setting 2) for 30 s. The mixes were then poured into plastic bottles for the tests.

2.3 COMPRESSIVE STRENGTH TESTS

Compressive strength is a measure of the structural integrity that cured grouts are expected to exhibit. A compressive strength sufficiently low to allow crushing of the cured grout would result in increased grout surface area and the possibility of increased leaching. Compressive strength tests were conducted in duplicate on each grout formulation.

The specimens for the compressive strength tests[5] were prepared by pouring freshly prepared grout into 2-in.-cube stainless steel molds and allowing the molds to stay in a humidity cabinet at room temperature for 28 d. Crushing strengths of the grout cubes were then determined by using a Model 60,000 Super "L" Tinius-Olsen Testing Machine. The freshly mixed grouts were prepared by following the same procedure as that used for the phase separation tests.

2.4 RHEOLOGICAL MEASUREMENTS

Rheological measurements were made similar to those reported in the past for hydrofracture[6] and Rockwell Hanford grouts.[7] It is recognized that the grouts for this in situ work will probably not need to be pumped;

nevertheless, measurements of (1) apparent viscosity, (2) 10-min gel strength, (3) critical velocity, (4) flow behavior index (n'), and (5) fluid consistency index (K') can be used to characterize the in situ grout. Thus, the above measurements were made for the more promising grout mixes.

The procedure for preparing the grout mixes for the rheological measurements was the same as that used for the phase separation test. Duplicate tests were conducted, and rheological measurements were made using a Fann Direct-Reading Viscometer, Model 35A. Freshly prepared grouts were added to the viscometer, and the shear stress was measured at shear rates from 600 to 0.9 rpm.

The data obtained in the tests were used to calculate (1) Reynolds numbers, (2) critical flow rate (flow rate required for turbulent flow in a 2-in.-ID pipe), (3) 10-min gel strength, and (4) frictional pressure drop per 100 ft of a 2-in.-ID pipe.

Although grouts can be readily pumped in the laminar-flow regime, the turbulent-flow regime is preferred for pumping grouts over long distances because radial components of velocity are present in turbulent flow. These components generate both resisting and driving forces and, therefore, promote mixing at the pipe wall. Pumping in the turbulent-flow regime will not eliminate caking at the pipe wall but should minimize it. This operational mode, in turn, minimizes the operational flushing requirements and excessive pressure drops.

2.4.1 Fluid Consistency Index, n', and Flow Behavior Index, K'

For non-Newtonian grouts, shear stress is dependent on shear rate and is represented by the power-law model,[8]

$$\log S_s = \log K' + n' \log S_r, \tag{1}$$

where

S_s = shear stress, lb_f/ft^2;*

K' = fluid consistency index, $lb_f \cdot s^{n'}/ft^2$;

S_r = shear rate, s^{-1}; and

n' = flow behavior index ($0 < n' < 1.0$), dimensionless.

*To convert to SI units: 1 lb_f/ft^2 = 47.9 N/m^2.

Values of K' and n' were evaluated from a least-squares fit of a given set of viscometer-shear-stress vs shear-rate data to the power-law model [Eq.(1)]. The values of n' and K' were then used for calculating Reynolds numbers and conditions for turbulent flow of the grouts.

2.4.2 Density

The density of each freshly mixed grout was measured directly, in lb/gal, at room temperature using a Baroid mud balance.

2.4.3 Apparent Viscosity

Viscosity in a grout varies with shear rate. The apparent viscosities in these tests were measured at 511 s^{-1} (300 rpm on the Fann Viscometer), which is the common practice.

2.4.4 Gel Strength (10-min)

The 10-min gel strength is indicative of the force required to restart the flow of grout in a pipe after it has been stopped for 10 min. The measurement is made in the Fann Viscometer with the same grout sample following the other rheological measurements. After the grout has been allowed to stand in the viscometer for 10 min without stirring, the instrument is turned on with the shear rate set at 3.0 rpm. The 10-min gel strength, in lb/100 ft^2, is read directly from the viscometer at the maximum deflection on the shear stress scale.

