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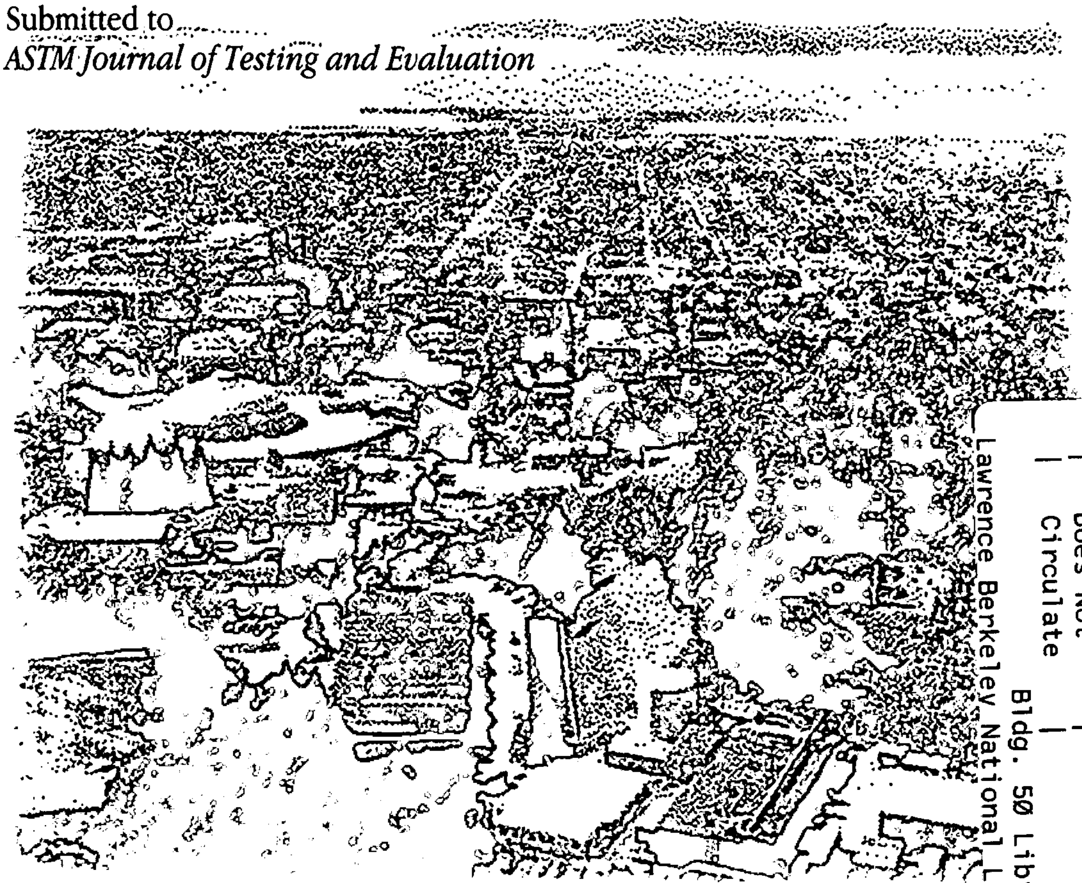

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EVALUATION TESTS FOR COLLOIDAL SILICA TO BE USED FOR GROUTING AT SAVANNAH RIVER SITE, SOUTH CAROLINA

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INTRODUCTION

Colloidal silica (CS) was planned to be injected into the soil beneath a pond that was formerly used as an unlined retention basin for contaminated water, to form a barrier to transport between the contaminated pond sediments and the underlying groundwater table. CS for this application must meet requirements of (i) low initial viscosity, (ii) low permeability after gelling, (iii) not requiring excessive injection pressure and (iv) controllable gel time in soil. Laboratory tests were devised to quantify these requirements, and were written into acceptance specifications. This paper describes the test methods and presents typical results.

