STABILIZATION OF HIGH SULFATE SOILS

By

NAGASREENIVASU TALLURI

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ABSTRACT

STABILIZATION OF HIGH SULFATE SOILS

Nagasreenivasu Talluri, Ph.D.

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Supervising Professor: Anand J. Puppala

Stabilization of expansive soils using lime and cement additives have been used by practitioners over the years. However, recent heaving and premature pavement failures in lime and cement-treated subgrades containing sulfates led to questioning the validity of calcium-based stabilization. When expansive soils containing sulfates are treated with calcium-based stabilizers, the calcium from the stabilizer reacts with soil sulfates and alumina to form the expansive mineral Ettringite. Formation and growth of the mineral Ettringite has been reported as the cause of severe heaving in several pavement failures. Under favorable environmental conditions, Ettringite transforms itself into another expansive mineral, Thaumasite. This heaving is termed as ‘sulfate-induced heave’ in literature.

Several theories have been proposed to understand the heaving mechanisms in sulfate bearing soils. Based on the theoretical background, researchers and practitioners have proposed various methods to treat sulfate soils. Applicability of these methods is mostly limited to soils containing sulfate content less than 8,000 ppm. Soils with sulfate content above 8,000 ppm are termed as ‘high sulfate’ soils, and chemical treatment of such soils is currently not considered. Hence there exists a research need to create better understanding of the heaving phenomenon in soils with higher sulfate contents and develop practical techniques for stabilizing such soils.

This research is designed to aid in understanding the heaving phenomenon in soils with sulfate contents above 8,000 ppm and to develop practical techniques to stabilize such soils. Six soils: four high plasticity clays, one low-plasticity clay soil and one high-plasticity silt, with sulfate contents varying from 200 ppm - 44,000 ppm, were considered for this research. Chemical and mineralogical tests were performed on the untreated soils to establish the clay mineral distribution and composition of the soils. Additional Gypsum was added to the soils with sulfate contents below 8,000 ppm so they could be considered as 'high sulfate'. These soils were treated with lime and mellowed for periods of zero, three and seven days. Following the mellowing, the samples were remixed, compacted and subjected to various engineering, mineralogical and chemical tests.

The present high-sulfate soils were treated lime stabilization with varying mellowing periods and treated soils after treatment were subjected to the engineering and chemical tests. Tests results were analyzed to understand the effectiveness of mellowing period on the heaving phenomenon of 'high sulfate' soils. Both Ettringite formation and crystal growth have contributed significantly to the overall swell of the treated soils. Swell trends observed in the treated soils at respective mellowing periods were attributed to the variability in sulfate levels and reactive alumina and silica contents. Treated soils at higher mellowing periods showed lesser sulfate induced heaving when sulfate levels are lesser than 30,000 ppm. At higher sulfate levels, the mellowing did not result in effective treatment of soils.

It was also observed that compaction void ratios and soil clay mineralogy have a significant impact on the swell behavior of chemically treated high-sulfate soils at different mellowing periods. Hence, mellowing effectiveness is explained using free energy and mass-volume approaches. Threshold void ratio framework comprising of natural soil void ratio and sulfate content was developed to predict Ettringite-induced heaving in chemically treated high sulfate soils at different mellowing periods.

Another treatment method using lime-fly ash treatment is also studied on two soils and the test results showed that the combined treatment has resulted in lesser soil heaving in these soils. The improvements here are mainly attributed to low amounts of calcium in the combined chemical additive used here.

In the final study, the rate of Ettringite formation and growth in the treated soils was indirectly assessed by measuring stiffness properties using the Bender Element tests. Bender Element tests revealed material softening and subsequent stiffness degradation in chemically-treated high-sulfate soils, and threshold stiffness loss values were established for the treated soils. This non-destructive study assessment can be used to evaluate the Ettringite induced soil heaving in sulfate soils under various chemical treatments.

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Natural expansive soils have been found in various regions across the globe (Chen, 1988). In the United States, several western and southwestern states have been reported to contain the most problematic expansive soils. These expansive soils undergo large volume changes upon seasonal wetting and drying, which eventually leads to severe damage of the structures built above them (Chen, 1988; Nelson and Miller, 1992). Lime and cement have been used widely in soil stabilization technology for treating expansive soils (Hausmann, 1990). When expansive soils are treated with lime/cement, the pH of the system is raised to 12.4 where the release of calcium and the dissolution of clay alumina and silica occur, leading to the formation of pozzolanic compounds. Formation of these pozzolanic compounds improves the volume stability, durability and stiffness characteristics of the expansive soils. Since calcium is the main ingredient of both lime and cement, these two stabilizers are called as calcium-based stabilizers. Due to its availability and reasonable cost, lime has been used as the subgrade stabilizer in most of the transportation infrastructure projects.

In the last two decades, many cases of severe pavement heaving and distress were reported when the expansive soils containing sulfates were treated with traditional calcium-based stabilizers (Sherwood, 1962; Mehta and Klein, 1966; Mehta and Wang, 1982; Mitchell, 1986; Hunter, 1988; Petry and Little, 1992; Puppala et al. 1999, 2003, 2012). Initially, it was assumed that the heave emanated from the swell movements of the underlying expansive soils upon wetting. Later it was confirmed by several researchers that the heave originated from the lime/cement-treated subgrades (Hunter, 1988; Perrin, 1992; Mitchell and Dermatas, 1992; Petry, 1994; Kota et al. 1996 and Puppala et al. 2000). Several pavements and infrastructure facilities that were built to last for decades experienced severe heaving issues shortly after construction. The heave and pavement distress were attributed to the complex reactions occurring in a moist environment between the calcium from the stabilizer, alumina, silica, and soil sulfates. This phenomenon is termed as “Sulfate – Induced Heave” in literature.

When sulfate-bearing soils are treated with calcium-based stabilizers at elevated pH conditions, calcium is released from the stabilizer, which reacts with soil sulfates, alumina and silica to form the highly expansive mineral, “Ettringite” ($Ca_6.[Al(OH)_6]_2.(SO_4)_3.26H_2O$). As the chemical composition indicates, the mineral Ettringite contains 26 molecules of water and is capable of swelling more than 137% of its volume (Little et al., 2010). The formation and growth of Ettringite causes expansive stresses in the soils. When these expansive stresses exceed the overburden pressure, heave and pavement failures occur. When the temperature of the system falls below 15^0C and carbonates are present in the system, Ettringite is transformed into Thaumasite through a series of reactions. Both Ettringite and Thaumasite are expansive minerals. Thaumasite has been reported as the cause of severe heaving of pavements and parking lots in Las Vegas, Nevada (Hunter, 1988).

Ettringite formation and subsequent heave is a complex phenomenon occurring in soils. It was reported that the formation of Ettringite is dependent on the availability of free alumina in soils, free access to water, temperature, humidity and site drainage conditions. In addition to these, forensic studies conducted by Hunter (1989) indicated that the observed swell is due to an increase in void ratio from the initial compacted state due to the mineral growth. Puppala et al. (1999) reported that under similar chemistry and environmental conditions, sulfate-induced heave is of greater concern in clays than in sands.

1.2 Current Guidelines in Practice

Based on previous sulfate heave case studies, researchers recommended that when significant amounts of sulfates are present in the native soil, lime/cement treatment must either be carried out with caution or be completely avoided (Mitchell, 1986). Berger et al. (2001) reported that soluble sulfates below 3,000 ppm are of little concern, sulfates between 3000-5000 ppm are moderate concern, 5000-8000 ppm are moderate to high risk and greater than 8000 ppm sulfate concentrations are of severe concern for stabilization using lime. Transportation agencies across United Sates use lime in most of the subgrade stabilization projects due to its cost and availability compared to cement. According to the guidelines developed by the Texas Department of Transportation (TxDOT), sulfate concentrations up to 3000 ppm can be stabilized by traditional lime with one day of mellowing. Soils with sulfate concentrations up to 8000 ppm can be stabilized by providing additional moisture, along with other chemical treatments including combined lime and fly ash treatments. When sulfate concentrations exceed 8000 ppm, alternative treatment such as remove—and-replace or blending in non-plastic soils is recommended. Remove, replace and blend non-plastic soils are not engineering solutions in view of the economy and sustainability since the transportation of foreign materials involves increased fuel emissions and costs associated with it.

The guidelines developed so far limited the threshold sulfate level for lime stabilization as 8,000 ppm. However, failures are particularly evident in sites where soil sulfates were 8000 ppm or more, which needs attention of the research community. Also, the soils with sulfate concentrations above 8,000 ppm are called as "High Sulfate Soils" and are deemed ineffective for lime stabilization. Hence, the main objective of the current research is to develop stabilization techniques for soils containing sulfate contents above 8000 ppm.

1.3 Scope of the Present Work

The main objectives of the current study are:

1. To understand heaving mechanisms in chemically-treated high sulfate soils and to study the time rate of Ettringite formation for quick assessment of sulfate-induced heaving.
2. To develop alternate stabilization techniques to suppress Ettringite-induced heaving in high sulfate soils and to assess the effectiveness of a combination of stabilizers such as lime and fly ash in treating high sulfate soils.
3. To address the effects of various soil compositional factors such as clay mineralogy, reactive alumina, silica contents, sulfate contents and compaction void ratios on the swell behavior of chemically-treated high sulfate soils. Also, to develop a threshold void ratio framework based on sulfate content and compaction densities.

1.4 Thesis Organization

This thesis is comprised of nine sections: introduction (chapter 1), literature review (chapter 2), experimental testing program (chapter 3), analysis of laboratory test results, Phase I (chapter 4), analysis of laboratory test results, Phase II (chapter 5), heave prediction models (chapter 6), time rate of Ettringite formation (chapter 7) and summary, conclusions and future research recommendation (chapter 8).

Chapter 1 provides the introduction to sulfate heaving, current guidelines in practice, limitations, scope of the current work and thesis organization.

Chapter 2 provides a summary of chemical stabilization of soils, problems in sulfate soils, introduction to sulfate bearing soils, sources of sulfates in soils, sulfate heave case studies from around the world, threshold sulfate levels and brief description of different techniques to measure soluble sulfate contents followed by sulfate heave mechanisms. A brief overview of different stabilization techniques, their limitation and challenges are presented here.

Chapter 3 presents soil selection criteria, stabilizer dosage selection, laboratory sample preparation, testing program, testing procedures used. Basic classification of the natural soils was determined and presented as per American Society for Testing Material (ASTM) standards and Texas Department of Transportation (TxDOT) methods and these results are included in this chapter.

Chapter 4 (Phase I) provides a summary and analysis of engineering test results on control soils and treated soils at different mellowing periods and possible mechanisms for the property variations. Tests covered in this chapter were subdivided as engineering, chemical and mineralogical tests. The engineering tests include 3-D volumetric swell, 3-D volumetric shrinkage, UCS, 1-D swell pressure tests, chemical tests include the determination of soluble sulfate contents and mineralogical tests include reactive alumina and silica measurements. Results of the testing were analyzed using Gibbs free energy approach and mass-volume calculations.

Chapter 5 (Phase II) describes a comprehensive analysis and summary of tests conducted on lime + fly ash treated soils with and without mellowing. The effect of lime fly ash treatment on Ettringite formation was also evaluated. Possible mechanisms between soil compositional factors and their interactions with chemical stabilizers in Ettringite formation and heaving are discussed.

Chapter 6 provides heave prediction models and threshold void ratio frame work developed in the current research study.

Chapter 7 provides summary of Bender Element studies conducted to measure the rate of Ettringite formation and subsequent material degradation in chemically treated high sulfate soils considered in the current study.

Chapter 8 provides summary of the research findings and future research recommendations.

Last section of this dissertation comprises of appendices listing standard proctor compaction curves, photographs of test specimens and equipment and references.

CHAPTER 2

LITERATURE REVIEW

2.1 Chemical Stabilization of Soils

The literature review presented in this chapter was collected by conventional library and electronic resources, as well as from previous research reports in the area of chemical stabilization of soils. An introduction to chemical stabilization of soils is given, followed by problems in sulfate-bearing soils and different sources of sulfates and their distribution. Past research on the threshold sulfate levels, various sulfate measurement methodologies and a description of each method was given in the preceding section. Ettringite formation reactions, chemical process and heave mechanisms were explained, followed by sulfate heave case histories around the world. The last section of the literature review is dedicated to the research conducted in the area of sulfate soil stabilization. A brief description of the guidelines developed by state DOT's was presented, followed by the different treatment techniques used by previous researchers and their merits and demerits. Limitations of the previous stabilization techniques and challenges were explained.

2.1.1 Chemical Stabilization of Soils

A brief background on chemical stabilization of soils is presented here.

2.1.1.1 Background

Chemical stabilization of expansive soils using lime/cement has been a favorite technique of practitioners over the years. As mentioned before, lime has been chosen more often due to its low cost and easy availability. Stabilization of soils using lime improves the plasticity characteristics, moisture stability and strength characteristics. Stabilization using lime proceeds through two stages; namely, soil modification and soil stabilization. When expansive soils are treated with lime, a series of reactions occur which leads to the plasticity and strength changes. These reactions are cation exchange, flocculation and agglomeration, pozzolanic reactions and carbonation reactions. The first two reactions are called modification reactions, which improve the plasticity, shear strength and workability. The degree of effect and amount of lime required to cause cation exchange reactions is based on the soil clay mineralogy and water conditions. These reactions lead to modification of lime-soil mixture and are short-term reactions. The last two reactions are called stabilization reactions, which impart long term strength gain due to the formation of a compound called pozzolan. Pozzolan is a finely divided siliceous or aluminous material which forms cemented products with the presence of water and calcium hydroxide. Clay mineral structure contains alumina and silica in its formation and is termed as a pozzolan. When expansive soils containing clay are treated with lime, lime elevates the pH of the system to 12.4, where clay alumina and silica are released into the system, combining with calcium and water, to from pozzolanic compounds. These reactions continue uninhibited as long as there is residual calcium available for release of alumina and silica to form pozzolanic compounds. These reactions are shortly summarized as:

$$Ca^{++} + OH^{-} + Soluble\ Clay\ Silica \rightarrow Calcium\ Silicate\ Hydrate\ (CSH) \qquad (1)$$

$$Ca^{++} + OH^{-} + Soluble\ Clay\ Alumina \rightarrow Calcium\ Aluminate\ Hydrate\ (CAH) \qquad (2)$$

Formation of the pozzolanic compounds and extended curing conditions lead to strength improvements in the order of several hundred pounds per square inch (psi). As the pozzolanic formation reactions indicate alumina and silica are released from clay which are absent in sandy soils. Research has shown that lime can be an effective stabilizer for sandy or silty soils containing as little as seven percent clay fraction (Little, 1995).

2.1.1.2 Problems in Sulfate Soils

Though lime stabilization improves the volume and strength charectersitics of the expansive soils, there are some limitations to lime stabilization. These limitations are the presence of organic carbon and soluble sulfates. It has been reported that the presence of organic carbon in excess of one percent can interfere with the pozzolanic reactions, leading to low strength gains. Compared to organic carbon, the presence of sulfates is of higher concern bacause lime treatement in these types of soils leads to excessive heaving and pavement failures (Mitchell 1986; Hunter 1988,1989; Puppala et al. 1999, 2003, 2012; Rajasekaran, 2005). These pavment failures lead to questions pertaining to the validity of lime stabilization itself. Figure 2.1 Shows the severe pavement failure on US 67 close to Dallas/Fort Worth, Texas (Burkart et al., 1999). It has been reported that when soils contain sulfate minerals such as gypsum ($CaSO_4.2H_2O$) and sodium sulfate (Na_2SO_4) in their natural formation and are treated with calcium based stabilizers, adverse reactions occur, causing severe heave and pavement distress. These adverse reactions are due to formation of expansive minerals, Ettringite ($Ca_6.[Al(OH)_6]_2.(SO_4)_3.26H_2O$) and Thaumasite ($Ca_6.[Si(OH)_6]_2.(SO_4).(CO_3)_2.24H_2O$). This phenomenon is termed as "Sulfate-Induced Heave" in literature. This phenomenon was first reported by Sherwood in 1962;however, the sulfate-induced heave phenomenon received little attention until the mid-1980's. Terzaghi's lecture by Mitchell in 1986 shed light onto the severity of the issue. Repair and re-construction of the the failed infrastructure is costing millions of dollars to the tax payers. Under favourable moisture, humidity and temperature conditons, these minerals grow, causing further swell. Researchers called lime treatment of expansive soils containing sulfate "man made expansive soil" (Puppala et. al., 2012). The next section gives an insight into the sulfate-bearing soils and sources of sulfates.

Figure 2.1 Severe Pavement Distress along US 67, Texas

2.2 Sulfate Bearing Soils

Sulfate-bearing soils are found all across the United States. Gypsum is found to be the most frequent occurring sulfate mineral in the western part of United States (Kota et al., 1996). Figure 2.2 shows the location of soils containing gypsum and gypsum mines in the US. Many sulfate-induced heave cases were reported in the state of Texas (Perrin, 1992; Puppala et al., 2010). Burkart et al. identified certain geologic formations that possess high sulfates, with gypsum being the most common sulfate in the Dallas area soils (Burkart et al., 1999). The most severe heaves in the Dallas/Fort Worth area are associated with the Eagle Ford formation. Harris et al. (2004) identified counties in Texas known to have sulfur-bearing minerals. These soils were found to contain sulfate concentrations ranging from 4000ppm to 27800ppm (Chen et al., 2005). Very high sulfate concentrations of 44,000ppm were reported in the Childress district (Puppala et. al., 2013). The geology of the state of Texas is given in Figure 2.3, showing the concentrations of the Eagle Ford shale and the presence of sulfate soils across the state.

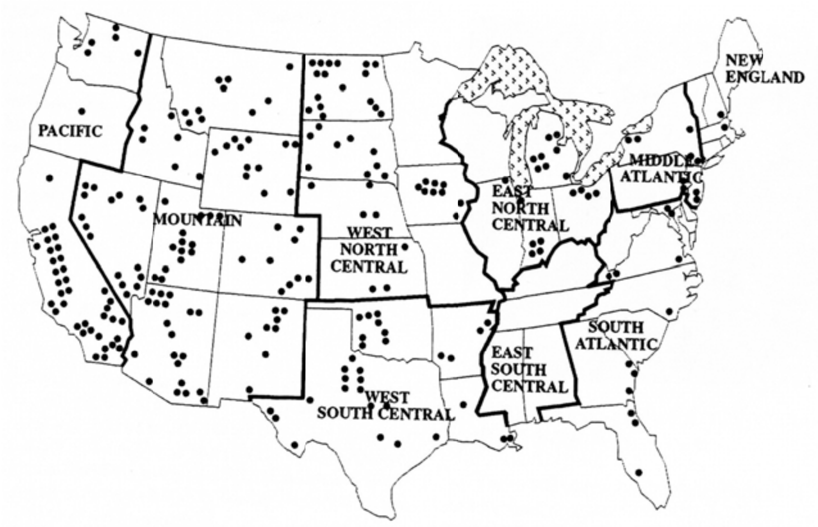

Figure 2.2 Location of soils containing Gypsum in United States (Kota et al., 1996)

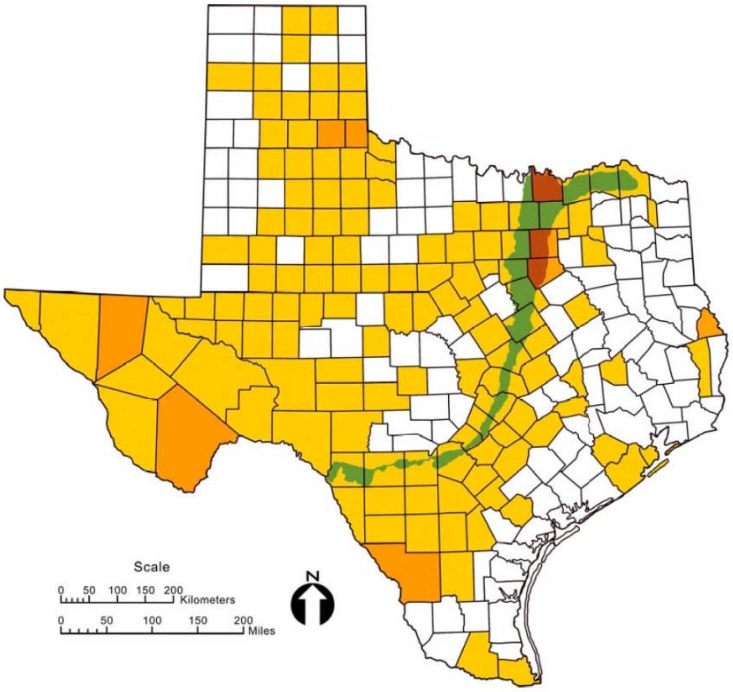

Figure 2.3 Geology of Texas (Harris, 2005)

2.2.1 Sources of Sulfates in Soils

There are several sources of sulfates in soils, produced from primary or secondary origin. Primary sources can be defined as the direct sources of sulfates in their natural form, as sulfate-bearing minerals such as gypsum while, the secondary sources are those that are not a direct source of sulfate but give out sulfates as a byproduct of oxidation or other forms of chemical interactions. The following section highlights these sources.

2.2.1.1 Primary Sources

The primary source of sulfates in soils is gypsum ($CaSO_4.2H_2O$). Sulfates are present in natural soils as calcium sulfate (gypsum), sodium sulfate and magnesium sulfate (Puppala et al., 2003). Natural sulfate minerals have different solubilities based on their dissolution rate. Due to gypsum's low solubility (2.58 gm/L) compared to both sodium sulfate (408 gm/L) and magnesium sulfate (260 gm/L), it acts as a steady source of sulfates in soils. The maximum solubility of gypsum occurs at 35°C-50°C. Sulfates are present in higher concentrations in clayey soils than in granular soils. This is due to the low permeability of clayey soils compared to granular soils which do not allow the dissolved sulfates to pass through them. In granular soils, these sulfates get leached out very easily due to their high permeability. Figure 2.4 shows the calcium sulfate and sodium sulfate crystals in their natural form.

Figure 2.4 Gypsum and Thenardite in Natural Form

2.2.1.2 Secondary sources

Other sources of sulfates in soils are pyrites (sulfides), which on oxidation reactions are converted into soluble sulfates. Many cases of pyritic oxidation and subsequent sulfate indueced heave were reported in UK (Floyd et. al., 2003). Floyd et. al., 2003 summarized pyritic oxidation reactions to form sulfate as:

$$2FeS_2\,(Pyrite) + 2H_2O + 7O_2 \rightarrow 2Fe^{2+}(Ferrous\ ion) + 4SO_4^{2-}(sulfate) + 4H^+ \tag{3}$$

$$4Fe^{2+} + 4H^+ + O_2 \rightarrow 4Fe^{3+}(Ferric\ ion) + 2H_2O \tag{4}$$

$$FeS_2 + 14Fe^{3+} + 8H_2O \rightarrow 15Fe^{2+} + 2SO_4^{2-} + 16H^+ \tag{5}$$

During the rainy season, sulfates in the top layers of the soil dissolve and move into the stabilized layers; whereas, during dry spells, due to evaporation, dissolved sulfates move upward into the top layers, increasing their concentration (Dermatas, 1995). As previously discussed, the sulfates can be from secondary origin. A forensic investigation study was conducted by Rollings et al. (1999) on the failure of a low-volume road in Georgia. Extensive bumps and longitudinal cracking were observed five months after construction on a pavement built on cement stabilized sand. The failure is attributed to the formation of Ettringite in the cement stabilized sand. While no sources of sulfate were found in the native soils, the investigation found that the water from a nearby well used for mixing cement and compacting was the source of sulfate in the soil. Also, the clay fraction of the sand contained the mineral Halloysite, which is rich in alumina. Though the native soil contained no sulfate, the sulfate transported from the mixing water, alumina released from clay fraction of the soils reacted with calcium from cement in the basic envrioment to form expansive mineral Ettringite. Another example of secondary sulfate contamination is the sulfates transported by ground water.

It can be understood from the above discussion that there are various sources of sulfates in soils; now, it is important to understand how these sulfates present in the soils induce heaving. The following sections details the threshold sulfate levles for lime stabilization folowed by process of sulfate induced heave along with the chemical reactions involved in the process.

2.3 Threshold Sulfate Levels

2.3.1 Past Research on Threshold Sulfate Levels

Based on the previous sulfate heave case studies, one can understand that when sulfates are present, lime treatment needs to be performed with caution or to be completely avoided. This is not true in all cases because soils with reasonable sulfate contents were stabilized successfully using lime. Hence, there needs to be a level of sulfates below which sulfate heaving is not a concern. These sulfate levels are called "Threshold Sulfate Levels". Researchers across the USA have reported different threshold levels of sulfates at which heave distress was recorded (Hunter, 1988; Mitchell and Dermatas, 1992; Petry and Little, 1992; Kota et al. 1996; Puppala et al. 1999; Viyanant, 2000; Little et. al., 2005). There are no conclusive threshold levels of sulfate that could be established, and this is primarily attributed to variability in soil type and site conditions of these case studies.

Petry et al., 1992 stated that if the level of soluble sulfate is below about 2,000 ppm or 0.2%, the development of expansive minerals will not be an issue in stabilized soils. Berger et al. (2001) indicated that soluble sulfates below 0.3 percent (3000 ppm of sulfates) are of no problem. Soluble sulfates between 0.3 and 0.5 percent represent a moderate risk of harmful reaction. Sulfates between 0.5 to 0.8 percent indicate moderate-to-high risk. Soils with soluble sulfates levels greater than 0.8 percent pose a serious threat to civil infrastructure facilities. Studies conducted at The University of Texas at Arlington (Puppala et. al., 2003) confirmed that at low sulfate levels, around 1000 ppm, lime stabilization plays an important role in reducing swelling of natural soils. At sulfate levels ranging from 0 to 2,500 ppm, the lime stabilization reactions and sulfate heave reactions occur simultaneously, but the magnitude and extent of heave depends on the lime concentration. At higher lime dosages, swell magnitudes were suppressed, indicating the dominance of stabilizing reactions. Also, when the sulfate concentrations exceeded 2500 ppm, the increase in lime dosage resulted in increased heaving due to increased amounts of Ettringite formed. Puppala et. al., 2003 reported that void ratio and compaction conditions play important roles in the sulfate-induced heaving phenomenon. If the void ratios are small, the soil matrix is dense and cannot accommodate any heave associated with Ettringite formation and growth leading to the pavement heave.

