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# Investigation of Electrokinetic Method to Reduce Sulfate Induced Heaving in Soils - A Laboratory Study

by

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## and

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### **Abstract**

Samples of soil from SH-118 near Study Bute were obtained with a nominal sulfate content of 27,000 ppm. Geotechnical characterization of the soil samples indicated that the soil is highly plastic in nature and consists of 13% (on an average) of soluble components. The chemical characterization in batches indicated sulfate contents of two to three times the nominal value.

Specimens of this soil compacted for a Liquidity Index of 50% were subjected to electroosmosis treatment. The test results indicated a removal rate of 7 mg/day of sulfates after the rate of removal becomes constant. The rate of removal will further decrease to a lower level as more sulfates are removed from the specimen. If this rate can be maintained throughout the treatment process, the completion of the clean up would require several years. This length of time is clearly excessive. Nevertheless, the major problem is posed by the large percentages of soluble components identified in the soil. The removal of all the sulfates would result in the removal of all soluble components. This would result in unallowable increases of the void ratio of the soil. The increase in void ratios would render any compacted layer inappropriate as a load-bearing layer. The main conclusion is that this soil can only be treated at the borrow area, before placement in the pavement structure. Nevertheless, the power and water consumption coupled with the long time delay needed for the treatment process precludes the application of this methodology in the removal of sulfates from the soil in SH-118 near Study Bute.

Another soil from the Fort Worth District with fewer sulfate contents was obtained. However, the evaluation of the soil indicated traces of sulfate. Thus, it was proposed to spike the soil with gypsum such that sulfate content of the soil would be from 1,000 ppm to 3,000 ppm. This amount of sulfate could be removed within a reasonable period. Four specimens were prepared with two water contents (95 and 50%) and two sulfate contents (1,000 and 3,000 ppm). The test results indicated that the sulfate removal was faster for 95% water content.

### **Executive Summary**

Samples of soil from SH-118 near Study Bute were procured with a nominal sulfate content of 27,000 ppm. Geotechnical characterization of the soil samples indicated that the soil is highly plastic in nature and consists of 13% (on an average) of soluble components. The chemical characterization in batches indicated sulfate contents of two to three times the nominal value.

Specimens of this soil compacted for a Liquidity Index of 50% were subjected to electroosmosis treatment. The test results indicated a removal rate of 7 mg/day of sulfates after the rate of removal becomes constant. The rate of removal will further decrease to a lower level as more sulfates are removed from the specimen. If this rate can be maintained throughout the treatment process, the completion of the clean up would require several years. This length of time is clearly excessive. Nevertheless, the major problem is posed by the large percentages of soluble components identified in the soil. The removal of all the sulfates would result in the removal of all soluble components. This would result in unallowable increases of the void ratio of the soil. The increase in void ratios would render any compacted layer inappropriate as a load-bearing layer. The main conclusion is that this soil can only be treated at the borrow area, before placement in the pavement structure. Nevertheless, the power and water consumption coupled with the long time delay needed for the treatment process precludes the application of this methodology in the removal of sulfates from the soil in SH-118 near Study Bute.

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The following can be concluded from this study: a) electrokinetic method can be used for removal of sulfates in the borrow pit area. It is not applicable for the soils in the existing roadways, b) soil should be mixed with de-ionized water to expedite the removal process, and c) the TxDOT procedure Tex 620-J should be modified. The sample from step *5* of procedure Tex 620-J should be split in two portions. One portion should be used as specified in Tex 619-J. The other portion should be used in Tex 619-J and skip steps 2 through 5. The difference in the precipitates obtained from these two portions should identify the actual amount of sulfates present in the soil.

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## **Implementation Statement**

The results of this study indicate that electrokinetics can be used in the cleanup of sulfates from soils. However, this process is not suitable for soils already on the roadway or the places where water is scarce especially de-ionized water. More laboratory and field studies are needed before electrokinetic process can be successfully used in the field.

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## **Investigation of Electrokinetic Method to Reduce Sulfate Induced Heaving in Soils - A Laboratory Study**

#### **Introduction**

The presence of soluble sulfates in natural soils has been linked to localized heaving of subbase clayey soils stabilized with lime and granular bases stabilized with Portland cement. The heaving is commonly attributed to the formation of ettringite due to the combination of tricalcium aluminate with calcium and sulfate ions. The formation of ettringite continues for as long as tricalcium aluminate and sulfate ions with sufficient moisture are available. This process continues during the hardening of the stabilized soils due to the pozzolanic reaction of lime and the clay minerals.

One potential approach, to eliminate or reduce the volume changes experienced by the stabilized soil, is to remove or reduce the sulfate ion concentration present in the natural soils whether before stabilization and/or after stabilization and compaction of the subbase.

A method based in the electromigration of charged ionic species toward electrodes, inserted in the soil, and subjected to a direct electric current is proposed as a possible field technique to be evaluated based on a laboratory program.

#### **Literature Review**

The cement research literature has amply documented attachment of the gypsum on Portland cement. This is commonly attributed to the formation of ettringite, by the combination of tricalcium aluminate with gypsum in the presence of water (Yan and Odler, 1995). Similar mechanisms have been documented in heaving granular soils (Hungtington et al, 1995; Dermatas, 1995) when stabilized with Portland cement or for lime stabilized clayey soils.

This section summarizes the most relevant information about electrokinetic treatment of soils and the occurrence of soluble minerals in soils.

#### *Electromigration*

Electrokinetics is the complex set of phenomenae that occur with the application of a direct electric current across a soil. One of the main effects is the electromigration of ionic species in the pore fluid toward the electrode of opposite signs. As the ionic species move, the hydration shells around the cation's move as well causing a net flow of pore water toward the cathode.

These phenomena have been under intense research consideration for application to the removal of soluble ionic species from contaminated waste sites, spill sites, etc. (Andrew and Probstein, 1993; Hamed, 1990; Renauld and Probstein, 1987; R. Lageman, 1993). Specifically, this technique has been used for applications dealing with the movement of sulfate ions in/out of sandy and clayey soils (Runnels and Wahli, 1993; Acaret al, 1997). Thus there is no question that as long as the sulfate salt dissolve in water and forms charged ionic species, these can be removed by electromigration.

