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ALTERATION OF HYDRAULIC CONDUCTIVITY DUE TO
BATCH ORGANO-MODIFICATION
OF AN AQUIFER MATERIAL

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Thomas F. Wolff
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ALTERATION OF HYDRAULIC CONDUCTIVITY DUE TO BATCH ORGANO-MODIFICATION OF AN AQUIFER MATERIAL

By

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ABSTRACT

ALTERATION OF HYDRAULIC CONDUCTIVITY DUE TO BATCH ORGANO-MODIFICATION OF AN AQUIFER MATERIAL

By

James Michael Grant

Falling-head permeability tests in a fixed-ring consolidometer were performed on Oshtemo B soil, an aquifer type material, before and after batch treatment with hexadecyltrimethyl ammonium chloride (HDTMA-Cl). Additionally, the hydraulic conductivity of the treated soil was explored at two different packing states, water saturated and air dry. It was found that batch treatment of the soil with HDTMA-Cl caused an initial reduction to the hydraulic conductivity at low effective stress states. Increasing the effective stress on an organo-modified soil sample caused the hydraulic conductivity to decrease at a lesser rate than that of an untreated soil sample. It was hypothesized that batch organo-modification imposed a higher degree of orientation, i.e. more parallel arrangement, on the clay fabric and that the hydrophobic clay assemblages existed predominately within the pore space created by the non-clay fraction of the soil. The implications of these results were discussed in terms of their relevance to engineered systems.

This thesis and all of the work that it represents, both previous to and during my graduate studies, is dedicated to my parents Harry and Judith Grant. I am grateful and indebted to them for their financial support and spiritual motivation and encouragement, without which I would not be where I am currently.

This work is also dedicated in part to Mr. James Wackerlin. If it had not been for his input of the current market place and opinions on the importance of, and lack thereof, higher degrees in the sciences, I may have never ventured into a graduate program. A decision that I know I would have regretted.

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There is no way that I can express my infinite appreciation for Dr. Thomas Wolff. I feel very fortunate to have wandered into his soil mechanics class as a misguided, undergraduate student with an undeclared engineering major. Dr. Wolff's wisdom of the geotechnical science and his command of its practical application sparked an interest in me that eventually led to a graduate degree and a young, confident civil engineer. He has helped me set professional and personal standards, both in and out of the classroom, that I will always be grateful for.

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I. INTRODUCTION

Volatile organic compounds (VOCs) present in groundwater and soil reportedly have a potential to produce adverse health effects. Natural deposits of low organic matter content soil, clay soil, and aquifer materials have little sorptive capacity to serve as temporary or permanent sinks for VOCs, especially for nonionic organic compounds (NOCs) that are poorly soluble in water. The native inorganic cations are strongly hydrated in the presence of water, creating hydrophilic clay surfaces and interlayers, hence a poorly water soluble organic molecule cannot compete for the mineral adsorption sites (Lee *et al.*, 1989). This results in the mobility and transport of organic contaminants in the subsurface, especially compounds that possess high water solubilities. The limited sorptive capacity of some such soils can be greatly improved by replacing the natural, metal cations with large organic cations through ion-exchange reactions (Lee *et al.*, 1989; Jaynes and Boyd, 1991). These organic cations fix on the clay giving it the surface properties of an organic phase which results from the alkyl hydrocarbon tails that project out from the clay surface (Jaynes and Boyd, 1991). The organic cations that have been studied most extensively are quaternary ammonium compounds (QACs) of the general form $[(CH_3)_3NR]^+$ where R is an alkyl or an aromatic hydrocarbon (Boyd *et al.*, 1991). When R is a relatively large alkyl hydrocarbon, as is the case for hexadecyltrimethyl ammonium (HDTMA) where R=$(CH_2)_{15}CH_3$, then the modified clays are referred to as organophilic clays due to the character that is imparted to the clay surfaces and the interlayers (Boyd *et al.*, 1991).

Organo-modification of a soil results in a greater sorption capacity for dissolved organic contaminants and hence improved retardation capabilities for low organic matter content soils and aquifer media. Mechanistically, the organic phase that results from the ion-exchange process behaves analogous to the partitioning observed in natural soil organic matter (Jaynes and Boyd, 1991). It has been shown that the organic phase derived from HDTMA treatment formed an effective partition medium that was 10 to 30 times more effective on a unit weight basis than natural organic matter (Lee *et al.*, 1989; Jaynes and Boyd, 1991). The enhanced sorption characteristics could be managed to intercept a migrating plume and retard the advective and the diffusive advance of a contaminant mass through a region. This implies that modified soils could become useful remedial tools for containment technologies.

Organo-modified soils have been suggested to have applications as containment barriers (Alther *et al.*, 1990; Boyd *et al.*, 1991), as *in-situ* sorptive zones (Boyd *et al.*, 1991; Burris and Antworth, 1992), for stabilization of hazardous wastes (Alther *et al.*, 1990; Boyd *et al.*, 1991; Brown *et al.*, 1992), for water and wastewater treatment (Srinivasan and Fogler, 1989; Alther *et al.*, 1990), and as an immediate response to terrestrial chemical spills to limit the extent of contamination (Boyd *et al.*, 1991). Each application requires the blockage of or the bulk transport of fluid through a treated soil region. An evaluation of the modified soil's hydraulic conductivity in different chemical environments is necessary to evaluate the advective transport characteristics and hence feasibility of potential organo-modified soil technologies.

Hydraulic conductivity is probably the most important parameter for assessing contaminant migration (Evans, 1991). Saturated hydraulic conductivity is a macroscopic property of porous media that depends on the medium properties as measured by the intrinsic permeability and the fluid properties of viscosity and specific weight. Intrinsic permeability captures the integrated influence of the pore geometry and depends on porosity and pore size distribution of the matrix. These properties are all macroscopic characterizations of the media which are dependent on other features of the soil matrix. Of importance are the grain size distribution and grain shape, clay type and content, structure, packing, mixing, cementation, stress state and chemical characteristics of the pore liquids. Allred and Brown (1994) used the Hagen-Poiseuelle equation to show the dependency of the hydraulic conductivity on the porosity, the effective pore radius, and the pore fluid properties:

$$K = \frac{n * r^2 * \gamma}{8 * \mu} \qquad (1)$$

where K is the hydraulic conductivity, n is the porosity, r is the effective pore radius, γ is the specific weight of the fluid, μ is the fluid viscosity, and 8 is a constant arising out of the geometry of the system. Gross volumetric parameters such as porosity cannot entirely account for changes in the hydraulic conductivity of fine grained soils as they are better related to pore-size distributions (Garcia and Lovell, 1981). When no microscopic data is available for soils, the effective-pore-size index (r^2) from equation (1) becomes a useful tool for making conjectures at this level. Its relative magnitude when comparing soils is a direct reflection of the integrated pore shapes and is an indication of whether a large number of small pores or a small number of large pores are contributing to the gross porosity.

Hydraulic conductivity is significantly influenced by the soil's structure which consists of both the geometric particle arrangement and the interparticle forces of a soil (Lambe, 1958a; Acar and Ghosh, 1986). The geometrical relationships established by the soil particles is known as the soil fabric. The clay fraction of the soil fabric can be characterized at two levels, a first-order and a second-order recognition pattern (Yong and Warkentin, 1975). The second-order recognition level describes the geometrical arrangement of single clay particles within individual, identifiable groups or group units called fabric units. The first-order recognition level describes the geometrical arrangement of the fabric units. The response behavior of clay soils to mechanical manipulation is through the fabric unit interactions and the various bonding mechanisms between these units; therefore, physical soil testing can only provide an evaluation of the soil's fabric at the first-order recognition level (Yong and Warkentin, 1975).

The response of a soil mass to changes in applied stress depends almost exclusively on the effective stresses in that mass. Effective stress is defined as the difference between an engineering total stress and a measurable pore water pressure. It is not a directly measurable, physical quantity and should not be confused as a grain-to-grain contact stress. The effective stress can be increased by increases in the total stress of a soil mass (i.e., increased bearing loads on a soil or greater depths of overburden material) and by decreases in the pore water pressure (i.e., fluctuations in the groundwater table). The porosity and effective pore radius, and hence the hydraulic conductivity, are dependent on the effective stress state; therefore, the fluid transport properties of a soil mass can be expected to change with varying effective stresses.

A fixed-ring consolidometer (Figure 1) is an apparatus that allows the measurement of a soil's macroscopic volumetric properties at different effective stress states. A controlled total stress can be applied to a soil sample and vertical deformations can be measured during and at the conclusion of primary consolidation (i.e., complete dissipation of the induced pore water pressure by drainage such that the total stress equals the effective stress). The attached vertical standpipe (Figure 1) can be employed to conduct falling-head permeability tests. This allows for the measurement of hydraulic conductivity at varying effective stresses. The consolidometer has the advantages of simulating ranges of overburden pressures that could be encountered in the field and of reducing the likelihood of any significant side-wall leakage, and hence channeling, during permeability testing.

The scope of this investigation is to provide data on the hydraulic conductivity alterations induced by batch organo-modification and the resultant response of the soil to differing stress states. A preliminary model of the soil fabric's alteration by batch treatment with the cationic surfactant (HDTMA) is developed. A fixed-ring consolidometer will be employed to impose varying effective stress states and measure