Research studies conducted by Harris et al. (2004) indicated that at or below 3000 ppm sulfate concentrations, sulfate heaving is of no concern and lime stabilization can be effectively implemented.

Also, between 3000 and 7000 ppm, sulfate concentrations lime stabilization can be performed in soils with some caution. In most of the cases, the sulfate levels to induce heaving ranged from 320 ppm to as high as 43,500 ppm (Puppala et al. 1999; 2003). The time for sulfate heave appearance after chemical stabilization ranged from a few days to 18 months. Also, soils that experienced this sulfate heave included sands to silts and clays with containing the significant amounts of clay fraction.

Overall, it can be seen that there is no conclusive threshold sulfate level above which heaving occurs. This is due to the fact that soil properties such as void ratios, environmental and site drainage conditions differ from site to site. Both crystal growth and mineral hydration and subsequent mineral expansion in a soil void space will eventually result in macro expansions at the surface levels that can distress transportation infrastructure. Another important reason for these variations is the differences in test methods to measure soluble sulfates. Several methods in the literature will yield different soluble sulfates for the same soil and sulfate conditions since they have different bases to determine the sulfate content. A brief overview and description of each of these methods is presented in the following section.

2.3.2 Sulfate Measurement Methodologies

In view of the sulfate heaving issues, transportation agencies across United States have mandated measurement of sulfates prior to subgrade stabilization. These agencies use different techniques to measure the subgrade sulfates, which are either gravimetric-based or turbidity-based. However, it was reported that these methods often fail to provide consistent and repeatable values (Viyanant, 2000). Also, under similar conditions, these methods provide different sulfate values (Harris et al. 2003 and Puppala et al. 2002). Gravimetric procedures determine the sulfate content based on the amount of sulfate precipitated upon the addition of Barium Chloride to soil-water solution. Turbidity-based procedures convert the turbidity caused by the presence of sulfates to sulfate concentration. A brief description of each of these techniques is given below.

2.3.2.1 Gravimetric Determination of Sulfates

The basis of gravimetric methods is precipitation of soluble sulfate on addition of barium chloride. Representative soil samples are collected and mixed with equal proportions of water and kept overnight to allow dissolution of sulfate into water. The soil-water solution is mixed, centrifuged and filtered thru a filter paper. The resultant solution is boiled, and barium chloride is added to the solution to precipate sulfate as barium sulfate. The resultant solution is filtered to get the weight of barium sulfate precipitated. The portion of sulfate (SO_4^{2-}) is calcuated and mutliplied with the weight of barium sulfate precipitated to obtain the sulfate concentration in ppm. There are two widely used gravimetric techniques for soluble sulfate determination. They are: Modified UTA method and AASHTO (T 290-95) method. A Brief discription of each of the methods is given below:

2.3.2.1.1 Modified UTA Method

Modified UTA method was developed by Puppala et al. (2002) and is a refined version of the UTA method proposed by Petry (1994). 1:10 soil water dilution was used in this method. Theoretically, 1:10 dilution ratio can extract sulfate concentrations up to 26,000 ppm. This method involves mixing of 10 grams of soil with 100 mL of distilled water and leaving it overnight. The next day, the sample is shaken for 30 minutes on an Eberbach shaker to disintegrate the sulfate salts in the soil matrix. Soluble sulfates are extracted from the solution by centrifuging the sample at 14,000 rpm using IEC-HT centrifuge machine. The higher rate of centrifuging allows the efficient extraction of soluble sulfates. The supernatant liquid is then filtered through 0.1μm VVLP type filter paper, and the filtrate is diluted to 200 mL. The pH of the dilutant is adjusted between 5 and 7 by adding diluted hydrochloric acid (1:9) HCl to facilitate the further dissolution of sulfates. The solution is then boiled until the bubbles appear. Warm $\mathrm{BaCl_2}$ (10%) solution is added slowly until the precipitation process is completed.

The precipitate is then digested in an oven at 80-90^{0}C for 12 hours. The precipitate is filtered through a 0.1μm membrane filter. While filtering, the precipitate is washed with distilled water to make it free from chlorides. Chlorides can be checked by silver nitrate-nitric acid reagent. The difference between the weights of dry filter paper and filter paper with barium sulfate precipitate gives the sulfate content in the soil. Puppala et. al., 2002 suggested the use of a finer filter paper and longer centrifuging time in the case of high montmorillonite soils since most of the soil particles sizes are smaller than 0.1 μm. The weight of the barium sulfate precipitated is obtained and multiplied with a factor to obtain the sulfate concentration in ppm (parts per million). Recent studies on diffferent soluble sulfate measurement methods indicated that the Modified UTA method gave repeatable and consistent results compared to other methods (Talluri et. al., 2012).

2.3.2.1.2 AASHTO Method (T 290-95)

The AASHTO method involves mixing 100 grams of soil sample with 300 mL of water. The mixture is then shaken and centrifuged to obtain a clear extract. This method does not specify the time and rate of centrifuging as Modified UTA method does. The resultant solution is filtered using 0.45μm membrane filter. 30 mL of clear sample containing sulfate ion equivalent to 20 - 50 mg of barium sulfate is pipetted. The extract is then diluted to 200 mL by adding distilled water. Hydrochloric acid (1:9) HCl is then added to the solution until the methyl orange end point is reached. Once the endpoint is reached, additional 10 mL of HCl is added to the sample. The solution is then heated until boiling, and then 5 mL of hot barium chloride ($\mathrm{BaCl_2}$) is added to the solution. Boiling is continued until the precipitation process is complete. The solution with precipitate is filtered through a fine ashless filter paper and washed with hot water until the washings are free from chloride. Instead of weighing the filter paper, it is placed in a platinum crucible and charred without flaming for one hour. A drop of sulfuric acid ($\mathrm{H_2SO_4}$) and few drops of hydrofluoric acid (HF) are added to expel the silica as silicon tetraflouride ($\mathrm{SiF_4}$). Then the sample is reignited and cooled in a desiccator to obtain the mass of barium sulfate. Silica and other insoluble materials interfere with the sulfate ion measurement. Provisions for removal of such interferences are given in this method. Concentration of sulfate ion in mg/Kg (ppm) is obtained from the following expression:

$$\mathrm{Sulfate\ in\ mg/Kg} = \frac{411500 \times \mathrm{W}}{\mathrm{S}}$$

Where, W = Weight of $\mathrm{BaSO_4}$ in grams and S = Weight of sample used in grams = 10

2.3.2.2 Turbidity Based Determination of Sulfates

Turbidity based techniques measure the amount of trubidity caused by presence of sulfates and translate it into the sulfate concentration. Two most widely used turbidity techniques are presented here. They are TxDOT method (Tex-145-E) and ASTM method (ASTM C1580).

2.3.2.1.3 TxDOT Method (Tex-145-E)

In this method. turbidity caused by sulfate is measured using a colorimeter. Colorimeter measures the degree of absorption of light transmitted through the sample by human eye (Harris et al., 2003). By using the relation between absorbance and concentration of a solution, the concentration of soluble ions can be obtained under the assumption that wavelength and optical path length of radiation remain constant. 10 g of representative soil sample is collected and mixed with 200 mL of distilled or de-ionized water corresponding to a dilution ratio of 1:20. The sample is shaken manually for 1 minute and kept for overnight. The next day, the sample is filtered through a Whatman No.42 filter paper and the filtrate is collected in a beaker. Using a clean pipette, 10 mL of the clear filtrate is extracted and placed in a sample vial. The sample vial is then cleaned using Kim wipe to remove any finger prints or dirt marks which interfere with the sulfate measurement. The colorimeter is switched on and the mode key is pressed until "SUL" method is displayed. The sample vial is then placed in the colorimeter and zeroed for initial calibration. The sample vial is taken out and a sulfate tablet is added to the solution and tamped with a white plastic rod. The sample vial is then placed in the colorimeter and the test key is pressed. The colorimeter displays a reading and an average of three readings is obtained. The results are multiplied with the dilution ratio to obtain the concentration in ppm.

With 1:20 dilution, the colorimeter can measure concentrations in the range of 100 - 4000 ppm. If the concentration exceeds the allowable limit of colorimeter an error message is displayed. Dilution ratio can be increased and the values obtained are multiplied with the corresponding dilution ratios. By changing the dilution ratios colorimeter can measure concentrations up to 44000 ppm. If the concentration is greater than 44000 ppm an error message is displayed and the sulfate value can be reported as > 44000 ppm.

2.3.2.1.4 ASTM Method (C1580)

ASTM method can measure sulfate concentration in the range of 0.02-3.33% (200-33,000ppm). This method involves preparation of a standard sulfate solution using 0.1479gm of anhydrous sodium sulfate (Na_2SO_4) diluted to 1 liter. This standard sulfate solution in volumes of 0.0, 2.0, 5.0, 10.0, 15.0, 20.0, 30.0 and 40.0 mL is collected and diluted to 100 mL using distilled water. These solutions correspond to sulfate concentrations of 0.0, 2.0, 5.0, 10.0, 15.0, 20.0, 30.0 and 40.0 mg/L respectively. These solutions are run in a photometer, and corresponding photometer readings are recorded to prepare calibration curves. In two 400 mL beakers labeled A and B, 30g and 3g of representative soil is collected and 250 mL of de-ionized water is added to each beaker and stirred for 1 hour using a magnetic stirrer. Once the stirring is over, the solutions are filtered through two dry medium-textured filter papers. pH of the resultant solution is adjusted using 0.1 N HCl or 0.1 N NaOH to 7±1. 10 mL and 20 mL of aliquot is collected from each solution (A and B) and diluted to 100 mL. 5.0 mL of conditioning reagent (containing 30 mL of HCl, 300 mL reagent water, 100 ml 95% ethanol, 75 g sodium chloride and 50 mL of glycerol) is added to each of these solutions and stirred using a magnetic stirrer. 0.3 g of barium chloride crystals are added to these solutions and stirred for 1 min. The solution is poured into the turbidity meter, and turbidity is recorded after 4 min. The sulfate concentration in mg is determined from the calibration curve.

$$P = \frac{2.5 * M}{A * W}$$

Where:

P= Percentage of SO_4 in dried soil; M=mg/L of SO_4 calculated from calibration curve; W= mass of soil placed in 400 ml beaker in g; A= volume of aliquot specimen, in mL

2.4 Sulfate Heave Mechanisms

2.4.1 Introduction

Sherwood (1962) reported that a high pH is required to generate the reaction between the clay fraction and sulfates present. When calcium-based stabilizers are used to treat expansive soils, the pH of the system rises to a value of 12.4. At higher pH levels, the solubility of silica and alumina in clays increase, and they are released into the solution. The sources of alumina in natural soils are amorphous hydroxyl aluminum, alumino silicate phases of soil and hydroxyl phases from smectite and montmorillonite. When the soils contain sulfates, sulfates react with calcium and alumina released from clay to form the expansive mineral Ettringite ($Ca_6.[Al(OH)_6]_2.(SO_4)_3.26H_2O$). Ettringite is highly expansive in nature and can expand up to 137% of its original volume (Little et al., 2010). Ettringite is a calcium-sulfate-aluminate-hydrate and has 26 molecules of water in it. Formation and growth of Ettringite has shown to improve the water retention capability leading to material softening. To study the effects of material softening, Puppala et. al., 2006 conducted experiments where they measured the stiffness properties of chemically-treated sulfate soils using Bender elements (BE). They found out that at sulfate levels around 1,000ppm, strength enhancements were observed; whereas, sulfate levels of 10,000 showed strength reduction behavior leading to destabilizing mechanisms. These destabilization mechanisms caused softening of small strain shear moduli properties.

Mehta et al., 1966 reported that the formation of Ettringite is favored in high alumina content whereas formation of monosulfate hydrate is favored in low alumina content. Also, the Ettringite formation is controlled by the molar ratio of Al_2O_3 and SO_4. When the molar ratio of Al_2O_3 and SO_4 is greater than one, formation of monosulfate hydrate is favored. When the ratio is less than one Ettringite formation is favored. Both monosulfate hydrate and Ettringite are stable phases in aqueous solution. It was established that at higher temperatures monosulfate hydrate is stable whereas at lower temperatures Ettringite is stable. When soils are initially treated with lime, lime increases the pH of the system and hence the solubilities of alumina and silica. At higher alumina contents monosulfate hydrate is formed first. As the rate of release of alumina into the solution decreases gradually Ettringite becomes a stable phase. If sufficient soluble silica and alumina are not available in the soil then calcium reacts with other minerals to form Gypsum, calcite and other pozzolanic compounds. The rate and amount of sulfate release in to the solution depends on the sulfate salt present in the soil. Sulfate salts with higher dissolution rates get dissolved easily and depleted early. If there is more alumina in the system than sulfates, the formation of monosulfate hydrates continues until the pH of the system drops to a level leading to slow release of alumina in to the solution. Ettringite becomes a stable phase once again. Once Ettringite is formed it continues to grow in its purest form until the temperature of the system drops below 15°C, above 15°C Ettringite is a stable phase (Kollman et al., 1977).

When the temperature of the system drops below 15°C, Ettringite transforms by series of reactions into Thaumasite ($Ca_6.[Al(OH)_6]_2.(SO_4)_2.(CO_3).24H_2O$) provided sufficient carbonates and humidity are present in the soil. This transformation takes place by isostructural substitution of silica for aluminum and carbonate for sulfate. The transformation of Ettringite to Thaumasite is followed by intermediate phases. Thaumasite is also an expansive mineral and was found in many of pavement failure cases (Hunter, 1988). Expansion capability of Thaumasite is similar to Ettringite. It was found that rate of Ettringite formation and subsequent growth would be greater during summer (Rollings et al., 1999). When the pH of the system falls below 10.5, the formation and growth of Ettringite is ceased. In this case, gypsum becomes a stable phase.

The literature on sulfate-induced heave in soils report different environments where heave may occur. Two different geochemical environments where heaving may occur are presented here. First, sulfide minerals oxidize and react with other soil minerals to form sulfate minerals (Dubbe et al., 1997). This transformation involves an increase in volume due to variations in atomic packing as well as the addition of water to the mineral structure. The second environment involves the formation of a mineral that expands up to 250% when completely formed (Berger et al., 2001). This mineral, called Ettringite, only forms under special circumstances; there have been many experiments that elucidate factors controlling the formation of this expansive mineral in concrete and more recently in soils (Sherwood, 1962; Ogawa and Roy, 1982; Hunter, 1989; Deng and Tang, 1994; Dermatas, 1995).

There are two geochemical environments under which Ettringite formation occurs. The first geochemical environment for sulfate-induced heave was due to the oxidation of sulfides. Pyrite and Marcasite, (both minerals are FeS_2 but the atoms are arranged differently) form under an 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 process is explained by the equation below:

$$4FeS_2\,(Pyrite) + 14H_2O + 15O_2 \rightarrow 4Fe(OH)_3 + 8H_2SO_4 \tag{6}$$

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 sulfur will either remain in solution or precipitate as gypsum if there is sufficient calcium present. The source of calcium is often limestone (CaCO3); limestone is very soluble in acids. Looking at the right side of equation 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 (equation 2), which will supply Ca^{2+} for the formation of gypsum (Equation 3).

$$CaCO_3 + H_2SO_4 \rightarrow\ Ca^{2+} + (SO4)^{2-} + H_2O + CO_2 \tag{7}$$

$$Ca^{2+} + (SO4)^{2-} + 2H_2O \rightarrow CaSO_4.2H_2O \tag{8}$$

The mineral transformation of pyrite to ferric hydroxide and gypsum results in an increase in volume. The oxidation of pyrite and the formation of gypsum are responsible for distress experienced in a few construction projects (Dubbe et al., 1997). 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 calcium aluminate sulfate hydrate mineral (Ettringite). The next section describes the chemical process of Ettringite formation and transformation to Thaumasite with equations.

2.4.2 Chemical Process

Hunter, 1988 summarized the series of reactions involving the formation and transformation of Ettringite to Thaumasite. These reactions are presented below. The initial reactions are normal pozzolanic reactions occurring in lime-treated soils. The type of clay mineral indicated here is Montmorillonite (Equation 11). The sole function of clay is to provide alumina and silica. In the presence of excessive sulfate, sulfate from any evaporate react with alumina released from dissolution of clay to form Ettringite.

$$CaO + H_2O \rightarrow Ca(OH)_2 \tag{9}$$

$$(Hydration\ of\ Quciklime)$$

$$Ca(OH)_2 \rightarrow Ca^{2+} + 2(OH)^- \tag{10}$$

$$(Ionization\ of\ Calcium\ Hydroxide\ as\ pH\ rises\ to\ 12.4)$$

$$Al_2Si_4O_{10}(OH)_2.nH_2O + 2(OH)^- + 10H_2O \rightarrow 2Al(OH)_4{}^- + 4H_4SiO_4 + nH_2 \tag{11}$$

$$(Dissolution\ of\ Clay\ Mineral\ at\ pH > 10.5)$$

$$2H_4SiO_4 \rightarrow 2H_3SiO_4^- + 2H^+ \rightarrow 2H_2SiO_4^{2-} + 2H^+ \tag{12}$$

$$(Dissociation\ of\ Silicic\ Acid)$$

$$5Ca^{2+} + 6H_3SiO_4^- + 4(OH)^- \rightarrow Ca_5(Si_6O_{18}H_2).4H_2.O + 6(OH)^- \tag{13}$$

$$(Formation\ of\ Clacium\ Silicate\ Hydrate\ Gel)$$

$$M_xSO_4.nH_2O \rightarrow XM^{Y+} + SO_4^{2-} + nH_2O \tag{14}$$

$$(Dissolution\ of\ Sulfate\ Minerals, x = 1; y = 2\ or\ x = 2; y = 1)$$

$$6Ca^{2+} + 2Al(OH)_4^- + 4(OH)^- + 3SO_4^{2-} + 26H_2O \rightarrow Ca_6[Al(OH)_6]_2.(SO_4)_3.26H_2O \tag{15}$$

$$(Formation\ of\ Ettringite)$$

$$CO_2 + H_2O \rightarrow H_2CO_3 \tag{16}$$

$$(Formation\ of\ Carbonic\ Acid)$$

$$CaCO_3 + H_2CO_3 \rightarrow Ca^{2+} + 2H^+ + 2CO_3^{2-} \tag{17}$$

$$(Dissolution\ of\ Calcite\ in\ Carbonic\ Acid)$$

$$Ca_6[Al(OH)_6]_2.(SO_4)_3.26H_2O + 2H_2SiO_4^{2-} + 2CO_3^{2-} + O_2$$

$$\rightarrow Ca_6[Si(OH)_6]_2.(SO_4)_{23}.(CO_3)_2.24H_2O + 2Al(OH)_4^- + SO_4^{2-} + 4OH^- + 2H_2O \tag{18}$$

$$(Iso - structural\ Substitution\ of\ Ettrngite\ to\ Thaumasite)$$

$$Ca^{2+} + SO_4^{2-} + 2H_2O \rightarrow CaSO_4.2H_2O \tag{19}$$

$$(Formation\ of\ Secondary\ Gypsum)$$

$$Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3 \tag{20}$$

$$(Formation\ of\ Secondary\ Calcite)$$

2.4.3 Ettringite Mineral Structure

The name of Ettringite is derived from the place Ettringen, Rhine province, Germany. The mineral occurs in cavities of metamorphosed limestone inclusions in volcanic flows. The term “Ettringite” was first introduced by Lehamann in 1874. The mineral structure of Ettringite is hexagonal prisms, often in elongated form. Ettringite can have different shapes: needle-like, lath-like, and rod-like. These shapes depend on the time and pH during the formation period. A photograph taken with the help of a Scanning electron microscope (SEM) shows the needle-like structure of Ettringite and is presented in Figure 2.5. Previous research concluded that when pH is very high, the rod-like Ettringite crystals are formed. Lath-like crystals are formed as concretions of smaller crystals aligned in the same direction and in low pH conditions, while needle-like crystals are formed in low pH conditions (Kollman, 1978). Figure 2.6 depicts a schematic of the mineral structure of Ettringite. The preceding section describes various schools of thought related to Ettringite formation and heaving.

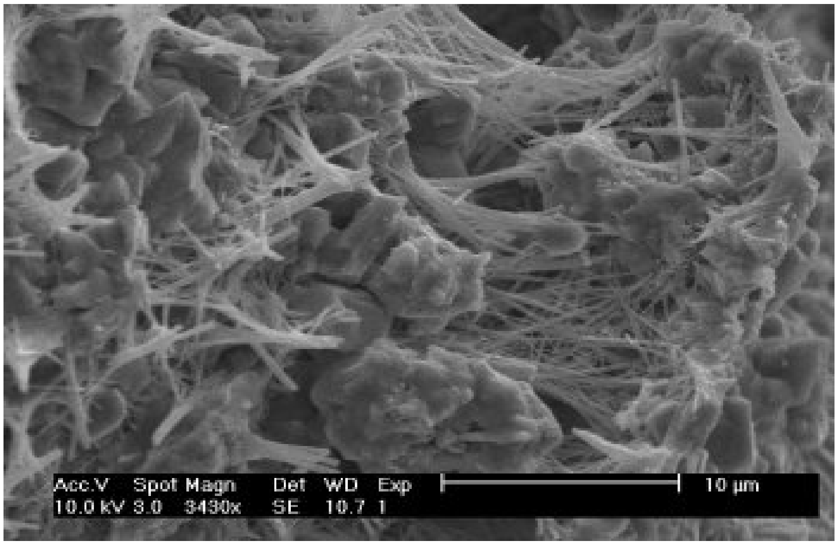

Figure 2.5 SEM photograph of needle-like Ettringite (Mallat, 2006)

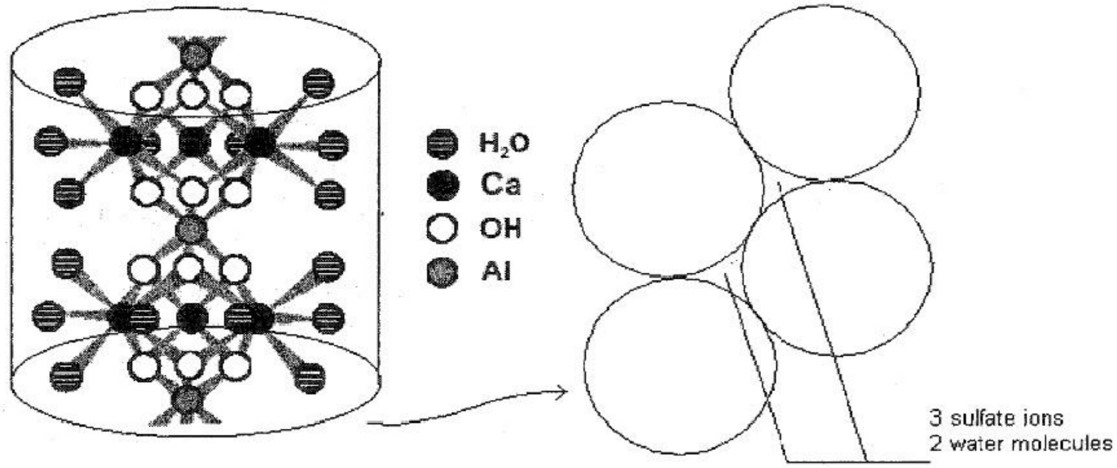

Figure 2.6 Schematic of the mineral structure of Ettringite (Intharasombat, 2003)

2.4.3 Heaving Mechanisms

The formation of Ettringite and its subsequent growth are explained by Cohen (1983). According to him, there are two different swell mechanisms associated with Ettringite formation and subsequent growth. These mechanisms are reported as crystal growth theory and swelling theory. The expansion theories presented in Cohen's paper are based on Ettringite formation and growth in cement concrete. Dermatas (1995) summarized the heaving mechanism in two categories. The first is a topochemical mechanism where expansion is related to formation and/or oriented crystal growth of Ettringite (Ogawa and Roy, 1982). As per the crystal growth theory, aluminum, calcium and sulfates can concentrate around

Ettringite nucleation sites and combine to form additional Ettringite. With Ettringite crystal growth, pressure is exerted on the restraining media. When this pressure exceeds the confinement or overburden, swelling occurs. The second is a thorough solution mechanism where expansion is related to swelling due to hydration.

2.4.3.1 Heaving due to Crystal Growth

Ogawa and Roy (1982) proposed a heave mechanism based on crystal growth. This results in a large nucleation rate which generates many small Ettringite crystals surrounding the aluminum-bearing particles. According to their theory, Ettringite forms around calcium aluminum sulfate particles in the early stages of cement hydration. This creates a reaction zone around every particle. In later stages, with the introduction of water into the system, the crystals assume a needle-like shape around particles from which they are formed. When the adjacent reaction zones intersect they exert mutual pressure on each other, leading to the swelling of the whole system. This mechanism is favored in high pH and sufficient availability of reactants. In soil-lime systems, the soil matrix is less rigid and contains larger void spaces compared to the cement concrete system. This allows the accommodation of initial Ettringite in soil voids. With introduction of additional water into the system in later stages, heaving occurs since the soil matrix can no longer accommodate the continuous Ettringite growth.

