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16. Abstract

Sulfate-bearing subgrade soils treated with calcium-based stabilizers often experience heaving problems (three-dimensional swell) due to chemical reactions with the sulfate and/or sulfide minerals. This project focuses on determining the sulfate content at which the deleterious chemical reactions occur and on evaluating the effectiveness of alternative construction practices aimed at reducing swell in high-sulfate soils. These practices include extended mellowing, double lime application, and increasing field moisture contents. To determine what concentrations of sulfate are too high for stabilization with lime in Texas, a soil from the Vertisol order that contained no detectable sulfates was selected for three-dimensional swell measurements. Two different sulfate compounds were added to the soil: sodium sulfate (Na_2SO_4) and gypsum ($CaSO_4 \cdot 2H_2O$). Samples containing sulfate concentrations of 0, 1000, 2000, 3000, 5000, 7000, and 12,000 parts per million (ppm) were then subjected to a three-dimensional swell test for a minimum of 45 days.

Results of these systematic swell experiments revealed the following: (1) sulfate contents up to 3000 ppm can safely be treated with traditional lime stabilization; (2) coarse-grained sulfates take longer to swell and form deleterious reaction products; (3) mellowing effectively treats sulfate concentrations up to at least 7000 ppm; (4) higher molding moisture contents (2% above optimum) reduce swell better than optimum moisture; and (5) single application of lime reduces swell better than double application. This study, using systematic laboratory experiments, confirmed empirical field observations of sulfate limits presented by other investigators.

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HYDRATED LIME STABILIZATION OF SULFATE-BEARING SOILS IN TEXAS

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DISCLAIMER

The contents of this report reflect the views of the authors, who are responsible for the facts and the accuracy of the data presented herein. The contents do not necessarily reflect the official view or policies of the Federal Highway Administration (FHWA) or the Texas Department of Transportation (TxDOT). This report does not constitute a standard, specification, or regulation. The engineer in charge was Tom Scullion, P.E. (# 62683).

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CHAPTER 1   INTRODUCTION

Sulfate-bearing subgrade soils have caused tens of millions of dollars in damage to Texas highways over the last decade. Many subgrade soils treated with calcium-based stabilizers experience heaving problems (Figure 1) due to chemical reactions with sulfate and/or sulfide minerals. Field observations indicate that the reactions can be very rapid and occur overnight following a single rainfall event. In other cases the reaction is delayed and it may take years for the problem to manifest itself in terms of excessive pavement roughness.

**Figure 1. Vertical Hezaves Generated during Construction of U.S. 67
near Midlothian.**

Sulfate problems in cement and concrete research have been reported for more than 70 years.[1] In 1962, Sherwood[2] reported problems with sulfates in lime and cement stabilization

of soils. However, reports of sulfate-induced heave in subgrade soils received little attention until the mid 1980s. Formation of ettringite was determined to be the cause of heaving in a case study from the southern United States.[3] Mitchell's Terzaghi lecture was the first time sulfate-induced heave received national recognition.[4] He used a parking lot in Las Vegas that experienced heave 2 years after construction as an example to stress the importance of physicochemical and biological changes in soil mechanics: Mitchell reported ettringite and thaumasite were the cause of failure.

Hunter explained many of the physicochemical details concerning sulfate heave.[5] Hunter's experiments determined that four ingredients (lime, clay minerals, sulfate ions, and water) are needed to generate sulfate heave at 77ºF (25ºC), with sulfate ions being the key ingredient.

Previous studies have focused on mechanisms of sulfate heave, although few studies examined swell caused by lime stabilization of sulfate-rich soils. Mitchell and Dermatas systematically added sulfates ranging from 3000 to 62,000 ppm to artificial kaolinite- and montmorillonite-rich soils.[6] This study focused on extremely high sulfate concentrations and extended curing times, generally 30 days. In 1999, another study evaluated the effects of ground granulated blastfurnace slag as a stabilizer in an artificial kaolinite and sulfate-rich soil and a natural sulfate-bearing Kimmeridge Clay.[7] This study examined extremely high sulfate concentrations (11,200 ppm sulfate was the lowest) as well.

At the time of this report, the Texas Department of Transportation (TxDOT) Dallas and Fort Worth Districts do not recommend using calcium-based stabilizers for subgrade stabilization if the sulfate levels are greater than 2000 ppm in a soil. This limit is based on empirical field observations and experience.

This research focuses on adding very low sulfate concentrations (0, 1000, 2000, 3000, 5000, 7000, 10,000, and 12,000 ppm) to a natural soil to measure three-dimensional (3-D) swell. With an understanding of the sulfate-heave mechanism, this study will identify the sulfate concentrations that cause unacceptable 3-D swell with lime stabilization.

The primary objectives of this study are as follows:

1. assess the 3-D swell potential of lime stabilized, sulfate-bearing, subgrade soils;

2. determine the sulfate level safe for traditional lime stabilization; and

3. assess the effectiveness of mellowing, double lime application, and increased moisture

content in reducing swell in high-sulfate soils.

A sulfate-deficient soil of the Vertisol order with a Plasticity Index (PI) of 24 was chosen as the soil to add selected concentrations of sulfate since that is what underlies a large portion of the Texas Coastal Plain, where most of the sulfate problems have been reported (Figure 2).[8]
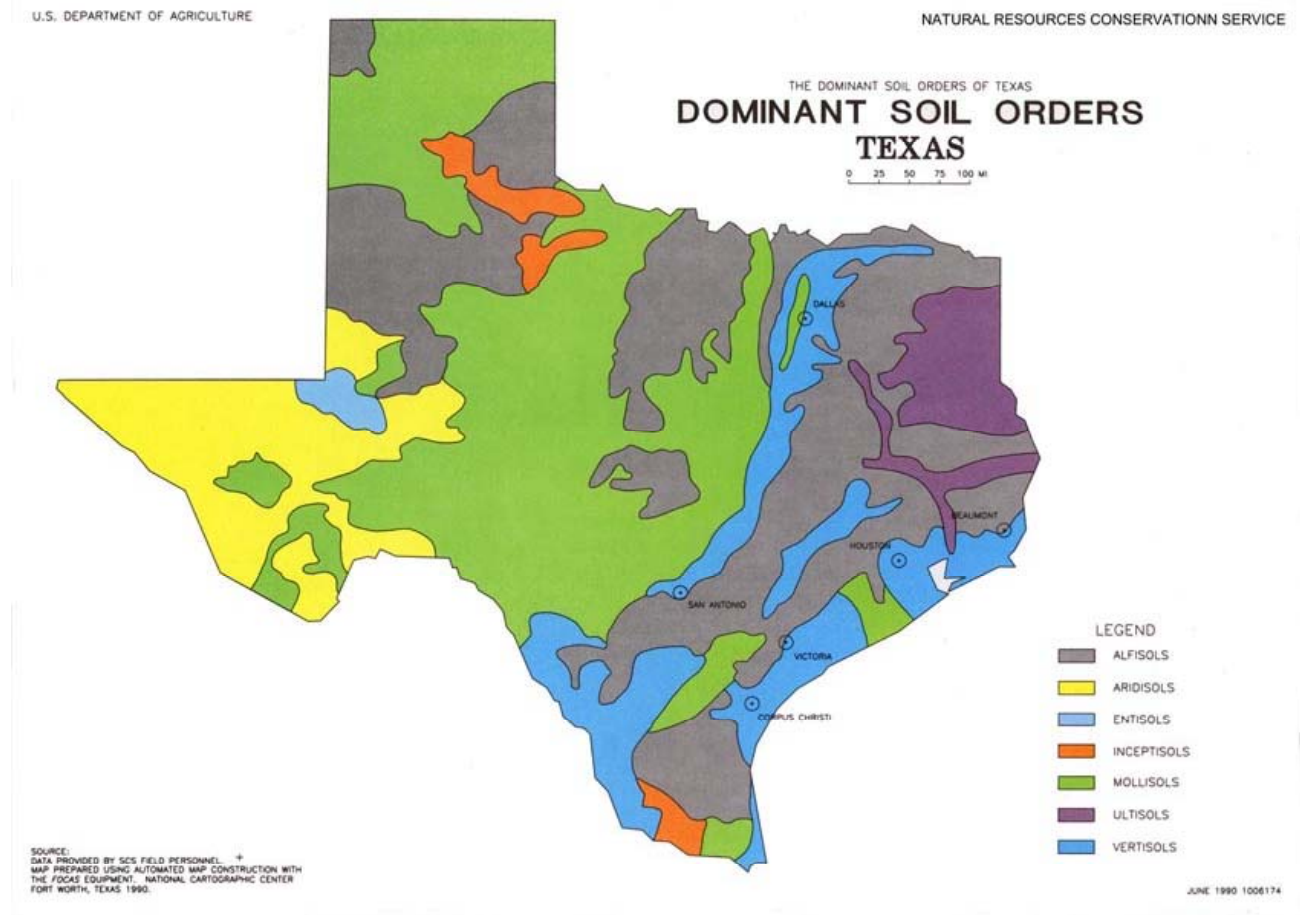
Figure 2. Map Showing Major Metropolitan Areas Constructed on Vertisols.