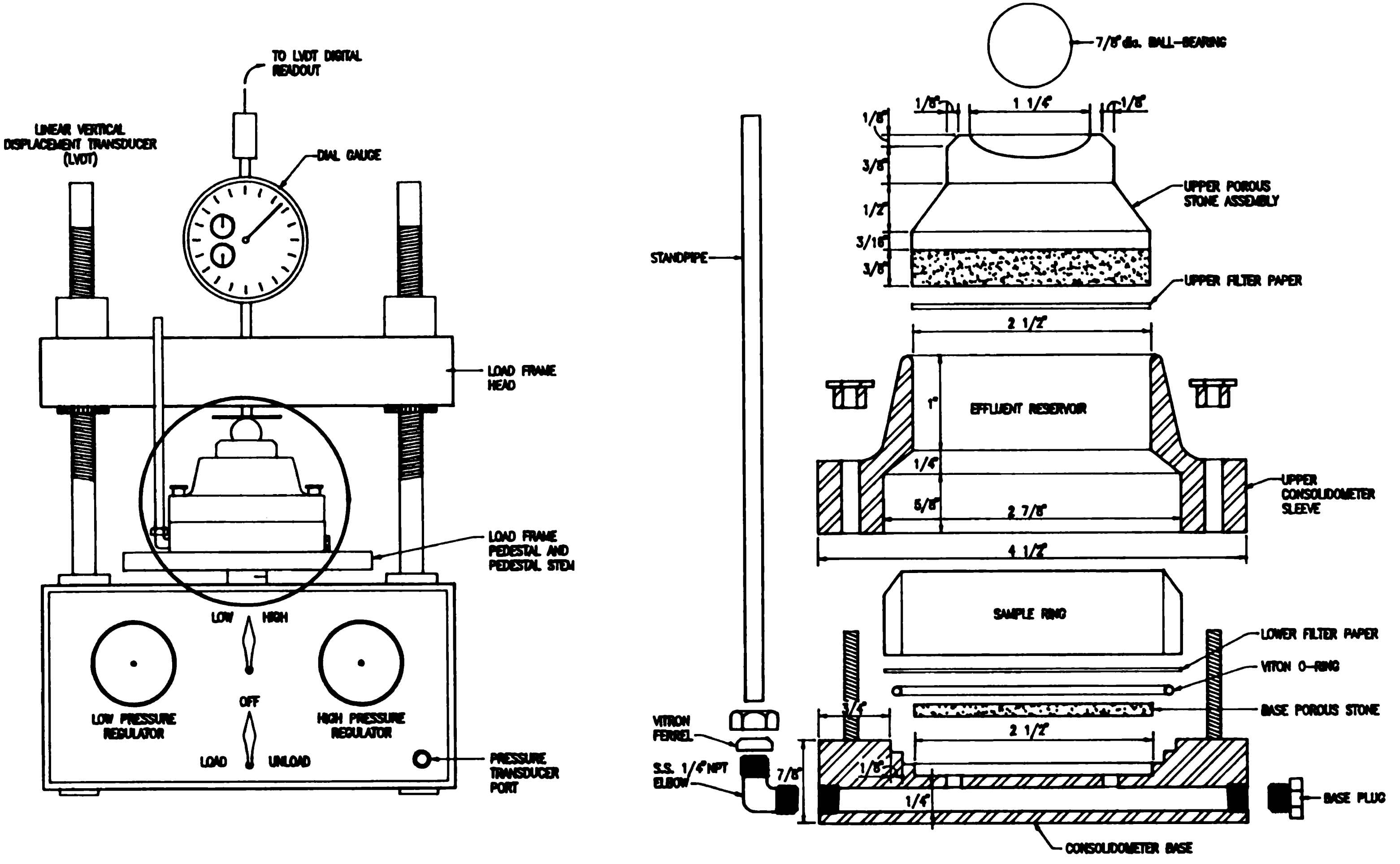


Figure 1: Consolidation Load Frame and a Cross-Section of the Fixed-Ring Consolidometer

gross volumetric parameters on an untreated and on a treated aquifer type material. The treated soil will be packed in a wet state and in a dry state to assess any changes in mechanical response induced by drying a hydrophobic soil. Falling-head permeability tests will be run at each stress state to measure the hydraulic conductivity's degree of dependence on the effective stress before and after modification. The macroscopic data that is collected will be used to draw conjectures regarding the alteration of the soil fabric at the first-order level as induced by the batch organo-modification process.

II. METHODS AND MATERIALS

2.1 Soil Batch Preparations. Approximately 50 kg (110 lbs.) of Oshtemo soil was obtained from a pit located at the Kellogg Biological Station (KBS) between Kalamazoo and Battle Creek, MI. It was taken from the B-horizon at a depth of approximately 60 cm (2 ft.) from the surface. The collected soil was air dried, sieved through a US standard #20 (0.84 mm), and then homogenized using the quartering method (ASTM C702-80). The homogenized Oshtemo B soil is a well graded , sandy loam with: 0.4% organic matter content; 78% sand, 19% clay, and 3% silt; 71.6% Ca^{2+}, 22.5% Mg^{2+}, 3.7% K^{+}, and 2.2% Na^{+} as exchangeable cations. The major clay minerals included one type of swelling clay (vermiculite) and three types of non-swelling clays (illite, kaolinite, and hydroxy-aluminum interlayered vermiculite). Three separate soil batches were prepared from this homogenized stock and were given the designations "untreated-dry", "treated-dry", and "treated-wet" where the term "treated" refers to the organo-modification of the soil and the "wet" or "dry" designation distinguishes the soil state at the time of packing.

The organo-modified soils were prepared by adding amounts of hexadecyltrimethyl ammonium (HDTMA) chloride equal to the maximum amount of inorganic exchangeable cations that can be replaced by HDTMA-Cl. This corresponds to the first plateau of the HDTMA-Cl adsorption isotherm (Figure 2) which is 46.1 mmole/kg (4.61 meq/100 g). 160 ml of HDTMA-Cl was added to batch suspensions of 840 ml high performance liquid chromatography (HPLC)-grade water and 25 g of

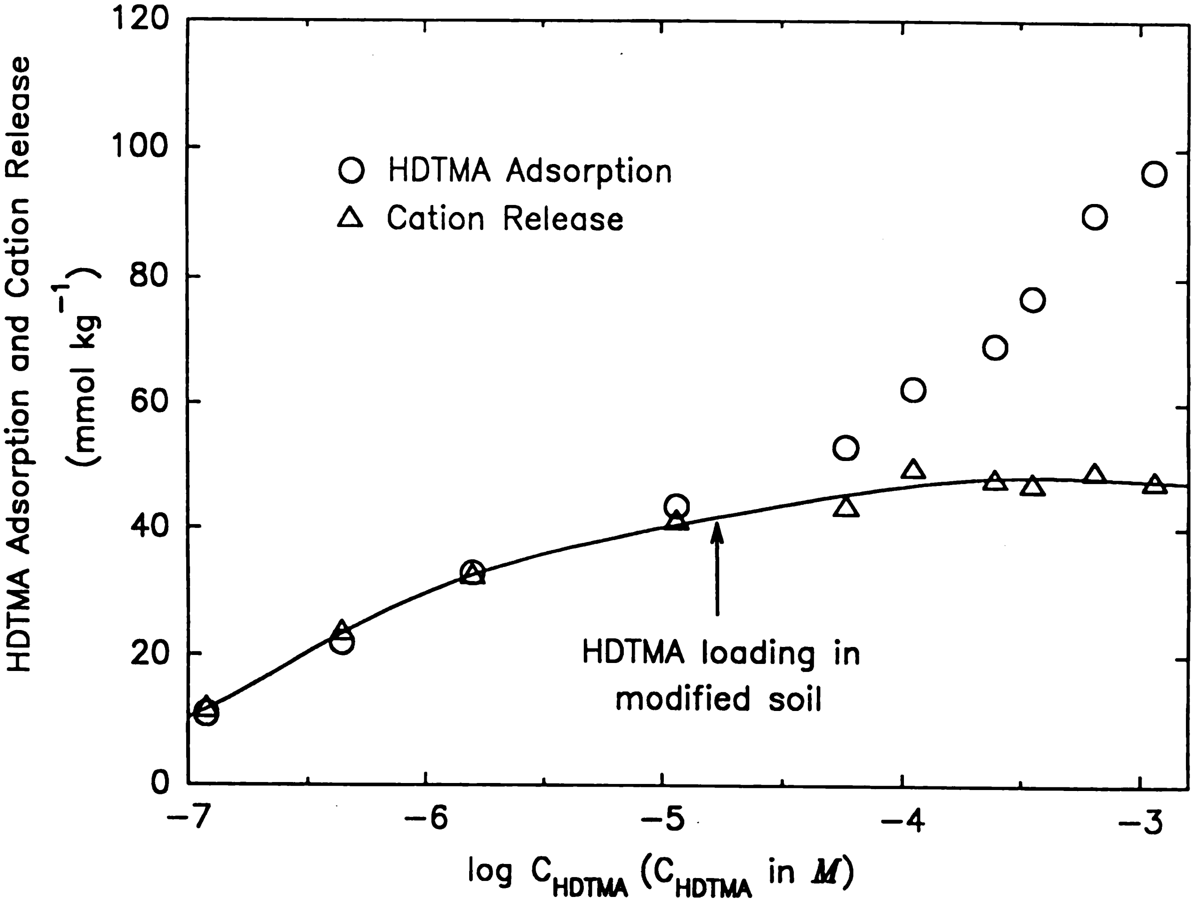


Figure 2: Oshtemo B Soil Adsorption of HDTMA-Cl

Oshtemo B soil and then stirred. A rapid mixing stage of 120 rpm for 1 minute and a slow mixing stage of 20 rpm for 30 minutes were imposed on the suspensions. Preliminary jar tests indicated that rapid mixing at 120 rpm for 1 minute produced the least turbidity when the soil was settled out of suspension, indicating a higher degree of flocculation. The organo-modified soil was settled out of suspension and then washed once with HPLC-grade water by decanting the HDTMA-Cl solution, filling and agitating the beakers with HPLC-grade water, and then decanting again. Insuring that no fines were lost, the treated Oshtemo B soil was transferred to a HPLC-grade water bath and mixed occasionally to prevent layering and to maintain complete water saturation of the soil. The treated soil was covered by approximately 10 cm (3.9 in.) of bath water at all times. The treated-dry batch was obtained by air drying soil from this bath.