3. METHODS OF CALCULATION

3.1 REYNOLDS NUMBERS

Reynolds numbers were calculated from the following expression:

$$N_{Re} = \frac{1.86\ V^{(2-n')}\ \rho}{K'(96/d_i)^{n'}}\ , \qquad (2)$$

where

N_{Re} = Reynolds number, dimensionless;

V = fluid velocity, ft/s (reference condition, 20.4 ft/s);

n' = flow behavior index ($0 < n' < 1.0$);

d_i = inside pipe diameter, in. (reference condition, 2 in.);

ρ = fluid density, lb/gal; and

K' = fluid consistency index, $lb_f \cdot s^{n'}/ft^2$.

3.2 CRITICAL FLOW RATE (FLOW RATE FOR TURBULENT FLOW)

The critical flow rate, V_c, is the flow rate required for turbulent flow of a grout through a pipe – in this instance, a 2-in.-diam pipe. Turbulent flow is assumed to occur at a Reynolds number ≥ 2100. The following expression for the critical velocity is obtained by rearranging Eq. (2) and setting $N_{Re} = 2100$:

$$V_c = \left[\frac{2100K'(48)^{n'}}{1.86\ \rho}\right]^{(2-n')^{-1}} . \qquad (3)$$

Values of V_c calculated from Eq. (3) are multiplied by 9.792 to convert the units from ft/s to gal/min in a 2-in.-diam pipe.

3.3 FRICTIONAL PRESSURE DROP

The frictional pressure drop for a grout during pumping can be defined as follows:

$$\Delta P_f = \frac{0.039 L \rho V^2 f}{d_i} , \qquad (4)$$

where

ΔP_f = frictional pressure drop through a straight pipe, psi;

L = pipe length, ft;

f = Fanning Friction Factor, dimensionless (f is a function of Reynolds number); and

ρ, V, and d_i represent the same variables as in Eq. (2). The value of L used in this case was 100 ft. The Fanning Friction Factor, f, decreases with increasing Reynolds number. The f values used in this work were obtained by dividing 16 by the Reynolds number.

4. TEST RESULTS AND DISCUSSION

The performance critera[7,8] tentatively used to investigate the acceptability of the various grouts are summarized below:

Criteria	Requirement
Apparent viscosity	<50 cP*
10-min gel strength	$\leq$100 lb_f/100 ft^2
28-d phase separation	0 vol %
28-d compressive strength	$\geq$60 psi;** expected 200–800 psi

A low apparent viscosity is needed to ensure that the grouts will effectively penetrate into voids in the trench waste matrix. The 10-min gel strength is a measure of the force that would be required to restart flow of a grout in a pipe after standing (nonflow) for an indefinite period. The limit on phase separation is for purposes of controlling water-soluble radionuclides and hazardous substances in place in the grout. It is important that the grouts exhibit a 28-d compressive strength $\geq$60 psi to prevent cracking and, in turn, exposure of increased surface to ground leach water.

The results of tests with Blends 1, 2, and 3 using ASTM Class C fly ash are shown in Tables 3, 4, and 5, respectively. The apparent viscosities for all of the mix ratios for Blend 3 were less than 50 cP, as were the 12-, 13-, and 14-lb/gal mixes for Blends 1 and 2. The 15-lb/gal mixes for Blends 1 and 2 exhibited apparent viscosities of ~55 cP. The 10-min gel strengths for all Blend 1, 2, and 3 mixes were <50 lb_f/100 ft^2, well within the acceptable range. The phase separation for all of the Blend 1 and Blend 2 mixes and for the 14- and 15-lb/gal mixes for Blend 3 was 0.0 vol % in periods of <28 d. The compressive strengths for all of the Blend 1 and Blend 2 grouts and for the Blend 3 grouts with mix ratios of 12 and 13 lb/gal ranged from 914 to 2953 psi, well above the 60-psi requirement.

Tests of the ASTM Class F fly ash-bearing grouts were conducted only to the scouting stage. Results of tests with Blends 4, 5, and 6 using mix ratios of 12.5, 11, and 10 lb/gal are shown in Table 6. Additional results for Blend 5 using 11.5-, 12.0-, 12.5-, and 13.0-lb/gal mixes are

---

*1 cP = 1 mPa·s.