CHAPTER 2   BACKGROUND

PREVIOUS INVESTIGATIONS

The most severe heaves reported in Texas were observed at Joe Pool Lake near Dallas.[9] Burkart et al. identified certain geologic formations that possess high levels of sulfates and determined that gypsum was the most common sulfate in Dallas area soils.[10] The most severe heaves in the Dallas/Fort Worth area are associated with the Eagle Ford Formation shown in Figure 3 below. Since the inception of this project, other areas with high sulfate concentrations have been identified around Texas. Counties known to have problematic sulfate concentrations are identified in Figure 3.
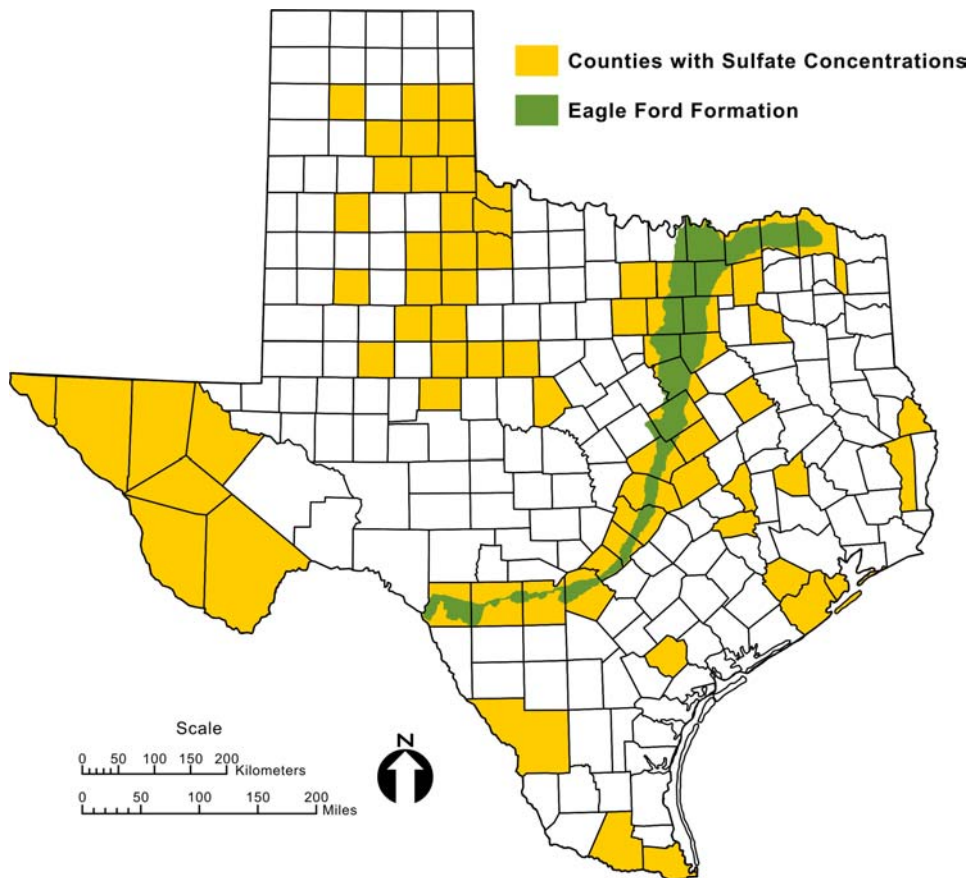
Figure 3. Map of Counties in Texas with Potential Sulfate Problems.

Researchers at Louisiana State University investigated the possibility of anhydrite ($CaSO_4$) converting to gypsum in a humid environment as the heave mechanism.[11] They determined that heave was actually due to formation of ettringite in the cement-stabilized soil.

It is also important to recognize that gypsum is not the only problematic mineral in soils. Pyrite (FeS_2) is a sulfide mineral that alters to gypsum ($CaSO_4 \cdot 2H_2O$) under the right conditions and creates similar problems. Dubbe et al. reported five case histories where pyritic shales oxidized to sulfates, causing heave and concrete deterioration.[12] Pyrite-derived sulfate was documented as the cause of heave in Portland cement-stabilized minestone.[13] Oxidation of pyrite-bearing Eagle Ford shale in north Texas is the source of sulfates in many soils in that region.[10]

Sulfate-Induced Heave Chemical Reactions

The literature on sulfate-induced heave in soils reports many mechanisms by which heave may occur. A detailed explanation on possible heave mechanisms is given in Mitchell and Dermatas;[6] however, only two will be discussed briefly as follows:

1. Sulfide minerals oxidize and react with other soil minerals to form sulfate minerals.[12] This transformation involves an increase in volume due to variations in atomic packing as well as the addition of water to the mineral structure.

2. The formation of the mineral ettringite, which only occurs under special circumstances, causes expansion of up to 250 percent when completely formed.[14] Hunter[5] performed extensive experiments to elucidate factors controlling the formation of this expansive mineral.

The first mechanism of sulfate-induced heave mentioned above is oxidation of sulfides. Pyrite and marcasite (both minerals are FeS_2 but the atoms are arranged differently) form under a reducing (oxygen deficient) environment and are not stable in an oxygen-rich environment. They are abundant in many coals, carbonaceous shales, and limestones. Often these rocks are exposed to the atmosphere during road construction. Upon exposure, $O_2(g)$ from the atmosphere serves as an oxidizing agent for pyrite and marcasite.

The following reaction (Rxn) illustrates what takes place:

$$4FeS_2 + 15O_2 + 14H_2O \rightarrow 4Fe(OH)_3 + 8H_2SO_4 \qquad \text{Rxn. 1}$$
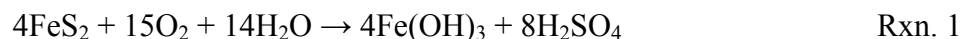
The iron and sulfur are oxidized by surface water that is enriched in atmospheric oxygen. The iron generally precipitates as a ferric hydroxide and the sulfate will either remain in solution or precipitate as gypsum if there is sufficient calcium present. The source of calcium is often limestone ($CaCO_3$), which is very soluble in acids. Looking at the right side of Rxn. 1, there are 8 moles of sulfuric acid (H_2SO_4) released in the weathering of 4 moles of pyrite or marcasite. This will make the surrounding environment very acidic and promote the dissolution of limestone (Rxn. 2) which will supply Ca^{2+} for the formation of gypsum (Rxn. 3).

$$CaCO_3 + H_2SO_4 \rightarrow Ca^{2+} + (SO_4)^{2-} + H_2O + CO_2 \hspace{2cm} \text{Rxn. 2}$$

$$Ca^{2+} + (SO_4)^{2-} + 2H_2O \rightarrow CaSO_4 \cdot 2H_2O \hspace{2cm} \text{Rxn. 3}$$
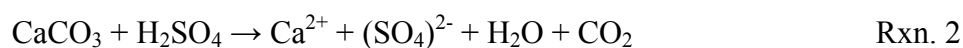
The mineral transformation of pyrite to ferric hydroxide and gypsum results in an increase in volume. The oxidation of pyrite and formation of gypsum alone is responsible for distress experien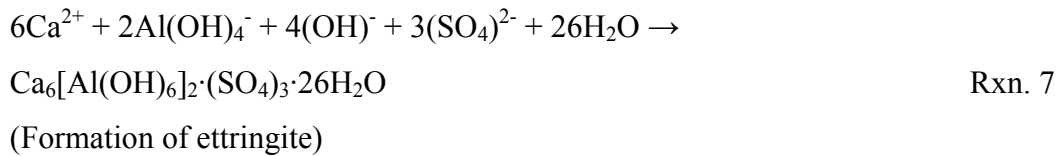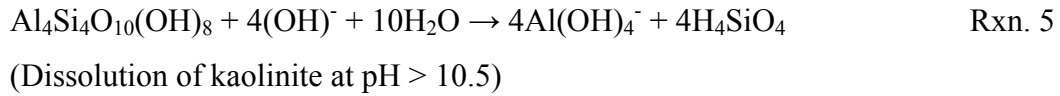ced in some construction projects.[12] In other projects, where traditional calcium-based stabilization is performed, other deleterious reactions may occur. Heave caused by calcium-based stabilizers in sulfate and clay-rich environments is mainly due to the formation of hydrous calcium-hydroxide-sulfate minerals.

The second mechanism mentioned on page 6 is the formation of ettringite, $Ca_6[Al(OH)_6]_2 \cdot (SO_4)_3 \cdot 26H_2O$, which requires unique conditions to form. At standard temperature (25ºC) the pH has to be above 10, and a source of water is critical for the 26 moles of water in the mineral structure; additionally, a source of aluminum, sulfur, and calcium are also required to form ettringite. When sulfur-bearing clay-rich soils are stabilized with lime or cement, then all of the above criteria are met. Lime and cement both raise the pH to above 12, which causes dissolution of clay minerals and releases aluminum into the system. Water may be supplied from a number of sources: during the stabilization process, as precipitation after stabilization, or from the groundwater or adjacent reservoirs. Calcium is released by the lime and cement during stabilization, and the sulfur is supplied from the sulfide- and/or sulfate-bearing soils or water.