The rate of ionic migration for a specific ion is a function of several factors such as: I) the electric potential gradient, 2) the concentrations of ions in the fluid (not only of the species of interest such as sulfate, but all the charged ionic species present), 3) the ionic mobility of the species of interest, and 4) the transference number of the ionic species of interest; this is basically the proportion of electricity carried by the species of interest relative to the amount of current carried by all other charged species in solution.

Perhaps the most important fact to realize is that all ionic species present will influence the rate of electromigration of the sulfate ions. This consideration suggests that the removal of sulfates from a soil although feasible might be quite inefficient when the sulfate ions are present in concentrations much smaller than those of the other ions present. In this sense, it has been suggested (Runnels and Wahli, 1993) that electromigration is best suited for the removal of major dissolved components rather than trace components. These conditions suggest that the electrolyte environment present in soil pores at the site of interest can be a very influential parameter in the applicability of electromigration to remove sulfate ions from the particular soil under consideration. Therefore, it is felt that the best alternative for this proposed laboratory evaluation would be to use a naturally occurring soil known to have presented heaving problems upon lime stabilization. Furthermore, these considerations would suggest that equilibrium batches at different ratios of soiVwater could be used to characterize the chemical species present in the soil in question.

One additional, seemingly very important, aspect that could also be addressed with the equilibrium batches would be the determination of soluble components that might exist in the soil more than their solubility limit. This aspect is related to the fact that some soil components have limited solubility (such as calcium carbonates, gypsum, etc.), thus, excess solid minerals can be found in equilibrium with a saturated pore-water solution. This excess mineral in solid form will then buffer a certain concentration of some ionic species in the pore fluid. Thus when the ionic species are removed by electromigration to the anode/cathode, the solid phase present will give more ions to replenish those removed. Thus in order to reduce the concentrations of these cations, maintaining the electromigration process would be necessary until all excess solid salt has been exhausted.

One aspect that limits the electric potential gradient application is the fact of the hydrolysis of water that takes place at the electrodes. At these sites, the water molecules dissociate into (H+) and (OH-). This process consumes energy that is not used to move the ions and, what is more important, it creates an acid environment (pH of about 2) at the anode and a basic environment (pH of about 12) at the cathode. These two environment form fronts that progress into the soil until the two fronts meet. The time required for this to happen has been shown (Hicks and Tondorf, 1994) to depend on the potential gradient. For potential gradients of 1 volt/inch and higher the typical time is only a few hours. Typically, the basic front penetrates about one third of the distance between electrodes and the acid front the remaining two thirds. The effect of these two environments is to induce drastic changes in the solubility of different electrolytes. The most common effect is that as the ions reach the high pH environment new products might precipitate depending on the solubility of the compounds of each ion. To reduce these pH changes and associated solubility changes in normal applications the imposed voltages are low and continuous flushing of the electrodes is normally provided.

One aspect that has not received much attention in the literature is the effect that the compaction of the soil might have on the rate of electromigration. Test performed on replicate specimens (Reddy, 1995; Kabir, 1996; and Reddy and Picomell, 1994) consolidated under different confining pressures show that the energy cost jumps by a factor of three as the confining pressure is increased from atmospheric pressure to 25 psi above atmospheric pressure.

In summary, all the considerations point to the convenience of using a natural clayey soil that has shown sulfate induced expansion upon lime stabilization. The results of the research project will indicate whether the removal of sulfate ions by electrokinetic would be effective for that particular soil.

The previous discussion also points to the fact that the removal of sulfate ions would be most effective before the soil is compacted. Furthermore, since there is a possibility of the presence of sulfate salts of limited solubility, it appears that a possible improvement in the process could be achieved by loosening the soil and increasing the water content before the application of the electric current. This would allow more sulfates to dissolve and the excess water would be removed during the electromigration phase.

Thus, the main effect proposed to be investigated at the present stage is that of the water content of the soil at the time of initiation of the electrokinetic process. Furthermore, it does not appear to be reasonable to investigate the application of the electrokinetic process to the lime stabilized soil. This is due to the large addition of soluble species caused by the stabilization and the fact of the very high pH of pore water in the stabilized soil. If electrokinetics were to be used under these conditions the low pH front generated at the anode will interfere with the pozzolanic reaction and the major ionic species would be  $Ca^{2}$  that would be removed by the electromigration process. Thus, major interference of the electrokinetic process with the stabilizing agent reaction would have to be expected.

#### *Soluble Minerals in Natural Soils*

Soils of arid and semiarid regions often contain evaporites because of little or no leaching. The chemical composition of extracts of the pore water of these soils indicated that the more common ionic species are Na<sup>+</sup>, Ca<sup>+2</sup>, Mg<sup>+2</sup> and Cl<sup>-</sup>, SO<sub>4</sub><sup>-2</sup>, HCO<sub>3</sub><sup>-2</sup>, and CO<sub>3</sub><sup>-2</sup>. The concentrations in percentage of soluble salts in alkali soils, (U.S. Salinity Lab Staff, 1954) ranges from 0.1% to 5%.

The sulfate minerals identified in soils (Doner, 1977) include gypsum  $(CaSO<sub>a</sub>2H<sub>2</sub>O)$ , hemihydrate (CaSO<sub>4</sub>·1/2H<sub>2</sub>O), mirability (Na<sub>2</sub>SO<sub>4</sub> 10H<sub>2</sub>O), thenardite (Na<sub>2</sub>SO<sub>4</sub>), epsomite (MgSO<sub>4</sub>)  $7H_2O$ , hexahydrite(MgSO<sub>4</sub>6H<sub>2</sub>O), and bloedite (NaMg(SO<sub>4</sub>)<sub>2</sub>4H<sub>2</sub>O). The solubility of these sulfate salts is about two orders of magnitude higher for the magnesium and sodium salts relative to the calcium sulfate salts (only  $0.2$  to  $0.3\%$  solubility). Due to the lower solubility of gypsum, the leaching of these salts requires higher availability of rainfall than for sodium or magnesium sulfates. Thus, in many alkali soils, gypsum is the major source of sulfate ions. The high solubility of  $MgSO<sub>4</sub>$ , NaSO<sub>4</sub>, and NaCl minerals causes their crystalline forms to be found only at the soil surface of extremely desiccated soils. These are not found at any appreciable depth in moist soil profiles (Kovda, 1946).