Colloidal silica is stabilized by a negative charge on the surface of the silica particles. This charge causes repulsion between particles which prevents them from approaching one another closely and forming interparticle bonds. However, if the ionic strength of the aqueous phase is increased (as by addition of brine), the electrical double layer surrounding each particle is compressed, which allows the particles to approach one another more closely, and the particles bond together. forming a gel. This gel can block water flow in pore space of a soil. In this work, the gel time was controlled by varying the concentration of added brine. Grouts were made by combining 5 parts by volume of the colloid with one part of brine.

The principal challenge in this work was the requirement for controllable gel time in soil. If the grout gels too rapidly in soil, excessive injection pressure will be required, but if it gels too slowly, control over grout placement is lost. Ideally, gel time should be unaffected by the soil. When this is not the case, it may be possible to adjust the brine concentration to compensate for effects of soil on gel time.

The testing procedures were designed to verify that the colloids satisfied the requirements listed above. Tests measured the gel time of the grout both with and without soil, the pressure required to inject grout into a packed soil column, and the gel time of grout after it was injected into the soil column.

Colloidal Silica Samples

Three samples of colloid and brine were tested. Gel-time curves provided by the manufacturers indicated the dilutions of brine to be mixed with the colloids to achieve desired gel times (both time to onset of gellation and time to final solidification). These curves necessarily were only valid for gelling the colloid in the absence of soil.

Soils

The soil samples were obtained from a trench near the pond. Two drums of soil were collected from the 5-10 ft depth interval, and two from the 10-20 ft depth interval. Only the 10-20 ft soil was used in this work. First the soil was sieved to eliminate saprolites and large lumps of kaolin. The -4 (smaller than 4.76 mm) fraction was homogenized. This is referred to as "native soil". The moisture content of this soil was approximately 18%. This soil is an ultisol, i.e., from a humid temperate environment.

In addition to the native soil, a clay-sand mixture was prepared to simulate the sandy layers or lenses into which grout is to be injected. To prepare this mixture, the native soil was dried. ground in a mortar and pestle, and sieved. Ten percent by weight of the -30 fraction of this dried and ground native soil was mixed with 90% # 30 Monterey sand. This soil is referred to as 10% clay and had negligible moisture content.

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LABORATORY TESTING

Laboratory testing was conducted to determine (i) whether the samples submitted for evaluation could be made to gel at controlled times, (ii) whether the gel time was significantly accelerated or retarded by the soils (iii) whether the grout could be injected into the soil without excessive injection pressures caused by uncontrolled gellation, and (iv) whether the grout gels in the soil at the desired schedule. The measurements conducted for this evaluation consisted of standard tests, gel-time jar tests with and without soil, and special tests designed to assess the ability of grout to flow and gel in the particular soil to be grouted. While the suppliers could be reasonably certain of the performance of the samples in standard tests, the gel time jar tests with soils and the special tests involved use of soil materials with which the manufacturers were not familiar; therefore the manufacturers could not anticipate with certainty the performance of the samples in these tests.

Standard tests

The pH, viscosity, and solids content (by evaporation) of each candidate colloid were measured. Grouts were prepared by mixing brine and colloid as described below, and combined with Monterey #30 sand in 2x4 inch cylinder molds. The hydraulic conductivity of the resulting core was measured by ASTM D-5084. Results of these tests are summarized in Table 1.

Table 1. Standard tests for evaluating colloidal silica

Colloid	specification	1A	2A	3A
solids content (wt % remaining after evaporation)	>30	32.2	31.8	27.8
pH	5-10	7.85	10.28	7.25
viscosity (cP)	<10	4.23	7.27	4.41
hydraulic conductivity of grouted sand (cm/sec)	<1E-8	1E-8	4E-9	9E-9