The following is an abbreviated geochemical reaction model from Hunter:[5]

$$Ca(OH)_2 \rightarrow Ca^{2+} + 2(OH)^- \qquad\qquad\qquad \text{Rxn. 4}$$

(Ionization of lime; pH rises to 12.3)

$$Al_4Si_4O_{10}(OH)_8 + 4(OH)^- + 10H_2O \rightarrow 4Al(OH)_4^- + 4H_4SiO_4 \qquad \text{Rxn. 5}$$

(Dissolution of kaolinite at pH > 10.5)

$$CaSO_4 \cdot 2H_2O \rightarrow Ca^{2+} + SO_4^{2-} + 2H_2O \qquad\qquad\qquad \text{Rxn. 6}$$

(Dissolution of gypsum)

$$6Ca^{2+} + 2Al(OH)_4^- + 4(OH)^- + 3(SO_4)^{2-} + 26H_2O \rightarrow$$
$$Ca_6[Al(OH)_6]_2 \cdot (SO_4)_3 \cdot 26H_2O \qquad\qquad\qquad\qquad \text{Rxn. 7}$$

(Formation of ettringite)

Rxns. 4 and 5 occur in any lime-stabilized kaolinite-bearing soil. Addition of lime to the soil causes the pH to rise to approximately 12.3, releasing large amounts of calcium to the soil. Clay minerals are unstable at a pH above 10.5, so the clays start breaking down into aluminum hydroxide and silicic acid. Sulfate ions (Rxn. 6) are supplied by the dissolution of gypsum. The only other elemental requirement for the formation of ettringite is water. Ettringite only forms in a high pH ≈ 10-12 environment. Once the pH drops below 10 ettringite stops forming. In this example, kaolinite is the aluminum source and gypsum is the sulfur source; aluminum may be derived from dissolution of any clay mineral and sulfur may be derived from any sulfur-bearing mineral as previously discussed. Gypsum is used as the sulfur-bearing mineral in this example, and it appears to be the dominant sulfur-bearing mineral responsible for sulfate-induced heave in Texas soils.[10]

Sulfate-Induced Heave Reaction Rates

The speed at which the aforementioned reactions proceed is controlled by a number of factors, namely the temperature, concentrations of reactants and products, and the rate of mass transfer into and out of a fixed reaction site.[15] An important aspect of the rate of mass transfer involves the grain size of the sulfate minerals. For example, a soil containing large gypsum

($CaSO_4 \cdot 2H_2O$) crystals will dissolve more slowly than a soil containing small crystals. Texas soils have a range of grain sizes that influence how rapidly sulfate heave reactions occur (Figure 4).
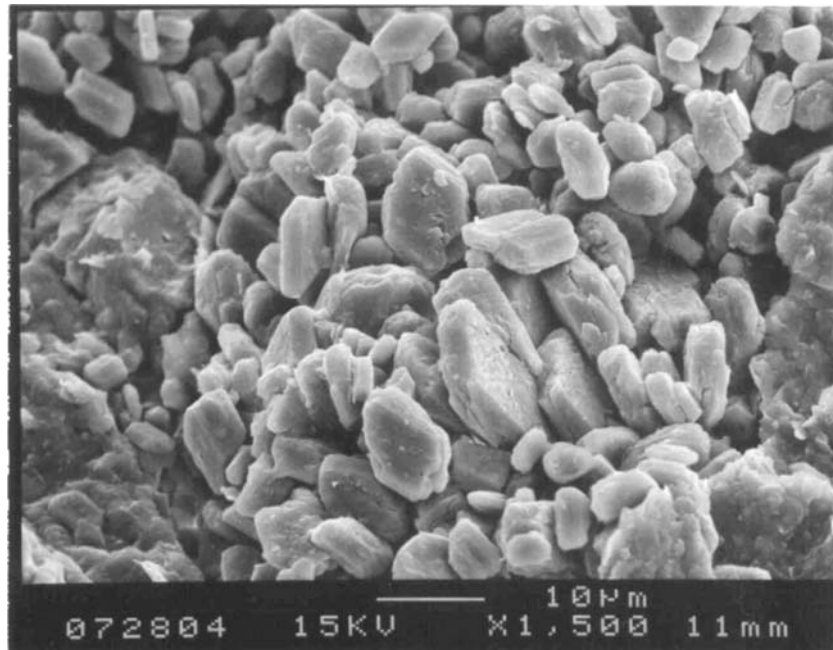
Figure 4. Variation in Grain Size for Gypsum Present in Texas Soils.

The top image in Figure 4 shows gypsum crystals in excess of 6 inches long. The bottom image is a scanning electron microscope (SEM) image of gypsum crystals smaller than 1 μm. One can imagine that it will take longer for the gypsum crystals in the top image of Figure 4 to dissolve than those in the bottom image because of the lower total surface area. Therefore, the size of the sulfate minerals plays a key role in determining how rapidly the reactions proceed in forming ettringite and generating sulfate-induced heave.

CHAPTER 3   TRADITIONAL LIME STABILIZATION

This chapter focuses on determining the sulfate content considered to be too high for traditional lime stabilization. Traditional lime stabilization is defined as lime mixed into the soil and immediately compacted without allowing the lime/soil mixture to sit/mellow for an extended period of time before compaction. The researchers developed the following laboratory testing program to identify the maximum sulfate concentration for lime stabilization without special construction procedures.

METHODS

A soil from the Vertisol order in College Station, Texas, was selected for swell measurements to determine what concentrations of sulfate are too high for stabilization with lime in Texas. Vertisols are present over large parts of the Texas Coastal Plain (Figure 2) and have high shrink/swell potential due to smectitic clay minerals.[8]   This soil was selected because it is typical of lime-stabilized soils in Texas and does not contain detectable sulfates greater than 100 ppm.

Samples were also selected from the Eagle Ford Formation (Figure 3) in Fort Worth, Texas, for comparison to the swell generated with the College Station soil. Construction projects on soils from the Eagle Ford Formation have generated a large percentage of the sulfate-induced heave problems in the Dallas/Fort Worth area. Samples from this particular location did not contain detectable sulfates as well.

Soil Processing

The soils were dried in a 140ºF (60ºC) oven to a constant weight and pulverized to pass a #4 sieve as outlined in American Society for Testing and Materials (ASTM) D 698. The engineering properties of the two soils were determined as follows:

1. For the College Station soil, a plasticity index of 24 was determined by ASTM D 4318; an optimum lime content of 6% determined by the Eades and Grim Test[16] or ASTM D 6276; and the optimum moisture content determined by modified Proctor (ASTM D 1557) using 6% lime is 22%.

2. The Fort Worth soil was determined to have a plasticity index of 42, an optimum lime content of 3 to 4% and an optimum moisture content of 19%.

3-D Swell Samples

The sulfate compounds, sodium sulfate (Na_2SO_4) and gypsum ($CaSO_4 \cdot 2H_2O$), were added to soil samples at concentrations of 0, 1000, 2000, 3000, 5000, 7000, and 12,000 ppm by four different techniques to represent scenarios observed in the field, as follows:

1. Sodium sulfate was added to the mixing water of some samples and dissolved.
2. Sodium sulfate was added to the water bath of other samples and dissolved to represent sulfates being added via an external water source (water truck) and groundwater, respectively.
3. Fine-grained (F.G.) gypsum passing the #200 sieve was added directly to the soil in a solid state.
4. Coarse-grained (C.G.) gypsum passing the #10 sieve was retained on the #40 sieve.

The fraction sizes in 3 and 4 above (shown in Figure 5 below) were chosen because they are representative of the more reactive sulfates found in natural soils in Texas. Grain size is an important issue because the larger the grains, the longer it takes for them to dissolve and react.
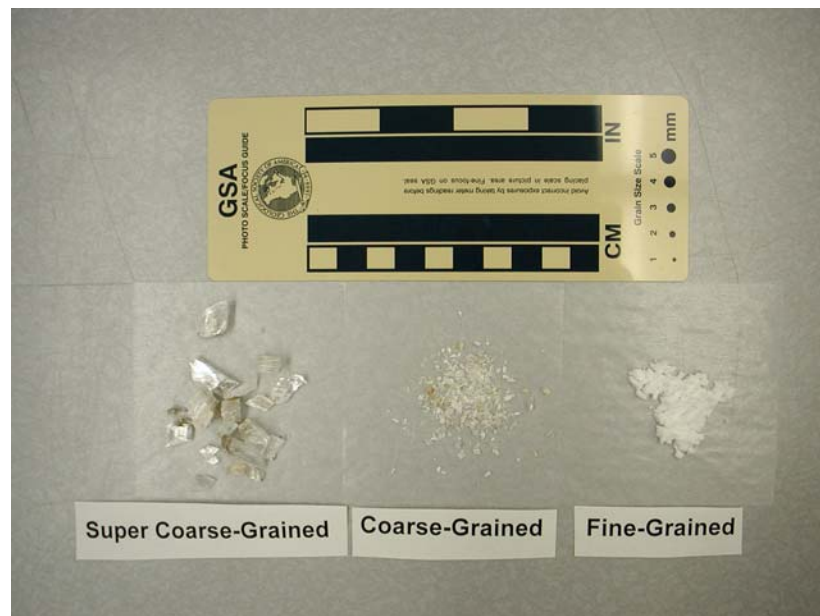
Figure 5. Gypsum Size Fractions Used in Samples Molded for 3-D Swell Tests.