This discussion highlights two main causes of concern. First the sulfate ion's concentration in the pore fluid of alkali soils will most probably be a small fraction of the total ionic population in the fluid. Thus one concern has to be the characterization of all soluble species present in the soil of interest to elucidate the relative abundance of sulfate ions and thus assess the applicability of electromigration.

The second cause of concern is the fact of the low solubility of calcium sulfates. These salts, if present in the soil, will be present in excess of their solubility limit (about 0.2 or 0.3 grams per 100 ml of solution). Thus, additional water will be needed to increase the total sulfate in solutions prior to the application of the electrokinetic process.

From this last point of view, it is believed to be convenient to prepare some samples, for the electrokinetic process, spiked with a known content of gypsum to elucidate the effect that the presence of the solid salt might have on the rate of clean up of sulfates, such as the extra time required for treatment.

#### **Research Objective and Organization**

The main objective of this report is to describe the findings of the tests performed on the soil samples containing soluble sulfates. First the test methods used in this research are reported. The selection of material and test results are described in the next section. The last section consists of summary, conclusion, and future research.

#### **Research Approach**

It is proposed to obtain a sample of a soil known to have experienced heaving upon stabilization with lime. The first step is then to characterize this soil sample with routine Geotechnical tests. The chemical make up of the pore water and possible buffer mineral contents are then determined from equilibrium batch solutions.

In this study, the main variable investigated was the water content of the soil at the time of molding specimens. For this purpose, specimens were compacted at different water contents ranging from the optimum water content to the liquid limit.

These specimens were then subjected to electrokinetic treatment. Daily monitoring of the electrode well waters was implemented. The test was continued until no measurable sulfate ion concentration was detected for several consecutive days at the anode well. Upon completion of the electrokinetic treatment, the specimens were then cut into slices approximately 1 em long and total sulfate concentration was determined using a small portion of each slice.

The test results can then be used to assess the feasibility of removing sulfate ions from the soil in question and perhaps select the appropriate pretreatment of the soil to permit the most effective rate of electromigration.

### **Test Methodologies**

#### *Geotechnical Characterization*

Geotechnical characterization of the soil sample was performed to identify the properties of soil samples. The main emphasis was to identify the Atterberg limits of the soil. Tex-104-E and Tex-105-E tests were performed to identify the Atterberg limits. The TxDOT procedure Tex-11 0-E was also performed to obtain the soil classification.

#### *Content of Soluble components*

To determine the presence of soluble salts in the soil, the soil samples are continuously washed until all the soluble salts are removed from the soil. The loss in weight is used to calculate the percent of soluble salts present in the soil.

In this method, a soil sample of known weight is placed in a beaker. Then de-ionized water is added to the beaker and the weight is recorded. The soil suspension is stirred for an hour. The beaker is weighed and covered with a saran wrap to prevent any loss of water. After a day or two, the soil settled down and clear supernatant floats on the top. The supernatant is decanted and the electrical conductivity is measured. The beaker is filled again with the de-ionized water and weight is recorded. This process is continued until the electrical conductivity of supernatant becomes constant. At the end of this process, samples of the clear supernatant are oven dried to determine the soluble solids present in the water. The weight of solids per kilogram of water should be below 0.05 grams. The process of filling and decanting is continued if the value is above 0.05 gm/Kg of water. At the end of this process, the beaker containing soil is then kept in the oven for drying and loss of weight is calculated. The loss in weight of soil after washes is the weight of soluble salts.

#### *Sulfates and Chloride Determination (TxDOT Procedure)*

Sulfates and chlorides present in the soil are determined using TxDOT procedure Tex-620-J. This test is performed as per the TxDOT' s guidelines with only exception of using a nickel crucible rather than a platinum crucible.

#### *Ion Content Determination*

Ion concentration of the soil is determined in this study using three different methods. All the three procedures are as follows:

1. **Bach Method:** is a easy and quick test to measure the sulfate concentration in the supernatant. However, this is an approximate method. This method consists of a turbidity test. The test method can measure sulfate concentration from 50 to 250 ppm. Highly concentrated samples (out of range) had to be diluted to determine their sulfate concentration. The results are obtained in mg/L of sulfate solution. These values are then transformed into mg/Kg of soil.

- 2. Ion-Chromatograph: test is performed to identify concentration of ions present in the solution. The equipment used in this study is manufactured by Dionex and the model used in this study is Dionex 4000i series. The first step is to test different standard solutions at different dilution factors to establish a relationship between the ion concentration and the retention times in the Ion-Chromatograph. Result for the different standard solution is used to identify the concentrations of ions in the soil samples.
- 3. Atomic Absorption Spectrophotometer: test is similar to Ion-Chromatograph test except only cation concentration can be measured with this method. The equipment used in this study is manufactured by Buck Scientific and the model of equipment is 200 A. A relationship between Atomic Absorption (AA) reading with standard solution is developed and then the relationship is used to identify the cation concentrations in solutions of unknown concentration.

#### *Electro-osmosis Test for Sulfate Clean-Up*

The removal of sulfates from soil specimens is performed by electro-osmosis process. The dimension of specimens used in this study is 2in.(Diameters) X 5in.(Length) or2in.(Diameters) X 2 in. (Length). The specimen is prepared by mixing de-ionized water to provide a water content corresponding to Liquidity Index of about 50% (i.e., a water content mid way between Plastic and Liquid Limit).

The soil is compacted inside an acrylic tube. Care is exercised during compaction process to prevent any entrapment of voids or air bubbles in the specimen. The specimen is then placed inside an electro-osmosis cell as indicated in the sketch shown in Figure 1. A constant voltage of 1 volts/inch is applied to two graphite electrodes. The cathode well is provided with continuous washing of approximately four to five gallons of water per day to prevent the formation of a basic front. The water in the anode well is collected on a daily basis and the amount of sulfate removal is monitored using Hach method.

The samples collected from the anode well are, at a later time, analyzed using Ion-Chromatograph to provide more precise measurements.