Gel time jar tests without and with soil

Colloidal silica is made to gel by adding 1 part by volume of brine to 5 parts colloid. The gel time is controlled by diluting the brine from its concentration as delivered. Twenty mL of colloidal silica was placed in a 2-oz glass jar. Four mL of brine of appropriate concentration were slowly added to the jar by syringe with moderate agitation. The mixture was then allowed to sit, unagitated, between readings. (In a separate study, agitation of the grout was found to delay gelling and weaken the gel.) The progress of gellation was recorded by assigning gel states according to Table 2, (Moridis et al 1993 ; modified from Sydansk, 1990). For each candidate colloid, gel-time jar tests were run using brine diluted to give four target initial gel times (i.e., time to reach state 2 as defined in Table 2 of 1, 2, 4, and 8 hr). Because the gel time in the soil (as well as *in vitro*) is an important consideration, the gel time tests were repeated with 20 g of soil added to the jar. All candidate colloids were tested at the four target gel times with both soils. Typical results of these tests are shown in Figure1.

SPECIAL TESTS

Drain-in Test

The drain-in test is used as a screening test to identify (and eliminate) colloids that gelled upon contact with the soil even though no brine was added to cause gelling. In this test, 100 g of soil is packed in a vertical column to a height of approximately 28 cm. Then 85 mL of colloid are poured onto the soil column and the height of the liquid is monitored as the colloid flows into the soil. If the colloid does not gel, most of the colloid will flow through. Figure 2 shows results of tests on 11 types of CS, some of which failed this test. For these soils, premature gellation in soil is less of a problem than delayed gellation.

Column Tests

These two tests are done sequentially in a packed column of soil. The first measures the pressure required to inject the grout into the soil and the second, the rate at which the grout gels in the soil.

Column Injection Test

In this test, soil is packed into a 1 inch diameter, 36" long column. First four pore volumes (PV) of water are pumped through the column, and the injection pressure (Pi,w) is monitored. The flow rate for all injections is 1 PV in 30 minutes. All injection pressure values are corrected by

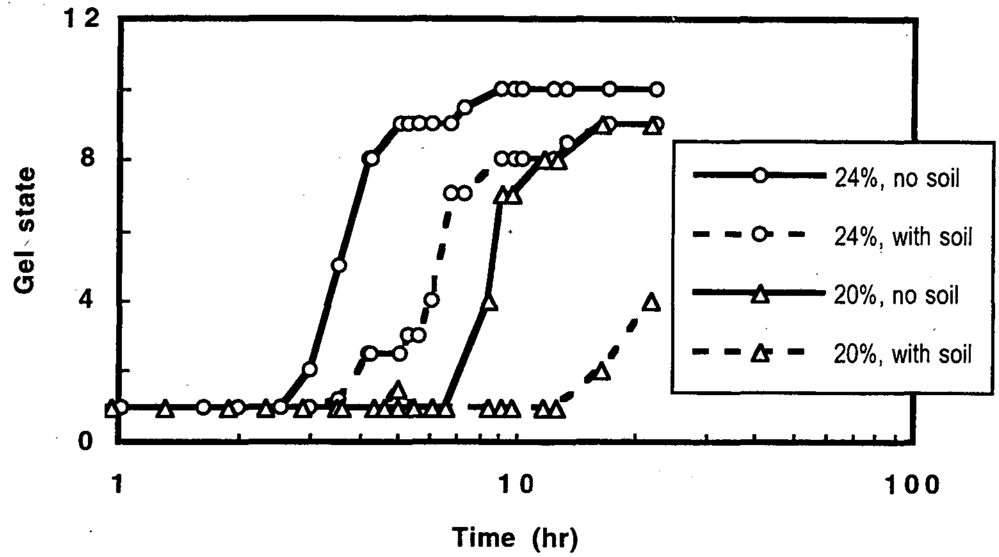

Figure1. Progress of gellation of one CS with two different brine concentrations, with and without added soil. The target times for gellation were 4 hr to state 2 and 8 hr to state 9 with 24% brine, and 8 hr to state 2 and 16 hr to state 9 with 20% brine.