Each sample was weighed and mixed separately using one of the four methods of sulfate application stated above. All samples were molded in duplicate at the density determined by modified Proctor in one lift with a Superpave Gyratory Compactor. The sample size was restricted to 4 inches in diameter by 4.5 inches tall (10.16 cm × 11.43 cm) due to the constraint of molding in one lift. The samples were then air dried for 3 days, placed in a 3-D swell test modeled after unpublished data by Tom Petry and wrapped in a paper membrane saturated with distilled water. Each sample utilized porous stones placed on the bottom and top of each sample, with a latex membrane placed over the sample. Duplicate samples were placed in a distilled water bath at 100 percent humidity and 77ºF (25º ± 2ºC) (Figure 6). The distilled water level was maintained just below the top of the porous stone located on the bottom of the sample to allow water to be drawn up into the sample by capillary action.

Figure 6. Sample Preparation for 3-D Swell Measurement.

Three-dimensional swell was measured by determining the height to the nearest 0.01 inch in three places 120º apart. The circumference was measured with a clear plastic tape to the nearest 0.0197 inch near the top, middle, and base of each sample. The three height and circumference measurements were averaged and the volume was calculated.

For swell testing, the National Lime Association recommends placing samples in a water bath for 3-D swell measurement immediately after molding;[14] however, these tests were conducted by air drying for 3 days after compaction then placing the samples in the swell test.

The National Lime Association procedures were not used nor the seven-day moist cure since neither technique is representative of field conditions. The typical scenario with heave in Texas has been that the subgrade is stabilized with lime, compacted to density, usually baked in the summer sun for a couple of days, and then saturated with water by a thunderstorm. Generally, subgrade heaves are observed the day after the storm.

Testing Plan

The researchers performed the testing in two phases as shown in Table 1. Phase I determined what sulfate content was too high for traditional lime stabilization (i.e., no mellowing) and is discussed further in this chapter. Phase II identified sulfate levels too high for modified lime stabilization techniques, which are discussed in Chapter 4.

After swell testing for at least 45 days, the samples were dried in a 140ºF (60ºC) oven until a constant weight was reached. The samples were then measured again for volume change.

To determine if deleterious reaction products were actually contributing to the swell of samples, X-Ray Diffraction (XRD) was performed on selected samples with a Rigaku X-ray diffractometer using Cuk_α radiation at a scan speed of 0.75º per minute with a step of 0.02 degrees. A bulk sample analysis was performed on selected samples to identify reaction products. A side-loading random powder mount reduced preferred orientation of minerals.[17]

Table 1. Sample Matrix for the Two Phases of Sulfate-Heave Experiments.

Sulfate content (ppm)*	Sulfate Application Method	Moisture content (%dry wt.)	Mellow Time (days)	Percent lime (Initial/final)
PHASE I				
0	1) Na_2SO_4 in	22	0	6/0
1000	water bath			
2000	2) Na_2SO_4 in			
3000	molding water			
5000	3) Gypsum (F.G.)			
7000	4) Gypsum (C.G.)			
12,000				
PHASE II				
5000	Na_2SO_4 in	22	1	6/0
7000	molding water	24	2	3/3
10,000			3	

*Two samples of each sulfate content were constructed.

A JEOL 6400 SEM with a Princeton Gammatech Energy Dispersive Spectrometer (EDS) was used to observe crystal habit (shape) and location of reaction products. The SEM was operated at a beam current of 15 kV and 10 mm (0.4 in.) working distance to maximize EDS results.

RESULTS

As previously stated in the Testing Plan on page 14, Phase I identifies the concentration of sulfates too high for "traditional" lime stabilization (i.e., no mellowing).

Traditional Lime Stabilization 3-D Swell Results

Figure 7 (top graph) shows three-dimensional swell progressing with time for the Fort Worth soil with fine-grained gypsum. The unstabilized control sample was molded at 17% optimum moisture and the lime-stabilized samples were molded at 19% optimum moisture. All samples were molded at 104 lb/ft^3 density. Negative swells from day 0 to day 3 are a result of air drying the sample for 3 days before placing it in the swell test. The bottom curve is a control with no sulfates and stabilized with 6% lime which still swelled more than 3%. The top curve is the unstabilized control; it swelled by about 30%. The curves between the two controls reveal a consistent trend of increasing swell with increasing sulfate level.

Figure 7 (bottom graph) shows three-dimensional swell progressing with time for the College Station soil with coarse-grained gypsum. All of the samples were molded at 22% moisture and 104 lb/ft^3 density. The bottom curve illustrates how lime treatment of the soil with no sulfates results in greatly reduced swell compared to the same soil with no stabilizer (top curve). This is exactly what would be expected for lime treatment and indicates that lime is doing its job. The curves between the two control samples show that increasing amounts of sulfate, from 3000 to 12,000 ppm in the form of coarse-grained gypsum, result in progressively more swell.

The trends observed for the two soils shown in Figure 7 are similar. The Fort Worth soil experienced larger swells, no doubt due in part to its higher PI value. Based on field experience, TxDOT and the National Lime Association have ranked soils containing less than 3000 ppm sulfates as soils with a low risk of lime-induced sulfate heave problems and soils containing

more than 8000 ppm sulfates are ranked as high-risk soils. The laboratory results presented in Figure 7 appear to support these views.

Fort Worth Soil (PI = 42)

**College Station Soil (PI = 24)**
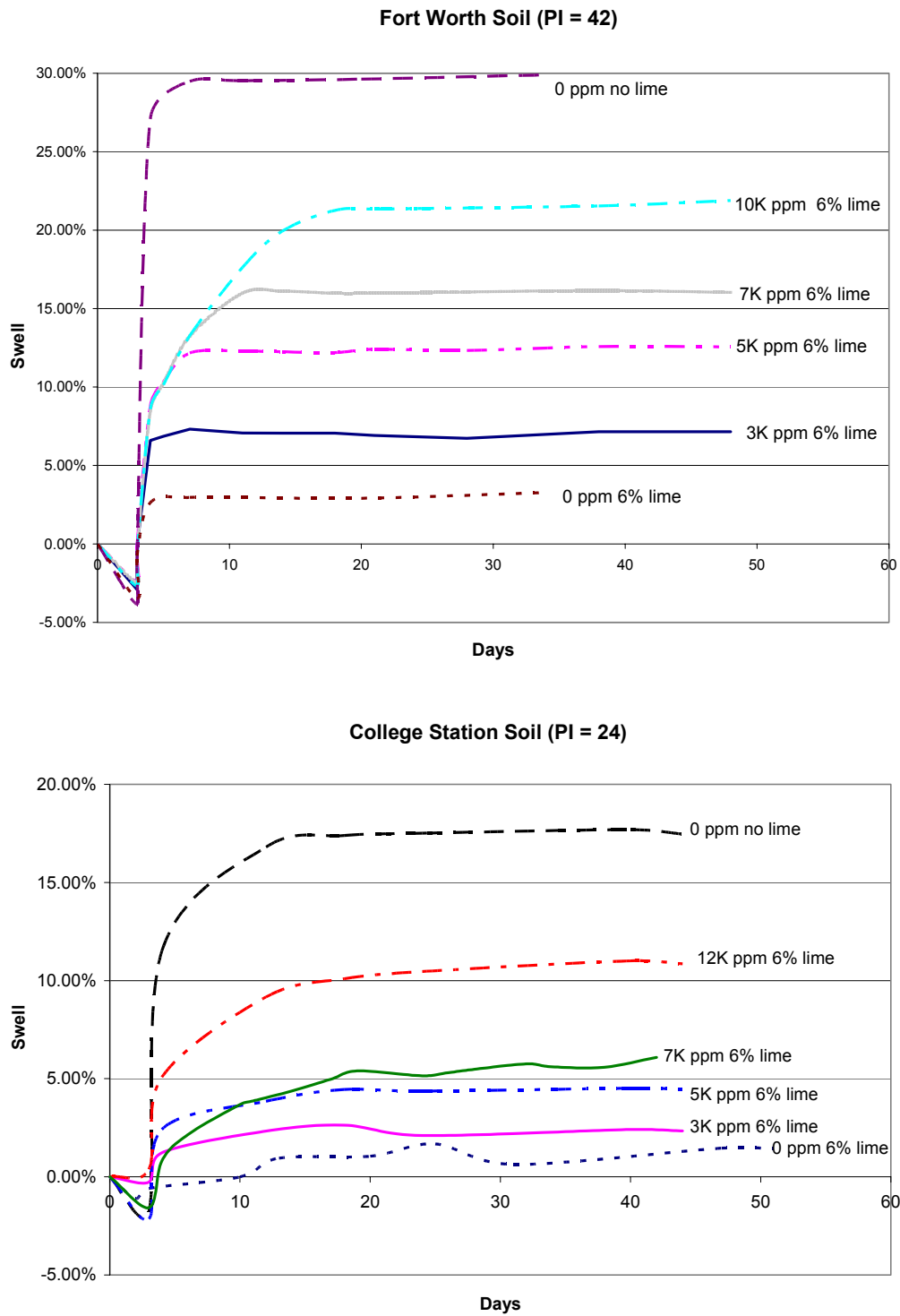
Figure 7. 3-D Swell for Selected Concentrations of Gypsum.