#### Site Selection and Test Results

#### *Site Selection and Sample Collection*

The selection of the site and the collection of soil samples was agreed with Mr. Raymond Guerra, Project Director in the first week of September 1998. After discussion with the Project Advisory Committee, Mr. Guerra selected the site to be investigated as State Highway 118 (near Study Bute,



Figure 1. Sketch of Electro-Osmosis Test Setup

Texas). Mr. Guerra, Mr. Richard Williamee and the UTEP researchers visited the sites and collected samples from four different sites. Two of the sites were near to the road while other two sites were from borrow pit areas. The collected samples were brought back to the laboratory and were kept in the laboratory for drying at room temperature.

#### *Geotechnical Soil Characterization*

The soil was characterized as per the TxDOT tests procedure. The Liquid Limit and Plastic Limit of the soil were found to be 65 and 24, respectively (A Plasticity Index of 41). The test results show that the soil is highly plastic in nature. The same tests were repeated on the soil after having been cleaned of soluble salts. The Liquid Limit decreased from 65 to 62. The Plasticity Index also decreased from 41 to 30. The only difference between the two soil specimens was presence of soluble salts.

Sieve and hydrometer analysis was also performed on the soil samples. The results of the analysis are shown in Tables 1 and 2 and in Figure 2. The test results indicate that the soil can be classified as CH as per the USCS classification and A-7-6 as per AASHTO Classification.

#### **Content of Soluble Components**

Soil was washed for several days to determine the quantity of soluble salts present in the soil. Various proportions of soil and water were evaluated to identify the amount of soluble salts. The test results are shown in Table 3. The test results indicate that the soil consists of more than 13% of soluble salts. Minimum amount of salts were observed in beaker no.7 and maximum in beaker number 5. The results are highly variable and one of the explanation could be small quantity of soil used for washing. For instance, the increase in soil weight from 5 grams to 50 grams reduced the variability. In the authors' opinion, may be 100 grams or more soil samples should be washed for finding the amount of soluble salts. Although the results show variability, amount of the soluble salts present in the soil is very high.

#### *Sulfate and Chloride Determination (TxDOT Procedure)*

Sulfate and Chloride determination was performed as per the TxDOT procedure and the results obtained are shown in Table 4. The results are shown in parts per million (ppm). The results indicate that approximately 30,800 ppm of soluble sulfates are present in the soil. The sulfate concentrations are similar to that observed by TxDOT before construction of SH 118. The average chloride concentration in the soil samples was approximately 750 ppm.

Sieve No.	<b>Sieve Size</b> (mm)	<b>Sieve</b> Weight <u>(gram)</u>	<b>Sieve and Soil</b> (gram)	<b>Soil Retained on</b> each sieve (gram)	<b>Cumulative Soil</b> <b>Retained</b> (gram)	$\overline{\mathcal{N}}_o$ <b>Retained</b>	$\overline{\mathcal{G}_{o}}$ <b>Passing</b>
	4.75	525.8	525.8	0.0 <sub>1</sub>	0.0	0.00	100.00
	2.36	715.0	715.8	0.8	0.8	0.32	99.68
16	1.18	416.3	420.0	3.7	4.5	1.80	98.20
30	0.06	569.7	573.8	4.1	8.6	3.44	96.56
50	0.03	380.8	383.9	3.1	11.7	4.68	95.32
$100\,\mathrm{l}$	0.015	405.1	407.6	2.5	14.2	5.68	94.32
200	0.0075	303.7	307.8	4.1	18.3	7.32	92.68
Pan		501.0	501.0	0.0		0.00	
		<b>Sum</b>		18.3			

Table 1. Sieve Analysis of Soil (SH 118)

Table 2. Hydrometer Analysis of Soil (SH 118)

<b>Time Elapsed</b>	<b>Water Temperature</b>	<b>Hydrometer</b>	<b>Hydrometer</b>	<b>Corrected</b>	<b>Effective</b>	$G_{w}$	<b>Water</b>	<b>Particle</b>	Percent
(minutes)	(C)	<b>Reading</b>	<b>Reading</b>	Hydrometer	Depth	water	<b>Viscosity</b>	<b>Diameter</b>	<b>Finer</b>
		(actual)	<b>Correction</b>	<b>Reading</b>	(cm)		(poises)	(mm)	$(9_0)$
0.25	21	32.0		33.0	11.1	0.9980	9.84E-03	0.091	91.8
0.5	21	32.0		33.0	11.1	0.9980	9.84E-03	0.064	91.8
	21	31.0		32.0	11.2	0.9980	9.84E-03	0.046	89.0
2	21	29.0		30.0	11.5	0.9980	9.84E-03	0.033	83.4
	21	27.0		28.0	11.9	0.9980	9.84E-03	0.021	77.9
10	21	26.0		27.0	12	0.9980	9.84E-03	0.015	75.1
15	21	25.0		26.0	12.2	0.9980	9.84E-03	0.012	72.3
30	20	24.0		25.0	12.4	0.9982	1.01E-02	0.009	69.5
60	20	24.0		25.0	12.4	0.9982	1.01E-02	0.006	69.5
240	20	22.0		23.0	12.7	0.9982	1.01E-02	0.003	64.0
1440	20	<u>11.0</u>		12.0	14.5	0.9982	1.01E-02	0.001	33.4



Figure 2. Sieve Analysis of Soil (SH 118)

 $10<sup>°</sup>$ 

<b>Beaker</b> <b>Number</b>	<b>Initial Weight</b> of Soil $gram$	<b>Initial Weight</b> of Water (gram)	<b>Average Weight of Soil After</b> <b>Washing and Drying</b> (gram)	Soluble Salts <sup>+</sup> ( %)		
	50.2	934	45.46	10.43		
$\overline{2}$	50.6	1627	44.96	12.54		
3	50.2	460	42.679	17.56		
$\overline{\mathbf{4}}$	50.2	3705	44.72	12.21		
5	5.1	3911	4.33	17.78		
6	10.1	3894	9.63	4.88		
7	4.9	1903	4.7	4.26		
Average Salt Concentration $(\%)^* = 13.18$						

Table 3. Amount of Soluble Salts Present in the Soil Samples {SH 118)

<sup>+</sup>Referred to non-soluble components

• Average is calculated based on first four samples

#### Table 4. Amount of Sulfate and Chloride Present in the SH 118 Soil Sample {TxDOT Procedure)





b) Chloride



#### *Ion Content Detennination*

Both anions and cations present in the soil were determined. The sulfate concentrations present in the soil were determined by Hach method while the presence of cations in the soil was determined using Atomic Absorption method. For both tests, approximately 200 grams of representative soil were selected and divided in four portions of 50 grams each. Each soil sample was placed in a beaker and different amount of de-ionized water was added. Four different proportions were selected for the analysis in this study. The volumes of water used were approximately 0.5, 1, 2, and 4 liters. Thus, the soil to water ratios was different for each beaker. The supernatant from each beaker was used for performing both test procedures.