2.4.3.2 Heaving due to Hydration

Swelling due to hydration was proposed by Mehta (1973). He suggested that formation of Ettringite follows a thorough solution mechanism. Also, in the presence of saturated $Ca(OH)_2$, the rate of hydration of aluminum decreases significantly. This causes the Ettringite to form gel-like and colloidal crystals. These colloidal crystals adsorb large quantities of water molecules due to their high surface area and net negative charge. Expansion of colloidal Ettringite is aided by external water supply. In the absence of lime, long rod-like crystals are formed. These crystals have low surface area. They absorb lesser water molecules, and hence no expansion is observed in this case. Colloidal or gel-like crystals are formed in high hydroxyl ion concentration; whereas, rod-like crystals are formed in low hydroxyl ion concentrations. Mehta and Wang (1982) conducted series of experiments and found that coarser Ettringite expands less than finer Ettringite. Scanning electron microscope studies revealed that in the presence of lime, Ettringite crystals are 1 micron in length and ¼ micron in width. In the absence of lime, longer (6 to 8 inches) and wider (1/2 to 1 micron) crystals are observed. He observed that the amount of expansion is associated with the amount of water adsorbed. The restraint offered by soil system is less compared to concrete system and hence even small swell pressures can cause expansions in soils.

The investigation of chemically treated soils in Las Vegas by Hunter (1989) identified a total swelling of 24% swell due to formation of ettringite/thaumasite, and he attributed the rest to an increase in voids from the soil initially being in such a heavily compacted state. Many investigators (Mitchell and Dermatas, 1992; Wild et al., 1999; Kota et al., 1996) have noted that low compaction density reduced the swell due to more void space which will allow the expansive minerals formation. Harris et al., 2004 concluded that reduced swell is due to a combination of more void space and a faster reaction rate removing more of the sulfates from the system before compaction.

2.4.4 Factors Influencing Ettringite Induced Heave

The following conditions needs to be satisfied for the formation of Ettringite and its growth:

1. High pH environment
2. High water supply
3. Relative humidity
4. Continuous supply of calcium

Ettringite formation is favored in high pH conditions. When the pH of the system drops below 10.5 the Ettringite formation/expansion is terminated. At pH<10.5, due to inadequate supply of dissolved clay or calcium ions or sulfate ions, the Ettringite/Thaumasite formation/expansion is terminated. Adequate supply of alumina is an important factor. The possible sources of silica and alumina in soils are clay minerals, quartz, feldspar, mica and other alumino-silicate minerals. The experience from Stewart Avenue suggested that a minimum of 10 % clay fraction is needed for Ettringite formation. Availability of alumina is strongly dependent on the quantity and type of clay mineral present in the soils. Kaolinite mineral releases more alumina compared to Montmorillonite mineral. At elevated pH conditions, Kaolinite releases alumina five times faster than Montmorillonite (Dermatas, 1995). This fact is supported by Mitchell and Dermatas (1992) investigation of lime treated specimens of sand mixed with Kaolinite and Montmorillonite. Kaolinite acts as a continuous source of alumina and hence Ettringite phase is stable. A more recent study using geochemical modeling further illustrates the importance of aluminum availability

(Little et al., 2005) in generating reactions causing sulfate-heave; furthermore, they speculate how the Al/Si ratio may be modified to mitigate sulfate heave reactions.

Formation of Ettringite is a necessary but not sufficient condition for heave to occur. Heave development is controlled by rate and quantity of Ettringite formation. Heave is a function of the crystal size, restraint of the system and ion mobility within the system. Due to lower rigidity of stabilized soil matrix compared to Portland cement, soils are more open systems where Ettringite can diffuse easily, resulting additional precipitation of Ettringite (Hunter, 1988). Continuous supply of water is necessary for growth of Ettringite and subsequent heave. In Stewart Avenue, Las Vegas case study in areas with sulfate contents as low as 700 ppm severe damage and heave were observed whereas in areas with sulfate contents close to 20,500 ppm no heave was observed. In the previous case those areas were close to a major source of water.

Relative humidity of the system is also an important factor. Sulfate-induced heave is dependent on the amount of sulfates present in the soil. Hydraulic and osmotic pressures in soils are responsible for a supply of additional sulfates in soils. Hydraulic and osmotic pressures assist the movement of sulfates from subgrade soil to the stabilized pavement layers. The greater the soluble sulfates, the greater is the risk of sulfate attack.

The amount and type of lime used plays an important role since, with increase in lime content, the amount of calcium available for Ettringite formation increases. At low lime contents, sulfate heave is not observed. Formation of Ettringite is effected by temperature (Mitchell et al., 1992). Temperature affects the rate of ongoing reactions, and hence the overall reaction process. The temperature conditions found in pavement sections (20-40^{0}C) are sufficient to cause Ettringite growth. Ettringite formation and growth is higher at warm temperatures. Other factors influencing the formation of Ettringite are water content and dry density of lime-treated soil.

2.5 Sulfate Heave Case Histories

In this section various case histories of sulfate induced heave are reported in brief. A summary of several case histories was presented at the end of the section in Table 1.

2.5.1 Stewart Avenue, Las Vegas, Nevada

Severe heaving was observed on Stewart Avenue Street, Las Vegas, Nevada two years after construction (Hunter, 1988). Distress was observed in asphaltic pavement, concrete and asphalt medians. The maximum distress observed was in the order of 12 inches (30 cm). Figure 2.7 shows the extent of distress on Stewart Avenue. The first evidence of distress appeared six months after construction. The pavement section contained 10 cm of asphaltic concrete layer followed by 13-20 cm of aggregate base on a 30 cm lime treated sub-base. The soils in the area are treated with 4 % of lime by dry weight of soil. The soils in the Stewart Avenue are sandy silts and sandy clays and are fairly homogenous. The mineralogy of the soils contained primary minerals (Quartz and Feldspar), clay minerals (Sepiolite and Montmorillonite) and evaporates (Calcite and Gypsum). Areas of major damage were found to be adjacent to an obvious source of water, areas of poor drainage and construction joints between asphaltic pavement and concrete median. The observed distress is shown in Figure 2.7.

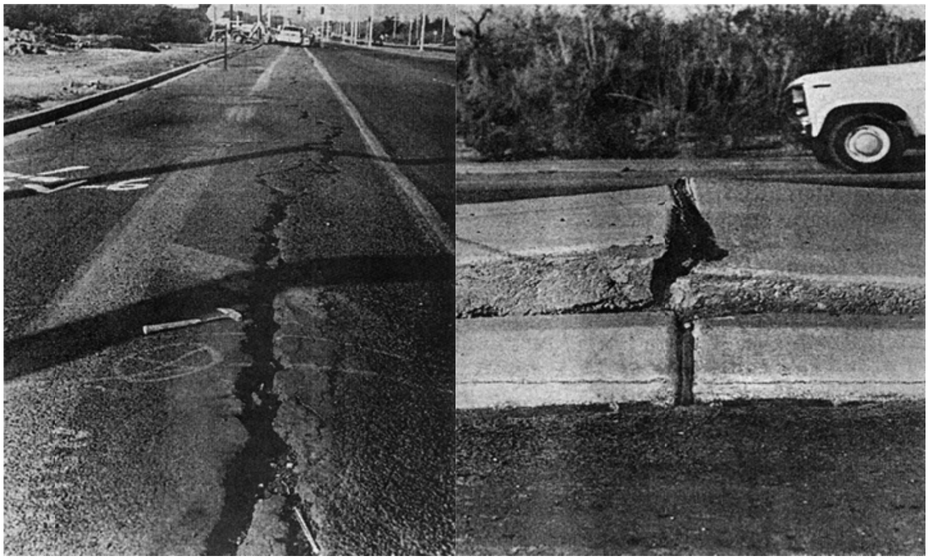

Figure 2.7 Longitudinal and Transverse cracks on Stewart Avenue (Hunter, 1988)

A forensic investigation was carried out to find the possible cause of the heaving. The results of the investigation showed that the clays present are slight to moderately expansive, but are not capable of 100 % swelling. The reactions between lime and sulfates were the causes of heaving. In addition, soils in this area contained gypsum as a source of sulfate. The influx of sulfates is provided by percolating water.

X-ray diffraction (XRD) and Scanning Electron Microscopy (SEM studies) indicated the presence of Thaumasite in the damaged sub-base layers. The temperatures recorded in the pavement areas were around 13°C. Formation and growth of Thaumasite is favored at this temperature, since below 15°C Ettringite gets converted into Thaumasite by isostructural substitution. Overall formation of expansive minerals Ettringite/Thaumasite, and their subsequent growth on soaking are the causes of swelling. It was also concluded that a clay fraction of 10% is sufficient to cause sulfate induced heave in soils

2.5.2 Bush Road, Georgia

In 1992, five months after its construction, a low volume road in Chatham County, Georgia developed severe bumps (Rollings et al., 1999). Reflective cracking in overlying asphaltic pavement and pavement surface heaving were observed in some locations. The maximum size of bumps was 3.1 m wide and 63 mm height. The low-volume road was constructed by 31 mm of asphalt concrete surface resting on 15 mm cement-stabilized sub-base. The soils in this area varied from fine sands to silt and clayey sands to sandy clays. The road was built on clayey sand stabilized with 5-6% Ordinary Portland cement. An investigation was carried out to find the probable causes of heaving. Core samples from areas of severe and no distress were sent to the laboratory, and mineralogical studies were performed. Figure 2.8 shows the distressed areas.

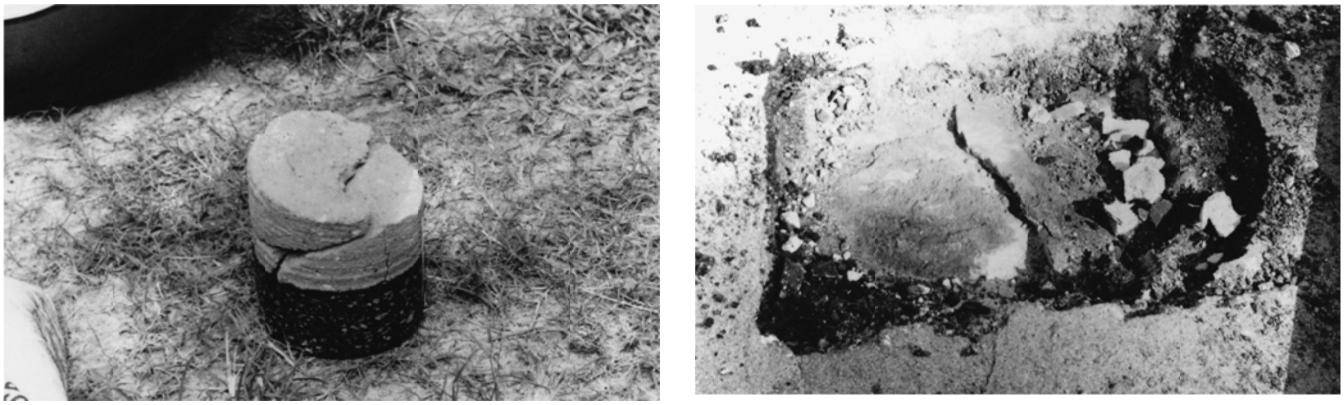

Figure 2.8 (a) Distress in Cement Stabilized Sand (b) Crack in Cement Stabilized Sand Base

Upon EDAX studies, an abundance of needle-like crystals were observed in areas with high bumps. These needles-like crystals were found to be Ettrnigite. Though the stabilized soil was sandy, it contained significant proportions of mineral halloysite, which is rich in alumina and is required for the Ettrngite formation. Also, the original soil did not contain enough sulfates to cause expansion, but the well water used for mixing soil and cement contained sulfates enough to cause Ettringite formation and heaving. Ettringite growth is supported by the moist climate of Georgia and ingress of water through the cracked joints of pavement. The temperature of the pavement is enough to cause heave reactions to take place. Sulfate concentrations as low as 700 ppm caused severe heave and damage in some areas due to the abundance of water. The authors classified this attack as a Type II attack in which the Portland cement hydration products provide calcium, clay fraction provide alumina which react with soil sulfates to from Ettringite. Findings of this case study cautioned against cement stabilization of sands with significant clay fractions since they are sometimes more susceptible to sulfate attack than lime treated materials.

2.5.3 Holloman Air force Base, New Mexico

Pavements and ribbed mat foundation on a project at Holloman Air force Base, NM developed heaving problems several years after construction (Rollings et al., 2006). All the areas in the project have 600 mm of crushed concrete as fill or base course. Severe heave was observed in the aircraft maintenance hangar, ramps and areas where the thickness of the floor is only 150 mm. Heave is mostly observed in areas where recycled concrete aggregate is used as fill or base course. The climate in NM is semi-arid. The water table at Holloman AFB fluctuates throughout the year, but it is often high. The soils in the area are CL-ML, ML, or CL in the Unified Soil Classification System, with plasticity indices varying from non-plastic to 15 and with about 50–75% passing through the number 200 sieve.

Large quantities of gypsum are found in the areas of Holloman AFB. Microscopy and X-ray diffraction of the samples from heaved areas indicated the presence of large quantities of Ettringite and Thaumasite. Intact core samples were collected near air craft maintenance hangar and tested for chemical composition. These cores contained gypsum as a re-cementing material on the intact cores, and gypsum was also found as coating around aggregate particles in the hangar area. The soils in the area contained Kaolinite and Chlorite as primary clay minerals, but they are not responsible for the heave. Figure 2.9 shows the heaving in flexible pavement near the northeast corner of the hangar.

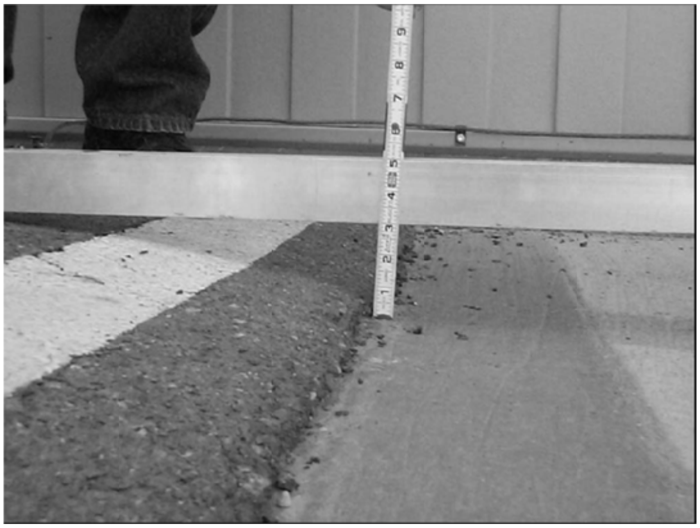

Figure 2.9 Heaving in flexible pavement near northeast corner of hangar

It was found that sulfate attack of the recycled concrete was the cause of the heaving at Holloman AFB. Crushed recycled concrete is more permeable and allows easy passage of water through it. The high water table level in this area assisted in seepage of water through crushed concrete layers. Fine soil particles are available within the recycled concrete through subgrade contamination during the reclaiming process. Although sulfate resistant cements were used in the concrete, which limit the availability of alumina in soils, crushing and placing allowed contamination of the soil. The reaction between alumina from fine fractions of soil, calcium from recycled concrete, and readily available gypsum resulted in the formation of expansive mineral Thaumasite. It was concluded from the forensic investigation that although recycled concrete is a valuable construction material, it cannot be used in pavement construction in areas of sulfate contamination. The use of fly ash, ground granulated blast furnace slag with recycled concrete can be implemented provided sufficient laboratory data is available.

2.5.4 Joe Pool Dam, Texas

In 1988 and 1989, several park roads in Joe Pool Lake experienced severe heaving problems (Perrin, 1992). The pavement sections contained 150mm thick subgrade layers stabilized with 5-6% lime. Soils in this area were lean clays and clayey sands, with less than three percent clay swelling clay minerals. The extent of heaving is depicted in Figure 2.10. These soils belonged to the Eagle Ford Shale formation. Though the soils contained barely detectable sulfate contents, the lime-treated base materials contained 2000-9000 ppm sulfates. Though the reason for the increase in sulfate content was not known, it was hypothesized that sulfates could have migrated from the surrounding soil through continuous supply of fresh water. Upon mineralogical investigation, it was found that Ettringite and Thaumasite were the causes of the heaving. The roadway was re-compacted, but the heaving issue continued. Finally, all the lime treated layers were replaced with gravel base and non-expansive fill, and no issues of heaving were observed.

Figure 2.10 Heaving in lime treated subgrade

2.5.5 Sulfate Attack on a Tunnel Shotcrete Liner, Dallas, Texas

Cracking and water leakage problems were observed in a tunnel shotcrete liner in Dallas, Texas (Puppala et al., 2010). The tunnel was found on limestone, on top of the Eagle Ford geological formation. On careful inspection, a white powder-like material and gel-like substance are found on the shotcrete liner. Samples of powder material and rock core samples from distress locations were collected and sent to The University of Texas at Arlington (UTA) geotechnical laboratories. These were collected from low distress regions (LDR), medium distress regions (MDR) and high distress regions (HDR). These samples were subjected to extensive mineralogical testing by XRPD and EDAX studies. Rock core samples were subjected to unconfined compressive strength (UCS), indirect tensile strength (ITS) and unconsolidated undrained (UU) tests. A photograph of the distress region is shown in Figure 2.11.

Figure 2.11 Distress region on tunnel lining (C-3)

Mineralogical studies of all the samples indicated the presence of Ettringite and Thaumasite. The core samples and powder samples collected from the field indicated presence of calcium, alumina, silica and sulfates. High moisture contents of approximately 15% were measured in high distress regions, and 4% were measured in low distress regions. Sulfate contents less than 1500 ppm and greater than 2000 ppm were observed in LDR and HDR areas. UCS testing of the rock samples indicated the decrease in UCS value with increasing distress magnitude. Similar trends were observed with UU tests and ITS tests. The presence of sulfates influenced the rock properties adversely. It was observed that the presence of sulfates and continuous moisture leakage from the joints could compromise the tunnel safety. It was concluded that continuous monitoring of the tunnel heave needs to be done. The use of sulfate resistant Type V cement as shotcrete material is suggested.

2.5.6 Sulfate Heave Issues at DFW Airport, Texas

One of the taxiway sections built at the Dallas/Fort Worth International Airport (DFW) showed signs of sulfate induced heave. Localized areas of heave distress were observed along both shoulders of the taxiway section. The shoulders were constructed of flexible asphalt pavement, which rest upon a lime-treated base soil. The main taxiway section did not show any signs of heaving since it was built with the rigid reinforced concrete pavement overlying a four to twelve inch lime-treated base course. The natural subgrade soil was shaley clay with sandy seams and occasional gypsum deposits. The base course consists of the native subgrade soil stabilized with lime. Figure 2.12 shows the distress pattern.

The shoulders exhibited pavement cracking associated with heave distress. At several locations, the amount of heave ranged from 0.05m (5cm). to as high as 0.3m (30cm). This heave pattern was irregular and sometime affected small localized areas of one to two feet in diameter. Other heave- related cracks in the asphalt pavement appeared near the junction between rigid concrete and asphalt concrete sections. Significant lateral movement of pavement edges had also occurred at certain locations. On the other hand, the rigid pavement was in good condition, with few minor shrinkage cracks at very few locations.

Reasons for the observed distress were location of drainage ditches near the shoulders and topography of the site which might have contributed to the increase in the heaving of lime treated soils. Heavy rain fall occurred in the last six months of 1996 and early 1997 may have raised the water levels under the pavement sections. This moisture may have contributed to the hydration necessary for the formation of Ettringite and Thaumasite compounds. These compounds, when further hydrated, may have resulted in the heaving of the flexible pavement section. The west shoulder exhibited more damage than the east shoulder. Part of this may be attributed to the water pooling near the base of the embankment which is located next to the west shoulder. Another factor for less distress on the east shoulder could be attributed to the better draining facilities of the east shoulder section due to topographical features.

Figure 2.12 Heave Distress Pattern on West Shoulder of Taxiway

2.5.7 US 82, Texas

The Texas Department of Transportation (TxDOT) observed heaving on a new construction project on U.S.82 (Chen et al., 2005). The pavement design included a 50 mm asphalt concrete over 300 mm flexible base followed by 200 mm lime treated subgrade. The heaving was observed on east side of the project, while no heaving was observed on the west side. Figure 2.13 depicts the observed heaving on the east side. The soils on both sides have tan colored soils, but the east side soils contained sparkly bits of gypsum. The subgrade soil from this project belongs to Eagle Ford formation. Soils in Dallas/ Fort Worth are rich in clay and gypsum.

A forensic investigation was initiated to determine the causes of heaving and possible solutions for future projects. Core samples of lime-treated subgrade and raw subgrade were collected and sent for testing by the Texas Transportation Institute (TTI). SEM analysis of the core samples confirmed the presence of long fibrous crystals. These were confirmed as Ettringite. These crystals were absent in raw subgrades. Further chemical analysis was performed to find the unreacted sulfate which could cause heaving in the future. In-situ conductivity and pH measurement tests were performed on soils from both the east side and the west side. The pH values were higher in both these cases since the soils are lime treated. Conductivity of samples from the east side were found to be higher than those from the west side. It was also concluded that a six-fold increase in conductivity is observed with an increase in depth. It was concluded that soluble gypsum seeps from the surface to depths, increasing the conductivity. The soils on the east side have sulfates in the range of 4,000 -27,800 ppm. The soils on the west side contain low amounts of sulfates. The west side of the project was treated with 3% of lime followed by a 72 hr. mellowing period and 3% of lime application. No heaving was observed on the west side. It was concluded from the study that conductivity measurement is a good indicator of sulfates in soils.

Figure 2.13 Heaved area on east bound side

2.5.8 US 287, Texas

Extensive longitudinal cracking and severe ride roughness were observed on US 287 near Childress, Texas (Zhiming, 2008). The pavement section consisted of 76.2 mm Type D asphalt hot mix , followed by a 254 mm fly ash stabilized crushed stone base underlain by a 178 mm flexible base and 228 mm hydrated lime-treated base. The maximum distress was observed in the north bound (NB) lane. Two raw subgrade soil samples from the north and south right of ways (ROW) were sent to the laboratory for testing. The soils in the NB ROW area were found to contain sulfates above 35,000 ppm. These soils are termed as high sulfate soils (HS). Soils in the south bound ROW are low sulfate soils (LS). The soils in NB ROW are classified as fine grained soils; whereas, soils in the SB ROW are classified as coarse grained soils. Higher clay content in the NB soils is the cause of the swelling. The heave observed in the NB area is sulfate induced. Figure 2.14 shows the longitudinal cracks on shoulder and swell.

A series of laboratory tests were done to characterize the unmodified soils. Atterberg limits tests, bar linear shrinkage tests, conductivity and colorimetry tests, moisture susceptibility tests, free-free resonant column tests, and unconfined compressive strength tests were performed after 10 days of soaking. Various alternative stabilizer treatments such as hydrated lime, Portland cement, lime and fly ash, lime and slag were conducted. Property enhancements with the addition of stabilizers were studied. It was found that a combination of lime and slag seemed to be the best choice for soils with moderate sulfate contents. The combination of lime and fly ash is most suitable for high sulfate soils in terms of swelling and retained unconfined compressive strength. The present study recommended replacing the lime-treated subgrade with select fill, or reworking with the stabilizers.

Figure 2.14 Longitudinal Cracks on Shoulder and Swell

2.5.9 Western Colorado

A commercial structure was constructed with monolithic cast-in-place footing on a lime treated structural fill blanket (Bagley et. al., 2009). The soils in the area were sandy clay to clay overlying sandy clay stone. Shortly after construction, the structure showed 28cm of differential elevation. Lime treatment of the on-site soils was selected for the structural blanket. Lime dosage was chosen as 5-10% by dry weight of soil. Low swell potential of soils, high moisture content and soft clay soils indicated that the water addition was not the cause of the movement. An investigation was begun to determine the causes of heaving. The post-construction investigation showed soluble sulfate contents ranging from 0.37 (3,700ppm) to 1.65 % (16,500 ppm). Surface water infiltrated due to poor drainage and landscape irrigation. Also, the ground water table was encountered at shallow depths. Lime-treated base layer thickness ranged from 0.3 to 0.82 meters. Upon stoichiometric calculations, authors calculated the maximum Ettrnigte induced heave to be 40% of the lime treated fill thickness (12cm to 33cm). Maximum measured heave was recorded as 30cm, which correlated well with the calculations. The south and south east portions of the foundation required complete replacement. The authors recommended precautions to prevent surface and subsurface water to lime treated base material.

Table 2.1 Sulfate Heave Case Studies

Location	Soil	Nature of	Lime/C	Sulfate	Heave
Parking Lots, Kansas, Southern California	N/A	Thaumasite and Ettringite	NA	NA	NA
Stewart Avenue, Las Vegas, Nevada	Silty clay	Ettringite	4.5%	43,500	6 months
Lloyd Park, Joe Pool Lake, Dallas, Texas	OC	Ettringite	5% (L)	2,000– 9,000	Immediately
Auxiliary Runway, Laughlin AFB, Spofford, Texas	Clays	Ettringite	6-9%	14,000– 25,000	2 months
Cedar Hill State Park, Joe Pool Lake, Dallas, Texas	Highly	Ettringite	6% (L)	21,200	2 months
Denver International Airport, Denver, Colorado	Expansi	Ettringite	NA (L)	2,775	NA
SH-118, Alpine & SH-161, Dallas, TX	Clayey	Ettringite	4% (C)	>12,000	6 to 18
Localities in Dallas-Fort Worth Region, Texas	Clays	Ettringite	6%-9%	233-18,000	Varies
Dallas – Fort Worth International Airport, Irving, Texas	Clay	Ettringite	5% (L)	320– 13,000	3 months
Near Shreveport, Louisiana	Aggregates	Ettringite	NA	NA	NA
Holloman Air Force Base, NM	Crushed	Ettringite	NA	NA	Several
U.S.82,TX	N/A	Ettringite	6%(L)	100-27800	Immediately
Baylor Creek Bridge, Childress, TX	Fine		5%(L)	6800-35000	Several
Western Oklahoma	Clays	Ettringite	0-	194-84000	NA
Pavements in Frisco, Arlington, Texas	Clays	Ettringite	6-8%	500-5000	1 month
DART Tunnel, Mocking Bird Station, Dallas	Clay	Ettringite	Ordinar	1700-2300	Several

2.6 Stabilization of Sulfate Bearing Soils

This section is dedicated to various techniques available to stabilize sulfate bearing soils and I their limitations.