The method by which sulfate is introduced to the stabilized layer has an impact on swell results as illustrated in Figure 8. The sulfates were introduced either in the molding water or as fine-grained or coarse-grained crystals with the water added prior to compaction. The three upper curves show samples containing 7000 ppm sulfate and the lower curve is the control (6 % lime stabilization, no sulfates). Figure 8 illustrates the following points:

1. The samples with dissolved sulfate reach equilibrium the fastest and produce the greatest swell (top curve).
2. The fine-grained sulfate (F.G., 6% lime) reaches equilibrium, or plateaus, much more rapidly than the coarse-grained samples (C.G., 6% lime).
3. The coarse-grained gypsum (C.G., 6% lime) resulted in the lowest swell.

The variations in the individual swell curves in Figures 7 and 8 are due to different researchers performing the 3-D swell measurements. A single researcher was assigned the task of performing all subsequent swell measurements and the results became more consistent. In general, the repeatability between duplicate samples was very good. The difference in swell measurements ranges from a minimum of 0.01% at the lowest sulfate content to a maximum of 2.76% at the highest sulfate content.
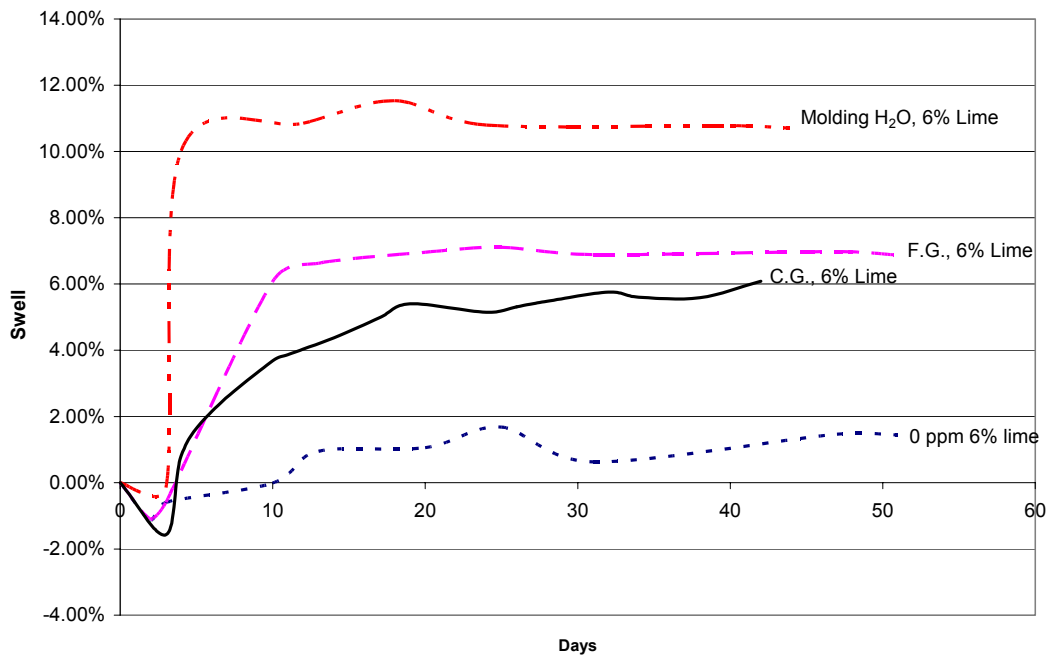
**Figure 8. 3-D Swell for Samples Containing 7000 ppm Sulfates.**

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DISCUSSION AND INTERPRETATION

The three-dimensional swell experiments show an unmistakable trend of increasing swell with increasing sulfate content. As discussed in Chapter 2, most sulfate-induced heave is attributed to the formation of ettringite and/or thaumasite. The question is: Can the swell we obtained in our experiments be attributed to formation of ettringite and/or thaumasite?

Factors Causing Swell

The experiments in this report were conducted in the range of 22º to 25ºC; therefore, the formation of thaumasite is ruled out since it requires temperatures below 15ºC to form.[5]

Selected samples were analyzed by XRD and SEM to determine if deleterious reaction products actually formed in the lime-stabilized samples. Figure 9 shows partial XRD patterns for an unstabilized gypsum-bearing sample (solid line) which contains a sharp peak at 7.51 Å. This peak confirms the presence of gypsum but is absent in the lime-stabilized sample which initially contained gypsum (dashed line). The broad peak at 7.16 Å is the (001) kaolinite peak and is present in both samples. The two peaks at 9.66 and 5.57 Å in the lime-stabilized sample are diagnostic of ettringite. The presence of these two peaks and the absence of the gypsum peak in the lime-stabilized sample illustrates that gypsum is being consumed to form the highly expansive mineral ettringite. These XRD patterns confirm that ettringite was formed in the swell tests.
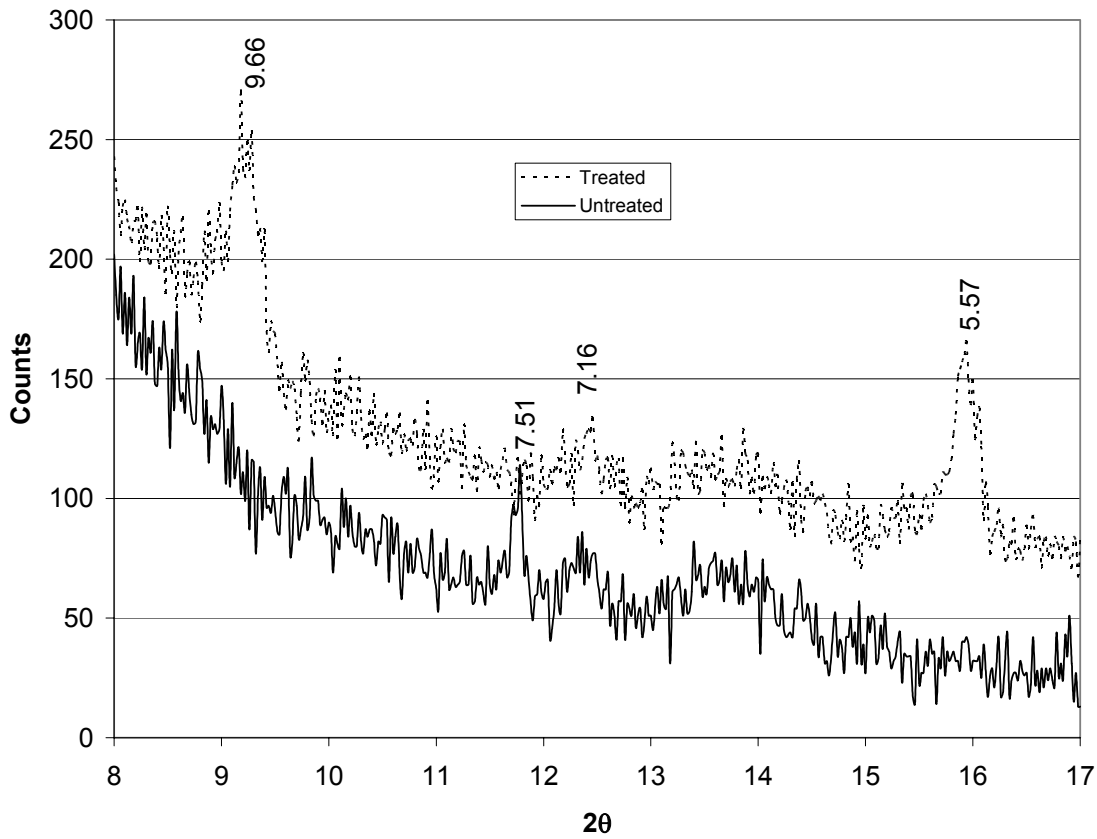
Figure 9. XRD Patterns for Lime Treated and Untreated Sulfate-Rich Soil.

To confirm the XRD results, SEM was used to analyze pieces of the same two samples represented in Figure 9 and the two images shown in Figure 10 are the result. The top image is of the unstabilized gypsum-bearing soil and the cornflake-appearing grains dispersed throughout the image are smectite clay minerals. The bottom SEM image is the lime-stabilized sample and the balls of radiating fibrous crystals evident in the lower image have the morphology of ettringite; EDS analyses of the radiating balls show the presence of calcium with lesser and subequal amounts of sulfur and aluminum, which confirms the presence of ettringite identified by XRD. Note the absence of ettringite in the unstabilized soil. Thus, based on XRD, SEM, and EDS analyses, formation of ettringite caused swell in the lime-stabilized samples.