The recorded concentrations of sulfate anions, using Hach Method, are shown in Table 5. The test results indicate that the sulfate content varied from 27,624 ppm to 72,238 ppm for four different soil to water ratios. However, the sulfate content as per the TxDOT procedure was 30,863 ppm. The tests performed by TxDOT, before construction of SH-118, suggested sulfate concentrations of 27,000 ppm. Approximately 500 ml of water was used to wash the soil in the TxDOT procedure. For trials 1 and 2 of Table 5, the soil to water ratio is quite similar to the one used for TxDOT procedure and, thus, produces very similar results (such as 27,624 ppm versus 30,863 ppm). Clearly, the Hach method provides results similar to the TxDOT, when the amount of deionized water used to mix the soil is the same.

The results on the remaining trials of Table *5* show that a decrease in the soil to water ratios increases the quantity of soluble sulfates. The main concern is the quantity of sulfates present in the soil much larger than originally expected.

The samples used for Hach method were subjected to determination of cation concentration using an Atomic Absorption Spectrophotometer. Four cation concentrations were measured in this study and the results are shown in Table 6. The sodium and calcium concentration showed an increase when the water is increased from 0.5 to 1 liter. However, the concentration did not change much further for decrease in soil to water ratios. On the other hand, the potassium and magnesium concentrations showed no relationship to change in soil to water ratios. Average sodium and calcium concentrations (if first reading is eliminated) were found to be 1,337 ppm and 6,037 ppm, respectively. Similarly, average potassium and magnesium concentrations were found to be 63 and 112 ppm, respectively.

#### *Electro-Osmosis Monitoring*

Two specimens were prepared in this study. One specimen was *5* inches long while the other specimen was 2 inches long. A complete set of all the data collected during electro-osmosis process is reported in Appendices A and B. The cumulative amount of sulfate removed from both specimens is summarized in Figures 3 and 4. The test results indicate that the sulfate removal is faster in the starting days and then slow downs as the electro-osmosis process is continued. The slope of the lines indicates that 7 mg of sulfate is removed per day after the rate of removal becomes constant, for both specimens. The time required to remove this much sulfate will be more than two years. This time could be further increased as the rate of removal is expected to reduce even further.

<b>Trial</b> No.	Weight of soil (gram)	Weight of Water (gram)	<b>Dilution</b> (ml/ml)	<b>Sulfate</b> <b>Reading</b> (mg/l)	<b>Sulfate</b> Concentration (mg/l)	<b>Sulfate</b> Concentration (mg/Kg)		
$\mathbf{1}$	50.17	568	1/21	125	2625	27,082		
$\overline{2}$	50.17	568	1/21	130	2730	28,165		
<b>Average Sulfate Concentration (ppm) = 27,624</b>								
3	50.20	1053	1/17	137	2329	46,515		
$\overline{\mathbf{4}}$	50.20	1053	1/17	150	2550	50,923		
<b>Average Sulfate Concentration (ppm) = 48,179</b>								
5	50.60	1755	1/15	100	1500	50,525		
6	50.60	1755	1/15	99	1485	50,020		
<b>Average Sulfate Concentration (ppm) = 50,273</b>								
7	50.18	3886	1/11	90	990	75,678		
8	50.18	3886	1/10	90	900	68,798		
	<b>Average Sulfate Concentration (ppm) = 72,238</b>							

Table 5. Sulfate Concentration Using Hach Method (SH 118)



### Table 6. Cation Concentrations Using Atomic Absorption (SH 118)





#### c) Magnesium



#### d) Potassium





Figure 3. Accumulated Removal of Sulfates from 5 inch Specimen (SH 118)



Figure 4. Accumulated Removal of Sulfates from 2 inch Specimen (SH 118)

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The quantity of sulfates removed was measured on daily basis using the Hach method. Since Hach is an approximate method, the same samples were tested using Ion-Chromatograph equipment. The results obtained from both tests are shown in Figure *5* (for *5* inch specimen). The test results indicate that the Hach method is providing higher sulfate concentrations than Ion-Chromatograph, thus, the rate of sulfate removal is slower and may take even longer time to clean up the soil.

Mr. Raymond Guerra (Project Director) was contacted and a meeting was held at the UTEP for future research directions. During the meeting, it was decided to discontinue the electro-osmosis of both specimens. The specimens were dismantled and cut into slices to identify the quantity of soluble sulfates left in the soil. The test results are summarized in the Figure 6. The amount of sulfate left in the specimens is less near the anodes and more toward the cathodes. This indicates that the sulfates available at the anodes are removed at first and then the sulfates from adjacent area start to move toward the anode, thus, increasing the quantity of sulfates present in the center slice. For 2 inch specimen (cell B) this phenomenon is not obvious because of few number of slices and the distance between anode and cathode. Approximately 7% of sulfates were removed from 5 inches specimen within 110 days and 20% of sulfates were removed from 2 inches specimen within 90 days of electro-osmosis.

Although higher quantities of the sulfate were removed especially from 2 inch specimen, the complete removal of sulfates will take longer time, as shown in Figures 3 and 4. Both figures show that much of the sulfates were removed in the starting and the slope of the curve becomes flat as the process continues. Thus, the time required to remove first 20% of sulfates will be quite less than removal of the rest of the 80% sulfates 9 (2 inch specimen).