2.6.1 Pre-Compaction Mellowing

The investigation of soils in Las Vegas showed that only 24% of the observed swell was caused by the formation of Ettringite and Thaumasite, and the rest of the swell is attributed to an increase in void ratio from the initial compacted state due to the mineral growth (Hunter, 1988). The experience from Stewart Avenue showed that an increase in the void space could potentially accommodate the Ettringite formation and growth. Several investigators noted that compaction to lower density means decreasing the maximum dry density which could create more void space to allow the formation of expansive minerals (Kota et al., 1996 and Mitchell et. al., 1992). Decrease in dry density of the treated mixes can be achieved by delaying the compaction after mixing with the stabilizers. This phenomenon is called "pre-compaction mellowing" in literature. Research studies in the UK have shown that mellowing soils for a certain period of time at constant moisture levels allows the lime to hydrate completely, diffusing through the soil and causing maximum changes in plasticity (West, 1959 and Sherwood, 1993). These plasticity changes create a non-plastic material which is workable. Though the final achieved density is slightly lower, maximum effects of lime stabilization can be achieved by using mellowing.

In the case of sulfate bearing soils, during mellowing, sulfate reactions and lime stabilization reactions occur simultaneously. The formation of deleterious compounds occurs before compaction in this case (Berger et al., 2001; Harris et al., 2004). During the remixing and re-compaction, already formed Ettringite is broken to prevent the formation of reaction zones for further Ettringite growth. As mentioned earlier, any further growth of Ettringite can be accommodated in the less dense material. Harris et. al., 2004 recommended provision of an additional 3-5% moisture above the optimum moisture content during the mellowing to allow faster dissolution of sulfates, leading to the early depletion of reaction compounds for Ettringite formation. Research studies conducted in Texas have shown that the magnitude of swell reduced due to a combination of more voids and a faster reaction rate, removing more of the sulfate from the system before compaction.

Harris et al., 2004 reported that using 3-day mellowing period resulted in acceptable swell in soils with sulfate contents around 7,000ppm. When the sulfate concentrations reached 10,000ppm mellowing did not give positive results in this case. Based on these research findings Texas Department of Transportation warranted use of lime treatment above 8,000ppm sulfate level.

In a study conducted by Berger et. al., (2001), sulfate soils from South Orange County, California were successfully stabilized using a pre-compaction mellowing technique. Soils containing 0, 5000ppm and 8000ppm sulfate concentrations were treated with 4% lime and 4% lime+8% fly ash and allowed to mellow for periods of one, three and five days. After the mellowing period, samples were cast into cylinders and strength and swell tests were conducted to see the effectiveness of stabilization. All the test soils passed the strength and allowable swell criteria, showing the dominance of pozzolanic reactions. The pH of the treated soils was determined to check possible Ettringite reactions. In all the cases, the pH was well below 10, indicating that Ettringite formation reactions are not possible at these pH conditions.

A second set of samples were fabricated using 6% lime and spiked with 14,000ppm (±1000ppm) sulfates to see the effect of a higher concentration of sulfates. All the soils were mixed with 6% lime and 6% lime and 12% fly ash and allowed to mellow for periods of one, three and five days. After the mellowing period, specimens were cast and moist cured for 60 days and tested for swelling in sulfate solution. All the soils showed positive effects of stabilization with minimal vertical swell. The pH of the test soils was observed to be higher than 10 in all cases, indicating the occurrence of both pozzolanic and sulfate reactions. Successful stabilization of all the soils using mellowing technique was attributed to the fact that all the expansion occurred during the mellowing period and sulfates were consumed during the mellowing period with no more sulfates available after compaction. Maximum effects of stabilization were achieved in this case.

2.6.2 Double Application of Lime

Double application of lime to treat sulfate soils is based on the assumption that first application of lime allows formation and expansion of Ettringite, whereas second application of lime accelerates the formation of pozzolanic compounds. These pozzolanic compounds bind the soil particles and improve soil strength. The time gap between the first and second application of lime is an important factor. If the time duration between successive applications of lime is less, the soil sulfates will not get dissolved completely leading to the formation of low-sulfate form of calcium-alumium-sulfate-hydrate (CASH) compounds. These compounds in later stages get converted in to Ettringite with release of sulfates by rain or oxidation of sulfides. A minimum of three days of curing is required between successive applications of lime. Adding more and more lime does not ensure heave arrest, since additional supply of sulfates by leaching and oxidation of pyrites is possible. In such cases heaving problem is still aggravated.

It was observed by Pat Harris et al. (2004) that double application of lime resulted in more heaving than single application. It was concluded that double application of lime is ineffective in soils with high sulfate contents and soils containing sulfides (pyrites). Soils with soluble sulfates up to 7000 ppm can be effectively stabilized with double application of lime (Kota et al., 1996). Double application technique can be used in low sulfate soils provided soils do not contain sulfides.

2.6.3 Combined Lime and Cement Treatment

It is well known that lime and cement treatment improves the workability and reduces volumetric changes of soils. Researchers from The University of Texas at Arlington used a combination of lime and cement to treat expansive soils from Arlington, Texas containing low to medium sulfates (Chakkrit et. al., 2008). As part of the laboratory studies, two high plasticity clay soils from Arlington were chosen and treated with 12% lime and a combination of 6%lime and 6% cement. Two curing periods, 2 days and 7s days, were considered in this study. Laboratory results indicated that the combination of lime and cement successfully provided strength enhancements and reduced swell and shrinkage characteristics. Also, the combination of lime and cement treatment proved to be more effective than the lime treatment alone. Findings from the laboratory study were implemented successfully in the field, and no issues of heaving were observed.

2.6.4 Stabilization Using Low Calcium Based Stabilizers

When soils containing sulfates are treated with calcium based stabilizers, sulfate-induced heave is observed. The most common calcium based stabilizers are cement and lime. The higher the calcium, the higher the heave associated with it. The availability of calcium can be limited by using low-calcium stabilizers. Fly ash is one of the calcium-based stabilizers. Fly ash is classified as class C fly ash and class F as per ASTM. The main difference between class C fly ash and class F fly ash is the availability of free calcium. The percentage of free calcium is higher in class C fly ash compared to class F fly ash. This is the reason why lime needs to be added to class F fly ash for soil stabilization. The factors which affect the reaction rate of fly ash in soils are soil type, specific surface areas of soils, chemical composition of fly ash admixtures and dosage of fly ash. It was concluded that the use of fly ash decreases the swell and shrinkage strains of the soils by decreasing the plasticity index of soil (Puppala et al., 2000).

The treatment of soils with low-calcium based stabilizers such as class C fly ash, class F fly ash has been attempted by various researchers (Wang et al., 2004; Puppala et al., 2006; Solanki et al., 2009, McCarthy et al., 2009). Treatment of soils with fly ash initiates short-term and long-term reactions. The short-term reactions include flocculation and agglomeration with increased ionic exchange. The long-term reactions include strength enhancements.

McCarthy et. al., 2009 studied the effect of lime +fly ash treatment on suppression of swelling associated with Ettringite formation. Their study consisted of two steps. One was a preliminary evaluation, and other was a main laboratory research program. As part of the preliminary evaluation, they used 5:1 quartz and Montmorillonite sand spiked with 20,000ppm sulfates (sodium sulfate) and stabilized using class F fly ash and GGBFS (ground granulated blast furnace slag). The dosages of fly ash and GGBFS were 3, 6, 9, 12% and 3, 6, 9% respectively. Test soils were mixed with different dosages of stabilizers, compacted and tested for swelling for a period of 28 days. Results of the testing showed that the fly ash dosage at 9% yielded the optimum results in suppressing the swelling.

The main research program included stabilizing sulfate bearing Kimmeridge clay using lime (3%) and different dosages of fly ash. As part of this investigation, they also studied the influence of mellowing on suppression of swelling in lime fly ash-treated soils. In this study, they used four different types of class F fly ash with varying coarseness. Test soils were mixed with lime and water, and Proctor curves were developed. Once the maximum dry density and optimum moisture content were established, soils were treated with lime and water and allowed to mellow for periods of 0, 1 and 3 days. After mellowing, the soils were mixed with fly ash (@6, 12, 18, 24% on dry weight basis) and Proctor curves were developed for a different dosage level. Compaction values from the Proctor curves were used in preparing specimens for swell and strength testing. Results indicated that mellowing was effective in suppression of swelling. A control level of swelling (below 5%) was achieved using 18% fly ash at one day mellowing and 12% fly ash at 3 day mellowing. Another important observation was that coarser fly ash was efficient in swell suppression due to the low maximum dry density achieved, which is indicative of more void space for Ettringite accommodation. For the sake of practicality, the authors recommended the use of 3% lime and 10-15% coarse fly ash, with one day mellowing.

In a research study conducted at UTA (Deepthi et al., 2007), three different types of fly ash were used to stabilize sulfate-rich expansive soils from the DFW (Dallas-Fort Worth) area and Arlington area. According to the Unified Soil Classification System (USCS), these soils are classified as low compressibility clays (CL), with medium plasticity index values. Stabilizer dosage levels used in the study are 0, 10, 15, and 20 % by dry weight of the soil. Three-dimensional free swell, shrinkage strain and pressure swell tests were conducted on treated soils. Based on three-dimensional swell test results, class F fly ash treatment was considered a better treatment method compared to bottom ash treatment. It was concluded that Class F fly ash provided maximum enhancements to the soil properties. Heave severity was reduced from a high level to a medium or low severity level by class F fly ash treatment. Maximum property enhancements were observed in combined stabilizer treatment with class F fly ash and nylon fibers.

A similar study conducted at the University of Oklahoma, using class C fly ash (CFA), supported the claim. The soils studied were sulfate-rich lean clays from Oklahoma. The soils were treated with 5, 10, & 15% dosage levels of class C fly ash, lime and cement kiln dust (CKD). The evaluation of stabilizer treatment was done on the long-term and short-term basis. Several engineering tests, including resilient modulus (MR), modulus of elasticity (ME), unconfined compressive strength (UCS), tube suction (TST) and three-dimensional swell tests, were performed. Mineralogical tests were conducted to identify the micro-structural developments. The addition of class C fly ash improved MR, UCS, ME after 28 days of curing. The moisture susceptibility was considerably decreased by the application of CFA. Overall, CFA showed better short-term and long-term performance compared to other stabilizers used in the present study.

A recent research study conducted by McCarthy et al. (2009) in the UK on fly ash treatment of high sulfate soils yielded satisfactory results. He concluded that swelling reduced by increasing fly ash contents and decreasing lime content. Use of coarser fly ash was found to be more effective than finer fly ash. Further, he recommended the use of 3 % lime, 10-15 % mass fly ash, and an allowance of one day mellowing period before application of fly ash and coarser fly ash.

2.6.5 Stabilization with Non-Calcium Based Stabilizers

There have been a number of studies, over the past 20 years, pertaining to the effectiveness of non-calcium based stabilizers for subgrade soil stabilization. The U.S. Forest Service, the U.S. Army Corps of Engineers, and researchers in Australia have studied many of the additives (Bolander, 1997; Giummarra et al., 1997). A review of some of the literature relating to this topic is presented below.

Marquart (1995) stabilized three Texas Vertisols (pH ranged from basic to acidic) with sulfonated naphthalene. Their study showed that higher amounts of stabilizer were required for basic soils (High pH soils) and that there was an optimal stabilizer content for swell reduction. The effects of a potassium-based stabilizer on expansive clay soils was investigated by Addison and Petry (1998). They determined that multiple, low concentration injections performed better than a single high concentration injection and that the treated sites should not be allowed to dry significantly before sealing with a foundation or slab . The effectiveness of three liquid soil stabilizers at changing the engineering properties of clay soils was evaluated by researchers at the University of Texas, Austin (Rauch et al., 2002).

Rauch et al. (2002) concluded that there were no significant changes in soil properties at the recommended stabilizer application rates. Santoni et al. (2002) studied the wet and dry strength gain of a silty-sand material stabilized with 12 nontraditional stabilizers, including: acids, enzymes, lignosulfonates, petroleum emulsions, polymers, and tree resins. They reported a large variation in results, with some stabilizers performing well and other stabilizers not performing at all for the experimental conditions and soil they used.

These studies, using nonstandard (noncalcium-based) stabilizers, were criticized by the manufacturers of the non-standard stabilizer products for measuring properties biased toward the lime/cement industries (i.e. strength gain and/or unrealistic swell tests) in a controlled laboratory setting and for not following the manufacturer's recommended mixing and curing procedures. Many of these products are reported to change the soil compaction characteristics and/or the clay minerals' affinity for water, resulting in less 3-D swell. Harris et al. (2006) evaluated many of the same stabilizers as previous researchers (Rauch et al., 2002; Santoni et al., 2002), but strict mixing and curing requirements imposed on lab testing by the stabilizer manufacturers were followed. Soils with sulfate concentrations above 10,000 ppm were stabilized with nine non-standard stabilizers (acids, emulsions, enzymes, and polymers) and evaluated with respect to 3-dimensional swell and unconfined compressive strength (UCS) because the Texas DOT is primarily concerned with swell reduction in plastic soils and strengthening the subgrade enough to serve as a working platform for construction. The research showed that many of the stabilizers were no more effective than adding water to the soil in the soils tested. Harris et al. (2006) showed that a polymer at six times the recommended application rate and an acid actually reduced the swell by eight percent. However, the strength was not improved. Both stabilizers proved to be more costly than removing and replacing the subgrade.

2.6.6 Ground Granulated Blast Furnace Slag (GGBFS)

Sulfate-induced heaving phenomenon is observed when soils containing sulfates are treated with calcium-based stabilizers such as lime and cement. Replacement of cement or lime by GGBFS has been studied by various researchers (Wild et al., 1998; Tasong et al., 1999; Puppala et al., 2005). Substituting part of the lime with GGBFS for high sulfate clay soil stabilization has been successfully implemented in the UK by researchers. Progressive replacement of the lime by GGBFS reduces the amount of available free lime, improves cementation properties due to slag hydration and reduces the pH of the soil system significantly (Wild et al., 1998, 1999). Experimental studies by Wild et al. (1999) on kaolinite and Kimmeridge clay illustrated that substitution of lime by GGBFS produces significant reduction in linear expansion of sulfate bearing clayey soils containing gypsum as a source of sulfate. In this study lime stabilization is partially replaced with 60 to 80 % of GGBFS. The addition of GGBFS in stabilized sandy and soft clayey soils reduces the permeability considerably. When soils are treated with insufficient quantities of GGBS, Ettringite-induced heave is observed. In such cases, soil sulfates react with calcium, alumina and silica, leading to the formation of Ettringite and subsequent heaving. Replacement of lime with GGBFS reduces the availability of calcium to react with soil sulfates, and the growth of Ettringite is severely inhibited.

Treatment of sulfate-bearing soils by GGBFS has been attempted in United States by various researchers (Harris et al., 2004; Puppala et al., 2005). In a research study conducted at UT Arlington, soils from north Texas were treated with various stabilizers such as class F fly ash + Type V cement, GGBFS, Type V sulfate resistant cement, lime + fibers and GGBFS. These soils are classified as low compressible clays (CL) with sulfate levels around 5000 ppm. Three different moisture contents were considered in the study. The dosage level of GGBFS is 20% by dry weight of soil. UCS, one dimensional swell and three dimensional swell tests were conducted on the soil samples to see the effect of stabilizers on soil property enhancements. Swell strains considerably decreased with the application of GGBFS, and a significant increase in UCS is observed in the present study.

In a similar study conducted by Harris et al. (2006), two stabilizer treatments were applied on College Station, Texas soils. The stabilizers studied were GGBFS + lime and class F fly ash. The soils were treated with 0 and 20,000 ppm sulfates. Several engineering and mineralogical tests were performed to see the effects of stabilizers. The use of 5% GGBFS with 1% lime yielded satisfactory results in reducing swell and improving strength. The cost of GGBFS plus lime treatment was similar to lime treatment alone.

2.6.7 Sulfate Resistant Cements

Cement has been used as a stabilizing material for expansive soils for many years. Soils stabilized with cement are called as “Cement-stabilized” soils (Hausmann, 1990). Different types of cements are available in the market to meet physical and chemical requirements for various applications. Type I cement is used for general RCC structures, Type II for soils with low sulfate contents, Type III for high strength in early stages, Type IV for dam structures and Type V for high sulfate soils. Cement treatment of soils provides strength enhancements and plasticity reductions through flocculation, cementation and pozzolanic reactions. In sulfate-resistant cement stabilization, the series of reactions involved are similar to ordinary cement stabilization. In general, tricalcium aluminate (C_3A) formed in the ordinary Portland cement concrete material provides enough alumina required for Ettringite formation

(Rollings et al., 1999). Sulfate-resistant cements inhibit formation of Ettringite by limiting the availability of reactive alumina. The tricalcium aluminate (C_3A) concentrations in sulfate resistant cements are low; hence, they reduce the amount of alumina available in concrete material to react with soil sulfates to form Ettringite.

Sulfate-resistant cements fail in cases where alumina is introduced ito the soil matrix by contamination. It was observed in the Holloman Air force Base, NM case study that although sulfate resistant cements are used in recycled concrete, they become ineffective through soils contamination during mixing and stock piling, resulting Ettringite formation and subsequent heaving.

Studies on sulfate-resistant cements in stabilization of sulfate soils have been conducted by various researchers (Griffin, 2001; Puppala et al., 2004). In an experimental study conducted by Puppala et al. (2004), Type I/II and Type V stabilizers were used to treat soils with sulfate levels varying from 1000- 5000 ppm and above. The stabilizer dosage levels were established as 5 and 10% by dry weight of the soil, as well as two moisture content levels, wet of optimum and optimum moisture content. Strength, stiffness, swell and shrinkage strain tests were carried out on the treated soils. In both treatments, plasticity indices reduced to an insignificant value. Treated soil samples compacted at wet of optimum yielded higher strength and lesser swell properties. Higher moisture contents at wet of optimum facilitated stronger hydration reactions between cement and soil particles. The strength enhancements due to cement treatment are attributed to the formation of pozzolonic compounds (Tobermorite, Jusite and Prehnite) and flocculation. XRD tests results confirmed that both treatments are effective in sulfate induced heave mitigation. Overall, both treatments yielded similar results since the soils involved in the study were low-to-medium sulfate.

A similar study was conducted by Deepti et al. (2007) at The University of Texas, Arlington. In this study, stabilization potentials of class F fly ash + Type V cement, GGBFS, Type V sulfate resistant cement, lime + fibers and GGBFS were studied on soils with medium sulfate levels. It was found that all the stabilizers improved the soil properties by reducing liquid limits, plasticity indices, swell and shrinkage strains, and increased UCS and MR values. Based on the ranking analysis, it was concluded that Type V cement showed the best performance among the other stabilizers.

Sarkar and Little (1998) successfully stabilized a crushed concrete base parking lot that was contaminated with sulfates, using a Type V cement and Class C fly ash. However, a 19% stabilizer was required to stabilize the base due to very high moisture content in the degraded base.

2.6.8 Use of Geosynthetics

A combination of geotextiles and geogrid can be used as an alternative for high plasticity soils with soluble sulfates. Geotextile acts as a separator between pavement and natural subgrade. The purpose of geotextile is to separate layers of pavement from the natural subgrade and to prevent migration of clay into the upper layers due to traffic loads. Geogrids were successfully used on stabilization of high sulfate soils. Mechanical stabilization by geogrid instead of chemical stabilization can be an alternative for high sulfate soils (Zhiming, 2008).

2.6.9 Pre-treatment with Barium compounds

Using barium compounds to pretreat the sulfate bearing soils was suggested by Ferris et al. (1991). Laboratory testing, using the California Bearing Ratio (CBR) test, indicated increased bearing strength values and decreased swell values when barium hydroxide or barium chloride are added to sulfate bearing soil before lime application. Tests were carried out on soil samples from California, Texas and Colorado. Two types of barium compounds; namely, barium hydroxide and barium chloride were used in this study. The soils were treated with the above mentioned chemicals before the application of lime treatment. The treatment of sulfate-bearing soils with barium compounds resulted in the formation of barium sulfates, removing all the sulfate ions available. With no sulfate ions available to form the water sensitive Ettringite mineral, no heaving was observed in the soil samples. A comparison study indicated the use of barium hydroxide over barium chloride to arrest Ettringite formation effectively. Since the cost of barium treatment is higher than lime treatment, this method did not enjoy much of popularity.

Walker Jr. (1992) used a barium-containing compound to treat soils with high sulfate contents. The dosage of the barium compound was chosen so that it effectively reacted with sulfates to form less soluble products to participate in the Ettringite reactions. Barium hydroxide, barium chloride and barium carbonate were used in his study to pre-treat the soils. Following the barium treatment, test soils were stabilized using lime to give maximum stabilization effects. Soils with sulfate contents up to 10,000 ppm were stabilized successfully using this method.

2.6.10 Use of Polymeric Fibers

The use of polymeric fibers in treating sulfate-bearing soils has been successfully implemented (Punthutaecha et al., 2006). Fibers are not affected by salts in soils, biological degradation, or ultraviolet degradation. They provide additional tensile strength to the soil by reinforcing it. In a study conducted by Punthutaecha et al. (2006), polypropylene (PP) and hydrophilic nylon fibers (N) were used with various forms of fly ash to stabilize sulfate bearing soils. The effect of the combined stabilizer treatment (fibers and ashes) is studied in the present research. The polymeric fiber dosages used in the present study are 0, 0.2, 0.4, and 0.6 % by dry weight of soils. Three-dimensional free-swell, shrinkage strain, pressure swell tests were conducted on all the treated soil samples. At dosage levels higher than 0.2 %, increase in swell behavior was observed in poly propylene treated soils; whereas, nylon fibers decreased the swell behavior of soils with an increase of dosage. Volumetric shrinkage strains decreased with an increase in stabilizer dosage. Inclusion of fibers was slightly effective in reducing swell pressures. It was observed that soils stabilized with combined stabilizers experienced lower heave movements compared to soils stabilized with either fibers or ashes. This is due to flocculation, chemical strengthening reactions between soils and ash stabilizers. Inclusion of fibers enhanced tensile and shear strengths of soils. It was concluded that Class F fly ash combined with nylon fibers provided maximum improvement to soil properties.

2.6.11 Amorphous Silica and Alumina

Researchers in Louisiana used amorphous silica (AS) and silica fume (SF) to stabilize soils containing sulfates (Wang et. al., 2003). Local roads in Louisiana experienced severe heaving and pavement failures when Winn Rock gravel containing gypsum was used as a surface course and stabilized using Portland cement. A laboratory stabilization program was designed to find a suitable stabilizer. A silty clay soil from Louisiana was mixed with Winn Rock containing soil (WRS) and was used for the laboratory evaluations. The soils were mixed with cement and blast furnace slag (BFS), class C fly ash, amorphous silica and silica fume at different proportions. The stabilizer dosage varied from 5-20%. Treated specimens were tested for strength and expansion characteristics. It was observed that soils treated with cement alone produced a material with no compressive strength, indicating the dominance of Ettringite reactions. The observed expansion in this case was high and directly proportional to the cement content. Cement and blast furnace slag-treated soils showed significant strength enhancements and low swell magnitudes. A mixture of cement, class C fly ash and amorphous silica performed best among the stabilizer combinations used with respect to strength enhancements and swell reductions. The authors also recommend the use of silica fume (SF) in place of amorphous silica (AS) due to its easy availability in the market.

The use of amorphous silica in reducing sulfate expansions was demonstrated by McKinnon et al., 1993. They used silicon dioxide at dosages of 0.2-6% by dry weight of the soils before treating with lime. The purpose of adding amorphous silica or silica compound is that silica becomes soluble due to the high pH generated by lime treatment and readily reacts with free calcium and alumina to form pozzolanic compounds. Early formation of pozzolanic compounds robs the constituents required for Ettringite reactions (free calcium and alumina) thereby causing stabilization of the soil. The authors also recommended the use of potassium hydroxide to maintain the high pH conditions to ensure release of free alumina to react with calcium from stabilizer and silica compounds. The authors successfully stabilized soils from Denver, Colorado containing sulfate levels of 6,000 ppm.

2.6.12 Several Other Techniques

The other methods include stabilizing the top portion of select fill, compacting to lower densities and use of polymeric fibers with soil. Soils with no soluble sulfates can be brought from different locations to use as select fill material over the subgrade. Stabilizing the selected fill material is equivalent to stabilizing the natural subgrade. Proper care should be taken to avoid migration of sulfates in to the select fill material, failure of which again leads to sulfate-induced heave. Compaction of the stabilizing layer to lower densities is another option available. Compacting at lower densities allows more void spaces in the soil matrix. This allows more room for the growth of Ettringite and its overall expansion.

2.7 Summary

Based on the literature, we can understand that sulfate-induced heave is inevitable when soils containing considerable amounts of sulfates are treated with calcium-based stabilizers. Cement and lime treatments proved to be effective in treating soils with low sulfate levels; whereas, both the treatments failed in treating high sulfate soils. It was also understood that the exact measurement of soil sulfates in in-situ conditions is not possible, given there is a chance of sulfate migration from external sources. Establishment of a generalized threshold sulfate level at which heaving occurs is difficult due to the limitations on available soil mineralogical data and non-homogeneity in the available data. Alternative stabilization techniques such as double application of lime, pre-compaction mellowing, non-calcium based additives, and low calcium based additives, blast furnace slag, amorphous silica and alumina, and addition of barium compounds have been tried with some degree of success. All the above mentioned methods have their own limitations with respect to sulfate content, cost, applicability in real field conditions and environmental concerns. Applicability of any of these methods is project and site specific since the site conditions and project specifications vary. Another important observation is that in most of the cases treated soils have sulfate concentrations well below 10,000ppm.