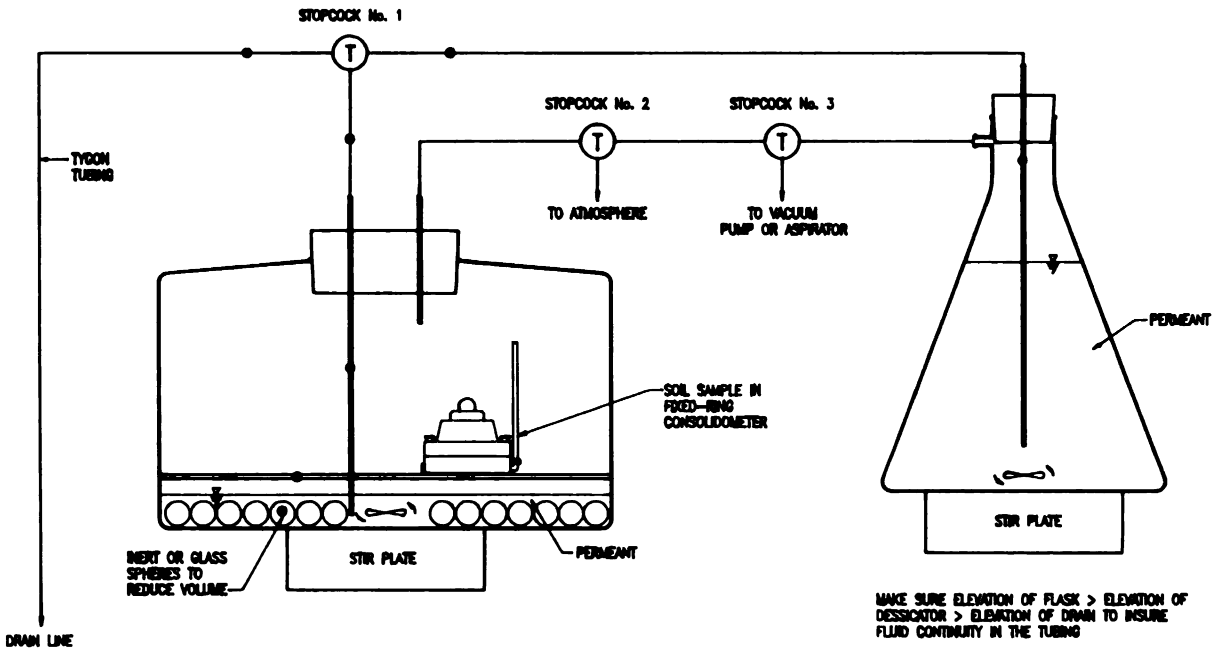
2.2 Sample Preparation. A fixed-ring consolidometer (Figure 1) was used for testing all of the Oshtemo B soil samples. This device allowed the total volume of a soil sample to be measured at different stress states. A dial gage and a linear vertical displacement transducer (LVDT) mounted on the load frame measured the change in thickness of the soil samples at each effective stress. The measured sample thicknesses at the conclusion of consolidation (L_{load}) were actually volumes since the soil was confined to a constant cross-sectional area (A). The specific gravity of solids (G_s) was measured using the pyncometer test (ASTM D854) and it was assumed that the volume of soil solids (V_s) remained constant; therefore, any measured changes of the sample volume (V) associated with varying the effective stress were attributed to void volume (V_v) changes. This allows for calculation of the soil's volumetric parameters at different stress states. The porosity (n) at each effective stress state was calculated using the following equation:

$$n = \frac{V_v}{V} = \frac{(V - V_s)}{V} = \frac{A * L_{Load} - \frac{M_s}{G_s}}{A * L_{Load}} \qquad (2)$$

Here M_s was the oven dry mass of the soil solids and the rest of the terms were as defined above.

The term "index" will be used throughout these procedures as reference to the process of taking dial gage readings at four points, each 90 degrees to the previous point, around the top of the upper porous stone assembly and then averaging these values to obtain an equivalent "height" for that specific assemblage of the fixed-ring consolidometer (Figure 1). Initially, a 2.54 cm (1 in.) "height" datum (L_{ref}) was established for a fixed position of the dial gage and load frame pedestal by inserting a machined, metal puck of 2.54 cm (1 in.) thickness and 6.35 cm (2.5 in.) diameter with filter paper on each surface within the completely assembled fixed-ring consolidometer and indexing. As long as the dial gage and load frame pedestal position was kept constant, the thickness of any soil within the sample ring of the consolidometer could be calculated, even if the assembly was removed from the load frame, by indexing against the 2.54 cm (1 in.) reference datum. The establishment of this datum was the first step for all of the sample preparations, regardless of the soil batch that was being tested.

The treated-wet samples were prepared by adding treated Oshtemo B soil slurry from the HPLC-grade water bath to a fixed-ring consolidometer within a desiccator system (Figure 3) that allowed for drainage and vacuum saturation. The empty consolidometer was assembled without the base plug, stand-pipe, or upper porous stone assembly and set within the desiccator. A vacuum was applied to the system to deair the HPLC-grade water that would be used for saturation and to vacuum saturate the base components of the consolidometer. The vacuum was collapsed on the desiccator only



MAKE SURE ELEVATION OF FLASK > ELEVATION OF DESSICATOR > ELEVATION OF DRAIN TO INSURE FLUID CONTINUITY IN THE TUBING

- VALVES 1, 2 AND 3 ARE 3-WAY STOPCOCKS
- ● INDICATES PIECES THAT SHOULD BE REPLACED BETWEEN GASOLINE AND WATER TRIALS

Figure 3: Vacuum-Dessicator System used to Saturate Oshtemo B Samples with Water

and the water within was drained by a siphon to a level equal to the top surface of the lower porous stone. Appendix A contains more detail on this procedure. Approximately 160 g (0.35 lb.) of the treated soil slurry was poured into the sample ring of the consolidometer. The slurry was scooped from random regions of the HPLC-grade water bath and transferred to a tared container before it was poured into the sample ring. Light tamping with a copper rod was implemented during pouring to avoid layering effects associated with differential settling velocities of the soil grains and to create a flat upper surface on the soil sample. Drainage was allowed until there was no standing water on top of the soil sample. An attempt was made to minimize the amount of fluid associated with the slurry. This seemed to result in greater reproducibility in the initial dry density of the samples and sped up the drainage process. The upper filter paper and porous stone assembly were set on the soil sample slowly to minimize tilt and the desiccator was resealed in preparation for the sample vacuum saturation procedure.

The treated-dry and untreated-dry samples were packed with the intention of replicating the initial conditions (dry density) of the wet samples as closely as possible. The compaction device employed had the following specifications: 1.47 kg (3.24 lb) hammer, 664 g (1.46 lb) hammer head, 7.5 cm (2.9 in.) hammer drop. The hammer head was machined to a 6.35 cm (2.5 in.) diameter and a 2.54 cm (1 in.) thickness so that it would just fit in the effluent reservoir and was the same diameter as the sample ring of the fixed-ring consolidometer, thus preventing the loss of soil during packing. After the initial indexing to establish L_{ref} was completed, the empty consolidometer was assembled without the base plug, stand-pipe, or upper porous stone assembly and either 130 g (0.29

lb.) of untreated-dry Oshtemo B soil or 140 g (0.31 lb.) of treated-dry Oshtemo B soil was placed in the ring. Best results were obtained when vacuum grease was used around the consolidometer's O-ring to prevent leakage. The untreated-dry samples were tamped 4 times and the treated-dry samples were tamped 2 times. The upper filter paper and porous stone assembly were set on the soil and the sample was indexed to obtain the packed height of the specimen (L_{pack}). The soil sample and the consolidometer were set inside the desiccator and it was resealed in preparation for the sample vacuum saturation procedure.

2.3 Vacuum Saturation. A vacuum is applied to the entire system to reestablish the negative headspace pressure in the desiccator for the treated-wet samples, and to deair the HPLC-grade water for the dry packed samples. While the vacuum was maintained on the desiccator only, it was flooded with deaired HPLC-grade water by opening stopcock no. 1 (Figure 3) to the flask. When the level was just below the lip of the consolidometer's effluent reservoir, flow was discontinued. Slowly removing the rubber stopper on the reservoir flask and exposing the headspace to atmospheric pressure increases the total head, and hence the differential head driving the flow and drastically reduces the time necessary to flood the desiccator. The vacuum was maintained on the desiccator over night (an average of 15 hours). Whenever there was any leakage at the consolidometer's O-ring seal there were small air bubbles drawn out at the steel-to-steel interface and the sample was repacked. After 15 hours, the water level within the effluent reservoir should have risen above the upper porous stone unless delivery of water was blocked through the base components of the consolidometer or the soil sample. Water was always observable within the effluent reservoir for this set of experiments; however, if this were not true it could be an indication of insufficient saturation of the soil sample. The vacuum within the desiccator was collapsed. The desiccator top was removed and the base plug and stand-pipe were attached to the consolidometer while it was still immersed. It was best to fill the bottom half of the stand-pipe with water before attaching to avoid introducing air bubbles into the base components of the consolidometer. Wrapping the base plug with Teflon tape was also helpful to prevent leakage. The consolidometer assembly was removed from the system, dried off, and then indexed to calculate the saturated height of the soil sample (L_{sat}). The difference between the saturated thickness and the packed thickness of the dry soil samples represents the change in volume due to swelling. Due to the uncontrolled nature of the swelling interface of the soil, it was difficult to maintain constant bulk densities during saturation. The untreated Oshtemo B soil displayed a greater tendency to swell due to its hydrophilic nature. The stainless-steel ball bearing was set into place in preparation for the consolidation sequence. Appendix A contains more detail on the vacuum saturation procedures.

2.4 Sample Consolidation and Hydraulic Conductivity Measurement. The load frame was tared for the consolidometer assembly then the saturated soil samples were set in the load frame (see Appendix B for calibration data) and subjected to a loading sequence from 24 kPa (0.25 ton/ft^2) to 766 kPa (8.0 ton/ft^2). Appendix A contains detailed procedures for taring the load frame. A load increment ratio (LIR) of unity, which means that the load was doubled each increment. The consolidation procedure was a modified form of the conventional consolidation test (ASTM D2435-90) that employed a rapid-loading sequence. Each load increment was imposed on the soil for one hour before a falling-head permeability test was performed and the next load was applied. Evidence suggests that for a LIR of unity, variable loading times greater than the time for 90% consolidation (t_{90}) have an insignificant affect on the void ratio vs. effective stress relationship as would be obtained using the classical approach of a 24 hour load duration (Olson, 1986). Taylor's square root of time fitting method was used to obtain t_{90} on the order of 1-2 minutes for the untreated Oshtemo B soil. It was assumed that the treated soil would possess a t_{90} on the same order of magnitude and that a load duration of one hour would still be adequate.

Once the soil sample had consolidated under each effective stress, a conventional falling-head permeability test with constant tail water level (ASTM D5084-90) was run . The fixed-ring consolidometers (Figure 1) were outfitted with a standpipe to allow permeability tests to be run. The inner diameters (a) were 0.258 cm^2 (0.040 in^2) for the glass and 0.335 cm^2 (0.052 in^2) for the plastic standpipes. This allowed adequate time scales to measure hydraulic heads at intermediate points and validate Darcy's Law. This was done for each falling-head test by plotting the logarithm of the hydraulic head vs. the cumulative time to insure that a linear relationship existed. This checked for the continuity of flow and that there was no system leakage during experimentation. The sample thickness was noted (L_{load}), the temperature of the permeant was measured, and then 2 or 3 tests, corresponding to 3 ml-8 ml of flushed deaired HPLC-grade water, were conducted at each state of stress. The total volume of permeant flushed through the soil by the end of the entire loading sequence varied from 34 ml to 48 ml which corresponds to approximately 1.0 to 1.5 pore volumes. The hydraulic conductivity for each test was corrected for temperature effects on the permeant's fluid properties using a correction factor (R_T). All values reported correspond to an equivalent hydraulic conductivity at 20 C (68 F). The hydraulic conductivity for each trial was calculated using:

$$K = \frac{2.303 * a * L_{Load} * R_T}{A * t} * \log\frac{(h_1 - \Delta h)}{(h_2 - \Delta h)} \qquad (3)$$

where Δh was the height of the tailwater datum, h_1 was the initial hydraulic head, h_2 was the final hydraulic head, and t was the elapsed time for falling head. All other terms are defined previously. The average hydraulic conductivity value was reported for each effective stress (Table 1). The measured hydraulic conductivity values from the falling-head tests in the consolidometer are actually equivalent values that account for headloss through the entire system. Experiments were conducted that determined the contribution of the porous stones to fluid resistance was negligible, hence the calculated hydraulic conductivity is representative of the soil sample only. A spreadsheet was constructed for the data reduction and error analysis of each experiment (see Appendix C for a sample output).