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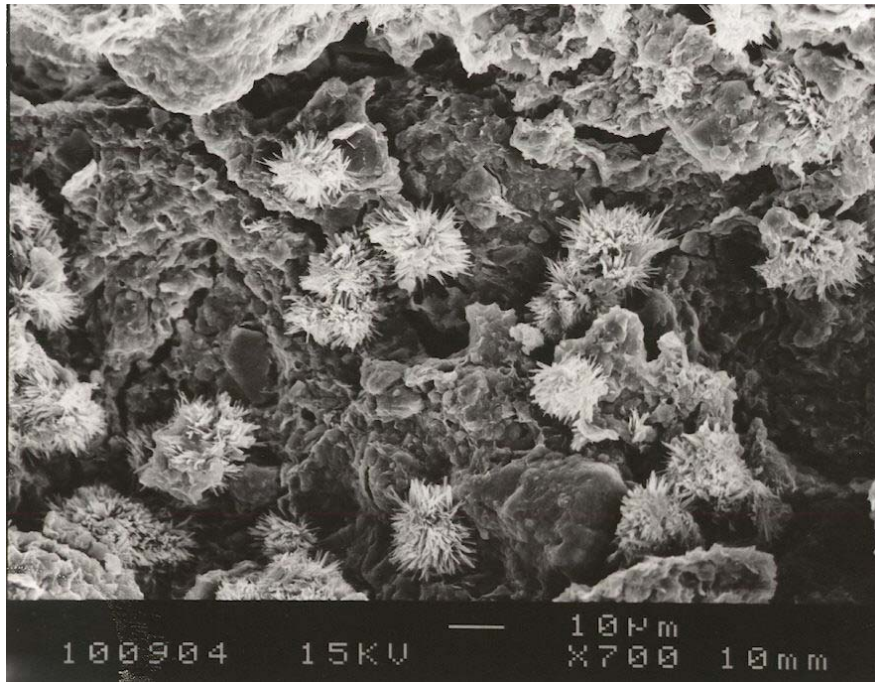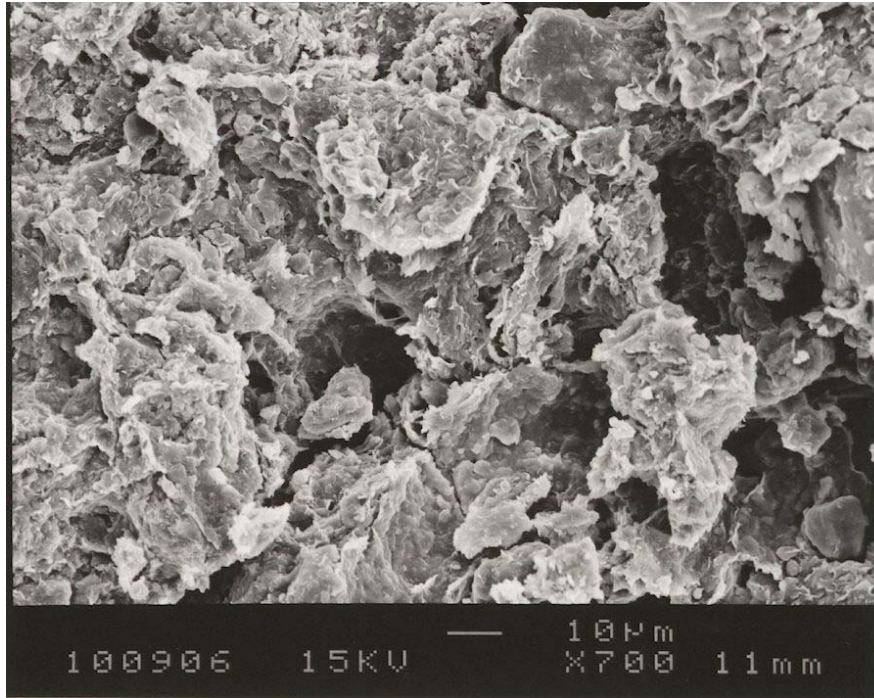
Unstabilized Soil

Stabilized Soil

Figure 10. SEM Images of Unstabilized and Stabilized Sulfate-Rich Soils.

Upper Sulfate Limit for Conventional Lime Stabilization

Upon consulting with TxDOT engineers it was determined that no criteria were available for an acceptable level of swell for lime-stabilized soils; therefore, it was decided to use the no sulfate swell level as the baseline cutoff.   Using the College Station soil, this was approximately 1-2% 3-D swell (Figure 7, bottom graph on page 16, and Figure 8 on page 17). A 3-D swell of 2-3% correlates with 3000 ppm sulfates (Figure 7 bottom graph), which implies that no modified construction techniques are required for sulfate concentrations of 3000 ppm or less; however, caution should be exercised for the following reasons.

First, there must be a limited supply of sulfate ions. For example, suppose there is a body of water adjacent to a construction site where sulfates at concentrations less than 3000 ppm have been detected. A full geotechnical investigation should be conducted to determine the source of the sulfate. In many instances, the level of sulfate increases dramatically with depth below the surface. It is feasible that sulfates could be carried upward to a treated layer via capillary action or by the rise and fall of water levels in a body of water. If sulfate is being supplied from a body of water, then sulfate could be continuously supplied to the stabilized layer and eventually result in a disruption of a pavement structure.

Second, the swell obtained with the samples in this report is different for each soil type. Soils with different reactive clay minerals yield different amounts of swell, which is not related to the sulfate content or the amount of ettringite formed. The Fort Worth soil has potentially more smectite, which has a higher shrink/swell capacity. In Figure 7 (top graph on page 16), the Fort Worth soil shows more shrinkage for the 3-day dryback as well as more swell than the College Station soil. Since the lime is reacting with the sulfates to form ettringite, the lime is not available to react with the clay minerals and reduce the shrink/swell capacity of the soil.

CHAPTER 4   MODIFIED LIME STABILIZATION FOR
HIGHER SULFATE CONTENTS

The National Lime Association[14]   recommends using modified construction techniques to stabilize soils with higher sulfate concentrations. For example, three practices which they advocate include the following:

1. mellowing: a process allowing lime-stabilized soil to remain in a soft, loamy state for a period of one to several days for chemical reaction before compacting to final density;

2. elevated moisture contents at least 3 to 5 percent above optimum; and

3. double lime application: a process that adds one-half of the optimum lime content, allowing it to react with the soil components for a specified time period, and then adding the other one-half of the lime before compacting to final density.

This chapter presents results of experiments designed to test these construction techniques for soils with sulfate levels greater than 3000 ppm.

METHODS

To evaluate the construction techniques recommended by the National Lime Association, 3-D swell samples were prepared using the same College Station soil and experimental procedures as in the traditional lime stabilization experiments. However, concentrations of 5000, 7000, and 10,000 ppm sulfate were the focus of these experiments using the techniques described below:

- mellowing of the lime/soil mixture to be molded by placing the mixture in a sealed bag to react for 1 to 3 days before compacting the mixture;

- single application, which adds all of the lime (in this case 6%) and allowing it to mellow for the specified time before compacting the mixture; and

- double application, which adds one-half the lime (in this case 3%) to the soil and allowing it to mellow for the specified time followed by addition of another 3% lime before compacting the mixture.

RESULTS

Effect of Mellowing on 3-D Swell

Following the results of the above swell measurements, the second phase of the testing was performed by mixing lime with the sample and allowing it to react (mellow) for a time of 1 to 3 days before compacting the sample. Figure 11 shows how 1 day of mellowing reduces the overall 3-D swell for samples bearing 5000 (5K) and 7000 (7K) ppm sulfates supplied via the molding water. Interesting points from Figure 11 are noted as follows:

1. Everything else equal, higher molding moisture contents (2% above optimum or 24% H_2O) reduces swell.

2. Single application of lime reduces swell better than double application (adding 6% lime [6/0] and mellowing for 1 day is better than adding 3% lime and mellowing for 1 day followed by another 3% lime [3/3] before compacting).

3. One-day mellowing results in less than 5% 3-D swell for 7000 ppm sulfates.
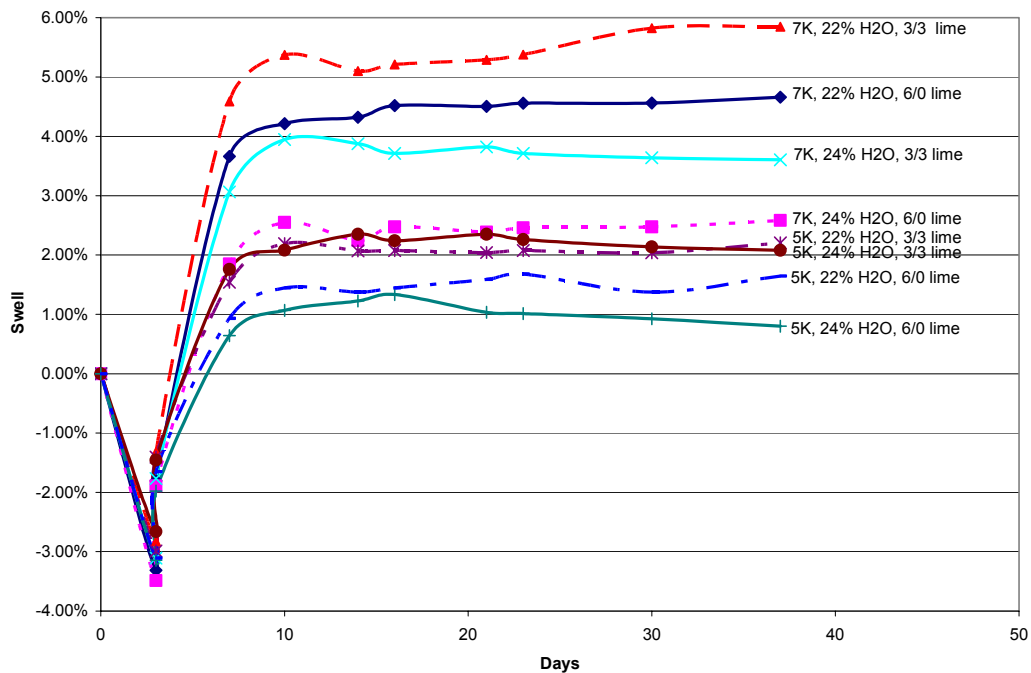

Figure 11. 3-D Swell with Sulfate Dissolved in Molding Water and One-Day Mellowing.