Current research focuses on the roles of clay mineralogy, reactive alumina and silica, compaction void ratio and moisture content availability on Ettringite formation. Also, the effect and role of pre-compaction mellowing period on Ettringite formation in soils with sulfate contents above 8,000 ppm will be studied. Finally, the impacts of lime and lime-fly ash treatments on swell, shrinkage behaviors of high sulfate soils will be addressed.

CHAPTER 3

EXPERIMENTAL PROGRAM

3.1 Introduction

An experimental program was designed to study the heaving mechanisms in chemically stabilized high sulfate soils. Lime is the main stabilizer for the current research due to its adaptability by several transportation agencies. Lime+ fly ash studies were also conducted on two soils to see the effect of lime + fly ash treatment on high sulfate soils. The current testing program has been subdivided into two phases. Phase I: lime treatment studies and Phase II: lime + fly ash treatment studies. The testing programs with both lime and lime + fly ash was designed to understand the reactions between calcium-based stabilizers, sulfate, and alumina contents at different sulfate levels, compaction densities and moisture contents. For the testing program in Phase I, six soils with varying sulfate concentrations from the state of Texas were chosen. Due to limited availability of high sulfate soils, one of the two soils used in Phase II testing was chosen from the Phase I soils

The following sections describe criteria for soil selection, physical and chemical properties of the control soils, testing materials, types of laboratory tests performed, test equipment used, and test procedures.

3.2 Soil Selection Criteria

Two criteria's were used in selection of tests soils. First criterion for selection of soils is the sulfate content. Tests were conducted to determine the soluble sulfates in the soils. Soluble sulfates were determined using the Modified UTA method. The results were consistent, with minimum differences. The sulfate test results are presented in Table 3.1. Based on the sulfate contents, the soils were categorized into three groups: soils with sulfate contents greater than 8000 ppm, soils with sulfate contents less than 8000 ppm and soils with negligible sulfate contents. Among the six soils, three soils showed sulfate contents above 8000 ppm (Austin, Childress and FM-1417). Two of the six soils have sulfate levels ranging from 5,200 ppm to 7000 ppm (Dallas and US82). One of the six soils is a control soil with negligible sulfate contents (Riverside). Additional sulfates in the form of gypsum will be added to these soils to make them high sulfate (> 8000 ppm).

The second criterion in the selection of test soils was based on the PI values. The determination of Liquid limit, Plastic limit and Plasticity index of the soils was carried out as per ASTM D-4318. The Atterberg limits of all six soils are shown in Table 3.2. The soils are grouped into four categories based on the PI values: Soils with PI values < 30, 30 < PI < 40, 40 < PI < 50 and PI > 50. Figure 3.1 depicts the test soils location on the Texas map. The following section describes the methodology adapted in the current study to treat high sulfate soils.

Table 3.1 Test Soil Locations and Soluble Sulfate Contents of the Selected Soils

	Soil Location	**Soluble Sulfates, ppm**
Sulfate soils	US-82 (Sherman)	5,200*
	Dallas	7,000*
	FM-1417 (Sherman)	24,000
	Austin	36,000
	Childress	44,000
Control soil	Riverside (College Station)	200*

* Sulfates < 8,000 ppm, additional sulfates were added in the form of Gypsum

Table 3.2 Atterberg Limits and Soil Classification

Soil	**Atterberg Limits**			**USCS Classification**
	LL	**PL**	**PI**	
Austin	76	25	51	CH
Childress	71	35	36	MH
Dallas	80	35	45	CH
FM-1417	72	30	42	CH
Riverside	35	11	24	CL
US-82	75	25	50	CH

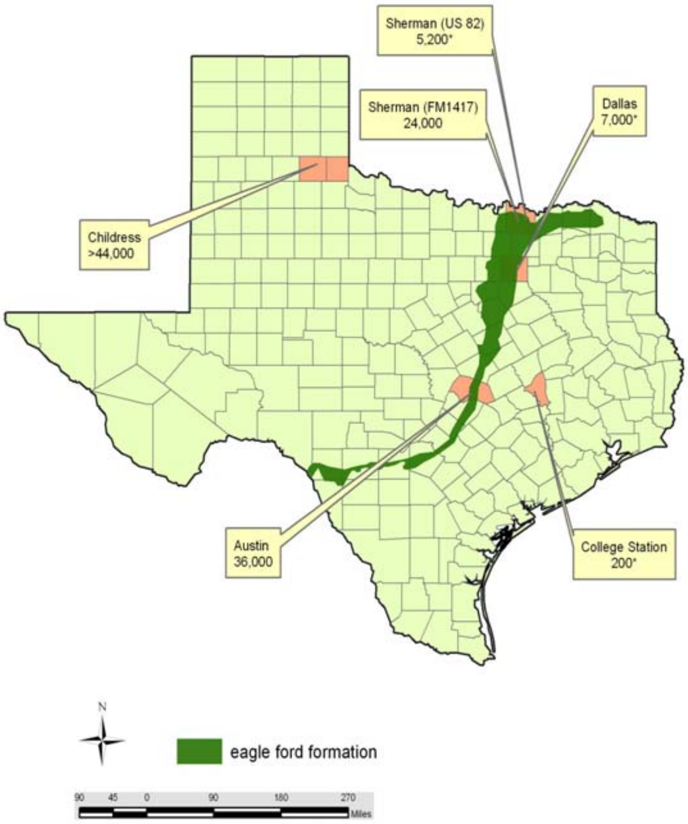

Figure 3.1 Map showing the location of test soils (Courtesy: Ahmed Gaily, 2012)

3.3 Methodology

The main intent of the current research is to study heave mechanisms in soils with sulfate contents above 8,000ppm. For this reason, soils with sulfate levels below 8,000ppm were spiked with gypsum to raise the sulfate content above 8,000ppm. The testing Program has been divided into basic tests, engineering tests and chemical and mineralogical tests. Basic tests include Atterberg limits, hydrometer, specific gravity and Proctor compaction tests. Engineering tests include 3-D volumetric swell strain (3-D swell), 3-D volumetric shrinkage strain (3-D shrinkage), 1-D pressure swell and UCS tests. Chemical and mineralogical tests include sulfate content and lime dosage and reactive alumina and silica, cation exchange capacity (CEC), specific surface area (SSA) and total potassium (TP). Cation exchange capacity, specific surface area and total potassium tests were conducted to determine the basic clay mineralogy as in the procedures outlined by Chittoori et al., (2011). The purpose of determining the clay mineralogy is that certain clay minerals release more alumina at elevated pH conditions compared to others, which is the critical component of both pozzolanic and Ettrngite formation reactions (Dermatas, 1995 and Puppala et al., 2005).

Followed the basic soil classification tests, all the soils were subjected to clay mineralogy tests to determine clay mineral distribution. Test soils were also subjected to reactive alumina and silica tests, using ICP_MS (inductively coupled plasma _ mass spectroscopy) to determine the amount of alumina and silica in the natural formation which upon chemical stabilization using lime leads to formation of both pozzolanic and deleterious compounds. Engineering tests were performed to determine the swell, shrinkage and strength characteristics which will be used as a datum for comparing with the treated soil properties.

As mentioned before, lime is used as the main stabilizer in the current research. In this study the "Pre-compaction mellowing" technique was used in stabilizing the high sulfate soils. Two mellowing periods were considered in the current study. They are 0 days and 3 days. Test soils were treated with lime and allowed to mellow in a moisture-controlled environment. Following the mellowing, the samples were mixed thoroughly and compacted. Engineering tests were conducted on the compacted soil samples. To study the effect of higher mellowing periods, an additional set of 3-D swell tests were conducted using a 7 day mellowing period. After the samples were subjected to swell tests, reactive alumina and silica measurements were conducted to determine the loss of alumina and silica during stabilizing and sulfate reactions.

3.4 Testing Variables

Testing variables include soils, sulfate contents, compaction moisture contents, mellowing periods and stabilizers. Six different soils from the state of Texas were chosen to include high plasticity clay (CH), low plasticity clay (CL) and high plasticity silt (MH), with different geological origins. Elevated sulfate contents in these soils varied from 12,000ppm to 44,000ppm. Two compaction moisture contents; namely, optimum moisture content (corresponding to maximum dry density, MDD) and wet of optimum moisture content (corresponding to 95% maximum dry density), were chosen. Also two mellowing periods, 0 and 3 days, were selected for stabilization of soils. The following section describes the test procedures in brief. Two stabilizers lime and lime + fly ash were used in the current study. Table 3.3 shows the testing variables.

Table 3.3 Testing Variables

Description	**Variable**
Soils	Six (Austin, Childress, Dallas, FM-1417, Riverside, and US-82)
Sulfate Contents	Five (12,000 ppm; 20,000 ppm; 24,000 ppm; 36,000 ppm; and 44,000 ppm)
Stabilizer	One (Lime)
Dosage	One (6%L)
Compaction Moisture Contents	Two (Optimum, OMC and Wet of optimum, WOMC moisture content)
Mellowing Periods	Three (0, 3 and 7 days)

3.5 Basic Tests

Basic tests include Atterberg limits, sieve analysis, hydrometer, specific gravity and Proctor compaction tests. Testing procedures are given below.

3.5.1 Atterberg Limits

Liquid limit (LL), Plastic limit (PL) and Plasticity index (PI) of the soils was determined per ASTM procedure D4318-10. These tests were conducted in order to determine the plasticity properties of the soils. Upon addition of water, the state of soil proceeds from dry, semisolid, plastic and finally to liquid states. The water contents at the boundaries of these states are known as shrinkage SL, plastic PL and liquid LL limits, respectively (Lambe and Whitman, 2000). Therefore, LL is calculated as the water content at which the soil flows, and PL is determined as the water content at which the soil starts crumbling when rolled into a 1/8-inch diameter thread. Figure 3.2 shows the apparatus used for Atterberg limits.

Figure 3.2 Atterberg Limits Apparatus

These Atterberg limits are very important to show a relationship between the shrink-swell potential of the soils and their relevant plasticity indices. The numerical difference between LL and PL values is known as plasticity index (PI), and this property is generally used to characterize the plasticity nature of the soil and its expansive potential. The water content of the samples during tests is measured using the microwave drying method based on the repeatable data as reported by Hagerty et al. (1990).

3.5.2 Specific Gravity

Soil consists of an accumulation of particles, which may be of a single mineral type or, more often, a mixture of a number of mineral types. This means that different soil types have different specific gravity values. In this research, the standard test as per ASTM D 854 was conducted to determine the specific gravity of the selected soils.

3.5.3 Hydrometer analysis

Hydrometer Analysis was carried out as per ASTM D422. The procedure involved taking 50 grams of the oven dried portion that passed through a No. 200 sieve and mixing with a solution containing a 4% deflocculating agent (Sodium Hexametaphosphate) and soaking it for about 8 to 12 hours. The prepared soil was thoroughly mixed in a mixer cup, and all the soil solids inside the mixing cup were transferred to a 1000 cc graduated cylinder and filled to the mark using distilled water.

The hydrometer readings were recorded at cumulative times of 0.25 min., 0.5 min., 2 min. 4 min., 8 min., 15 min., 20 min., 2 hr., 4 hr., 8 hr., 12 hr., 24 hr., 48 hr., and 72 hr. After taking the readings initially for the first 2 minutes, the hydrometer was taken out and kept in another cylinder filled with distilled water. Necessary temperature corrections, zero corrections and meniscus corrections were made to the hydrometer readings as per procedure. Table 3.4 summarizes the basic soil classification and particle size distribution.

Table 3.4 Summary of Basic Tests

Soil	**Gradation, %**				**G_s**	**Elevated Sulfate Content (after adding Gypsum)**	**USCS Classification**
	Gravel	**Sand**	**Silt**	**Clay**			
Austin	0	21	39	40	2.61	36,000	CH
Childress	0	23	69	8	2.51	44,000	MH
Dallas	0	4	32	64	2.71	12,000	CH
FM-1417	0	9	20	71	2.66	24,000	CH
Riverside	0	32	30	38	2.56	20,000	CL
US-82	0	5	35	60	2.65	12,000	CH

3.5.4 Standard Proctor Compaction Tests

In order to determine the compaction moisture content and dry unit weight relationships of the soils in the present research program, it was necessary to conduct standard Proctor compaction tests. The optimum moisture content (OMC) of the soil is the water content at which the soils are compacted to a maximum dry unit weight condition. Specimens exhibiting a high compaction unit weight are best at supporting civil infrastructure since the void spaces are minimal and settlement will be less. Compaction tests were conducted on all types of soil to determine moisture content and dry unit weight relationships. ASTM D698 procedure was followed to determine the maximum dry density (MDD) and corresponding optimum moisture content. This test requires use of a hand held rammer, which has a 50 mm diameter face with a weight of 2.5 kg, and a cylindrical mold, which has internal dimensions of 105 mm in diameter and 115.5 mm in height. This gives a volume of 1000 cm^3 (or 1/30 ft^3). Figure 3.3 shows the proctor compaction apparatus.

Figure 3.3 Standard proctor compaction test apparatus

Soil mix was compacted in three layers by applying 25 blows per layer of rammer dropping from the controlled height of 300 mm. After compacting, the compacted soil was weighed, and the moisture content was measured. The procedure was repeated after each increment of water was added to the soil mix. Test results were commonly presented in the form of a compaction curve, which depicts a relationship between dry unit weight and water content. Water contents at 95% of the maximum dry density conditions representing wet of OMC were selected for testing. Figure 3.4 depicts the typical Proctor curve for FM-1417 soil. Proctor test results on natural, lime treated and lime +fly ash-treated soils are presented in Table 3.5, Table 3.6 and Table 3.7 respectively. All the natural and treated Proctor curves are presented in Appendix.

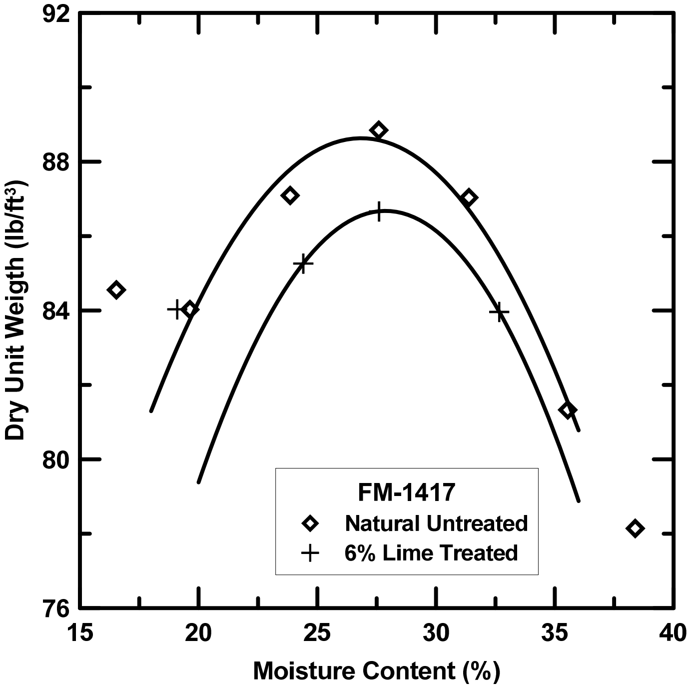

Figure 3.4 Standard proctor Compaction Curve for FM-1417 Soil

Table 3.5 Summary of Proctor Tests on Natural Soils

Soil Type	Natural (Untreated) Soil			
	Moisture Content (%)		Dry Density (lb/ft³)	
	OMC	WOMC	OMC	WOMC
Austin	18	23	106	100
Childress	21	25	103	98
Dallas	28	33	92	87
FM-1417	27	34	89	84
Riverside	20	25	99	94
US-82	24	32	91	86

Table 3.6 Summary of Proctor Tests on Lime Treated Soils

Soil Type	**6% Lime Treated Soils**			
	Moisture Content (%)		**Dry Density (lb/ft³)**	
	OMC	**WOMC**	**OMC**	**WOMC**
Austin	21	29	95	90
Childress	22	28	96	91
Dallas	29	39	87	83
FM-1417	28	34	87	83
Riverside	21	25	98	93
US-82	26	32	89	85

Table 3.7 Summary of Proctor Tests on Lime + Flyash Treated Soils

Soil Type	**Natural**		**Lime (4%) + Flyash (8%)**	
	Moisture Content (%)	**Dry Density (lb/ft³)**	**Moisture Content (%)**	**Dry Density (lb/ft³)**
FM-1417	27	89	21	90
Bells	23	96	20	91

3.6 Engineering Tests

Engineering tests include 3-D swell, 3-D shrinkage, 1-D swell pressure and UCS tests. Following section describes the procedures followed.

3.6.1 Three Dimensional Swell Test (3-D swell)

Free swell test commonly refers to the conventional one-dimensional free swell test, which was conducted on soil specimens that were 2.5 in. diameter with 1 in. thickness. This test is only used to measure the maximum amount of heave in the vertical direction of a confined soil specimen. To examine the maximum vertical, radial, and volumetric swell potential, three-dimensional free swell test was conducted in this research. Three dimensional volumetric swell tests were conducted using the double inundation technique to determine the maximum possible radial and vertical swell. Double inundation represents the worst possible scenario in a field where 100% saturation of the soil is achieved after a continuous rainfall event. Maximum expansive heave is observed in this case. The double inundation technique for measuring the volumetric swell was used successfully in the past by various researchers across the United States and the UK. It was observed in the current study that 3-D swell tests, using the capillary rise method, yielded lower values of swell, indicating the underestimation of swell; whereas, the swell determined using "double inundation" technique was on the higher side, representing the actual field swell.

The following paragraphs describe the soil sample preparation method and testing procedures used. Oven-dried soils were pulverized and mixed at targeted moisture content levels. Both control and treated soil specimens were mixed and then compacted by using Gyratory Compactor Machine at two moisture content levels. Figure 3.5 illustrates the Gyratory Compacting Machine and sample after extraction.

Figure 3.5 Gyratory Compacter Machine, (b) Soil sample after extraction

Samples which were 4 in. (101.6 mm) in diameter and 4.6 in (116.8 mm) in height were covered by a rubber membrane. Porous stones were placed at the top and bottom of the specimens, which facilitated the movement of water to the soil specimen. The specimen was fully soaked under water in a large container. It is common practice to perform swell tests on chemically-treated sulfate soils under moisture inundation from both ends of the soil specimen. Sulfate soils need the presence of moisture content that will facilitate reactions for Ettringite crystal formation and its hydration. Hence, swell tests here were performed under full soaking conditions. The amount of soil heave in both vertical and diametrical directions was monitored until there was no significant swell for 24 hours. Radial measurements were then taken at the top, middle, and bottom circumferences of the soil samples and averaged at a frequency similar to the Consolidation Test. Figure 3.6 shows the 3-D swell test setup. The percent values were calculated based on the original dimensions of the soil specimen. Table 3.8 gives the vertical, radial and volumetric swell strains of natural soils. Figures 3.7 thru 3.12 show the vertical swell strain vs. elapsed time for untreated soils considered in the current study.

Figure 3.6 Three Dimensional Swell Test Setup

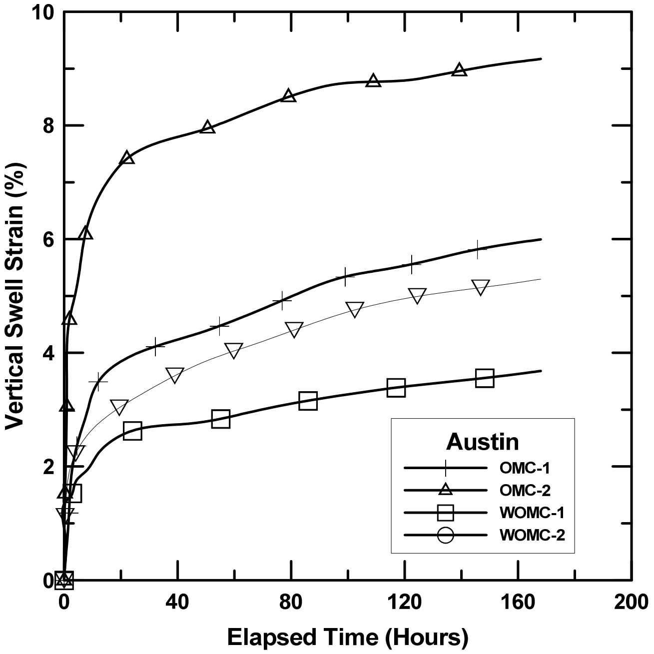

Figure 3.7 Vertical Swell Strain vs. Elapsed Time (Austin Soil)

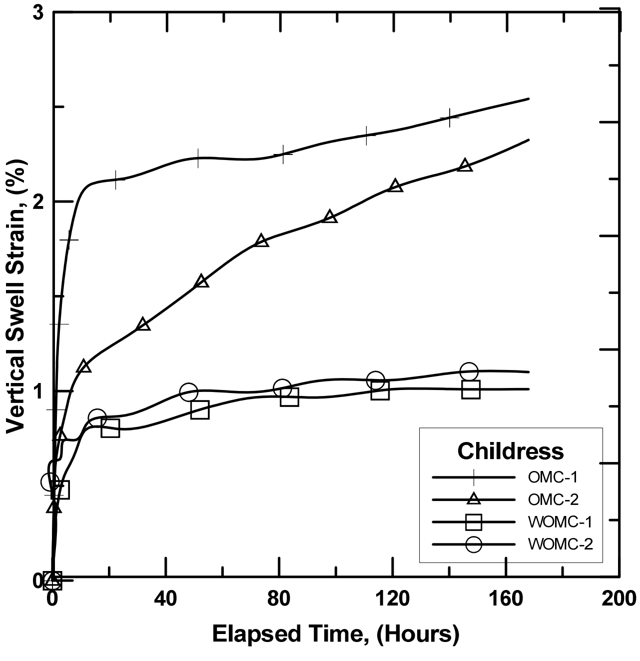

Figure 3.8 Vertical Swell Strain vs. Elapsed Time (Childress Soil)

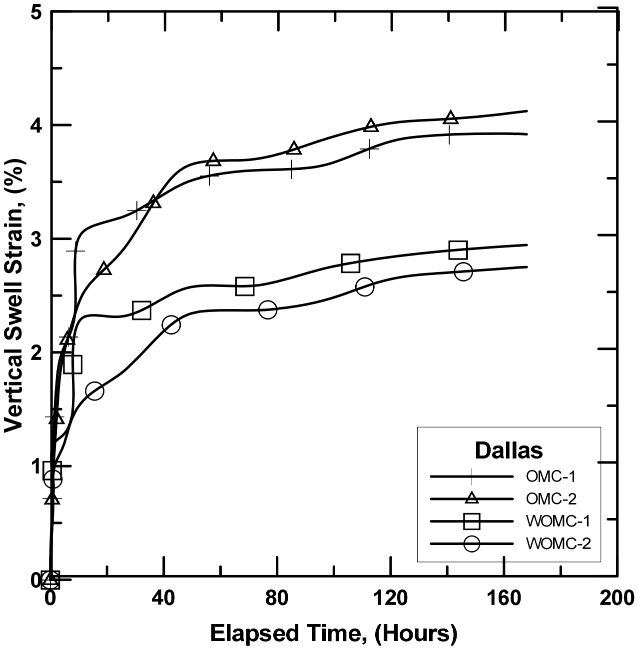

Figure 3.9 Vertical Swell Strain vs. Elapsed Time (Dallas Soil)

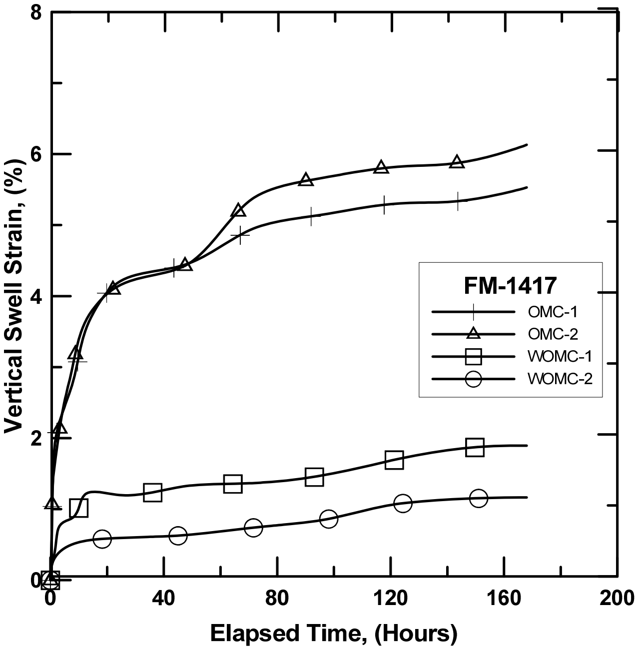

Figure 3.10 Vertical Swell Strain vs. Elapsed Time (FM-1417 Soil)

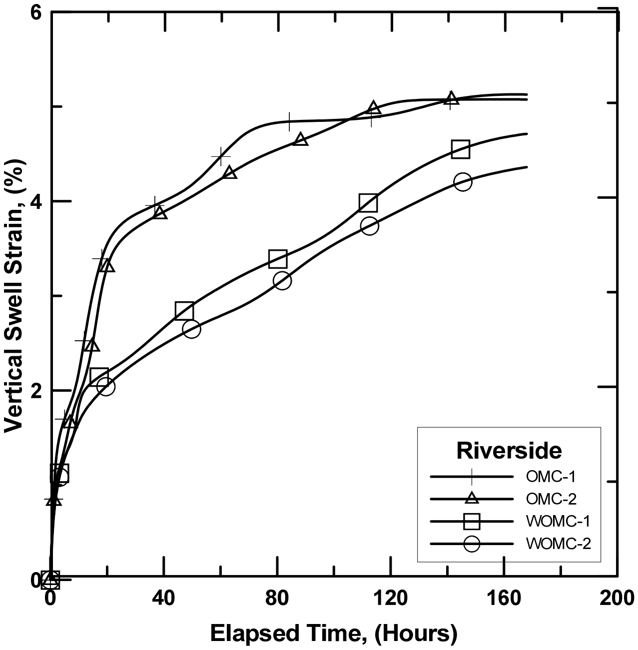

Figure 3.11 Vertical Swell Strain vs. Elapsed Time (Riverside Soil)

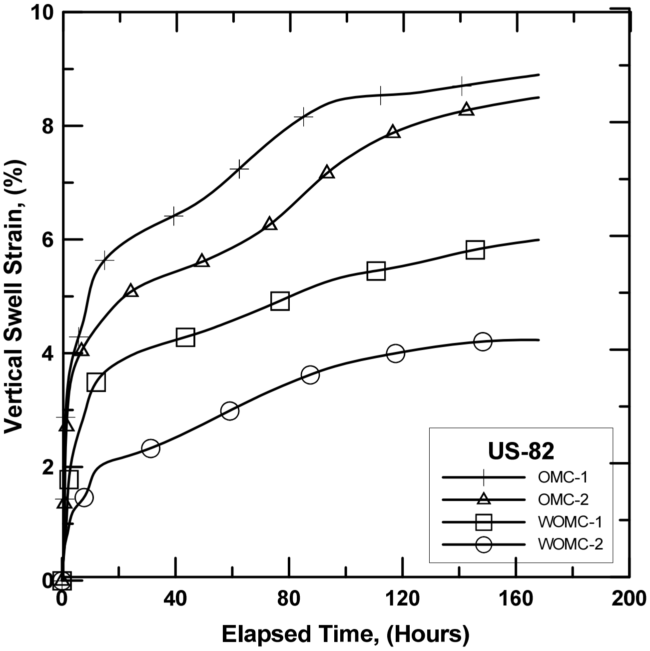

Figure 3.12 Vertical Swell Strain vs. Elapsed Time (US-82 Soil)

Table 3.8 Summary of 3-D Swell Tests on Natural Soils

Soil Type	**Vertical Strain (%)**		**Radial Strain (%)**		**Volumetric Swell Strain (%)**	
	OMC	**WOMC**	**OMC**	**WOMC**	**OMC**	**WOMC**
Austin	7.6	3.6	4.5	2.5	16.6	8.7
Childress	3.9	0.9	1.8	1.4	7.5	3.7
Dallas	3.8	2.6	3.7	2.6	11.0	7.8
FM-1417	6.0	6.1	5.1	1.3	16.2	8.9
Riverside	4.2	4	2.9	3.1	10.0	10.2
US-82	7.2	4.9	5.5	1.5	18.1	7.9

3.6.2 Three Dimensional Shrinkage Test (3-D shrinkage)

Due to limitations in the linear shrinkage bar test, researchers at University of Texas at Arlington (UTA) proposed a new test method using cylindrical compacted soil specimens and subjecting them to a drying process and then measuring the volumetric, axial and radial shrinkage strains. This test offers several advantages over the conventional linear shrinkage bar test such as reduced interference of boundary conditions on shrinkage, larger amount of soil being tested, and simulation of compaction states of moisture content - dry density conditions. The 3-D shrinkage test was conducted to measure the shrinkage characteristics of the expansive natural soils followed by a long dry spell. This method was published in the ASTM Geotechnical Testing Journal (Puppala et al., 2004), which signifies the importance of this method being accepted by the researchers and practitioners.