**1 psi = 6.89 kPa.

Table 3. Properties of grouts prepared from Blend 1[a] at mix ratios of 12, 13, 14, and 15 lb/gal

(Values are averages based on duplicated measurements.)

Mix ratio (lb/gal)	12	13	14	15
Apparent viscosity (cP)	23.5	31	41.8	55.5
10-min gel strength (lb_f/100 ft^2)	23	30.5	33.5	40
Density (lb/gal)	13.44	13.71	14.00	14.26
Fluid consistency index, K', ($lb{\cdot}s^{n'}/ft^2$)	0.0054	0.0085	0.0125	0.0189
Flow behavior index, n'	0.6021	0.5768	0.5629	0.5389
28-d phase separation (vol %)	0 (21d)	0 (6d)	0 (2d)	0 (1d)
28-d compressive strength (psi)	1109 ± 230	1506 ± 135	2145 ± 54	2684 ± 146
At reference conditions:[b]				
Reynolds number	2100	2100	2100	2100
Frictional pressure loss per 100 ft of pipe (psi)	1.8	2.8	4.4	6.4
Critical flow rate (gal/min)	29.5	36.6	44.8	53.8
Pump head pressure (psi)	3.8	5.1	5.6	6.7

[a]Type I Portland Cement, 36 wt %; ASTM Class C fly ash, 58 wt %; bentonite, 6 wt %.

[b]2-in.-ID pipe arbitrarily chosen.

Table 4. Properties of grouts prepared from Blend 2[a] at mix ratios of 12, 13, 14, and 15 lb/gal

(Values are averages based on duplicated measurements.)

Mix ratio (lb/gal)	12	13	14	15
Apparent viscosity (cP)	25	31.5	43	55
10-min gel strength ($lb_f/100\ ft^2$)	22.5	26	40	44.5
Density (lb/gal)	13.36	13.91	14.08	14.33
Fluid consistency index, K', ($lb \cdot s^{n'}/ft^2$)	0.0065	0.0087	0.0128	0.0189
Flow behavior index, n'	0.5754	0.5714	0.5611	0.5346
28-d phase separation (vol %)	0	0	0	0
28-d compressive strength (psi)	1476 $\pm$ 288	1725 $\pm$ 263	2238 $\pm$ 96	2953 $\pm$ 165
At reference conditions:[b]				
Reynolds number	2100	2100	2100	2100
Frictional pressure loss per 100 ft of pipe (psi)	2.0	2.8	4.4	6.4
Critical flow rate (gal/min)	30.7	36.1	45.1	53.9
Pump head pressure (psi)	3.8	4.3	6.7	7.4

[a]Type I Portland cement, 41 wt %; ASTM Class C fly ash, 54 wt %; bentonite, 5 wt %.

[b]2-in.-ID pipe arbitrarily chosen.

Table 5. Properties of grouts prepared from Blend 2[a] at mix ratios of 12, 13, 14, and 15 lb/gal

(Values are averages based on duplicated measurements.)

Mix ratio (lb/gal)	12	13	14	15
Apparent viscosity (cP)	16	19.5	26	34
10-min gel strength ($lb_f/100\ ft^2$)	13.5	21.5	25	30
Density (lb/gal)	13.30	13.63	13.95	14.22
Fluid consistency index, K', ($lb \cdot s^{n'}/ft^2$)	0.0026	0.0039	0.0053	0.0074
Flow behavior index, n'	0.6572	0.6338	0.6233	0.6082
28-d phase separation (vol %)	2.9	0.92	0	0
28-d compressive strength (psi)	914 ± 19	1258 ± 163	ND[c]	ND
At reference conditions:[b]				
Reynolds number	2100	2100	2100	2100
Frictional pressure loss per 100 ft of pipe (psi)	0.92	1.4	2.0	2.9
Critical flow rate (gal/min)	21.1	25.8	30.5	36.3
Pump head pressure (psi)	2.3	3.6	4.2	5.0

[a]Type I Portland cement, 30 wt %; ASTM Class C fly ash, 70 wt %; bentonite, 0.0 wt %.

[b]2-in.-ID pipe arbitrarily chosen.