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Figure 12 further illustrates how lime application technique (single/double) affects 3-D swell. Samples represented in this graph were all treated with 7000 ppm sulfate in the molding water and molded at a moisture content of 22%. A single lime application rate of 6% (6/0) resulted in less swell than double application (3/3) of lime in all samples. Another point illustrated in Figure 12 is longer mellowing times yielded smaller volume increases.
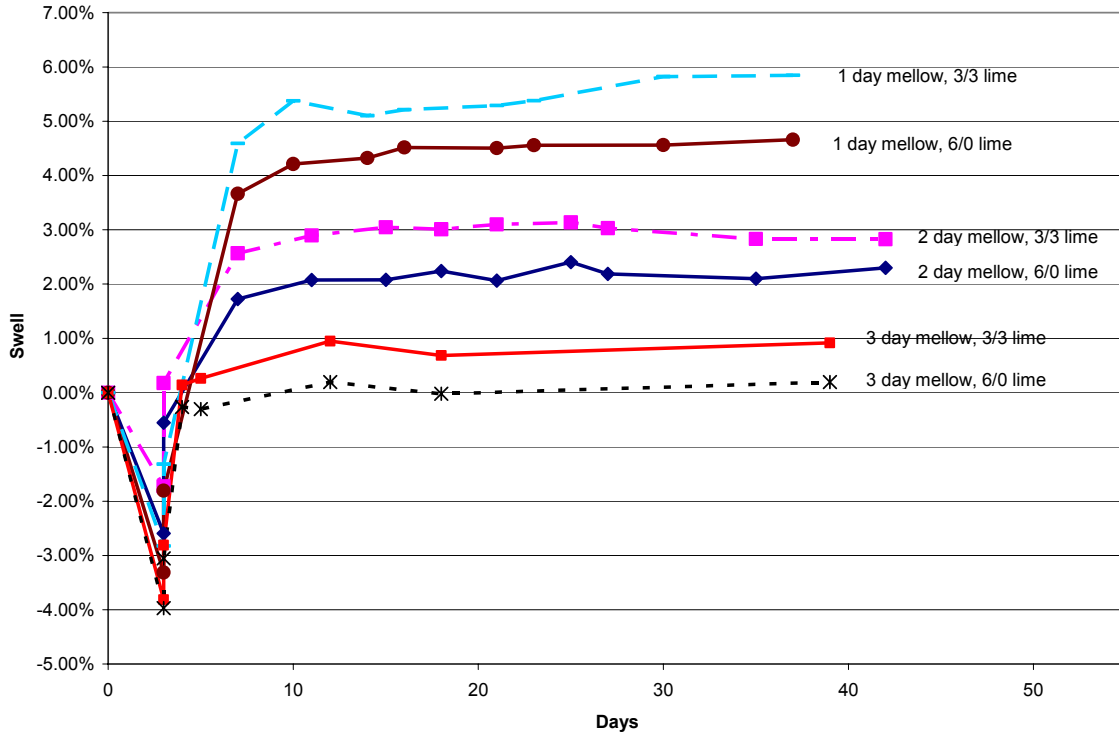

Figure 12. 3-D Swell with 7000 ppm Sulfates Dissolved in Molding Water.

DISCUSSION AND INTERPRETATION

Effect of Mellowing on Upper Sulfate Limit

From the many swell experiments conducted, researchers determined that 7000 ppm sulfates can be safely treated by adequately mellowing the soil after lime treatment (Figures 11 and 12). However, 3 days mellowing time produced the lowest 3-D swell. One day mellowing produced the lowest swell for 5000 ppm sulfates (Figure 11). In conclusion, at least 7000 ppm sulfates can be treated by mellowing the soil after lime treatment. Samples containing

10,000 ppm sulfates were also tested (data not shown) but swell was excessive using 6% lime, 24% moisture, and up to 3 days mellowing. This leads to speculation that increasing sulfate content requires more time to form deleterious reaction products; this would also be true for coarser grained sulfates.

Figure 13 shows how the soluble sulfate content decreases with time (mellowing). The top graph indicates that for up to 2 days mellowing the three lime treatments similarly reduced the soluble sulfate, but after 2 days the curves deviate. After 2 days the 3% lime at 24% moisture treatment ceased to remove sulfate because all of the lime had reacted. At day 3, another 3% lime was added and sulfate started reacting again, which explains why double application of lime resulted in more swell than single application. It is evident that 3% lime was not enough to remove all of the sulfates, so addition of the lime and compaction restarted the sulfate reaction, resulting in swell. The curve for 6% lime at 22% moisture also takes longer to react all the sulfates, which is consistent with observations that lower moisture contents result in more swell. The 6% lime at 24% moisture removed the soluble sulfate more rapidly and was enough to react with all of the sulfates. The researchers speculate that the additional water dissolved the sulfate more rapidly and reacted with the lime to form ettringite. The bottom graph in Figure 13 shows the effect coarse-grained gypsum has on mellowing. After 8 days mellowing there were still 6000 ppm sulfates available for reaction, illustrating that sulfate grain size has a profound impact on how rapidly the lime/sulfate reactions occur.
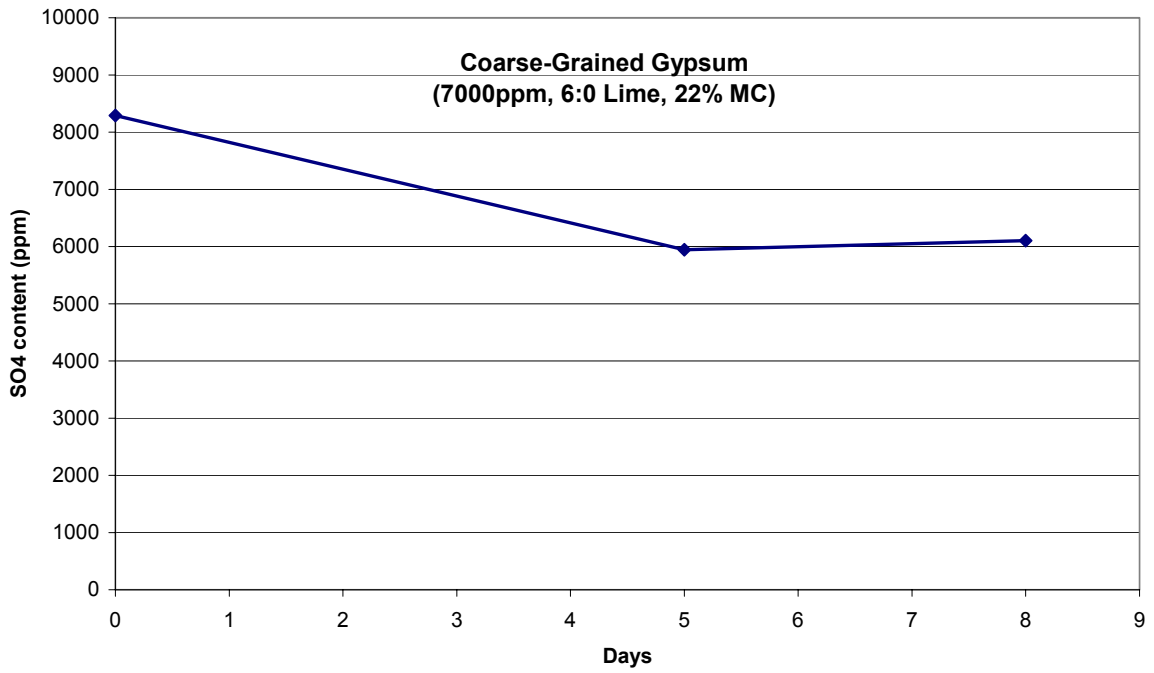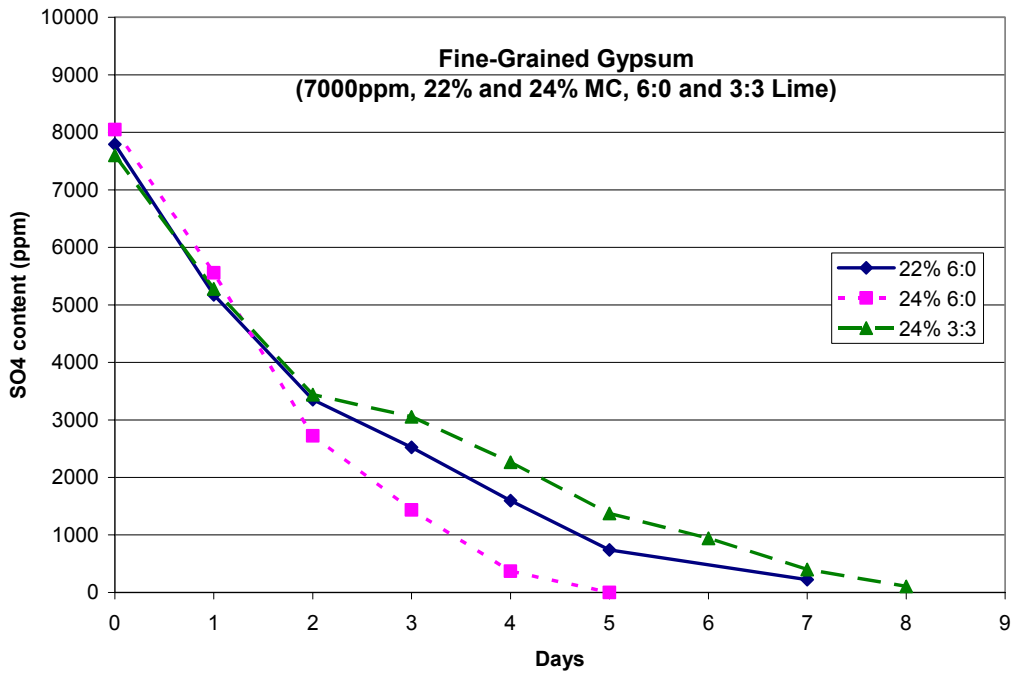
Figure 13. Effect of Lime Treatment on Sulfate Content.