Volumetric shrinkage tests were conducted to measure the decrease in the total volume of soil specimens due to loss of moisture content from predetermined initial moisture content to a completely dry state. Another reason for conducting these tests was to study shrinkage strain potentials of treated soils with Ettringite formed. Two different initial moisture contents (optimum, wet of optimum) were used as initial compaction conditions, and tests were conducted as per the procedure outline in Puppala et al. (2004). Specimen preparations were performed by mixing the dry clay with the appropriate amount of water to achieve the designed water contents, then compacting the soil specimens in 2.26 in. (57 mm) diameter and 5 in. (127 mm) height mold, and measuring the initial height and diameter of the specimen. The specimens were then extracted and left at room temperature for 12 hours before being transferred to an oven set at a temperature of 220° F for 24 hours. Upon removal from the oven, the average height and radial dimensions of the dried soil specimens were manually measured. The volumetric shrinkage strain was calculated from these measurements. A summary of 3-D shrinkage tests on natural soils is presented in Table 3.9. The 3-D shrinkage setup is shown in Figure 3.13. 3-D shrinkage results of natural soils are compared with treated soils.

Table 3.9 Summary of 3-D Shrinkage Tests on Natural Soils

Soil	**Average Volumetric Shrinkage Strain (%)**	
	OMC	**WOMC**
Austin	-8.8	-11.3
Childress	-14.1	-15.7
Dallas	-19.7	-26.2
FM-1417	-15.6	-20.6
Riverside	-12.5	-14.0
US-82	-13.7	-20.2

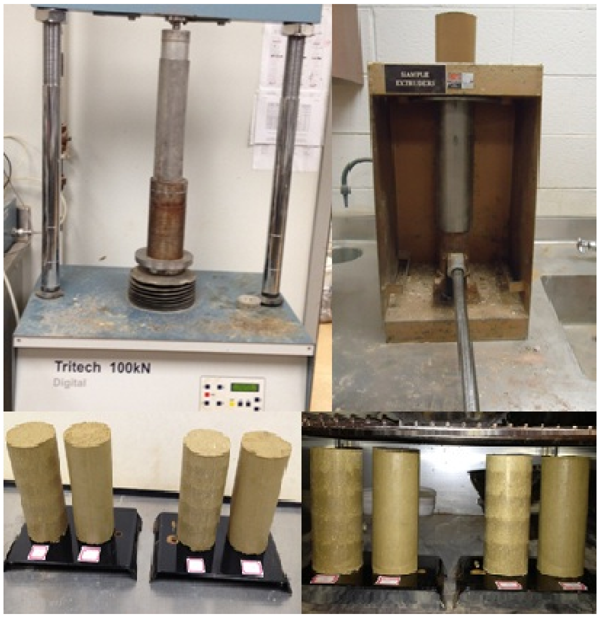

Figure 3.13 3-D Shrinkage Test

3.6.3 One Dimensional Swell Pressure Test (1-D swell pressure)

The pressure swell test was conducted as per ASTM D-4546 specification (Standard Test Methods for One-Dimensional Swell or Collapse of Cohesive Soils). The swell pressure test measures the amount of overburden pressure necessary for preventing expansion behavior of soils. The objective of this test was to determine the maximum swelling pressure of the soil samples at which no volume change of samples was allowed to anticipate. The preparation procedure and test set up are the same as mentioned before in the one dimensional swell test section. Same steps were followed to initiate soil heaving process. However, vertical loads were added to a hanger at regular hourly intervals in order to reduce the elapsed vertical swell strains to zero magnitudes. Test was continued until the sample did not exhibit any heaving movements for at least 24 hours. The final load at the end of the test was determined and used in the calculation of swell pressures. 1-D swell pressure test set up is shown in Figure 3.14. A summary of swell pressure test results is presented in Table 3.10.

Figure 3.14 1-D Swell Pressure Test Setup

Table 3.10 Summary of 1-D Swell Pressure Tests on Natural Soils

Soil	**Average Swell Pressure (psi)**	
	OMC	**WOMC**
Austin	46.2	22.4
Childress	6.7	2.2
Dallas	40.7	15.1
FM-1417	37.1	16.2
Riverside	8.3	1.8
US-82	48.9	40.1

3.6.4 Unconfined Compressive Strength Tests (UCS Test)

The Unconfined Compressive Strength (UCS) tests were conducted as per ASTM D 2166 method. This test was conducted on the soil samples under unconfined conditions. The test was conducted on compacted soil specimens of 2.8 inches in diameter and 5.6 inches in height. The soil specimen was first placed on a platform and then raised at a constant strain rate, using the controls of the UCS set up until it came in contact with top plate. Figure 3.5 shows the unconfined compressive strength test set up and the computer system used for data acquisition. Once the specimen was intact, it was loaded at a constant strain rate and, as the load approached the ultimate load, failure cracks began to appear on the surface of the specimen. Both deformation and corresponding axial loads on the specimen were recorded using a Data Acquisition System (DAS). The data retrieved contained load (Q) and deformation (δ) data, and the same were analyzed to determine the maximum unconfined compressive strength (q_u) in psi or kPa. The following expressions show the computation of stress (σ) and strain (ε) corresponding to the load-deformation data.

$$\varepsilon = \frac{\delta}{L}\ ;\ \sigma = \frac{Q}{A_c}\ ;\ A_c = \frac{A}{1-\varepsilon}\ \text{and}\ q_u = \sigma_{max}$$

Where, δ = change in length, L = total length of specimen, A_c = corrected area of cross section of the specimen and A = initial area of cross section. A typical Stress-Strain curve obtained from UCS test is presented in Figure 3.15. Figure 3.16 shows the UCS test setup. Summary of UCS tests on natural soils is presented in Table 3.11.

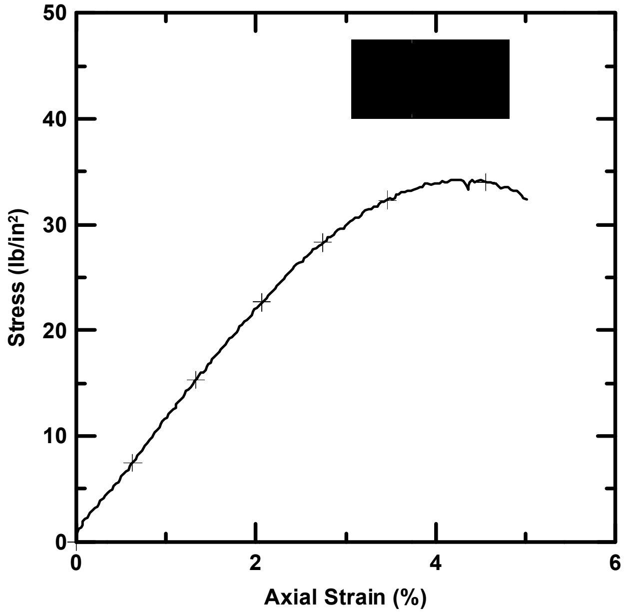

Figure 3.15 Stress vs. Axial Strain, US-82, OMC, Natural

Figure 3.16 UCS Test Setup

Table 3.11 Summary of UCS Test Results

Soil	**Average UCS (psi)**	
	OMC	**WOMC**
Austin	28	21
Childress	22.8	15.9
Dallas	16	9.9
FM-1417	32.3	18.5
Riverside	29.5	17.2
US-82	30	18.2

3.7 Chemical and Mineralogical Tests

Chemical and mineralogical tests include soluble sulfate determination, lime dosage, reactive alumina and silica, cation exchange capacity, specific surface area and total potassium tests. Following section describes the procedures followed.

3.7.1 Soluble Sulfate Determination (Modified UTA Method)

The basis of the current method is the standard Gravimetric Method outlined in the seventeenth edition of Standard Methods for the Examination of Water and Wastewater by Clesceri, Greenberg and Trussell (1989). Puppala et al. (2002) modified the existing UTA method proposed by Petry (1994). A soil water dilution ratio of 1:10 is used in this method. Theoretically, 1:10 dilution ratio can extract sulfate concentrations up to 26000 ppm. In this method, 10 grams of soil is mixed with 100 mL of distilled water and left overnight. This allows the dissolution of water soluble sulfates into the solution. The sample is then shaken for 30 minutes on an Eberbach shaker to disintegrate the sulfate salts. Soluble sulfates are extracted from the solution by centrifuging the sample at 14000 rpm using the IEC-HT centrifuge machine. A higher rate of centrifuging allows the efficient extraction of soluble sulfates. The supernatant is then filtered through 0.1μm filter paper, and the filtrate is diluted to 200mL. Use of the 0.1μm filter paper stops the migration of suspended particles in the soil and de-ionized water solution. This is an improvement over the previous UTA method. The pH of the dilutant is adjusted between 5 and 7 by adding diluted hydrochloric acid (1:9) HCl to facilitate the further dissolution of sulfates. The solution is then boiled until the bubbles appear. Warm $BaCl_2$ (10%) solution is added slowly until the precipitation process is completed.

The precipitate is then digested in an oven at 80-90^{0}C for 12 hours. The precipitate is filtered through a 0.1μm membrane filter. While filtering, the precipitate is washed with distilled water to make it free from chlorides. Chlorides can be checked by a silver nitrate-nitric acid reagent. The difference between the weights of dry filter paper and filter paper with barium sulfate precipitate gives the sulfate content in the soil. Weight of the barium sulfate is multiplied with a factor 41,156, representing the portion of sulfate in barium sulfate to obtain the concentration in ppm. It is also suggested to use a finer filter paper and longer centrifuging time in the case of high montmorillonite soils since most of the soil particles sizes are smaller than 0.1 μm. In a recent study conducted at The University of Texas at Arlington, two soils with different classifications and origins were spiked with different sulfate concentrations and tested using the Modified UTA method, AASHTO method and TxDOT method. Results indicated that the Modified UTA method gave repeatable and accurate values compared to the other methods (Talluri et. al., 2012). A pictorial representation of the filtration setup used and final barium sulfate precipitate on filter paper is given in Figure 3.17. Figure 3.18 shows the step-by-step procedure followed in soluble sulfate determination by the Modified UTA method.

(a) Filtration Setup (b) Barium Sulfate Precipitate

Figure 3.17 Modified UTA Method for Determination of Soluble Sulfate Content

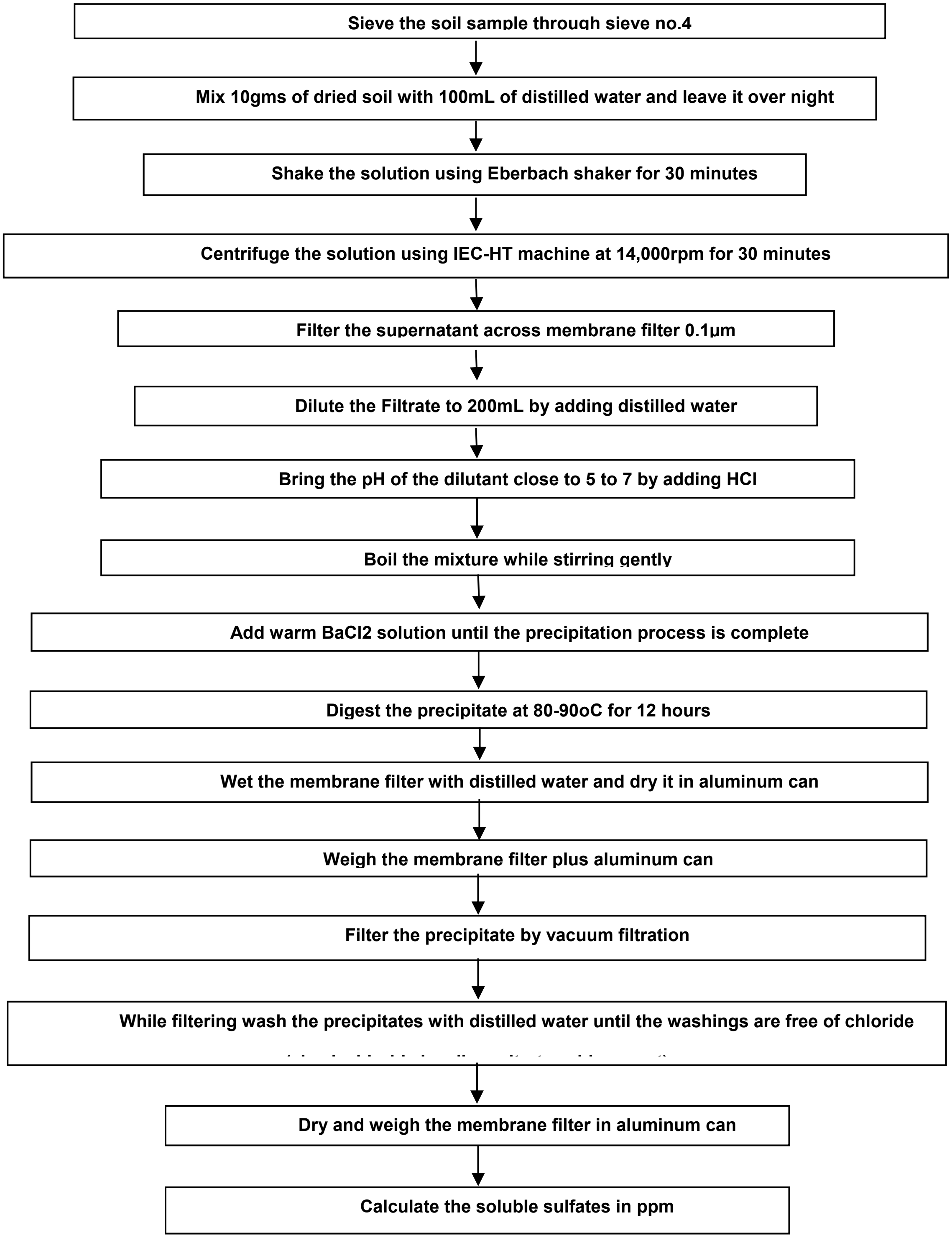

Figure 3.18 Soluble Sulfate Determination (Modified UTA method)

3.7.2 Lime Dosage Determination

Lime content is determined as per the "Eades-Grim" test (Eades and Grim, 1966), and is fully described in ASTM D 6276 procedures. The basic objective of this method is to add sufficient lime to the soil to ensure a pH of 12.4 for sustaining the strength-producing lime-soil pozzolanic reaction. The lowest percentage of lime in soil that produces a laboratory pH of 12.4 is the minimum percentage for stabilizing the soil. The procedure is described below.

Weigh to the nearest 0.01gm a series of 30 gm soil samples and place them in separate containers. Add a series of lime equivalent to 0, 2, 4, 6, 8 and 10% of dry weight of soil. Add one of the lime percentages to each of the containers, followed by the addition of 150ml of water. Stir the samples every 15 minutes for about an hour. At the end of hour, record the temperature of the mixture and adjust the pH meter to that temperature. Standardize the pH meter with distilled water to 7.0. Clean the electrodes with distilled water. Check the pH of the soil-lime solutions and record. Plot the pH on y-axis and lime dosage on x-axis. From the curve, read a pH value of 12.4 and record the corresponding lime dosage. If the pH readings are 12.4 or higher, take the lowest percentage of lime that gives a pH of 12.4. If the pH readings do not go beyond 12.3 or higher for two different lime dosages, further testing is required to establish the lime dosage. The minimum strength criterion for lime content is based on an unconfined compressive strength of 150 psi for bases and 50 psi for soils. As mentioned before, the lime dosage for all the test soils in the current study is 6% (by dry weight of soil). A graphical representation of the test is given in Figure 3.19. Table 3.12 shows the results of the pH testing.

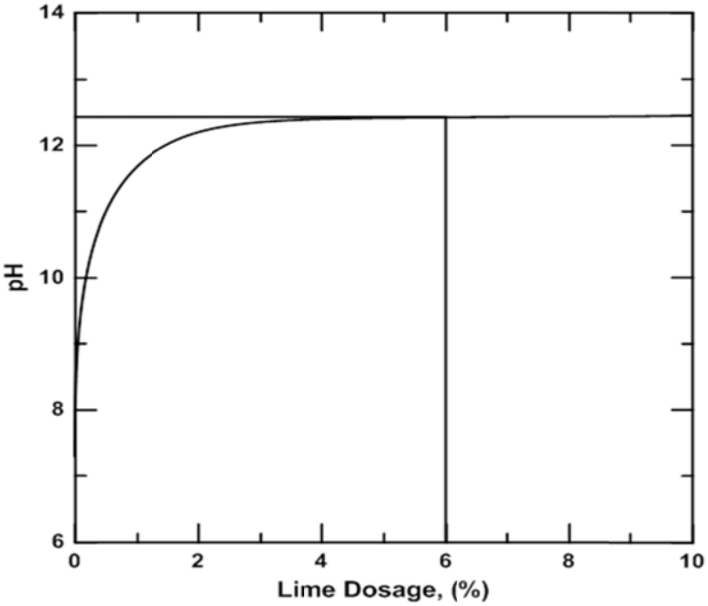

Figure 3.19 Lime Dosage Determination for Austin Soil

Table 3.12 Lime Dosage Determination (Eades and Grim Method)

Soil	Lime Dosage (% by dry weight)
Austin	6
Childress	6
Dallas	6
FM-1417	6
Riverside	6
US-82	6

3.7.3 Cation Exchange Capacity (CEC)

Cation exchange capacity or CEC can be used to determine the mineral composition of a given soil. For example, a soil with a high CEC value of 100 meq/100gm to 120 meq/100gm indicates a high amount of expansiveness due to the presence of the clay mineral Montmorillonite' whereas, a low CEC indicates the presence of non-expansive clay minerals such as Kaolinite. CEC of a soil can be defined as the capacity or the ability of the soil to exchange free cations that are available in the exchange locations.

One of the earliest methods proposed by Chapman (1965) is the most commonly used method in the field and the method selected for the current research. The method involves the addition of a saturating solution and then removal of the adsorbed cations, using an extracting solution. The saturating solution used here is ammonium acetate (NH_4OAc) at pH 7. This solution was added to a prepared soil specimen (preparation involves treating for organics with 30% hydrogen peroxide (H_2O_2) and set aside for 16 hours after shaking for half hour, to ensure that all the exchange locations are occupied by the ammonium ion (NH^{4+}). Then the solution was filtered through a Buchner funnel and washed with 4 different 25 mL additions of NH_4OAc. This step is to bring out all the cations from the soil sample solution that has been replaced by ammonium ions. Excess NH_4OAc was removed by the addition of 8 different 10 mL additions of 2-propanol. Now, all the cation places are replaced by the ammonium ion and excess ammonium is also removed. The CEC of the soil sample can be obtained by measuring the amount of ammonium ions that replaced all the exchange locations. This was done by washing the sample with 8 different 25 mL additions of 1M potassium chloride (KCl) solution. The concentration of NH^{4+} ion in the KCl extract gives the CEC of the soil. Photographic representation of the different steps involved is presented in Figure 3.20.

Soil sample saturated with NH_4OAc

25 mL 1 M NH_4OAc additions

25 mL Ethanol additions

25 mL 1 M KCl additions

Spectrophotometer

Colorimetric determination of ammonia concentration

Figure 3.20 Photographs of the various steps involved in the determination of CEC

3.7.4 Specific Surface Area (SSA)

Specific surface area or SSA of a soil sample is the total surface area contained in a unit mass of soil. This property of the soil is primarily dependent on the particle size of the soil. Soils with smaller particle sizes have higher specific surface areas. It should be noted here that a soil with a high specific surface area has a high water holding capacity and greater swell potential.

The most commonly used method in the field of agronomy is adsorption by Ethylene Glycol Monoethyl Ether (EGME) (Carter et al., 1986) and is implemented in this research. This involves saturating prepared soil specimens, equilibrating them in vacuum over a calcium chloride – EGME ($CaCl_2$-EGME) solvate, and weighing them to find the point when equilibrium is reached. Specific surface is then determined from the mass of retained EGME in comparison to the amount retained by pure montmorillonite clay, which is assumed to have a surface area of 810 m2/g (Carter et al., 1986). Test procedures typically take two days to complete. They also indicated that the procedure is repeatable and gives reliable results. Photographic representation of the different steps involved is presented in Figure 3.21.

Dried soil sample

Addition of 3 mL of EGME

Desiccator containing $CaCl_2$

Desiccator containing $CaCl_2$-EGME Solvate over $CaCl_2$

EGME mixed soil sample placed in the desiccator placed

Apply vacuum to the desiccator

Figure 3.21 Photographs of the various steps involved in the determination of SSA

3.7.5 Total Potassium (TP)

Potassium is the inter-layer cation in the clay mineral Illite (Mitchell and Soga, 2005). Hence, measuring the amount of potassium ion in the soil gives a direct indication of the presence of the mineral Illite. The test procedure, formulated by Knudsen et al. (1982), was followed to obtain the amount of total potassium present in the soil. The method involves a double acid digestion technique developed by Jackson (1958), which uses two acids (Hydrofluoric acid and Perchloric acid) to break own the mineral structure of the soil and extract the potassium ions from the structure. Once the potassium is extracted, its concentration in the solution can be obtained with the help of a spectrophotometer or any other suitable device.

The test started by taking 0.1gm of soil in a Teflon digestion vessel. The original method recommended the use of platinum vessels as the hydrofluoric acid used has the ability to dissolve silica and glass is 90% silica. However the usage of platinum vessel was not possible due to cost constraints; hence, other possible alternatives were looked at and a Teflon vessel was found to have resistance to the acids that are being used in the current test procedure (Hydrofluoric acid, Perchloric acid and Hydrochloric acid) and high temperature tolerance (200ºC). The Teflon vessel was therefore finally selected.

An amount of 5 ml of Hydrofluoric acid and 0.5mL of Perchloric acid were added to 0.1gm of the soil sample. Hydrofluoric acid dissolves the silicate mineral structure and releases the interlayer cations; Perchloric acid was used as an oxidizing agent to oxidize the organic matter in the soil sample. Then the vessel was placed on a hot plate and heated to 200ºC and then cooled, and another addition of HF and $HClO_4$ was made and reheated on the hot plate. The sample was then heated until it was dry. The process was repeated to make sure all the interlayer cations were released, and then, finally, 6N HCl was added and the amount of potassium in this solution was obtained by using a spectrophotometer. Photographic representation of the different steps involved is presented in Figure 3.22.