Since the electrokinetic process will be a time consuming and costly solution to the soil from SH<sub>118</sub> soil, the project director obtained another soil from Fort Worth District. The soil samples from two different sites were obtained. Various tests were performed, similar to the Study Bute soil, on the Fort Worth District soil. The results of the testing are summarized in the following sections.

#### **Highway 67 Soil from Fort Worth District**

#### *Geotechnical Soil Characterization*

The soil was characterized as per the TxDOT tests procedures. Two different soils from Highway 67 were provided by the Fort Worth District. One soil was borrowed from location B-1 and another one from location B-7. Both soils were tested for geotechnical characterization and the results are shown in Table 7. The test results show that the soil is highly plastic in nature.

Sieve and hydrometer analysis was also performed on the soil samples. The results of the analysis are shown in Figures 7 and 8. The test results indicate that both soils can be classified as CH as per the USCS classification and A-7-6 as per AASHTO Classification.


Figure *5.* Comparison of Hach and Ion-Chromatograph Methods (SH 118)

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Figure 6. Amount of Sulfate not Removed by the Electroosmosis (SH 118)



Figure 7. Sieve Analysis of Soil B-1 (Hwy. 67)

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Figure 8. Sieve Analysis of Soil B-7 (Hwy. 67)

<b>Soil Sample</b>	<b>Liquid Limit</b>	<b>Plastic Limit</b>	<b>Plasticity Index</b>
$R-$			
R.			

Table 7. Atterberg Limit Test Results of Hwy. 67 Soil

*Sulfate Determination (TxDOT Procedure)* 

Sulfate determination was performed as per the TxDOT procedure and the results obtained are shown in Table 8. The results are shown in parts per million (ppm). The results indicate that 1,750 and 3,700 ppm of soluble sulfates are present in the soils B-1 and B-7, respectively. The sulfate concentrations are lower for soil B-1 and B-7 in comparison to the results reported by the TxDOT. The sulfate contents reported by the TxDOT are 4,000 and 4,800 ppm for B-1 and B-7 soils, respectively.

**Table 8. Amount of Sulfate Present in the Soil Sample (TxDOT Procedure)** 

Soil Type	Я.	Sample Weight   Weight of Crucible ደ.	Crucible plus Residue .g	Residue $\left( {\bf g}\right)$	Sulfate (ppm
$B-1$	80.0	148.250	148.279	0.029	1,754
$B-7$	80.0	148.275	148.311	0.036	3,703

#### *Ion Content Determination*

Ions present in the soil were also determined using an Ion-Chromatograph. Approximately *500* gram of representative soil was selected from both soils and two trial tests were performed on each soil to determine the presence of ions in the soil. The results are summarized in Table 9. The test results indicate that the soil has insignificant amount of soluble salts present in both soils. The amount of Nitrate ions in both soils is less than 3 ppm. However, the presence of sulfate ions is less than *50* ppm for both soils. The results obtained from Ion-Chromatograph are in direct conflict with the results obtained using the TxDOT procedure (i.e., 50 ppm versus 3,700 ppm).

Since Ion-Chromatograph is an accurate test method, it was decided to investigate whether the sulfates, found by the TxDOT method, were clays rather than sulfates. Therefore, the TxDOT procedure to determine sulfate ions was followed again without adding barium chloride to the sample (specifically steps 2,3,4 and 5 in TxDOT procedure 619-J were omitted). H barium chloride is not added, there should not be any precipitate of barium sulfate. Thus, the TxDOT procedure would have to indicate zero sulfates. The samples were filtered and washed with hot de-ionized water to remove the chlorides, if any. Then, the filter was placed into the nickel crucible and oven-dried for one hour. Steps 6 through 11 ofTxDOT procedure were followed and the results are shown in Table 10. The results indicate that both samples do not have sulfates and the higher sulfate contents predicted by the TxDOT procedure are errors caused by the inability of the filter paper to retain all the clay particles and, thus, produce a false reading of sulfate content.



#### Table 9. Ion-Chromatography Test Results

#### b) Nitrate



Table 10. Amount of Clay Present in the Soil Sample (Modified TxDOT Procedure)

Soil $T$ ype	Sample Weight g)	Weight of Crucible g,	Crucible plus Residue g,	Residue g)	Clay (ppm)
$B-1$	80.0	148.225	148.265	0.04	2,421
$B-7$	80.0	148.231	148.265	0.034	3,498

#### *Electro-Osmosis Monitoring*

The amount of sulfates in both soils was minimal, thus, there was no need of cleaning of soil from Forth Worth District. It was decided to spike the soil by adding gypsum to the soil after discussing with the project director. Since both soils had similar properties, it was decided to use soil B-7. The gypsum was added such that the sulfate content in the soil would be 1,000 and 3,000 ppm. Again the samples were prepared with water contents near Liquid and Plastic limits. Thus, four specimens were prepared for electro-osmosis monitoring and the specimen information is summarized in Table **11.** All four specimens were 5 inches in length and 2 inches in diameter. All specimens were tested for a total of 76 days. A complete set of all the data collected during electro-osmosis process is reported in Appendices C through F.

Cell Designation	Water	<b>Estimated Initial Sulfate Content</b>		Specimen	Specimen	
	Content $\mathscr{C}_b$	ppm	mg	Dry Weight (g)	Water Weight (g)	
C	96	1,116	166	149	142	
D	44	1,116	269	241	105	
E	95	2,790	423	152	144	
F	50	2,790	617	221	102	

**Table 11. Summary of Specimens Prepared from Hwy. 67 Soil** 

The cumulative amount of sulfate removed from both specimens is summarized in Figure 9. The test results indicate that the sulfate removal is faster in the starting days and then slow downs as the Electro-osmosis process is continued. The slopes of the lines indicate that removal rate is less than 2 mg of sulfates per day. The cells with higher water contents (Cell C and E) showed higher cumulative removal of sulfates than cells with lower water contents (Cell D and F). The test results indicate that the water content affects sulfate removal from the specimens.