Effect of Molding Moisture on Swell

The National Lime Association recommends adding 3 to 5% above the optimum moisture content to give plenty of water for the sulfate to react and to compact the soil at this higher moisture content as well (Eric Berger, unpublished data). The Project Monitoring Committee determined that 2% above optimum moisture was the practical limit for construction purposes; therefore, the researchers evaluated mellowing and compaction at 2% above optimum. As evident in Figure 11 on page 24, the higher moisture content always resulted in less 3-D swell.

Hunter's[5] investigation of soils in Las Vegas identified that 24% of the swell was due to formation of ettringite/thaumasite and the remainder to an increase in voids since the soil was initially in a heavily compacted state. Many investigators[6,7,18] have noted that decreased density may reduce swell by allowing the expansive minerals to form in the additional void space. An observation, from Figure 14, is that at the higher moisture content the samples could not be compacted to maximum density resulting in more void space into which the deleterious minerals can expand. A one-tail t-test showed that the mean densities are statistically different with a P value of 3.4×10^{-12}. This is analogous to air entrainment in cement paste to allow deleterious reaction products to expand into the voids without disrupting the concrete. However, the top graph in Figure 13 on page 27 shows that higher moisture contents result in more rapid removal of soluble sulfates, so the researchers conclude that reduced swell is due to a combination of more voids and a faster reaction rate removing more of the sulfate from the system before compaction.
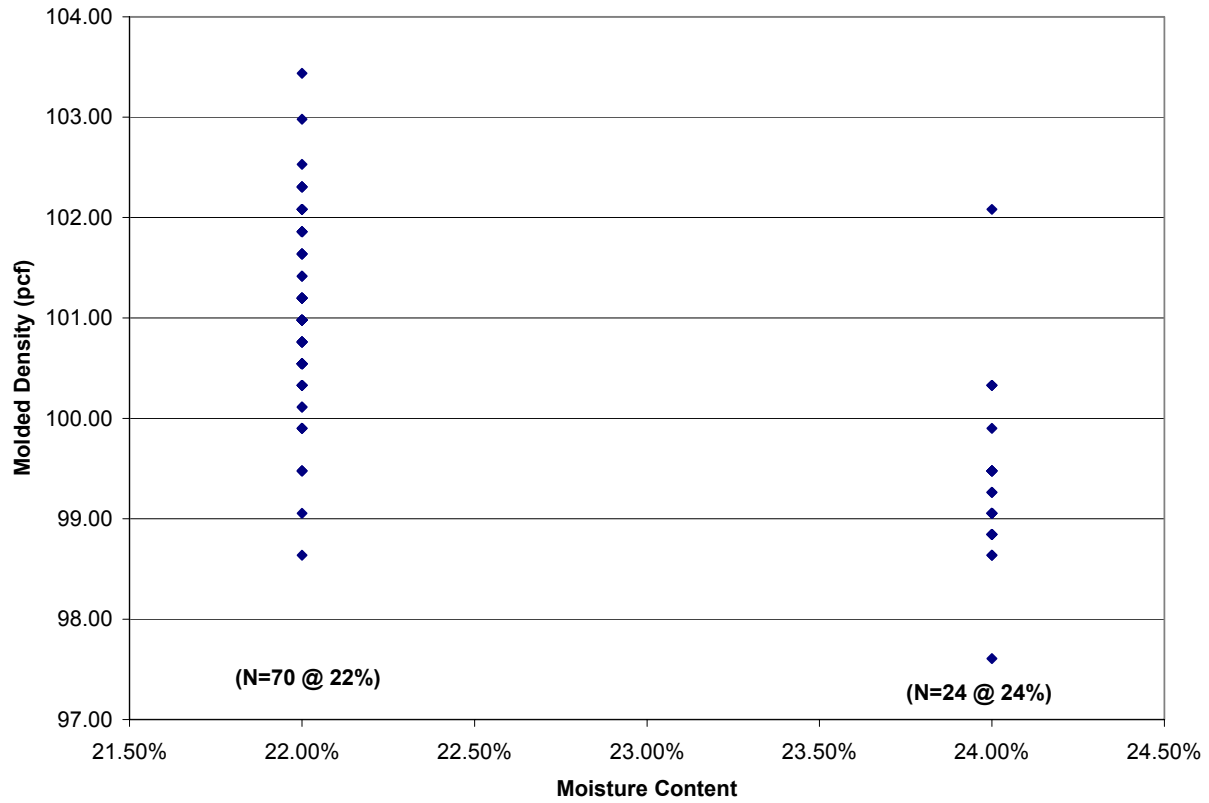
Figure 14. Density Comparison for Samples Molded at Different Moisture Contents.

CHAPTER 5   CONCLUSIONS

The findings of this research confirm thresholds identified by experiences of other investigators.[9,14]   Based upon their field experiences with sulfate heave, researchers Berger, Little, and Graves[14] have stated sulfate thresholds for lime stabilization as follows:

- below 3000 ppm sulfates there is little concern,

- 3000 to 5000 ppm sulfates are of moderate concern,

- 5000 to 8000 ppm sulfates pose a moderate to high risk, and

- greater than 8000 ppm sulfates are generally too high for lime stabilization.

Utilizing systematic addition of sulfates of known concentration to a Vertisol followed by lime stabilization reveals the following:

- Using a typical east Texas soil, the sulfate cutoff for traditional lime stabilization (lime is mixed into the soil and compacted) is 3000 ppm. This agrees with other researchers' recommendations.[9,14]

- Coarse-grained sulfates require more time to form deleterious reaction products than fine-grained sulfates, as illustrated in the swell tests and mellowing graph (Figures 7, 8, and 13).

- A mellowing period up to 3 days after lime application is effective for concentrations up to at least 7000 ppm for fine-grained sulfates.

- Mellowing of 3 days at 2% above optimum moisture and 6% lime did not result in acceptable swell with 10,000 ppm sulfates.

- Using a moisture content above optimum results in lower swell due to a combination of lower compaction density (more voids are available for reaction products to form in) and faster removal/reaction of sulfates.

- A single application of all the lime followed by mellowing reduces 3-D swell better than adding half of the lime and mellowing followed by addition of the other half of the lime and compaction.

- Mellowing time is a function of sulfate concentration and size (Figures 11, 12, and 13).

The sulfate limits mentioned above may vary for soils of different mineralogical composition.

CHAPTER 6 RECOMMENDATIONS

Sulfate problems have been reported from the Texas Panhandle along with regions of west, central, south, and east Texas. Many other states have reported sulfate heave problems as well, so identification of sulfate thresholds for lime stabilization is imperative. Based on the experiences of other researchers[9,14] and the numerous experiments performed for this study, the researchers have the following recommendations:

1. The use of lime to stabilize high-PI clay soils containing sulfates at or below the threshold of 3000 ppm should be safe and sulfate heave should be of little concern. Special construction techniques at this threshold are not indicated; however, it should be emphasized that construction water, streams, drainage water, groundwater, and site conditions should be investigated to ensure that total sulfates do not exceed the sulfate threshold of 3000 ppm. The rapid field test procedures set forth in Project Report 4240-1 are recommended for this verification.

2. The use of lime to stabilize high-PI clay soils that contain 3000 to 7000 ppm sulfates should include the mellowing test developed as part of this research to determine how quickly the sulfates will react with the lime before compacting to prevent excessive sulfate heave.

3. Mellowing should be accomplished by mixing a single application of the optimum amount of lime to the soil at 2% above optimum moisture and allowed to stand uncompacted for a period of time appropriate to the concentration and grain size of the sulfate.

4. The use of lime to stabilize high-PI clay soils that contain more than 7000 ppm sulfates is not recommended, except for special projects, since it will not be considered economical for roadway construction due to additional time, materials, and special construction techniques.

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