Table 2 — Jar-Test Gel State Codes (Modified from *Sydansk* 1990]

1	No detectable gel formed. The gel appears to have the same viscosity (fluidity) as the original polymer solution and no gel is visually detectable.
2	Highly flowing gel. The gel appears to be only slightly more viscous than the initial polymer solution.
3	Flowing gel. Most of the obviously detectable gel flows to the bottle cap upon inversion.
4	Moderately flowing gel. A small portion (about 5 to 15%) of the gel does not readily flow to the bottle cap upon inversion—usually characterized as a "tonguing" gel (i.e., after hanging out of the bottle, gel can be made to flow back into the bottle by slowly righting it
5	Barely flowing gel. The gel slowly flows to the bottle cap and/or a significant portion (> 15%) of the gel does not flow upon inversion.
6	Highly deformable nonflowing gel. The gel does not flow to the bottle cap upon inversion (gel flows to just short of reaching the bottle cap).
7	Moderately deformable nonflowing gel. The gel flows about halfway down the bottle upon inversion.
8	Slightly deformable nonflowing gel. Only the gel surface deforms slightly upon inversion.
9	Rigid gel. There is no gel-surface deformation upon inversion.
10	Ringing rigid gel. A tuning-fork-like mechanical vibration can be felt or a tone can be heard after the bottle is tapped.
11	Rigid gel no longer ringing. No tone or vibration can be felt or heard, because natural frequency of the gel has increased.

subtracting the gravity head so that only viscous head loss is measured. The value of $P_{i,w}$ during the four PV of water injection is recorded ($P_{i,w}$ was constant during the four PV of water injection). This provides a measure of the hydraulic conductivity of the soil pack. Then two PV of grout are injected, and the injection pressure ($P_{i,g}$) is monitored. The maximum value of $P_{i,g}$ during the two PV of grout injection is recorded. ($P_{i,g}$) is expected to be greater than ($P_{i,w}$) because the viscosity of the grout is initially greater than that of the water, and also increases as the grout gels

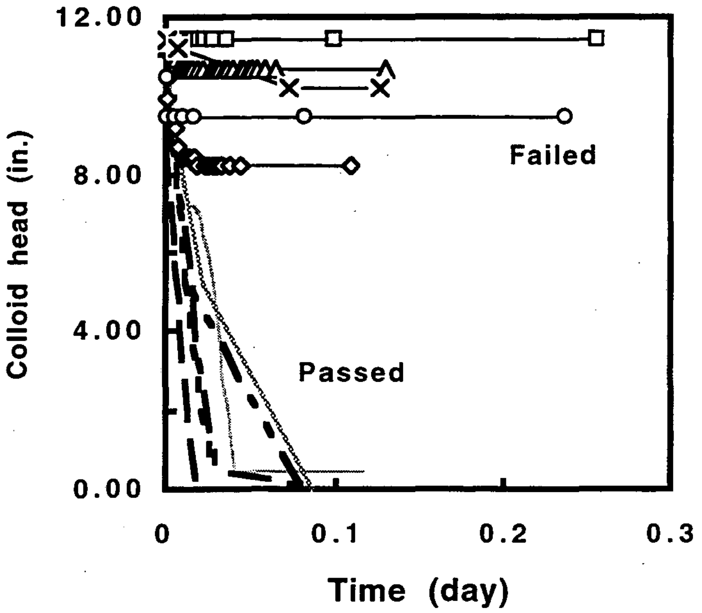

Figure2. Results of drain-in tests on 11 colloids, in native SRS soil.