After 76 days of testing, specimens were dismantled and cut in the slices to identify the quantity of sulfates left in the soil. The amount of sulfate in each slice was added to identify the total amount left in each specimen. The results obtained from each specimen are reported in the Appendices C through F and are summarized in Table 12. This analysis again indicated that more sulfates could be removed when the water content was nearly 95%. Approximately 80% of sulfates were removed from Cell C and 61% of sulfates were removed from Cell E (both had 95% water content). The test results also indicate that estimated sulfate contents are somewhat different than the recovered total contents obtained from the effluent of the electroosmosis cell and from the specimen slices after the treatment. Some of this difference probably can be explained by the large number of determinations that were needed to accumulate the sulfate recovered in the effluent and the treated specimen.



Figure 9. Accumulated Removal of Sulfates from Cells C Through F (Hwy. 67)

The results of the study indicate that water content should be near the liquid limit to increase the sulfate removal rate. Also, the electro-osmosis process can only be used in the borrow area and not feasible for on road applications.





#### **Closure**

#### *Summary*

Samples of soil from SH-118 near Study Bute were procured with a nominal sulfate content of 27,000 ppm. Geotechnical characterization of the soil samples indicated that the soil is highly plastic in nature and consists of on an average of 13% of soluble components. The chemical characterization in batches indicated sulfate contents of two to three times the nominal value.

Specimens of this soil compacted for a Liquidity Index of 50% were subjected to electroosmosis treatment. The test results indicated a removal rate of 7 mg/day of sulfates after the rate of removal becomes constant. The rate of removal will further decrease to a lower level as more sulfates are removed from the specimen. If this rate can be maintained throughout the treatment process, the completion of the clean up would require several years. This length of time is clearly excessive. Nevertheless, the major problem is posed by the large percentages of soluble components identified in the soil. The removal of all the sulfates would result in the removal of all soluble components. This would result in unallowable increases of the void ratio of the soil. The increase in void ratios would render any compacted layer inappropriate as a load-bearing layer. The main conclusion is that this soil can only be treated at the borrow area, before placement in the pavement structure. Nevertheless, the power and water consumption coupled with the long time delay needed for the treatment process precludes the application of this methodology in the removal of sulfates from the soil in SH-118 near Study Bute.

Another soil from the Fort Worth District with fewer sulfate contents was obtained. However, the evaluation of the soil indicated traces of sulfate. Thus, it was proposed to spike the soil with gypsum such that sulfate content of the soil would be from 1,000 ppm to 3,000 ppm. This amount of sulfate could be removed within a reasonable period. Four specimens were prepared with two water contents (95 and 50 %) and two sulfate contents ( 1,000 and 3,000 ppm). The test results indicated that the sulfate removal was faster for 95% water content.

#### *Conclusions*

The following can be concluded from this study:

- Electrokinetic method can be used for removal of sulfates in the borrow pit area. It is not applicable for the soils in the existing roadways.
- Soil should be mixed with de-ionized water to expedite the removal process. Thus, this method is not feasible for the sites like Study Bute because of the water problem.
- The TxDOT procedure Tex 620-J should be modified. The sample from step *5* of procedure Tex 620-J should be split in two portions. One portion should be used as specified in Tex 619-J. The other portion should be used in Tex 619-J and skip steps 2 through 5. The difference in the precipitates obtained from these two portions should identify the actual amount of sulfates present in the soil.

#### *Recommendations for Future Research*

The results of the study indicate that the sulfates can be removed from soils in the laboratory. A future study should be performed to evaluate the effects of stabilization on heaving of the soil. Soil known to have problems of heaving should be cleaned for eliminating the soluble salts present in the soil. This soil should be spiked with known amounts of sulfates. After spiking, the soil should be divided in two proportions. One proportion of the soil should be subjected to electrokinetic process. Both portions can then be stabilized with lime for further evaluation of heaving. This proposed research can probably identify the effectiveness of the electrokinetic process in terms of reduction of heaving.

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Appendix A

Electro-Osmosis Data For Cell A (Five Inch Specimen)

#### Effluent Wt of Cathode pH E.C. **Current** Date Days Elapsed of Anode Well Bottle Anode Cathode Anode Cathode Cathode Cathode Cathode Cathode (mS) (ml) (lbs) (mS) (mS)  $(mA)$ 0.384 2110/98 21 60 24.8 2.604 6.107 2.0 0.4 0.550 2111/98 22 68 22.6 6.901 7.860 1.9 0.5 1.015 2112/98 23 66 29.0 2.605 7.810 2.0 0.5 0.635 2113/98 24 67 28.2 2.714 5.237 2.1 0.5 1.425 2117/98 28 76 21.1 2.004 8.295 2.5 1.0 0.761 2118/98 29 70 23.9 2.545 8.023 2.2 0.5 0.741 2119/98 30 68 18.5 2.701 7.198 2.1 0.6 0.706 2120/98 31 68 25.2 2.343 7.560 2.2 0.5 1.080 2124/98 35 69 18.2 2.369 7.860 2.2 0.7 0.981 2126/98 37 65 22.3 2.746 8.115 2.2 0.8 0.673 2127/98 66 23.9 2.283 6.551 2.1 0.7 3/2/98 41 62 25.5 1.858 6.154 2.7 0.6 2.280 3/3/98 42 70 23.6 2.338 9.860 2.1 0.7 0.637 3/6/98  $\overline{45}$ 1.010 69 22.0 2.576 5.888 2.0 0.5 2.800 319/98 60 27.2 1.889 7.348 2.6 0.6 1.070 3/10/98 70 20.0 2.328 9.913 2.2 1.2 2.190 3112198 51 66 29.6 2.424 8.597 2.4 0.8 1.110  $\frac{3/13/98}{2.2}$  52 70 22.3 2.657 9.193 2.2 1.0 0.981 3/16/98 66 38.8 2.453 8.119 2.3 0.7 0.948 3/17/98 56 74 22.6 2.662 6.728 2.1 0.7 0.973 3118/98 57 74 18.9 2.824 6.320 2.1 0.7 0.777 3/20/98 59 66 39.4 2.750 6.341 2.4 0.7 0.923 3/24/98 63 64 19.7 3.053 5.239 2.3 0.9 0.852 3/25/98 64 71 22.7 2.92 5.318 2.2 0.7 0.673 3/26/98 65 70 20.1 2.856 6.223 2.1 0.6 1.466 3/27/98 66 72 21.5 2.924 5.862 2.1 0.6 3/30/98 69 64 16.5 2.244 9.649 2.5 0.6 2.750 0.980 4/1/98 71 66 43.7 2.355 6.409 2.4 0.6 1.790 4/3198 73 66 18.5 2.363 8.492 2.5 1.1 4/8/98 78 68 15.6 2.281 5.707 2.5 0.9 1.130 4/16/98 86 68 27.8 1.863 10.272 2.8 1.3 2.920 1.086 4/17/98 87 66 14.2 2.145 9.364 2.3 0.8 1.421 4/20/98 90 61 36.5 2.042 8.732 2.7 1.0 1.078 4/21/98 91 70 13.4 2.223 8.981 2.3 0.7 1.658 4/23/98 93 66 25.8 2.363 9.126 2.5 0.8 1.017 4/24/98 94 70 14.2 2.175 8.971 2.2 0.6 3.340 4/28/98 98 60 33.0 1.964 8.915 3.3 0.7 0.526 4/30/98 100 70 14.5 2.43 8.959 2.2 0.7 0.950 5/1/98 101 70 16.7 2.468 8.649 2.2 0.5 5/4/98 104 66 31.6 2.079 8.944 2.5 0.6 0.622 0.288 5/6/98 106 65 27.1 2.176 8.303 2.4 0.4 0.231 5/8/98 108 64 24.7 2.312 8.353 2.4 0.4 5/11/98 111 62 35.7 2.099 7.885 2.5 0.5 0.467