2.5 Sample Disassembly. At the conclusion of the loading sequence and the last falling-head permeability test, the final dial gage reading was taken and the fixed-ring consolidometer was removed from the loading frame. The air pressure to the load frame assembly was vented by turning the load dial to the "unload" position. The treated-wet samples were removed from the consolidometer and placed in an oven to dry at 110 C.

This would allow for calculation of the dry weight of soil solids (W_s). The oven-dried, treated-wet samples and the dry packed samples were visually inspected for channeling along the sample ring wall and for migration of fines. Ultrasonic baths were used to clean the porous stones at the conclusion of each experiment. It was assumed that no microbial growth was incurred during stone storage to contribute to flow resistance in subsequent experiments.

III. RESULTS AND DISCUSSION

Hydraulic conductivity is dependent on many factors which can be broadly defined as fluid property effects or as soil matrix effects. The intrinsic permeability is a soil property that represents the integrated influence of the pore geometry on the fluid conductance of the material. When the effective stress is varied on a soil mass, the geometric alteration is captured in this macroscopic parameter which, as defined earlier, depends on porosity and pore size distribution. The dominance of particular clay aggregate arrangements can be associated with certain types of engineering behavior (Collins and McGown, 1974), thus the mechanical response of the treated and untreated soil to increasing effective stress will be used to develop a hypothesis of the effect that batch HDTMA-Cl treatment has on the Oshtemo B soil's hydraulic conductivity and geometric particle arrangement. The experimental data are presented in Tables 1 and 2, and in Figures 4 through 6.

The measured hydraulic conductivity of a sample in the fixed-ring consolidometer has some uncertainty associated with it. Uncertainty is a quantity that indicates the variability of the best estimated value due to calibration errors, data acquisition errors, sample variability, data reduction errors, and other uncontrolled errors. As shown in Table 1, hydraulic conductivity was measured with a maximum relative uncertainty of approximately 4% for one standard deviation. In many experiments the uncertainty was lower. Uncertainty was estimated for each experiment (see sample spreadsheet output in

Table 1: Variation of the Measured Hydraulic Conductivity

Soil Type	Load (tsf)	Hydraulic Conductivity (K) (cm/s)			Average K (cm/s)	Relative Uncertainty (%)
		Trial #1	Trial #2	Trial #3		
Untreated Dry	0.25	1.84e-3	2.26e-3	2.25e-3	2.12e-3	3.33
	0.50	1.31e-3	1.32e-3	1.28e-3	1.30e-3	2.94
	1.0	6.68e-4	7.04e-4	6.40e-4	6.71e-4	2.79
	2.0	2.62e-4	2.29e-4	2.72e-4	2.54e-4	2.73
	4.0	1.09e-4	8.07e-5	8.80e-5	9.26e-5	2.67
	8.0	4.29e-5	2.96e-5	3.28e-5	3.51e-5	4.31
Treated Dry	0.25	6.89e-4	6.91e-4	8.02e-4	7.27e-4	2.93
	0.50	5.81e-4	5.91e-4	6.66e-4	6.13e-4	2.87
	1.0	4.54e-4	4.51e-4	5.14e-4	4.73e-4	2.84
	2.0	3.30e-4	3.24e-4	3.63e-4	3.39e-4	2.88
	4.0	2.29e-4	2.22e-4	2.48e-4	2.33e-4	2.87
	8.0	1.50e-4	1.50e-4	1.56e-4	1.52e-4	2.87
Treated Wet	0.25	2.26e-4	2.05e-4	2.49e-4	2.27e-4	2.70
	0.50	1.83e-4	1.65e-4	1.99e-4	1.82e-4	2.73
	1.0	1.31e-4	1.21e-4	1.50e-4	1.34e-4	2.70
	2.0	8.59e-5	8.26e-5	1.04e-4	9.08e-5	2.72
	4.0	5.02e-5	5.11e-5	6.61e-5	5.58e-5	2.72
	8.0	2.55e-5	2.93e-5	3.79e-5	3.09e-5	2.72

Appendix C) based on the assumptions that the measurement error was a random variable with a normal probability distribution function and an expected value of zero, and that the absolute uncertainty associated with an individual measurement was small enough to permit the analytical function to be approximated by the first linear term of a Taylor series expansion. The hydraulic conductivity was most sensitive to uncertainty in the hydraulic head measurements made during a falling-head permeability test. This could be improved by providing a standpipe with a greater cross-sectional area for slower drops and having more precise increments for the head measurements.

The measured volumetric parameters of a soil sample at a certain stress state also had uncertainty associated with it. The relative uncertainty was estimated for each experiment (see Appendix C) based on the same assumptions as previously mentioned, and are presented in Table 2 for the dry density calculations. The volumetric parameters were most sensitive to variances in the measured total volumes of the soil samples at each effective stress. This could be improved by a procedural refinement. The total sample volume was dependent on the sample thickness at a particular effective stress (L_{load}). The absolute uncertainty associated with L_{load} was dominated by the uncertainty in the measured initial sample thickness (L_{init}). It was assumed that the soil sample's initial thickness after the load frame head was set in place was unchanged from the calculated L_{init} by indexing. The indicator used to prevent sample compression was the displacement of a marked line on the load frame's pedestal stem (Figure 1). This line could only realistically be judged for displacement with an accuracy of 0.1 mm.

Table 2: Variation of the Measured Dry Density

Soil Type	Load (tsf)	Dry Density (γ_d) (g/cm^3)			Average γ_d (g/cm^3)	Relative Uncertainty (%)
		Trial #1	Trial #2	Trial #3		
Untreated Dry	0	1.35	1.32	1.31	1.33	0.33
	0.25	1.50	1.46	1.50	1.49	0.37
	0.50	1.56	1.53	1.58	1.56	0.39
	1.0	1.62	1.59	1.66	1.62	0.40
	2.0	1.68	1.68	1.73	1.70	0.42
	4.0	1.74	1.74	1.80	1.76	0.44
	8.0	1.79	1.80	1.87	1.87	0.46
Treated Dry	0	1.58	1.51	1.46	1.52	0.36
	0.25	1.62	1.60	1.60	1.61	0.37
	0.50	1.64	1.62	1.64	1.64	0.37
	1.0	1.67	1.66	1.68	1.67	0.38
	2.0	1.71	1.70	1.73	1.72	0.39
	4.0	1.75	1.75	1.78	1.76	0.40
	8.0	1.80	1.81	1.84	1.82	0.42
Treated Wet	0	1.48	1.49	1.53	1.50	0.44
	0.25	1.64	1.59	1.60	1.61	0.48
	0.50	1.67	1.62	1.63	1.64	0.49
	1.0	1.71	1.67	1.66	1.68	0.50
	2.0	1.76	1.71	1.69	1.72	0.52
	4.0	1.82	1.76	1.73	1.77	0.53
	8.0	1.87	1.82	1.77	1.82	0.55

Initially, the batch treatment of Oshtemo B soil with HDTMA-Cl caused a reduction in the soil's hydraulic conductivity to water at low effective stress states. Figure 4 shows that the hydraulic conductivity decreased by approximately 50% when comparing the untreated-dry and the treated-dry samples, and by as much as 90% when comparing the untreated-dry and the treated-wet samples at an effective stress of 24 kPa (0.25 tsf). This implied that the initial handling of the treated sample, which involved chemical treatment and physical compaction, had already caused a bulk reduction in the pore space available for fluid conductance. Figures 5 and 6 supported this by illustrating the initial reduction in porosity and effective-pore-size index at low effective stresses.

Increasing the effective stress had a greater effect on the untreated soil such that the treated-dry Oshtemo B soil had a larger hydraulic conductivity at higher effective stress states. Figure 4 shows that the hydraulic conductivity of the untreated soil decreased almost two orders of magnitude over the same effective stress increase for which the conductivity of the treated soil decreased only one half to one order of magnitude. The amount of rearrangement and compression that took place in the soil matrix as the effective stress increased was dependent on its rigidity, which is ultimately a function of the soil structure. The data implied that the batch modification somehow altered the soil matrix to reduce its compressibility. The reduction in the medium's compressibility suggested that the treated soil did not have as much opportunity to rearrange due to physical compression because its fabric had already been altered by chemical treatment and compaction. This was reflected in the lesser degree that