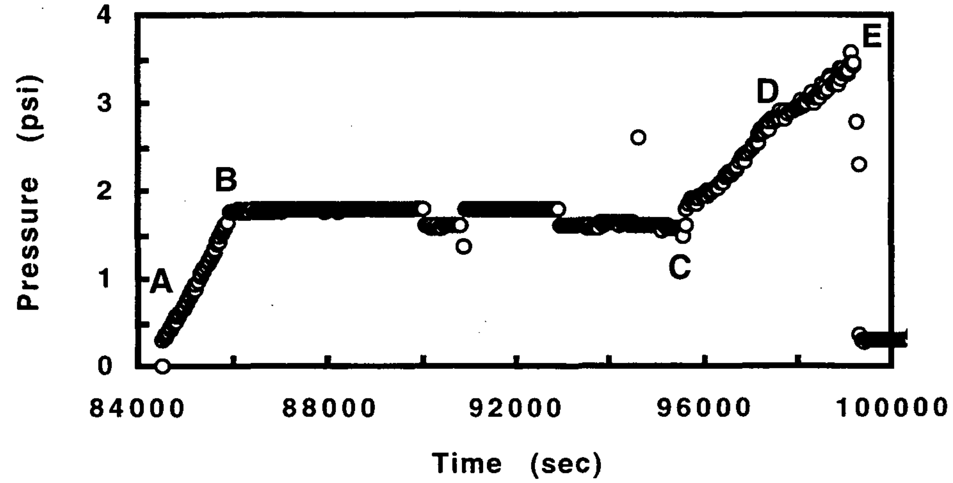

Figure 3. Column Injection test. Test starts at time A with column packed with soil, but not saturated. Water is injected into the bottom of the column, with effluent eventually emerging at time B. Injection pressure is steady from time B to time C as three more PV of water are injected. At C injection is switched to grout, and injection pressure increases as grout replaces water in column due to increased density and viscosity. At time D grout breaks through; pressure increases until injection is stopped after 2 PV at time E.

(the grout was pre-mixed as a batch before injection). An unexpectedly high $P_{i,g}$ indicates that premature gelling is occurring in the soil, or that the injection of grout has caused some change in the soil that decreases its hydraulic conductivity, such as swelling of clays. The criterion for success is that $(P_{i,g}/P_{i,w})(\mu_w/\mu_g)$ not exceed 2.5. Figure 3 shows a typical injection pressure test.

Column Gel-Time Test

Immediately following the column injection pressure test, the gel time of the grout in the soil is measured by monitoring the mobility of the grout in the grouted soil column. The grouted column is removed from the injection manifold and connected to flexible tubes filled with water. By moving one or both of the flexible tubes, the water levels in the two tubes can be made to differ. This imposes a hydraulic gradient across the grouted soil column. As long as the grout remains mobile, this gradient will decay to zero, as in a falling head permeability measurement. By monitoring the heights of the two water columns as the gradient decays, a measure of the mobility of the grout is obtained.