[c]ND = not determined

Table 6. Properties of grout mixes prepared from Blends[a] 4, 5, and 6 at ratios of 12.5, 11, and 10 lb/gal

Blend	4	5	6
Mix ratio (lb/gal)	12.5	11	10
Apparent viscosity (cP)	ND[b]	35.5	ND
10-min gel strength ($lb_f/100\ ft^2$)	ND	26.5	ND
Density (lb/gal)	ND	13.03	ND
Fluid consistency index, K', ($lb \cdot s^{n'}/ft^2$)	ND	0.0103	ND
Flow behavior index, n'	ND	0.5614	ND
28-d phase separation (vol %)	1.7	2.9	5.3
28-d compressive strength (psi)	1213 ± 67	1229 ± 38	1315 ± 33

[a]Dry-solids blend contents are shown in Table 2.
[b]ND = not determined.

shown in Table 7. It can be seen that phase separation was greater for the grouts prepared with ASTM Class F fly ash than with the grouts prepared with ASTM Class C fly ash, even though the latter contained smaller amounts of bentonite clay. The 1213-, 1229-, and 1315-psi compressive strengths reported in Table 6 respectively for Blends 4, 5, and 6 are acceptable. The apparent viscosities and 10-min gel strengths for all of the tests (shown in Tables 6 and 7), although acceptable, were greater than would be preferred. They were generally greater than those obtained using blends with Class C fly ash; for example, the apparent viscosity of the Blend 5 (12-lb/gal) mix, 46.5 cP, was significantly greater than the value for the Blend 2 mix, 25.0 cP. Similarly, the 10-min gel strength of the Blend 5 mix, 33.5 $lb_f/100\ ft^2$, was considerably higher than the value for the Blend 2 mix, 22.5 $lb_f/100\ ft^2$. Therefore, the tests using ASTM Class F fly ash were discontinued, based on the phase separation and apparent viscosity results.

Table 7. Properties of grout mixes prepared from Blend 5[a] at ratios of 11, 11.5, 12, 12.5, and 13 lb/gal

Mix ratio (lb/gal)	11.0	11.5	12.0	12.5	13.0
Apparent viscosity (cP)	34	40.5	46.5	ND[b]	ND
10-min gel strength ($lb_f/100\ ft^2$)	24.5	27	33.5	ND	ND
Density (lb/gal)	12.98	13.11	13.24	ND	ND
Fluid consistency index, K' ($lb \cdot s^{n'}/ft^2$)	0.0047	0.0126	0.0174	ND	ND
Flow behavior index, n'	0.5650	0.5509	0.5271	ND	ND
28-d phase separation (vol %)	1.4	1.2	0	0	0

[a]Type I Portland Cement, 49.5 wt %; ASTM Class F fly ash, 41.5 wt %; bentonite, 9.0 wt %.

[b]ND = not determined.

5. SUMMARY

The conditions used and the results obtained thus far in the study being conducted by the Chemical Technology Division can be summarized as follows:

1. The principal ingredients of the grouts included in this investigation were: Type I Portland cement, ASTM fly ash (Class C or F), bentonite, and water.
2. Tests conducted included: phase separation, 28-d compressive strength, apparent viscosity, 10-min gel strength, and various rheological tests.
3. Tentative performance criteria:

Criteria	Requirement
Apparent viscosity	<50 cP
10-min gel strength	$\leq 100\ lb_f/100\ ft^2$
28-d phase separation	0 vol %
28-d compressive strength	≥ 60 psi

4. Acceptable grouts were prepared from two dry-solid blends (1) containing 36 wt % Type I Portland cement, 58 wt % ASTM Class C fly ash, and 6 wt % bentonite; and (2) 41 wt % Type I Portland cement, 54 wt %, ASTM Class C fly ash and 5 wt % bentonite. Acceptable grouts were obtained when these blends were mixed with water at ratios of 12, 13, and 14 lb/gal.
5. Grouts of composition and mix ratio similar to those discussed above, but without the bentonite, also show promise.
6. Grouts prepared containing ASTM Class F fly ash exhibited phase separation, high apparent viscosity, and 10-min gel strength problems when compared with grouts prepared containing ASTM Class C fly ash.

6. ACKNOWLEDGMENT

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