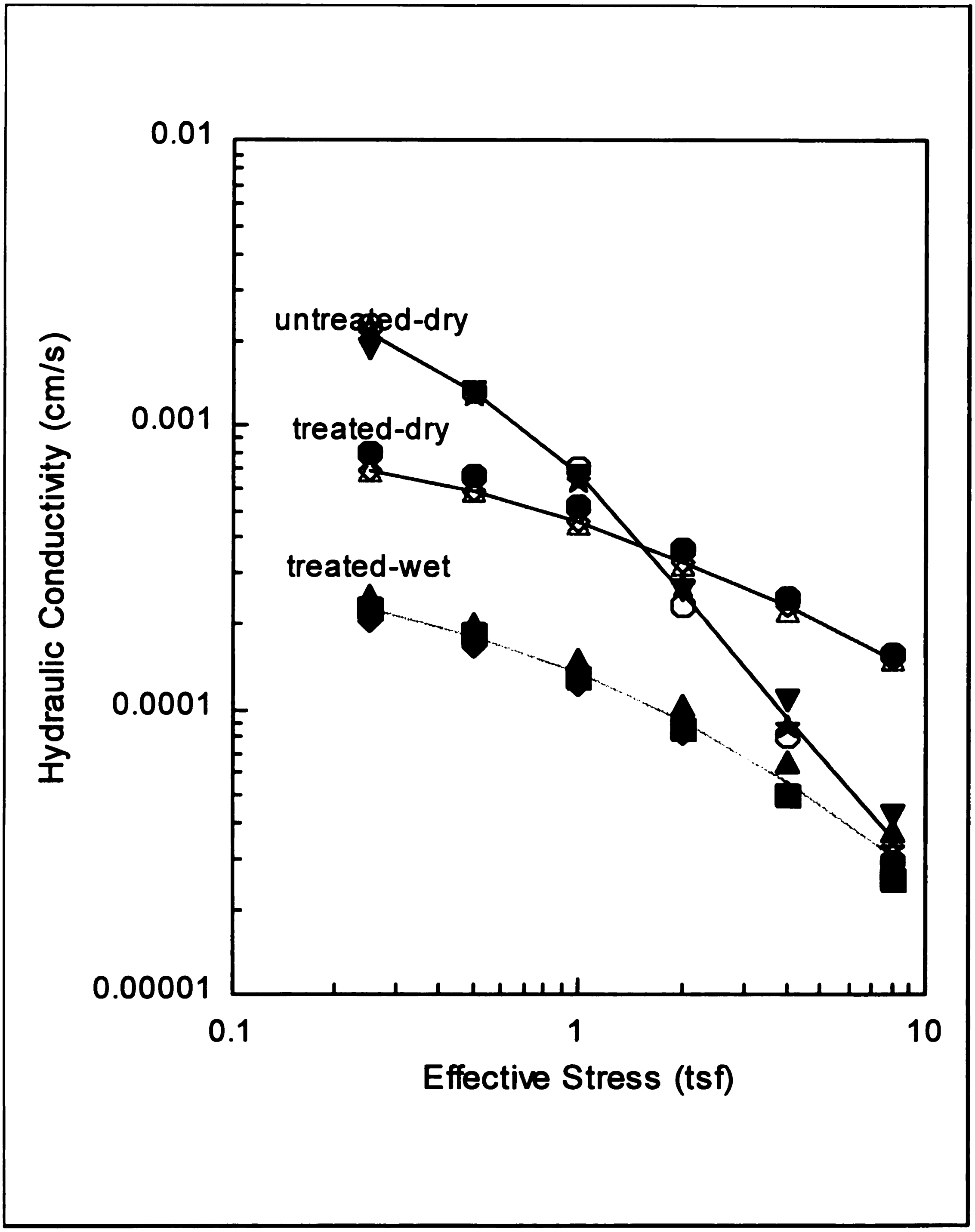


Figure 4: Hydraulic Conductivity vs. Effective Stress for Treated and Untreated Oshtemo B Soil

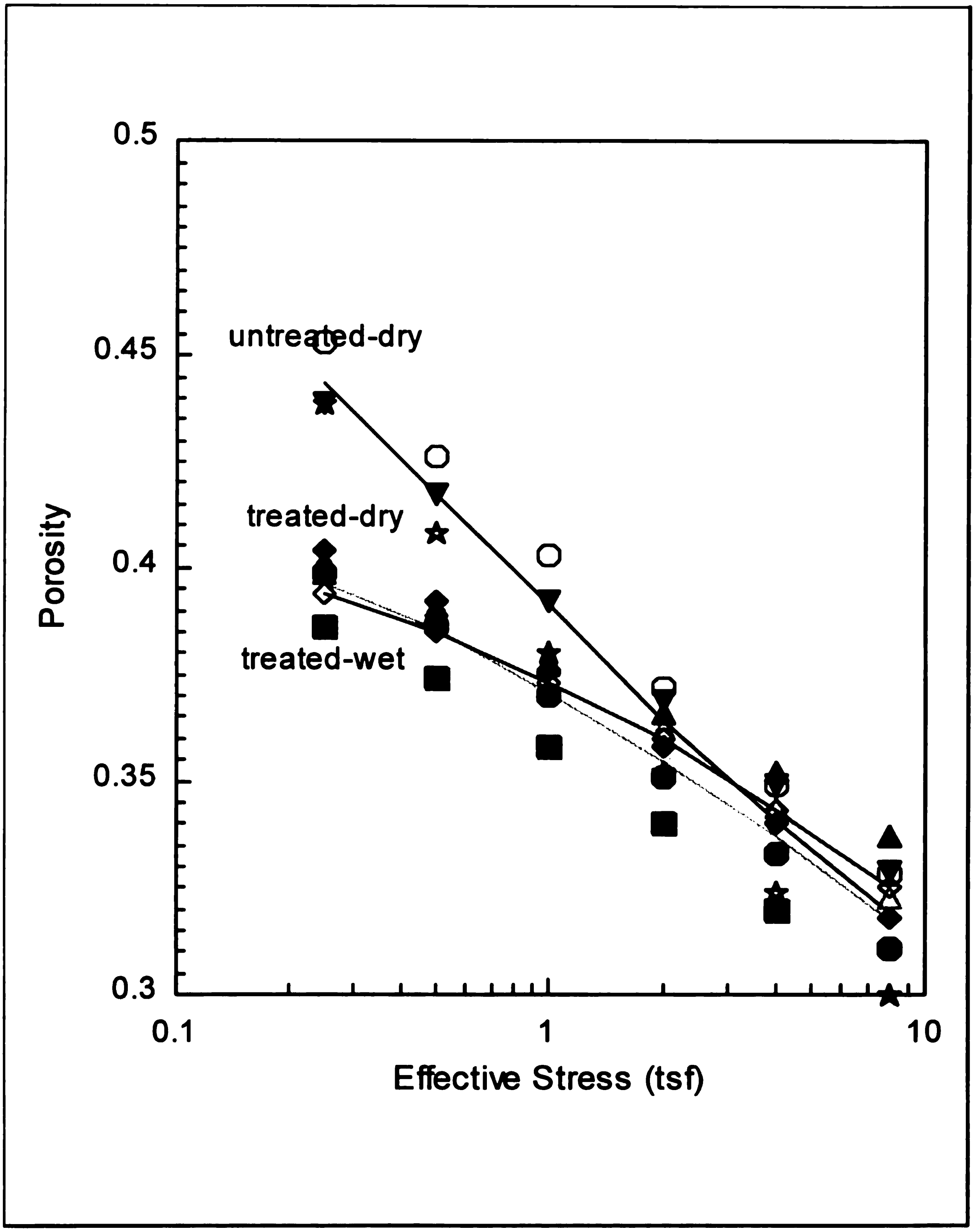


Figure 5: Porosity vs. Effective Stress for Treated and Untreated Oshtemo B Soil

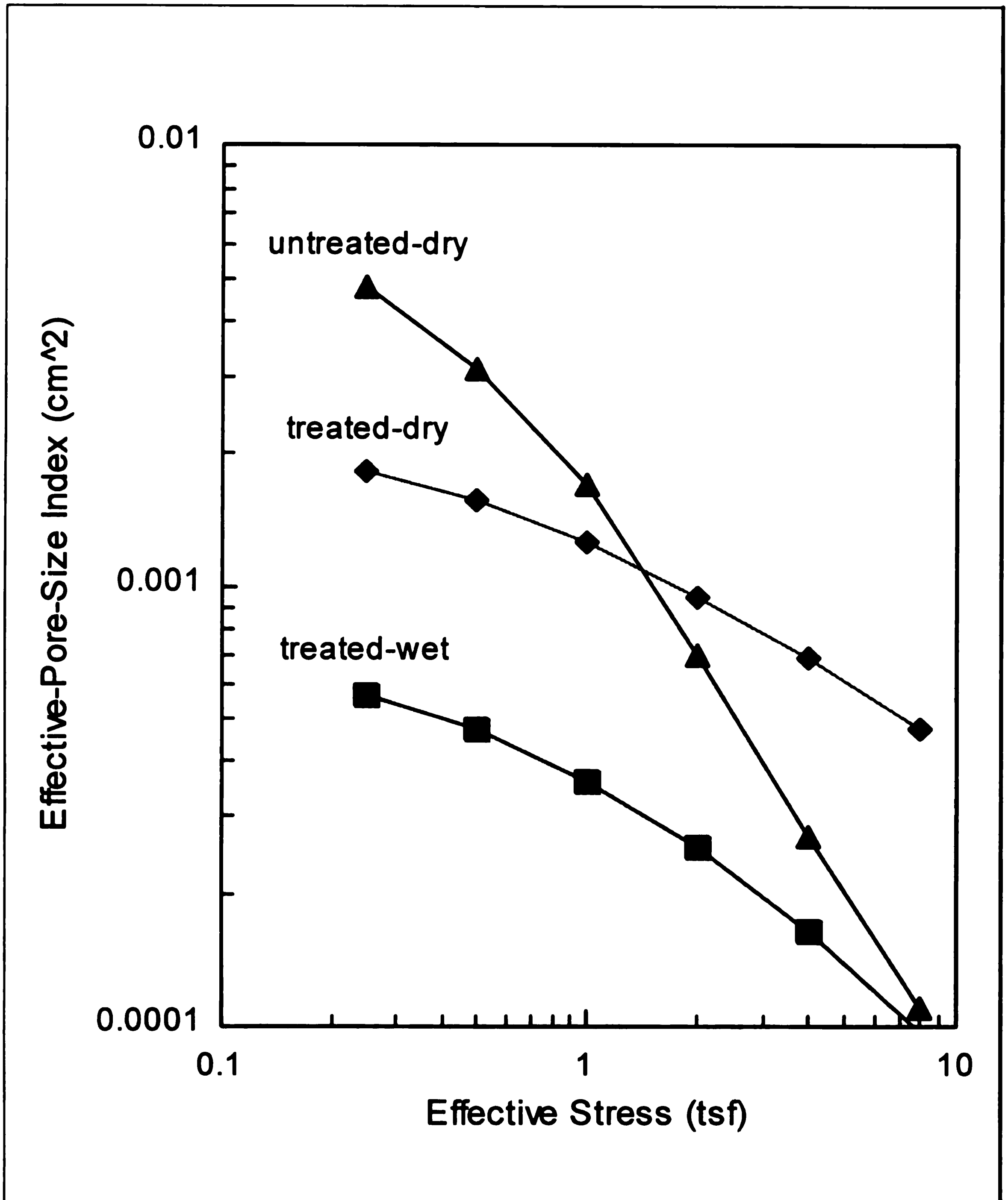


Figure 6: Effective-Pore-Size Index vs. Effective Stress for Treated and Untreated Oshtemo B Soil

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decreases in the batch treated soil's porosity (Figure 5) and other effects (Figure 6) occurred as the effective stress increased.

As indicated by equation (1), the hydraulic conductivity is proportional to the porosity (n) and to the effective-pore-size index (r^2); therefore, the observed decrease in conductivity as effective stress is increased can be discussed in terms of changes in these parameters. Porosity is simply a ratio of the void volume to the total soil volume (equation (2)) and contains no information describing the pore space geometry. Effective-pore-size index describes the distribution of space within the void volume, which the hydraulic conductivity is more dependent on. It should be noted that the data reported in Figure 6 is actually K/n and has incorporated the fluid specific weight and viscosity and the geometric constant from equation (1) into the effective-pore-size index term. This is based on the assumption that the fluid properties remained constant during experimentation, which is not unreasonable to make since correction factors are used to adjust all of the reported hydraulic conductivities to an equivalent value at 20 C.