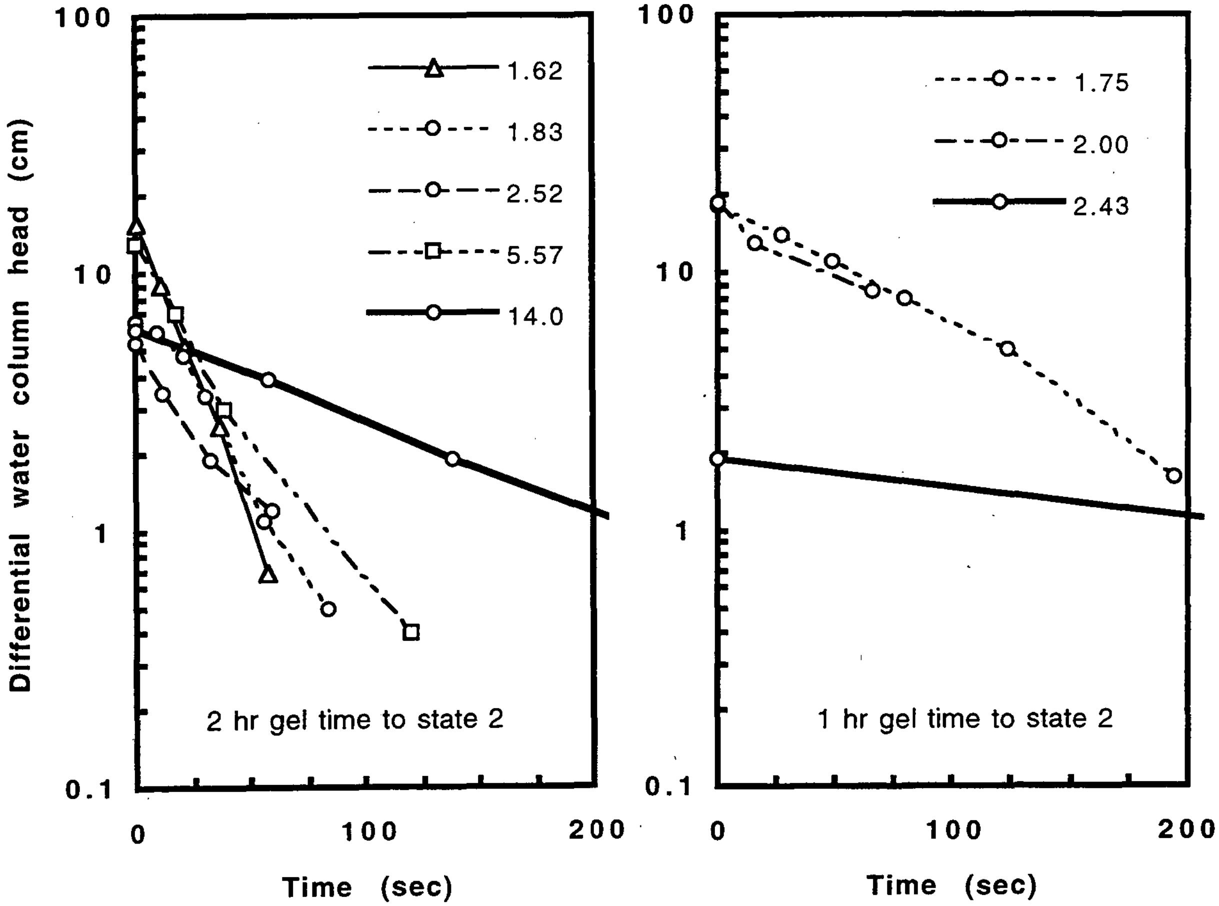

Figure 4. Gel-time column test for a colloid that, in absence of soil, would gel to state 2 (Left side, a) in 2 hr. (Right side, b) in 1 hr. Numbers are time (hr) after grout was mixed. Test (a) followed immediately after the injection shown in Figure3. Grout mobility decreased during 4 hr, but the grout was still mobile in the soil after 14 hr. and finally became immobile after 23 hr. In (b) grout mobility decreased during 0.7 hr after injection ceased and grout became immobile 3.8 hr after mixing.

The procedure for measuring the grout mobility is to impose a hydraulic gradient across the grouted soil column, and record the heights of the two water columns as the gradient decays. When equilibrium is reached, the height of the two water columns are not equal, because the density of the grout is greater than that of water. The equilibrium height difference is recorded and used to correct the readings. Darcy's law requires that the corrected height difference decay exponentially, and this is confirmed by the straight semi-log plots shown in Figure 4. The mobility of the grout is proportional to the absolute value of the slopes of the lines, and as the mobility decreases (i.e., as the grout gels), the lines approach horizontal. Finally, when the grout has gelled sufficiently to prevent any water movement, the imposed hydraulic gradient is maintained and no longer decays. The criterion for success in this test is that the grout remain mobile 2 hours after mixing (and 1 hr after injection ceases) but that it become effectively immobile within four hours after mixing. Results for one colloid, tested with two different brine concentrations, are shown in Figure 4.

Drip test

Another test to measure gel time in soil, called the drip test, was devised as a more convenient alternative to the column gel time test. In the drip test, a vertically mounted 30-cm long, 2.5-cm i.d. column is prepared with an open top and a screened and capped 1/8 inch tube fitting at the bottom. 100 mL grout is poured into the column, and 100 g of soil is poured into the grout. This ratio of soil to grout will result in a packed bed with no free liquid over the top of the soil column. If needed, the amount of soil or grout can be adjusted to make the two heights equal. At intervals the cap is removed to allow grout to drain from the column, and the time needed for 10 drops of grout to drain is recorded. This volume is sufficient to gauge the flow rate without changing the head. The drained grout is returned to the top of the column.