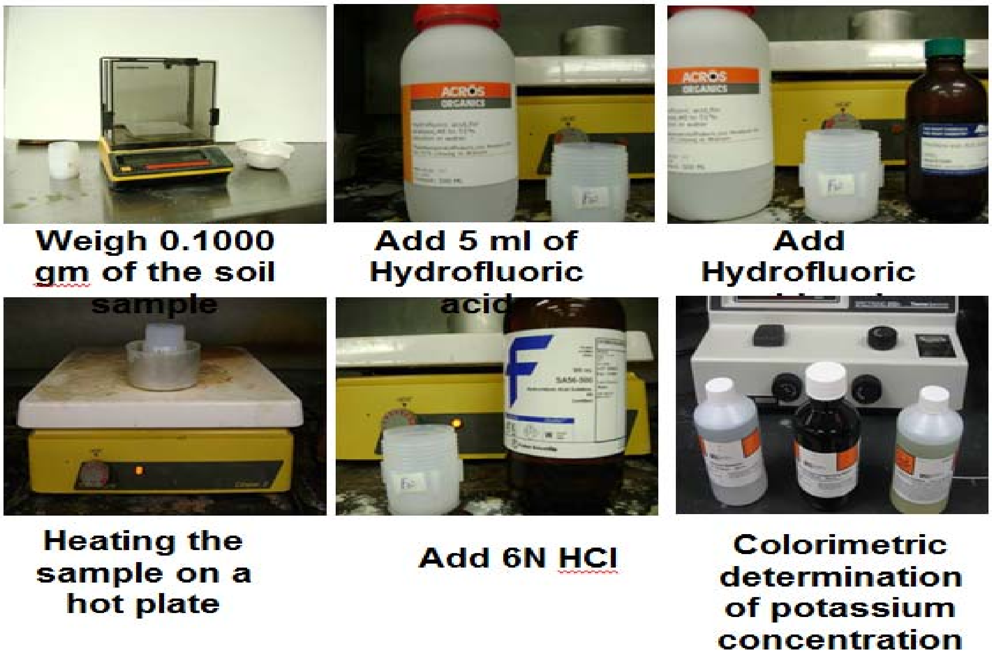

Figure 3.22 Photographs of the various steps involved in the determination of TP

Based on the cation exchange capacity, specific surface area and total potassium measurements the percent clay mineral distribution of the soils is obtained. Summary of mineralogical tests is given in Table 3.13. The clay mineral distribution of test soils is presented in Table 3.14.

Table 3.13 Summary of Mineralogical Tests

Soil	**Cationic Exchange Capacity (meq/100g)**	**Specific Surface Area (m^2/g)**	**Total Potassium (%)**
Austin	78	242	0.8
Childress	28	140	1.1
Dallas	55	236	0.9
FM-1417	65	280	0.8
Riverside	35	173	1.3
US-82	66	284	0.8

Table 3.14 Percent Clay Minerals Observed for Soils under Study

Soil Region	**% Illite**	**% Kaolinite**	**% Montmorillonite**
Austin	14	49	37
Childress	18	66	16
Dallas	15	35	50
FM-1417	13	20	67
Riverside	21	59	20
US-82	14	39	47

3.7.6 Reactive Alumina and Silica

Reactive alumina and silica are the aluminum and silica present in amorphous or poorly crystalline Al/Si phases, including amorphous alumino silicate, organically complex alumina and hydroxyl-Al polymers present in montmorillonite interlayers. These measurements were important since alumina and silica constitute the compositions of Ettringite and Thaumasite respectively. Reactive alumina and silica measurements were conducted using Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) analysis on natural and treated soils at different mellowing periods. These measurements were conducted by a procedure modified after Foster (1953). To determine the reactive alumina and silica, 15gm of soil was mixed with 150mL of 0.5 N NaOH and boiled. Once boiling was over, the solution was centrifuged at 8000rpm and filtered using a 0.1μm membrane type filter paper. The extract obtained was stored in a plastic bottle, and the ICP analysis was performed on the clear extract.

ICP analysis requires a clear solution. If the resultant extract is dark colored, it could be due to organics or iron oxides in the soils. Organics can be removed by treating the solution with hydrogen peroxide (H_2O_2). It was reported that iron oxides (Fe_2O_3) coat the clay surface, which prevent clay from releasing alumina to react with lime to form pozzolanic compounds (Joffe, 1949). Iron oxides can be removed by treating 10 ml of the solution with 1mL of 6N HCl and agitating every hour. The solution is left overnight and filtered using 0.1μm membrane-type filter paper the next morning. Once a clear extract was obtained, ICP analysis was performed on the natural and treated soil samples at different dilution ratios. Table 3.15 gives the reactive alumina and silica of natural soils. Figure 3.23 shows the flow chart for reactive alumina and silica measurements. Figure 14 shows the ICP_MS system used in the current study. Figure 16 shows the colored extract due to presence of organics and iron oxides.

Table 3.15 Reactive Alumina and Silica Measurements

Soil	Reactive Al (ppm)	Reactive Si (ppm)
Austin	58.9	15.4
Childress	75.8	12.6
Dallas	289.9	231.2
FM-1417	279.2	137.3
Riverside	297	379.8
US-82	323.3	187.1

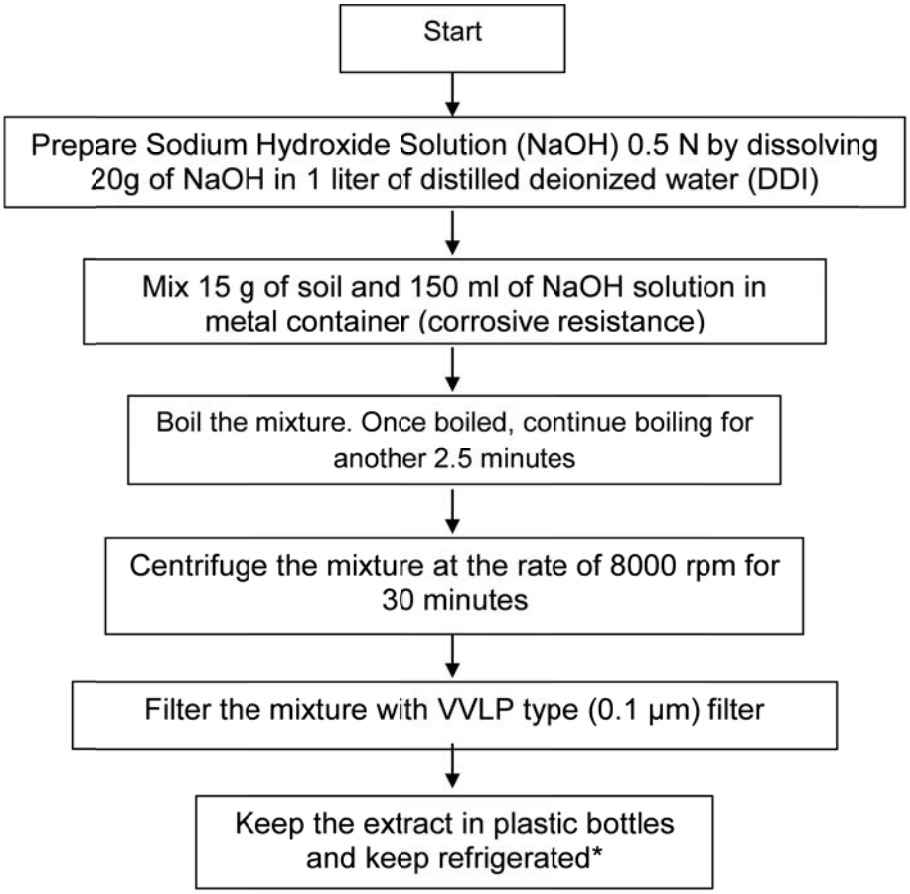

Figure 3.23 Reactive Alumina and Silica Measurements
(*If the extract is dark colored further treatment is required)

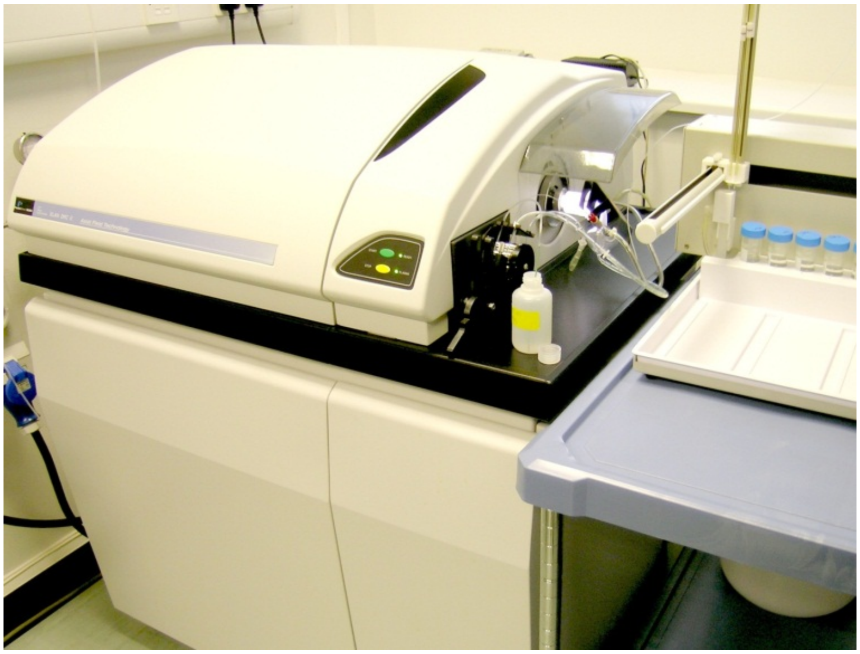

Figure 3.24 ICP_MS Set up

Figure 3.25 Dark Colored Extract Due to Presence of Organics and Iron Oxides

3.8 Summary

This chapter provides a summary of initial properties of natural soils, the experimental program, test procedures and equipment used in this research study. Testing variables and methodology followed were explained. Typical particle size distribution, engineering properties and chemical and mineralogical details of the soils were presented.

CHAPTER 4

ANALYSIS OF LABORATORY TEST RESULTS (PHASE I)

4.1 Introduction

In this chapter, physical, engineering and mineralogical test results obtained from the entire laboratory testing program are provided. The effects of chemical stabilizer, mellowing period, soluble sulfate content, soil clay mineralogy, reactive alumina and silica content and compaction moisture contents on the swell and shrinkage characteristics of treated high sulfate soils are explained and discussed. The discussion findings are based on the majority of the trends noticed in test results of both untreated (control) soils and treated soils at different mellowing periods. Results of the testing program are discussed in the subsequent sections.

4.1.1 Volume Change Characteristics

Three-dimensional volumetric swell and shrinkage tests were conducted on untreated soils and were used as a reference for comparing with volumetric strain changes in treated high sulfate soils at different periods of mellowing; i.e., the onset of sulfate and stabilization reactions. The following sections describe the results of the testing program. In the current research study, for each variable condition (moisture content and mellowing period) two set of tests were conducted. It was observed that the results were repeatable with minimum standard deviation. An average of the two readings is presented in the following test results.

4.1.1.1 Three Dimensional Volumetric Swell Test (3-D swell)

The 3-D free swell test measures the potential of the soil to swell in three (3) directions when soaked under water. The three (3) values measured were vertical, radial, and volumetric strains. Vertical swell was measured using a dial gauge placed at the top and radial measurements were taken using pi tape at the end of test. Two identical specimens are used for each variable condition (OMC and WOMC). Table 4.1, 4.2 and 4.3 show the average vertical, radial and volumetric swell strains of the lime treated soils at 0, 3 and 7 day mellowing periods. Volumetric swell strains for lime treated soils at 0, 3 and 7 day mellowing periods is shown in Table 4.4, 4.5 and 4.6 respectively. Table 4.7 shows the summary of the volumetric swell tests. Vertical swell vs. elapsed for natural and treated soil samples at different mellowing periods are presented in Figure 4.1 thru 4.6 at OMC condition. Volumetric swell strains of natural and treated soils at different mellowing periods are shown in Figures 4.7 thru 4.12.

Table 4.1 Vertical, Radial and Volumetric Swell Strains of 6% Lime Treated Soils @ 0 day mellowing

Soil Type	**Avg. Vertical Strain (%)**		**Avg. Radial Strain (%)**		**Avg. Volumetric Swell Strain (%)**	
	OMC	**WOMC**	**OMC**	**WOMC**	**OMC**	**WOMC**
Austin	6.4	5.2	1.2	1.15	8.8	7.5
Childress	5.3	5.0	4.6	4.05	14.6	13.1
Dallas	10.3	8.9	7.1	7.4	24.4	23.7
FM-1417	8.5	5.7	6.8	5.2	22.0	16.1
Riverside	5.6	4.6	5.2	4.0	16.0	12.7
US-82	9.2	8.9	8.5	7.8	26.1	24.4

Table 4.2 Vertical, Radial and Volumetric Swell Strains of 6% Lime Treated Soils @ 3 day mellowing

Soil Type	**Avg. Vertical Strain (%)**		**Avg. Radial Strain (%)**		**Avg. Volumetric Swell Strain (%)**	
	OMC	**WOMC**	**OMC**	**WOMC**	**OMC**	**WOMC**
Austin	5.3	4.2	3.2	2.8	11.6	9.8
Childress	6.5	5.2	1.8	1.6	10.2	8.5
Dallas	5.0	4.0	2.7	1.2	10.4	6.4
FM-1417	4.7	2.9	2.8	2.0	10.2	7.0
Riverside	3.9	3.7	2.55	2.35	9.0	8.4
US-82	3.6	3.7	3.8	1.5	11.3	6.70

Table 4.3 Vertical, Radial and Volumetric Swell Strains of 6% Lime Treated Soils @ 7 day mellowing

Soil Type	Avg. Vertical Strain (%)		Avg. Radial Strain (%)		Avg. Volumetric Swell Strain (%)	
	OMC	WOMC	OMC	WOMC	OMC	WOMC
Austin	7.6	4.5	5.3	3.8	18.2	12.1
Childress	5.2	4.3	3.0	2.8	11.2	9.9
Dallas	4.0	3.1	2.5	1.9	9.0	6.9
FM-1417	3.7	2.6	1.5	1.4	6.7	5.4
Riverside	3.6	2.3	1.8	1.5	7.2	5.3
US-82	4.1	3.1	2.85	1.6	9.8	6.3

Table 4.4 Volumetric Swell of Treated Soils @ 0 day mellowing

Soil Type	Sample 1		Sample 2		Avg. Volumetric Swell Strain (%)	
	Volumetric Strain (%)		Volumetric Strain (%)			
	OMC	WOMC	OMC	WOMC	OMC	WOMC
Austin	9.0	N/A	8.6	N/A	8.8	7.5
Childress	13.8	13.9	15.4	14.3	14.6	13.1
Dallas	23.6	22.8	25.2	24.6	24.4	23.7
FM-1417	22.4	16.0	21.6	16.2	22.0	16.1
Riverside	16.4	11.9	15.5	13.5	16.0	12.7
US-82	25.8	24.4	26.5	24.4	26.1	24.4

Table 4.5 Volumetric Swell of Treated Soils @ 3 day mellowing

Soil Type	Sample 1		Sample 2		Avg. Volumetric Swell Strain (%)	
	Volumetric Strain (%)		Volumetric Strain (%)			
	OMC	WOMC	OMC	WOMC	OMC	WOMC
Austin	11.8	9.9	11.4	9.7	11.6	9.8
Childress	9.8	8.5	10.4	8.5	10.2	8.5
Dallas	11.2	6.5	9.6	6.3	10.4	6.4
FM-1417	9.8	6.3	10.6	7.7	10.2	7.0
Riverside	9.1	8.5	9.3	8.3	9.0	8.4
US-82	10.8	6.2	11.8	7.6	11.3	6.70

Table 4.6 Volumetric Swell of Treated Soils @ 7 day mellowing

Soil Type	Sample 1		Sample 2		Average Volumetric Swell Strain (%)	
	Volumetric Strain (%)		Volumetric Strain (%)			
	OMC	WOMC	OMC	WOMC	OMC	WOMC
Austin	17.8	11.3	18.4	12.9	18.2	12.1
Childress	10.6	9.8	11.8	10	11.2	9.9
Dallas	8.5	7.3	9.5	6.5	9.0	6.9
FM-1417	6.6	5.2	6.8	5.6	6.7	5.4
Riverside	7.1	5.0	7.3	5.6	7.2	5.3
US-82	9.9	6.1	9.7	6.5	9.8	6.3

Table 4.7 Summary of Volumetric Swell

Soil	Natural Soil		6% lime, 0 day mellowing		6% lime, 3 day mellowing		6% lime, 7 day mellowing	
	Volumetric Swell Strain (%)							
	OMC	WOMC	OMC	WOMC	OMC	WOMC	OMC	WOMC
Austin	16.6	8.7	8.8	7.5	11.6	9.8	18.2	12.1
Childress	7.5	3.7	14.6	13.1	10.2	8.5	11.2	9.9
Dallas	11.0	7.8	24.4	23.7	10.4	6.4	9.0	6.9
FM-1417	16.2	8.9	22.0	16.1	10.2	7.0	6.7	5.4
Riverside	10.0	10.2	16.0	12.7	9.0	8.4	7.2	5.3
US-82	18.1	7.9	26.1	24.4	11.3	6.70	9.8	6.3

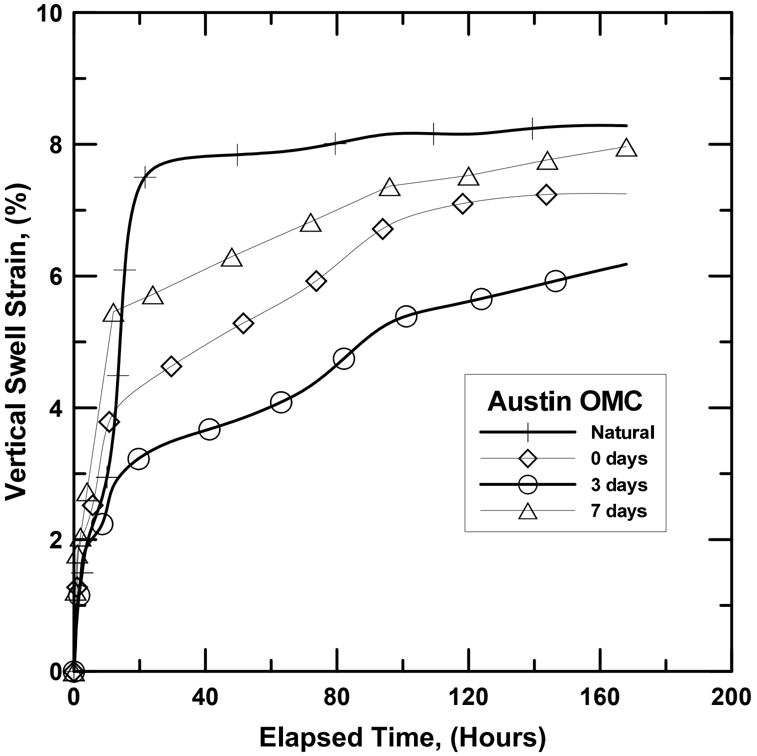

Figure 4.1 Vertical Swell vs. Elapsed Time, Austin Soil (36,000 ppm sulfates)

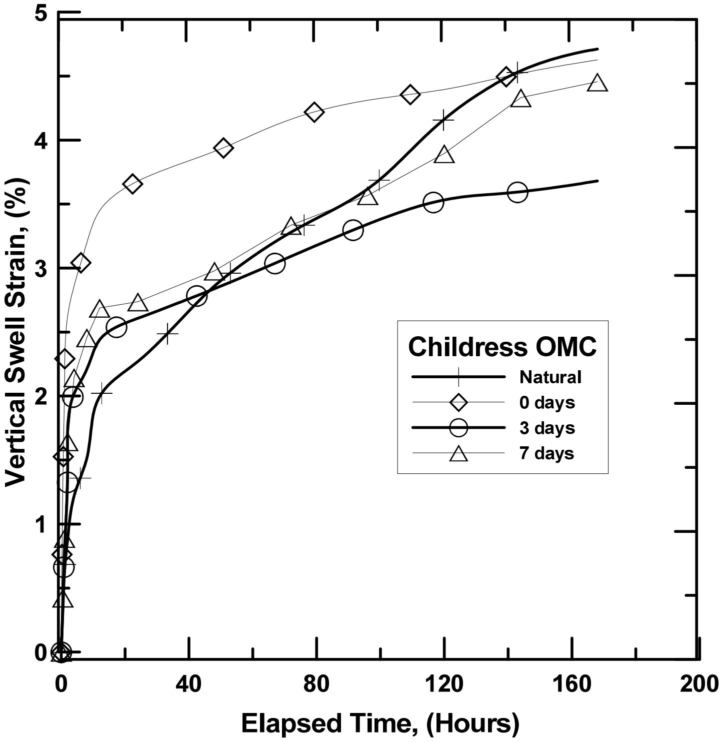

Figure 4.2 Vertical Swell vs. Elapsed Time, Childress Soil (44,000 ppm sulfates)

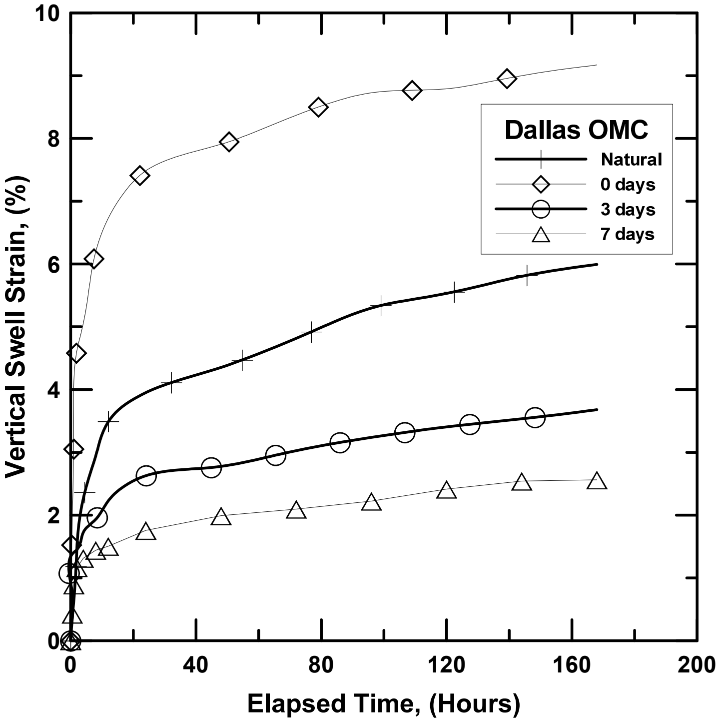

Figure 4.3 Vertical Swell vs. Elapsed Time, Dallas Soil (12,000 ppm sulfates)

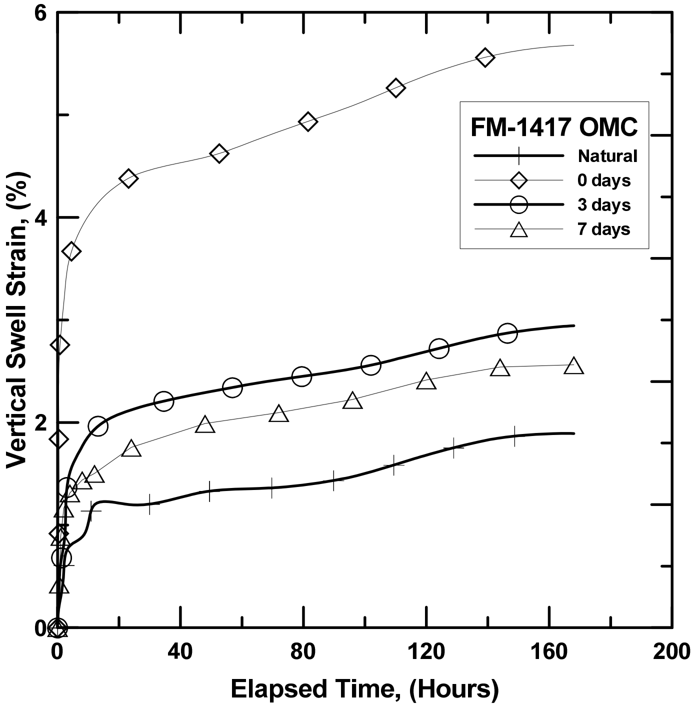

Figure 4.4 Vertical Swell vs. Elapsed Time, FM-1417 Soil (24,000 ppm sulfates)

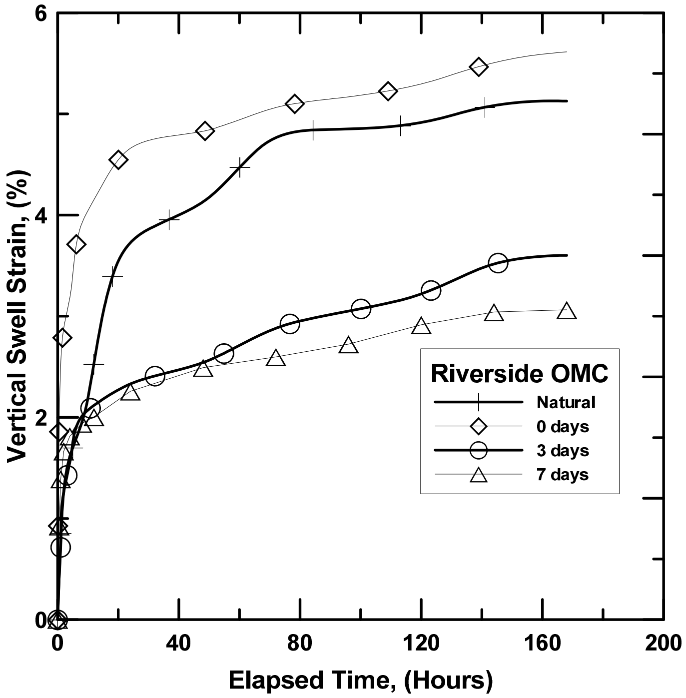

Figure 4.5 Vertical Swell vs. Elapsed Time, Riverside Soil (20,000 ppm sulfates)