The results presented from this investigation are collected using physical soil testing procedures in a fixed-ring consolidometer. Mechanical soil testing provides a macroscopic evaluation of the material; therefore, the data gathered by such means can provide only an indirect assessment of soil fabric as it relates to the first-order characterization of the soil matrix geometry (Yong and Warkentin, 1975). Conjectures beyond the general orientation of the clay fabric units, i.e. oriented or random arrangements, is not possible without an analysis involving microscopy procedures to verify the clay particle interactions.

The proposed interpretation was that the batch organo-modification procedure caused flocculation of the clay fabric at the first-order characterization into a more parallel arrangement. The preparation of the dry packed samples provided the initial evidence that the clay fabric units in the batch treated soil were more oriented. Table 2 shows that initially the untreated-dry sample had a lower dry density than the treated-dry sample even though a greater compactive effort per unit mass (4 tamps/130 g for untreated vs. 2 tamps/140 g for treated) was employed in the packing of the untreated Oshtemo B soil. The compactive effort measured the mechanical stabilization or densification energy imposed on a sample. The soil's resistance to this energy input provided an indication to the degree of randomness of the soil's clay matrix. It has been shown that the more random the clay fabric array is, the greater the resistance to compaction is (Lambe, 1958b; Haug and Wong, 1992). The lower dry density of the untreated Oshtemo B soil sample reflected the greater ability of the soil's clay fraction to resist compaction relative to that of the treated sample. The untreated Oshtemo B soil was expected to have a predominately flocculated clay fabric due to more than 90% of the exchangeable sites being occupied by divalent cations (71.6% Ca^{2+} and 22.5% Mg^{2+}). The treated-dry Oshtemo B soil was noticeably "slippery" to the touch and configured itself into a denser packing when compacted, indicating that the chemically induced arrangements of the clay aggregates facilitate grain slippage. This was consistent with observations that the more parallel the clay units are, the less stable and more readily remolded a soil was upon compaction (Lambe, 1958b; Haug and Wong, 1992). This sug

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suggested that the flocculation that resulted from batch HDTMA-Cl addition to the Oshtemo B soil was of a more parallel nature than the untreated soil's clay fabric.

Comparing the untreated-dry and the treated-dry results (Figure 5) showed that about 10% of the initial loss of conductivity may be attributed to the lost porosity. The remaining reduction in conductivity at an effective stress of 24 kPa (0.25 tsf) should be attributed to the change in the effective-pore-size index as shown in Figure 6. The initially larger porosity and effective-pore-size index of the untreated soil were indications that a more random arrangement, and hence a greater void volume, were present. This also suggested a greater potential for rearrangement of the granular matrix due to the unstable state of a random array. The greater response of the untreated soil's hydraulic conductivity as effective stress was increased was attributed to this rearrangement which was facilitated by the alignment of the initially random clay fabric. It has been shown that one dimensional compression of hydrophilic soils tends to align the clay fabric normal to the direction of pressure application and to decrease the spacing between adjacent fabric units (Lambe, 1958a,b; Leonards, 1962; Yong and Warkentin, 1975). The batch treated Oshtemo B soil exhibited a less dramatic reduction in hydraulic conductivity because the chemical alteration of the soil fabric seemed to be at the expense of the larger pores as indicated by the reductions in porosity (Figure 5) and effective-pore-size index (Figure 6). The treated soil's response to increasing effective stress supported the conjecture that the organo-modification process imposed a higher degree of orientation on the clay fabric. It appeared from Figure 5 and Table 2 that the compressive behaviors of the treated and the untreated soils converged at approximately 4 tsf as their macroscopic volumetric parameters became equal. This suggested that once all of the soil's clay fabric was predominately oriented and parallel to one another that the mechanical behavior was similar as effective stress increased.

The chemical rearrangement of the clay fabric resulted in macroscopically measured differences between the treated and the untreated soil response to initial compaction, one-dimensional consolidation, and permeation. At the microscopic level, modification of Oshtemo B soil with HDTMA-Cl has been shown to cause both aggregation and dispersion of the layered silicates, depending on the HDTMA loading levels (Xu and Boyd, 1994a,b). At loading levels below the cation exchange capacity (CEC), adsorbed HDTMA-Cl replaced the inorganic exchangeable cations (Ca^{2+}, Mg^{2+}, K^{+}, and Na^{+}) and compensated the negative charges of the layered silicates. A face-to-face, parallel type association developed due to the mutual attraction between the alkyl tails of the adsorbed HDTMA-Cl on the faces of adjacent clay sheets, and due to the tendency of the hydrophobic moieties of the adsorbed HDTMA-Cl to be removed from the water phase (Xu and Boyd, 1994a,b). At loading levels above the CEC, HDTMA adsorption by hydrophobic bonding caused the development of positive charges on the clay surface and ultimately dispersion (Xu and Boyd, 1994a,b). In the experiments reported herein the soil was treated at an HDTMA-Cl loading of about 100% the CEC, corresponding to the first plateau of Figure 2. Therefore, the face-to-face association should have been extensive because most of the accessible exchange sites were saturated with HDTMA-Cl, with little or no significant HDTMA-Cl adsorption by hydrophobic bonding. This face-to-face association imposed a higher degree of orientation on the clay fabric at a microscopic level. The measured macroscopic properties (volumetric parameters, hydraulic conductivities, and effective-pore-size indices) for a HDTMA-Cl loading of 100% the CEC were consistent with these microscopic observations

Drying the treated soil after the batch modification procedures altered the clay fabric such that the hydraulic conductivity of the treated-dry samples was always greater than the treated-wet samples (Figure 4). Initially the treated-dry Oshtemo B soil had a hydraulic conductivity one half order of magnitude greater than the treated-wet sample. Figures 5 and 6 show that the mechanical response and loss of pore volume of the treated-dry soil were indistinguishable from that of the treated-wet soil; however, the effective-pore-size index was larger. This indicated that the differences in hydraulic conductivity were due minimally to the total void volume that was available and essentially all due to differences in the distribution of pore space. Figures 4 and 6 suggested that drying the batch treated soil caused the clay aggregates to arrange themselves such that larger spaces were able to conduct fluid transport than when the treated soil remained water saturated. There was a shift from a large number of smaller pores in the treated-wet samples to a small number of larger pores in the treated-dry samples. This data also supported the conjecture that the treated clay fabric existed predominately within the pore space created by the granular skeleton as opposed to adhering to the soil minerals like a hydrophilic, natural clay would. The data showed that only the void space distribution was affected by the alteration of the treated clay fabric induced by drying (Figure 6), not the bulk mechanical response to effective stress increases (Figure 5). The non-clay fraction of the soil, i.e. sand and silt particles, were assumed to be unaffected by the ion exchange of

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HDTMA-Cl and therefore could be expected to be surrounded by films of water. It is hypothesized that the hydrophobic nature of the organo-modified clay limits its adherence or interaction with the sand and silt; therefore, limiting the treated clay to preferentially exist within the granular pore space until compression was significant enough to mask this effect.

Finally, a question can be raised regarding the effect of the permeant on the clay fabric. The HPLC-grade water that was used for these experiments had a low electrical conductance and hence a low ionic strength. The degree of clay flocculation in an untreated soil is dependent on the ionic strength of the solution flowing through it (Shackelford, 1994). Initially, after saturation, the inorganic salts that were precipitated on the dry soil have diffused into solution to satisfy some equilibrium between the pore water solution and the soil . There is some ionic strength associated with this equilibrium. As the falling-head permeability tests were carried out, the pore space was flushed with a solution that had a lower ionic strength. The lower the ionic strength of the solution, the more dispersion that occured during continued flushing as the clay structure attempted to reach a new chemical equilibrium. Back-to-back falling-head permeability tests indicated a decreasing hydraulic conductivity with increasing time at a constant stress state, which is consistent with a dispersion effect due to flushing. An analysis of the conductivity lost due to dispersion relative to that lost due to consolidation showed that the permeant effect contributed to less than 10% of the overall measured decrease in hydraulic conductivity for all of the experiments conducted.

IV. ENGINEERING SIGNIFICANCE

Superfund sites are predominately contaminated by organic chemicals. Of the ten treatability groups of contaminants found in soils, nine are groups of organic chemicals. The organic chemicals that possess high water solubilities, namely one- and two-carbon chlorinated solvents (i.e., trichloroethylene, carbon tetrachloride) and the aromatic hydrocarbon constituents of gasoline (i.e., benzene, toluene, xylene), become of special interest due to their low retardation factors and hence high mobility *in-situ*. Organo-modified soils become viable alternatives as remedial tools because they possess an increased potential to contain such contaminants within an engineered treatment zone and to act as barriers to further migration. The attractiveness of treated soil technologies is further enhanced by the commercial availability of large quantities of cationic surfactant (QACs) at reasonable costs (i.e., $0.50 to $1.00 per lb.), the wide variety of QACs available allowing for versatility in the production of organo-clays and adaptation to differing site characteristics, and the wide usage of QACs in other applications (i.e., detergents, fabric softeners, antistatic sprays, swimming pool additives) suggesting environmental acceptance (Boyd *et al.*, 1991).

Underground injection of cationic surfactants to increase the sorptive properties of subsurface materials has been suggested as a potential technology for greatly reducing the mobility of an organic contaminant plume (Burris and Antworth, 1992; Boyd *et al.*, 1991). A constructed sorption zone (Figure 7) in itself does not provide a final solution for containment of the contaminant mass; however, the retardation of such constituents

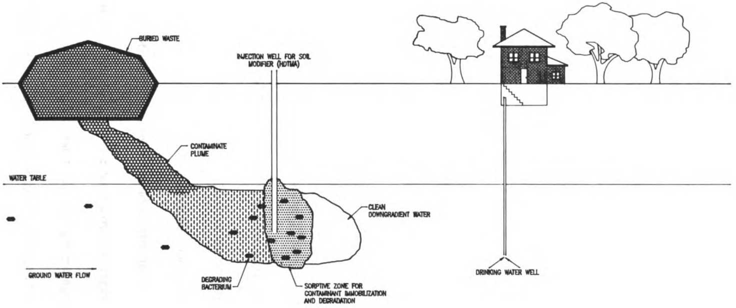

Figure 7: Schematic Representation of an In–Situ Sorption Zone Constructed by the Injection of a Cationic Surfactant Solution

within a defined treatment zone suggests that a manageable remediation technique is feasible when coupled with contaminant degradation mechanisms (Burris and Antworth, 1992). *In-situ* remediation, whether biotic or abiotic, can often be a slow process thus the enhanced retardation of a migrating plume provides time for slow reactions to occur. The advective transport of the dissolved contaminant to the sorption sites becomes important, hence the hydraulic conductivity of an aquifer material before and after modification is an important parameter for assessing the feasibility of such a technology. System failures due to hydraulic bypassing may result if the conductivity to the contaminated groundwater were reduced significantly. This could cause flow across the sorptive zone's face or around the zone entirely, allowing large fractions of the contaminant mass to never contact the interior sorption sites of the zone. Insignificant retardation would result and the integrity of the sorption zone would be lost.