#### **Electro-Osmosis Treatment- Cell A - 5 Inch Specimen Raw Data**

## pH Measurement of Anode and Cathode Wells Cell A - 5 Inch Specimen



**Daily Current Measurements** Cell A - 5 Inch Specimen



## **Electro-Osmosis Treatment - Cell A - 5 Inch Specimen Sulfate Removed from Specimen Calculated by Hach Method**







## **Electro-Osmosis Treatment -Cell A - 5 Inch Specimen Sulfate Removed from Specimen Calculated with lon Chromatograph**





## Sulfate Amount in Each Slice of Cell A After Electro-Osmosis Treatment

S04 Concentration Left in Specimen 'Cell A'



SO<sub>4</sub> Amount Left in Specimen 'Cell A'



Appendix **B** 

Electro-Osmosis Data For Cell B (Two Inch Specimen)

 $\mathcal{A}^{\mathcal{A}}$ 



## **Electro-Osmosis Treatment .. Cell B - 2 Inch Specimen Raw Data**





## **Daily Current Measurements** Cell B - 2 Inch Specimen



## **Electro-Osmosis Treatment - Cell B • 2 Inch Specimen Sulfate Removed from Specimen Calculated with Hach Method**

 $\mathcal{A}$ 





 $\mathcal{A}$ 

#### **Sulfate Amount in Each Slice of Cell B After Electro-Osmosis Treatment**







# SO<sub>4</sub> Amount Left in Specimen 'Cell B'



Appendix C

 $\mathcal{L}^{\text{max}}_{\text{max}}$ 

 $\sim 10^{-1}$ 

Electro-Osmosis Data For Cell C

## **Electro-Osmosis Treatment - Cell C - 5 Inch Specimen Raw Data**





Daily Current Measurements Cell C - 5 Inch Specimen



## **Electro-Osmosis Treatment - Cell C - 5 Inch Specimen Sulfate Removed from Specimen Calculated with Hach Method**




## Electro-Osmosis Treatment - Cell C- 5 Inch Specimen Sulfate Removed from Specimen Calculated with lon Chromatograph







## **Sulfate Amount in Each Slice of Cell C After Electro-Osmosis Treatment**





# 504 Amount Left in Specimen 'Cell c•



AppendixD

Electro-Osmosis Data For Cell D

## **Electro-Osmosis Treatment .. Cell D .. 5 Inch Specimen Raw Data**





pH Measurement of Anode and Cathode Wells

Daily Current Measurements Cell D - 5 Inch Specimen



#### **Electro-Osmosis Treatment • Cell D - 5 Inch Specimen Sulfate Removed from Specimen Calculated with HACH method**





## Electro-Osmosis Treatment - Cell D - 5 Inch Specimen Sulfate Removed from Specimen Calculated with lon Chromatograph





## **Sulfate Amount in Each Slice of Cell D After Electro-Osmosis Treatment**





S04 Concentration Left in Specimen 'Cell D'

Slice Number

SO<sub>4</sub> Amount Left in Specimen 'Cell D'



Appendix E

 $\sim 10$ 

Electro-Osmosis Data For Cell E

#### **Electro-Osmosis Treatment - Cell E • 5 Inch Specimen Raw Data**





pH Measurement of Anode and Cathode Wells Cell E - 5 Inch Specimen

## Daily Current Measurements Cell E - 5 Inch Specimen





## **Electro-Osmosis Treatment .. Cell E .. 5 Inch Specimen Sulfate Removed from Specimen Calculated with HACH Method**



## **Electro-Osmosis Treatment - Cell E - 5 Inch Specimen Sulfate Removed from Specimen Calculated with lon Chromatograph**







## **Sulfate Amount in Each Slice of Cell E After Electro-Osmosis Treatment**



# SO<sub>4</sub> Concentration Left in Specimen 'Cell G'

**Slice Number** 

# SO<sub>4</sub> Amount Left in Specimen 'Cell E'



Appendix **F** 

 $\mathcal{L}^{\text{max}}_{\text{max}}$ 

Electro-Osmosis Data For Cell F

## **Electro-Osmosis Treatment - Cell F - 5 Inch Specimen Raw Data**





pH Measurement of Anode and Cathode Wells Cell F - 5 Inch Specimen

## **Daily Current Measurements** Cell F - 5 Inch Specimen





#### **Electro-Osmosis Treatment • Cell F - 5 Inch Specimen Sulfate Removed from Specimen Calculated with HACH method**



## Electro-Osmosis Treatment - Cell F - 5 Inch Specimen Sulfate Removed from Specimen Calculated with lon Chromatograph






## **Sulfate Amount in Each Slice of Cell F After Electro-Osmosis Treatment**





## SO<sub>4</sub> Amount Left in Specimen 'Cell F'