As the grout gels, the drain rate slows and eventually stops. The criterion for success in this test is that the grout remain mobile 2 hours after mixing but that it become effectively immobile within four hours after mixing. Typical data from a set of drip tests are shown in Figure 5.

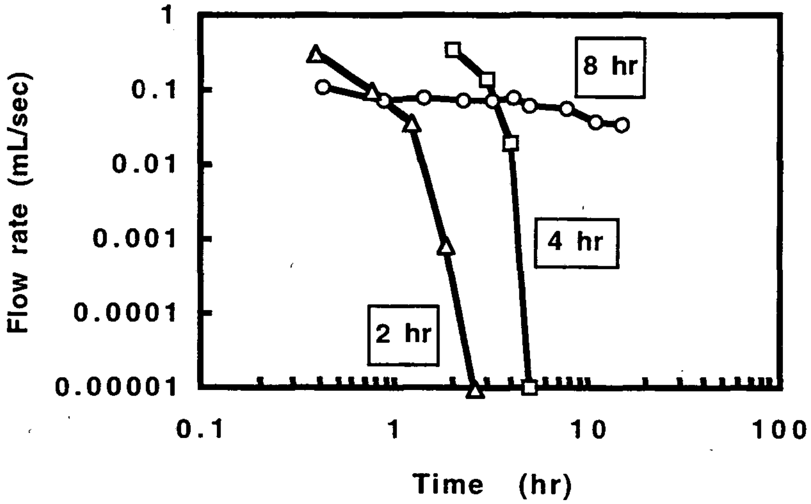

Figure 5. Flow rates in drip test for one colloid with three different concentrations of brine that, in absence of soil, would give gel time (to state 2) of 2, 4, and 8 hr. Drip rate decreases as the colloid gels in the soil.

SUMMARY AND DISCUSSION

A suite of tests for screening candidate colloidal silica grouts for use at a particular site were developed and used to test samples for conformity to specifications. These tests show the necessity of evaluating CS grout for every site. When CS is considered for use in permeation grouting of soils, this suite of tests can provide the operator with necessary knowledge of the suitability of various kinds of CS for use at any particular site. No specialized equipment is needed to run these tests.

The ability of gelled colloidal silica to block flow in porous media was confirmed, as shown by the measurements of hydraulic conductivity in Table 1. Of the three of the colloids studied in detail, all had acceptably low initial viscosities and could be injected into the test soils from this site without experiencing problems of premature gellation, as shown by the fact that they all passed the drain-in test and (when the brine concentration was selected to give gel state 2 in 2 hr) the column injection test.

The native soil significantly retarded gellation, as shown for example in Figure 2. In these tests it was possible to compensate for the retardation by increasing the brine concentration. However, such compensation would not necessarily avail for the combined column-injection and column-gel-time tests. In some cases, grouts that would gel to state 2 in 2 hr could pass injection pressure portion of the test, but the grout in the packed soil failed to meet the criterion for adequately rapid gelling in the soil. This is shown, for example in Figure 4a, where grout was still mobile after 14 hr. When the same colloid was tested with a more concentrated brine, calibrated to reach state 2 in 1 hr, the injection pressure during injection was unacceptably high, as the premixed grout was already increasing in viscosity before 2 PV had been injected.

The specific cause of gel retardation by soil is not known but is suspected to be organic compounds in the soil or pore water. This contrasts with experience with alkaline soils from arid sites e.g., at Hanford, Washington and Los Banos, California, where gel acceleration was caused by ion exchange between the grout and clay in the soil. (Persoff *et al.* 1994, Moridis *et al.* 1995).

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