Preliminary results from this investigation (Figure 4) indicated that batch treatment of an aquifer type material with HDTMA reduced the hydraulic conductivity by about 90% at low effective stresses. As the effective stress increased, the differences between the untreated and the treated soil's hydraulic conductivity decreased. It was hypothesized that the observed decrease in conductivity due to organo-modification was due, in part, to a reduction in the pore size caused by rearrangement of the clay aggregates in the pore space. These results allowed some conjectures to be made with regard to the feasibility of sorptive zones. First, the reduced hydraulic conductivity may cause hydraulic bypassing at shallow depths (i.e., less than 4 tsf of overburden) for aquifer material of a similar nature to Oshtemo B soil. Some kind of a modeling effort

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would be necessary to evaluate if the magnitude of the reduction was significant enough to direct the streamlines completely around the sorptive zone *in-situ*. It may be possible to compensate for the decrease in hydraulic conductivity by constructing a larger zone to intercept the contaminant plume. Second, the convergence of the treated-wet and the untreated-dry soil's hydraulic conductivity values at higher effective stresses suggested that at sufficient depths or high external loads, *in-situ* treatment can be implemented without producing decreases in the hydraulic conductivity. Third, the associated loss of porosity (Figure 5) due to organo-modification indicated that preferential flow paths may develop *in-situ* as a result of local settlement effects. This could promote faster routes through the sorptive zone and again reduce the mass of contaminant delivered to the interior sorptive sites.

It is important to note the limitations of the conjectures being made based on the measured data of this investigation. These are preliminary observations of the batch HDTMA-Cl treatment effects on a sandy loam material. The observed reductions in the hydraulic conductivity and in the effective-pore-size index may, in part, be induced by abrasive effects that occur during batch stirring procedures. *In-situ* applications such as the introduction of cationic surfactant solutions by underground injection wells would not involve such mixing and would involve more complex chemical dynamics. The efficiency of the ion-exchange process and the soil fabric alteration by abrasive motions of mixing come into question.

The abrasive motions induced by the stirring process used in this investigation were more directly related to currently practiced *in-situ* and *ex-situ* soil mixing techniques. With some adaptation, these systems could become useful as organo-modification tools. Soil mixing applications that could benefit most directly from the modified soil's increased sorption capacity and reduced hydraulic conductivity are vertical and horizontal barrier technologies. The more commonly designed vertical barrier systems are implemented using excavation-and-replacement methods that involve *ex-situ* soil mixing or using jet grouting methods that accomplish the soil mixing *in-situ*. The most common horizontal barrier systems are compacted clay liners such as those used for hazardous and solid waste landfills and for underground storage tanks (USTs).

Slurry trench cutoff walls are the most common vertical barrier technology (Evans, 1991). Typically a well-graded soil is mixed *ex-situ* with a bentonite clay and then placed in a trench to form a barrier to horizontal groundwater flow. The bentonite swells in the presence of water, plugging up the pore space and reducing the hydraulic conductivity. This serves to redirect the groundwater in a region of contaminated soil, preventing clean groundwater from entering the zone or any pumping, extraction, and treatment systems. In the presence of contaminants the swelling of bentonite is a reversible process (Evans, 1991). At sites where contaminant migration may destroy the barrier, modified soil becomes an attractive candidate as an admixture material. Evidence from this investigation indicated that a batch treated soil slurry (treated-wet samples) had a reduced hydraulic conductivity to water flow (Figure 4) and could thus contribute to the plugging of the pore space. Additionally, the enhanced sorptive capabilities of a modified clay would induce swelling in the presence of organic contaminants thus maintaining the integrity of the barrier wall. It is suggested that organo-modified clays or soils should be investigated as feasible components of slurry trench cutoff walls to enhance their resistance to flow in the presence of organic contaminants.

Jet grouting is an *in-situ* shallow and deep soil mixing technology. It involves the injection of pumpable materials into a soil and is commonly used for chemical stabilization processes in clays and for structural and water control processes in sands. The schematic diagram presented in Figure 8 suggests the feasibility of *in-situ* injection and mixing of soil with an abrasive action that is similar to that of the batch stirring used in this investigation. The "cement" and "additives" in Figure 8 could be replaced by an aqueous cationic surfactant solution and a natural soil to create a treated soil slurry, or the aqueous surfactant solution alone could be used as the pumpable suspension. The modified soil's hydraulic properties could be such that the region could act as a barrier or as a sorptive zone depending on the classification of the native soil and on the chemical composition of the injected suspension. It is implied that jet grouting, with some adaptation, could provide a means for *in-situ* construction of vertical barriers or sorption zones.

The use of compacted clays to minimize advective transport is well established. The most common uses of compacted natural clays are in the construction of hazardous and municipal landfills as shown in Figure 9. There is evidence suggesting that waste-soil interactions can have a negative effect on barrier technologies, causing the compacted clay fabric to flocculate and allow routes of exposure (Shackelford, 1994). This is reflected in the redundancy in the design of the barrier layers and the leachate collection systems, along with the EPA's concern that both geomembranes and natural clay liners

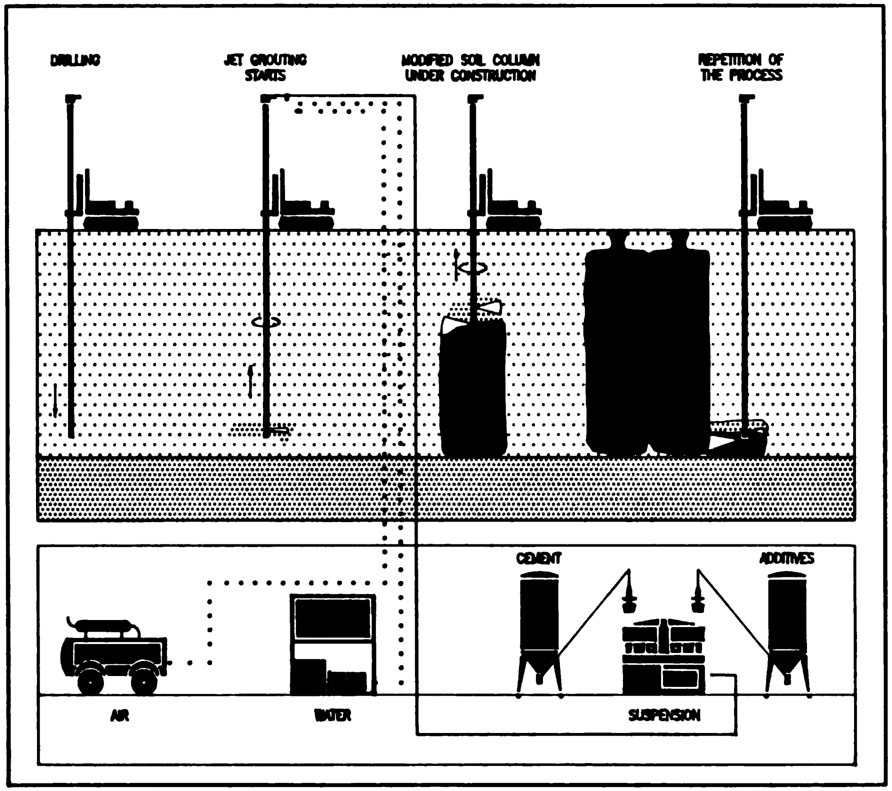

Figure 8: Schematic Illustration of a Typical Jet Grouting Operation

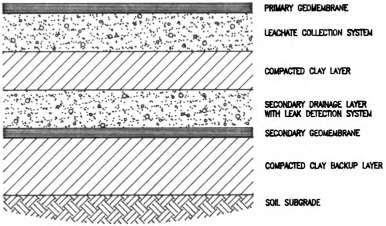

Figure 9: Typical Triple Liner System Showing the Redundancy in the Design of Horizontal Barriers

may be subject to degradation in the presence of organic contaminants (Alther *et al.*, 1990). To reduce the uncertainty in long-term effectiveness it becomes necessary to minimize the rates of contaminant migration by minimizing the hydraulic conductivity and rate of diffusion, thus maximizing the retardation. Organo-modified clays possess the properties to minimize the rate of organic contaminant transport. The additional adsorptive capacity of the treated clays becomes a useful tool for the collection of organic contaminants that migrate past the geomembrane in response to concentration induced diffusive fluxes or to advective fluxes through liner defects. Conceptually a composite barrier layer could be constructed that would include a modified clay component, or an actual modified soil layer could be incorporated to reduce the redundancy in design. Swelling is induced as the organic chemicals partition into the organic phase of the modified clays which serves to further reduce hydraulic conductivity and increase the time for chemical breakthrough. Furthermore, evidence from this investigation indicates that reductions in the hydraulic conductivity and the bulk density of treated soil could be accomplished with less compactive effort than that required for the untreated soil. The model that is hypothesized suggests that the batch organo-modification process oriented the clay fabric in a more parallel array, thus a more compact assemblage is attained with less compactive effort when compared to the unmodified soil. This suggests that a compacted, modified clay liner could be constructed in the field with less mechanical manipulation, and hence in less time, than a compacted, natural clay liner. It is proposed that organo-modified clays would be an effective attenuating component of a composite liner system.

V. CONCLUSIONS

The batch organo-modification of an aquifer type material with HDTMA-Cl caused an initial reduction in the soil's hydraulic conductivity to water at low effective stress states. The experimental results showed evidence that the loss of hydraulic conductivity resulted from a loss of porosity and an alteration of the pore size distribution at low effective stresses.

The batch treatment process also altered the compressibility properties such that treated soils exhibited less loss of hydraulic conductivity as the effective stress was increased. At higher effective stress states, the treated soil possessed a greater hydraulic conductivity if it was packed dry and an equal hydraulic conductivity if it was packed wet relative to the untreated soil that was packed dry.

The treated soil could not rearrange due to physical compression or conduct fluid flow as significantly as the untreated soil because its fabric had been altered. It was speculated through the engineering response of the natural and the modified soils that batch treatment processes changed the initially random clay fabric of the untreated soil to a more oriented, parallel type arrangement.

Additionally, the collected data showed that the void space distribution of the organo-modified soil was dependent on the state at which it was packed. The change in effective-pore-size index with an unchanging porosity between the wet and the dry packed treated samples indicated that the geometry within the pore space varied. The measured behavior suggested that the treated clay aggregates existed predominately within the pore space created by the non-clay fraction, i.e. the sand and silt grains.

APPENDIX A

APPENDIX A

PROCEDURAL DETAILS

The treated-wet samples were prepared using a fixed-ring consolidometer that was marked on the outside at the height corresponding to the top surface of the lower porous stone (the base of the soil sample). This assembly was placed within the desiccater system (Figure 3) without the consolidometer's base plug, stand-pipe, or upper porous stone. Best results were obtained when vacuum grease was used around the consolidometer O-ring to prevent leakage. HPLC-grade water was added to the 4000 ml vacuum flask until it was just below the vacuum spigot and to the desiccater until the level was just above the elbow joint for the stand-pipe. This insured that the static water level was above the top surface of the lower porous stone. The top of the desiccater was set in place and the 3-way stopcocks were sealed from the atmosphere by closing all the channels of stopcock no. 1 and closing the "to atmosphere" channel of stopcock no. 2. A vacuum was applied to both the desiccater and the flask for 1 hour. This allowed for deairing of the water within the system and to vacuum saturate the base components and lower porous stone of the fixed-ring consolidometer. Magnetic stir bars were used for agitation to promote faster deairing. The vacuum was collapsed on the desiccater only by opening stopcock no. 2 such that the flask was still isolated from atmospheric pressure. The top of the desiccater was removed and the water within the sample ring was drained by a siphon to a level equal to the outer marking on the consolidometer. The treated soil slurry was added to the consolidometer as described previously.

Once the treated soil slurry was in place, stopcock no. 3 was adjusted so that the channel to the flask was closed, then stopcock no. 2 was sealed from the atmosphere by closing the "to atmosphere" channel. This isolated the flask from atmospheric pressure and exposed only the desiccater to the vacuum. A vacuum was applied to the desiccater for 30 mins. to reestablish the negative headspace pressure. Vacuum saturation procedures then proceeded as described previously with the flooding of the desiccator assembly.

For the dry packed samples, HPLC-grade water was used to fill the 4000 ml reservoir flask to a level just below the vacuum spigot and to fill the desiccator to a level flush with the base plate. The soil sample and consolidometer were set inside the desiccator. The top of the desiccator was put into place and all the stopcock spigots were sealed from the atmosphere. A vacuum was applied to the system for 1 hour to deair all of the HPLC-grade water that would be used for saturation. Stir plates were used for agitation to promote faster deairing. Stopcock no. 3 was adjusted to isolate the reservoir flask from the vacuum, but to continue application to the desiccator. Stopcock no. 1 was opened to flow from the flask, allowing the deaired HPLC-grade water to flood the desiccator to a level just below the lip of the consolidometer's effluent reservoir, at that time stopcock no. 1 is resealed. The vacuum was then maintained on the desiccator overnight and the procedures continued as previously described.

The load frame had to be tared for the mass of the consolidometer and the soil sample before beginning the loading sequence. The consolidometer assembly was set on the load frame pedestal and the low pressure regulator was increased until the pedestal just began to rise. The pressure was then backed off a little to stabilize the pedestal in a stationary position. The load dial was set in the "off" position and the pressure transducer was zeroed. The load frame head was then lowered to just sit snug on the stainless-steel ball bearing, being very careful not to apply excessive mass to the consolidometer and induce some settling, and tightened into place. The displacement dial gage was tightened into position such that the tip contacted the consolidometer and there was enough play left in the stem to allow movement as the sample consolidated. The initial dial gage reading was noted from the linear vertical displacement transducer (LVDT). The first load increment was dialed using the low pressure regulator (see Appendix B for calibration data on each pressure transducer-load frame assembly that was used). At time zero the load dial is set on "load" to apply each pressure increment.

APPENDIX B

APPENDIX B

LOAD FRAME CALIBRATION DATA

NOTE: The calibration data was obtained from the load frame manufacturer.

<u>LOAD FRAME #1:</u> Serial No. = 270
Model No. = S-450

Table B1: Load Frame #1 Calibration Data

Pressure Transducer (psi)	Load Cell (lbs.)
0.80	10.00
1.20	20.00
2.00	40.00
4.80	100.00
9.50	200.00
18.80	400.00
37.30	800.00
56.00	1200.00

regression analysis: $Y(\text{lbs.}) = 21.5339284*X(\text{psi}) - 4.7530335$
$r^2 = 0.999988$

based on a 2.5 in. diameter soil sample:
$Z(\text{tsf}) = 0.31585362*X(\text{psi}) - 0.069720584$

This equation was used to calculate the required transducer pressure that would correspond to the desired sample pressure.

LOAD FRAME #2: Serial No. = 269
Model No. = S-450

Table B2: Load Frame #2 Calibration Data

Pressure Transducer (psi)	Load Cell (lbs.)
0.80	10.00
1.30	20.00
2.20	40.00
4.90	100.00
9.70	200.00
18.70	400.00
37.50	800.00
56.30	1200.00

regression analysis: Y(lbs.) = 21.4663518*X(psi) - 6.334829
$r^2 = 0.99997$

based on a 2.5 in. diameter soil sample:
Z(tsf) = 0.314862428*X(psi) - 0.0929175

This equation was used to calculate the required transducer pressure that would correspond to the desired sample pressure.

APPENDIX C

APPENDIX C

SAMPLE SPREADSHEET OUTPUT FOR DATA REDUCTION AND ERROR ANALYSIS

	A	B	C	D	E	F	G	H	I	J	K	L
1	Data Reduction and Error Analysis for 02/22/94 Experiment (untreated-dry pack):											
2							UD02/221 Data Red.&Err.					
3	SAMPLE DATA	st.pipe area =	0.335	cm^2 (plastic)								
4		sample area =	31.7	cm^2	constant = C	2.43E-02						
5												
6		ref. LVDT(in)=	0.2096	0.2097		E(Lref.) =	0.20965					
7		sat. LVDT(in)=	0.4057	0.4043		E(Lsat.) =	0.405					
8												
9			Expected Value	Variance (sq.units)								
10		init. hgt.(cm) =	3.036189	1.00E-04								
11		Wt. of Soil(g) =	130	2.50E-05								
12		Sp. Gravity =	2.67	4.00E-04								
13		Soil Vol(cm^3) =	48.68913858	1.33E-01								
14		Total Vol(cm^3)=	96.2471913	1.00E-01								
15												
16												
17												
18	LOAD DATA	Load (tsf)	Datum (cm)	Var (head)	Temp.	Dial Gage (in)	Smpl. Hgt.(cm)	Var (Smpl Hgt)	Void Ratio	Porosity	Dry Density	
19				(cm^2)	Correction			(cm^2)			(g/cm^3)	
20		0				0.071	3.036189	1.00E-04	0.976769237	0.494124058	1.35068877	
21		0.25	16.8	2.50E-03	1.018	0.189	2.736469	1.00E-04	0.781630767	0.438716473	1.49862702	
22		0.5	16.7	2.50E-03	1.018	0.2288	2.635377	1.00E-04	0.715812876	0.417185863	1.55611374	
23		1	16.7	2.50E-03	1.025	0.2719	2.525903	1.00E-04	0.644537723	0.391926384	1.62355655	
24		2	16.7	2.50E-03	1.025	0.3078	2.434717	1.00E-04	0.585169324	0.369152566	1.68436265	
25		4	16.7	2.50E-03	1.032	0.338	2.358009	1.00E-04	0.535227106	0.348630573	1.73915637	
26		8	16.7	2.50E-03	1.032	0.3648	2.289937	1.00E-04	0.490907521	0.329267586	1.79085554	
27												
28												
29		Load (tsf)	h1 (cm)	h2 (cm)	t(initial) (s)	t(final)1 (s)	t(final)2 (s)	t(final)3 (s)	Var (t(final))	Hyd.Cond.#1	Hyd.Cond.#2	Hyd.Cond.#3
30									(sec^2)	(cm/s)	(cm/s)	(cm/s)
31		0.25	1	11	0	16	16	16	0.083333333	1.84E-03	1.84E-03	1.84E-03
32		0.5	1	11	0	22	22	23	0.083333333	1.31E-03	1.31E-03	1.25E-03
33		1	1	11	0	41	42	44	0.083333333	6.76E-04	6.60E-04	6.30E-04
34		2	1	11	0	101	103	105	0.083333333	2.65E-04	2.59E-04	2.55E-04
35		4	1	9	0	166	170	173	0.083333333	1.10E-04	1.08E-04	1.06E-04
36		8	1	7	0	279	282	none	0.125	4.31E-05	4.27E-05	#VALUE!
37												
38												
39	Sensitivity Analysis of the Hydraulic Conductivity Measureands:											
40										(one std. dev.)		
41		Load (tsf)	Sample Height	Time	Initial Head	Final Head	Datum		Expected	Relative		
42									Value of k	Uncertainty		
43		0.25	1.34E-05	3.26E-04	5.29E-05	3.92E-04	1.57E-04		1.84E-03	3.07%		
44		0.5	1.44E-05	1.67E-04	5.24E-05	3.97E-04	1.61E-04		1.31E-03	2.82%		
45		1	1.57E-05	4.65E-05	5.24E-05	3.97E-04	1.61E-04		6.68E-04	2.59%		
46		2	1.69E-05	7.85E-06	5.24E-05	3.97E-04	1.61E-04		2.62E-04	2.52%		
47		4	1.80E-05	2.89E-06	1.06E-04	4.40E-04	1.14E-04		1.09E-04	2.61%		
48		8	1.91E-05	1.59E-06	2.32E-04	6.08E-04	8.87E-05		4.29E-05	3.08%		
49												
50												
51	Sensitivity Analysis of the Volumetric Measureands:											
52						Relative Uncertainty		(one std. dev.)				
53		Load (tsf)	Total Volume	Var(Total Vol.)		Void Ratio	Porosity	Dry Density				
54			(cm^3)	(cm^6)				(g/cm^3)				
55		0	96.2471913	1.00E-01		1.66%	0.84%	0.33%				
56		0.25	86.7460673	1.00E-01		1.90%	1.07%	0.37%				
57		0.5	83.5414509	1.00E-01		2.01%	1.17%	0.38%				
58		1	80.0711251	1.00E-01		2.16%	1.31%	0.40%				
59		2	77.1805289	1.00E-01		2.31%	1.46%	0.41%				
60		4	74.7488853	1.00E-01		2.47%	1.61%	Page 1 0.42%				
61		8	72.5910029	1.00E-01		2.63%	1.77%	0.44%				

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