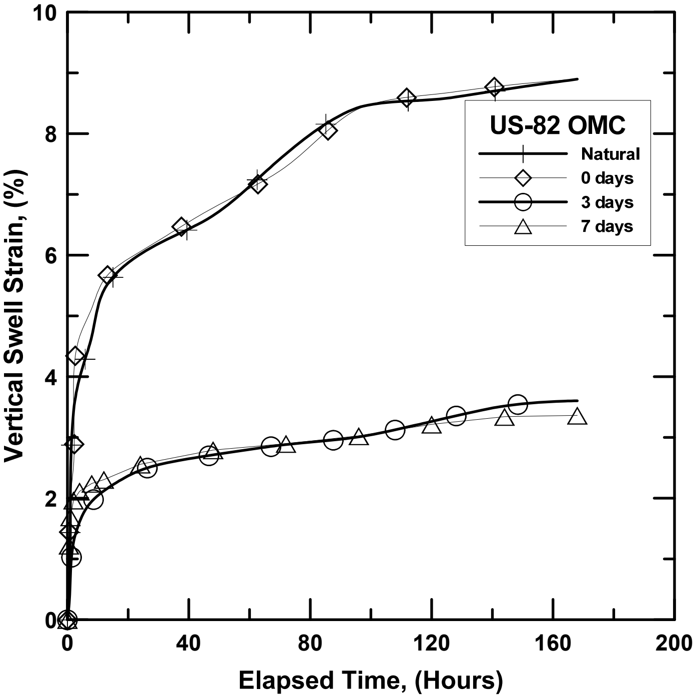

Figure 4.6 Vertical Swell vs. Elapsed Time, US-82 Soil (12,000 ppm sulfates)

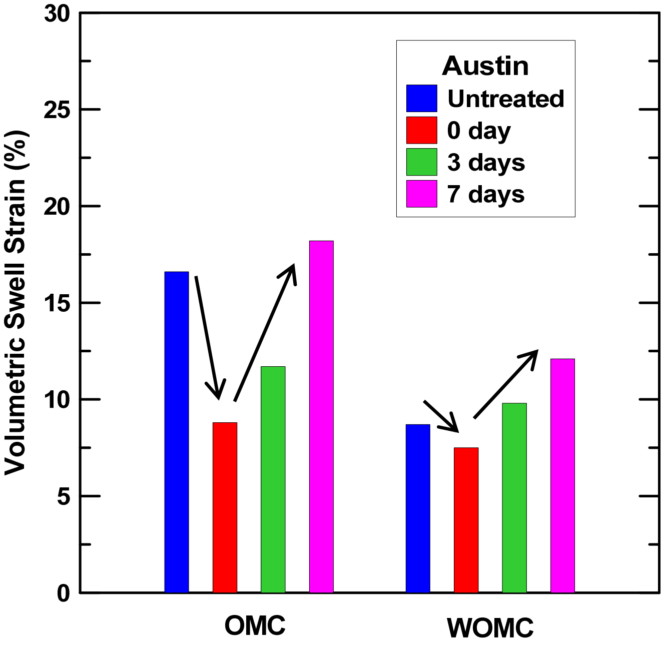

Figure 4.7 Volumetric Swell: Natural and 6% Lime Treated, Austin Soil (36,000 ppm sulfates)

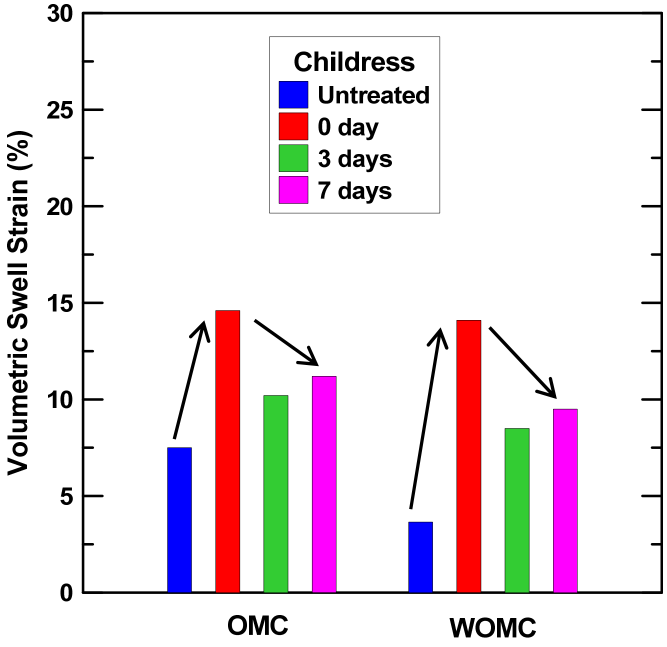

Figure 4.8 Volumetric Swell: Natural and 6% Lime Treated, Childress Soil (44,000 ppm sulfates)

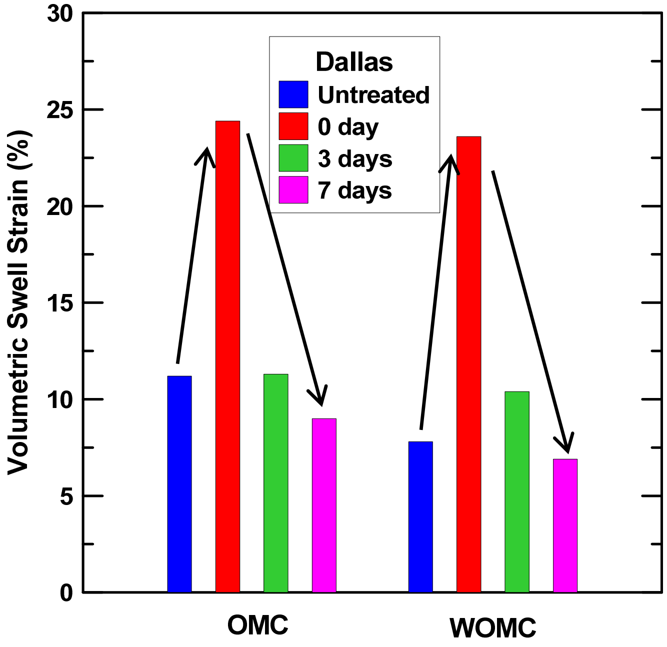

Figure 4.9 Volumetric Swell: Natural and 6% Lime Treated, Dallas Soil (12,000 ppm sulfates)

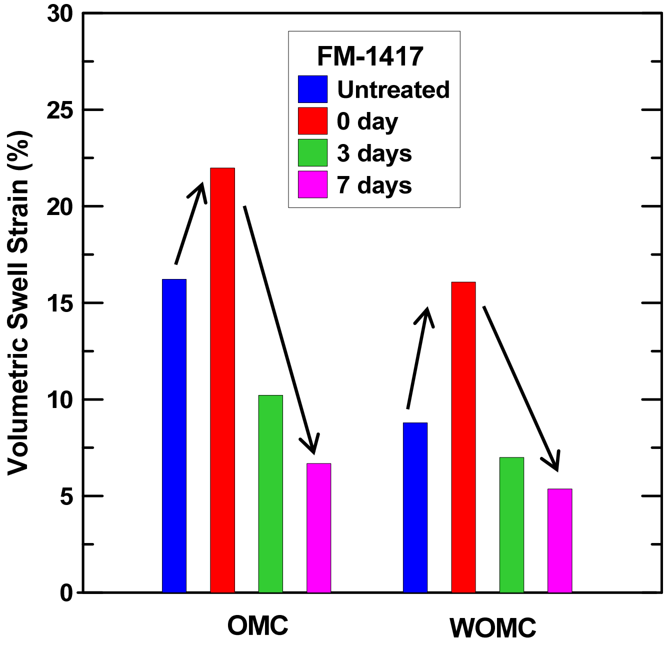

Figure 4.10 Volumetric Swell: Natural and 6% Lime Treated, FM-1417 Soil (24,000 ppm sulfates)

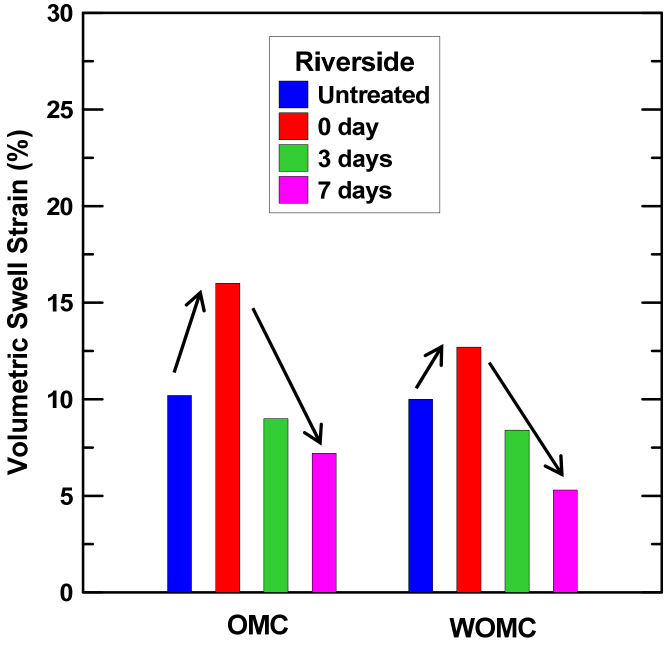

Figure 4.11 Volumetric Swell: Natural and 6% Lime Treated, Riverside Soil (20,000 ppm sulfates)

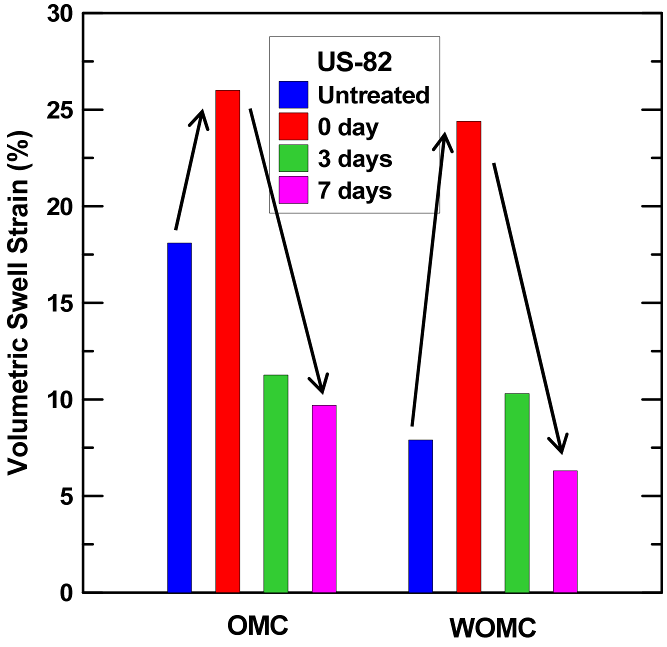

Figure 4.12 Volumetric Swell: Natural and 6% Lime Treated, US-82 Soil (12,000 ppm sulfates)

It can be seen that in untreated condition, Austin, Dallas, FM-1417 and US-82 all have high swelling potential. These four soils haves a volumetric swell strain (for the OMC condition) value greater than 10%, and this value indicates a high degree of expansion potential as per the problematic volumetric swell characterizations mentioned by Chen (1988). In addition, these four soils exhibited a PI value greater than 40.

With 0 day mellowing, the highest swell magnitude was observed in the US-82 soil (26%), whereas Austin soil showed the lowest swell (8.8%). The volumetric swell increased due to lime treatment at 0 day mellowing in all the soils, except for the Austin soil, where the volumetric swell strain value reduced from 16.6% for natural soil to 8.8% after lime treatment. In Austin soil, mellowing effectively reduced the swell to a level below 10%. In the rest of the soils, the swell increased beyond the natural level with 0 day mellowing, indicating the occurrence of sulfate reactions. In 3-day mellowed samples, the volumetric swell reduced below the natural level for four of the soils tested. Swell magnitudes in these soils were less than the natural swell, showing the effects of stabilization. All the four soils which responded positive to 3-day mellowing have sulfate contents below 30,000 ppm. The two soils which could not be stabilized by 3-day mellowing are Austin and Childress, both with sulfate levels higher than

30,000 ppm. With 3-day mellowing, the swell increased in Austin and Childress soils, both with sulfate contents above 30,000 ppm.

An additional set of 3-D swell tests was performed with a 7-day mellowing period to see the effect of the increased mellowing period on the swell behavior of high sulfate soils. For the 7-day mellowed soils, 5% more moisture was provided to cater for the moisture loss during mellowing. With 7-day mellowing, further reduction in swell was observed in the Riverside, Dallas, US-82 and FM-1417 soils. The observed volumetric swells in these soils were 7.2%, 9.0%, 9.8% and 6.7% respectively. It can be observed that in these soils, with a 7-day mellowing period, the volumetric swell reduced to a level below 10%, which is considered non-problematic and indicating that mellowing was effective in these soils. In Austin and Childress soils, swell further increased beyond the natural swell with 7-day mellowing, showing the dominance of sulfate reactions with increased mellowing periods. The observed swells at 7-day mellowing period and optimum moisture content were 18.2% and 11.2% respectively.

From the on-going discussion, it is evident that mellowing was ineffective in soils with sulfate contents above 30,000 ppm. Reasons for this behavior are addressed in the discussion section.

4.1.1.2 Three Dimensional Volumetric Shrinkage Test (3-D shrinkage)

Volumetric shrinkage tests were conducted to measure the decrease in the total volume of soil specimens due to the loss of moisture content from the compacted state to a completely dry state. 3-D shrinkage tests were conducted on all six treated soils at different mellowing periods. Volumetric shrinkage at 0 day and 3 day mellowing is presented in Tables 4.8 and 4.9 respectively. A summary of volumetric shrinkage strains is presented in Table 4.11. Variation of volumetric shrinkage with mellowing is depicted in Figures 4.13 thru 4.18.

Table 4.8 Volumetric Shrinkage Strain Test Results (0 day mellowing)

Soil	Sample 1		Sample 2		Avg. Volumetric Shrinkage Strain (%)	
	Volumetric Shrinkage Strain (%)		Volumetric Shrinkage Strain (%)			
	OMC	**WOMC**	**OMC**	**WOMC**	**OMC**	**WOMC**
Austin	-4.7	-8.6	-4.9	-8.6	-4.8	-8.6
Childress	-2.2	-2.4	-2.6	-2.8	-2.4	-2.6
Dallas	-8.6	-10.8	-9.0	-11.3	-8.8	-11.0
FM-1417	-7.5	-8.9	-7.7	-9.0	-7.6	-8.9
Riverside	-3.0	-3.2	-3.3	-2.7	-3.1	-3.0
US-82	-6.9	-8.4	-7.3	-8.6	-7.1	-8.5

Note: Negative sign indicate shrinkage.

Table 4.9 Volumetric Shrinkage Strain Test Results (3 day mellowing)

Soil	Sample 1		Sample 2		Average Volumetric Shrinkage Strain (%)	
	Volumetric Shrinkage Strain (%)		Volumetric Shrinkage Strain (%)			
	OMC	**WOMC**	**OMC**	**WOMC**	**OMC**	**WOMC**
Austin	-5.4	-9.6	-5.5	-9.8	-5.4	-9.7
Childress	-2.9	-4.0	-3.2	-4.0	-3.1	-4.0
Dallas	-8.5	-10.8	-8.6	-11.2	-8.6	-11.0
FM-1417	-6.4	-9.6	-6.1	-9.3	-6.3	-9.4
Riverside	-4.7	-3.9	-4.5	-3.7	-4.6	-3.8
US-82	-9.8	-10.5	-9.7	-10.4	-9.8	-10.4

Note: Negative sign indicate shrinkage.

Table 4.10 Summaries of Volumetric Shrinkage Strains

Soil	**Volumetric Shrinkage Strain* (%)**					
	Natural		**0 day mellowing**		**3 day mellowing**	
	OMC	**WOMC**	**OMC**	**WOMC**	**OMC**	**WOMC**
Austin	-8.8	-11.3	-4.8	-8.6	-5.4	-9.7
Childress	-14.1	-15.7	-2.4	-2.6	-3.1	-4.0
Dallas	-19.7	-26.2	-8.8	-11.0	-8.6	-11.0
FM-1417	-15.6	-20.6	-7.6	-8.9	-6.3	-9.4
Riverside	-12.5	-14.0	-3.1	-3.0	-4.6	-3.8
US-82	-13.7	-20.2	-7.1	-8.5	-9.8	-10.4

***all the results are average of two trials; Note: Negative sign indicate shrinkage.**

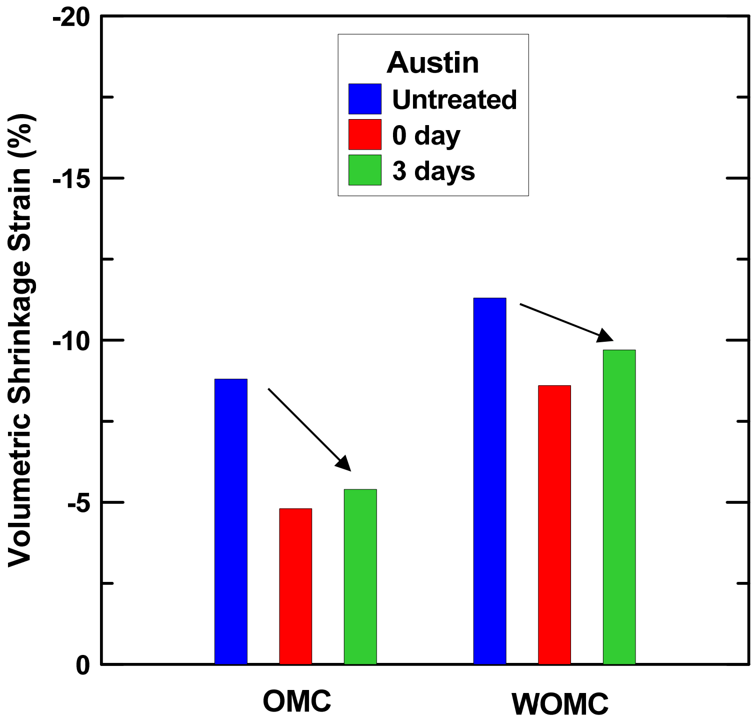

Figure 4.13 Volumetric Shrinkage, Austin Soil (36,000 ppm sulfates)

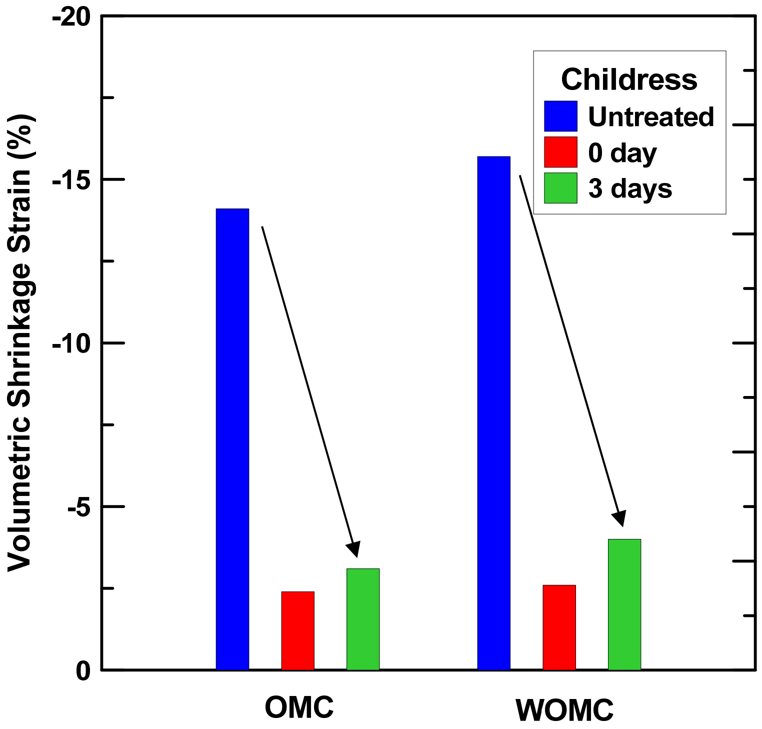

Figure 4.14 Volumetric Shrinkage, Childress Soil (44,000 ppm sulfates)

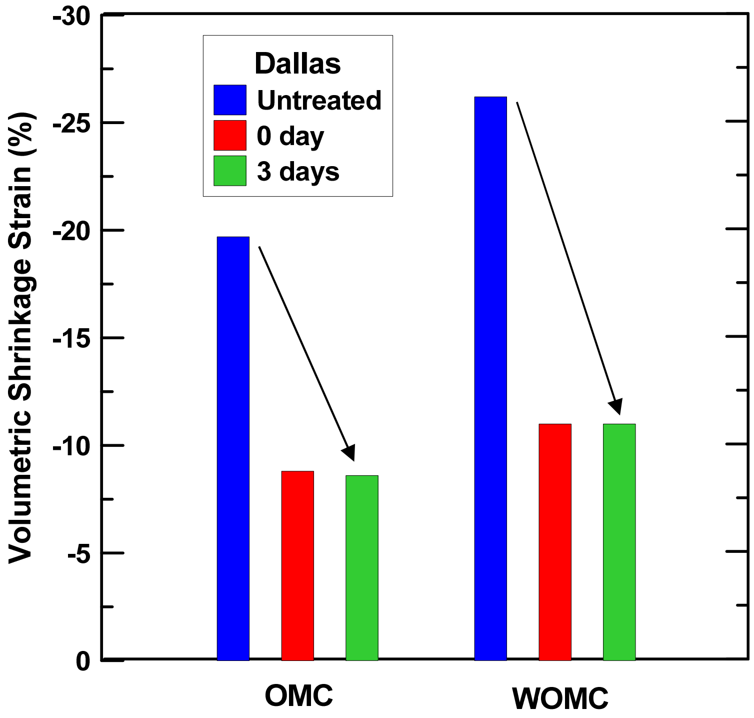

Figure 4.15 Volumetric Shrinkage, Dallas Soil (12,000 ppm sulfates)

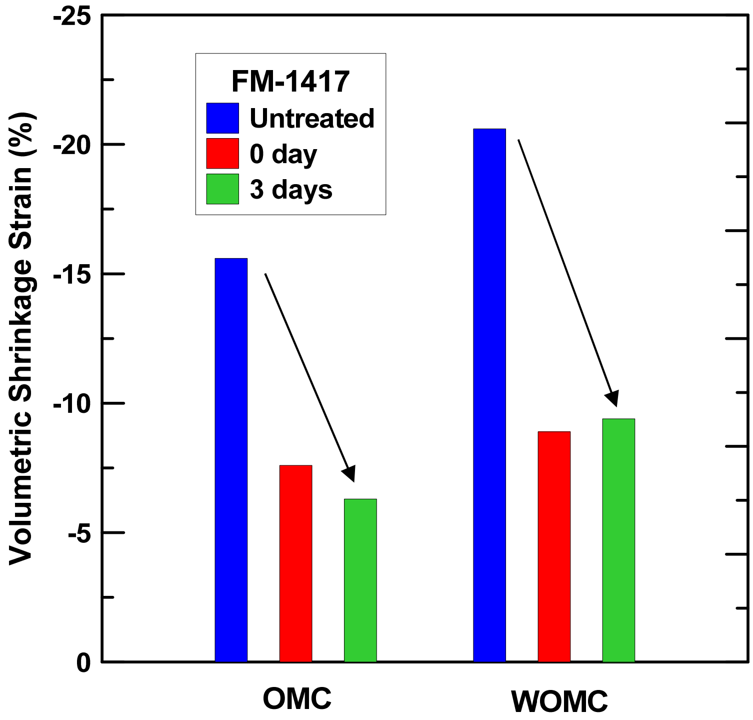

Figure 4.16 Volumetric Shrinkage, FM-1417 Soil (24,000 ppm sulfates)

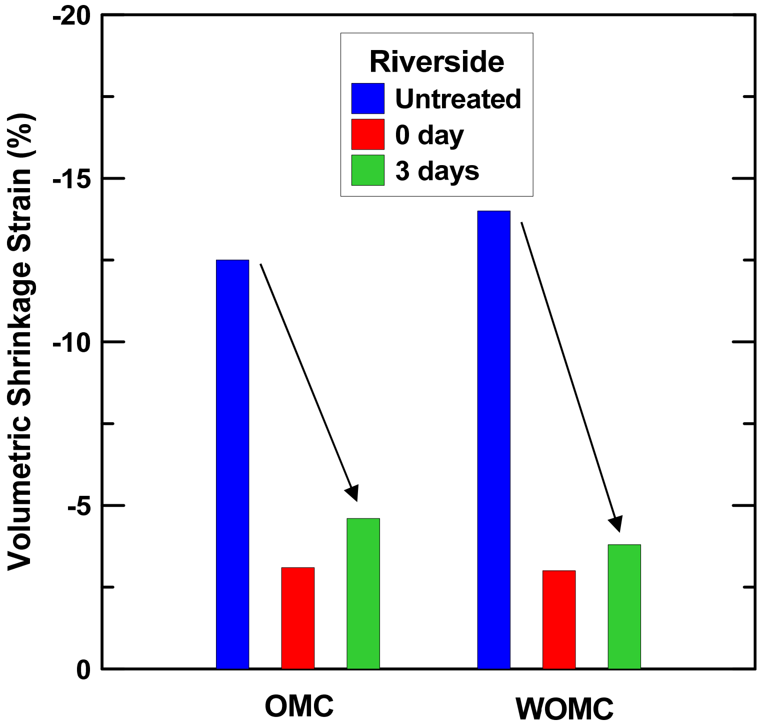

Figure 4.17 Volumetric Shrinkage, Riverside Soil (20,000 ppm sulfates)

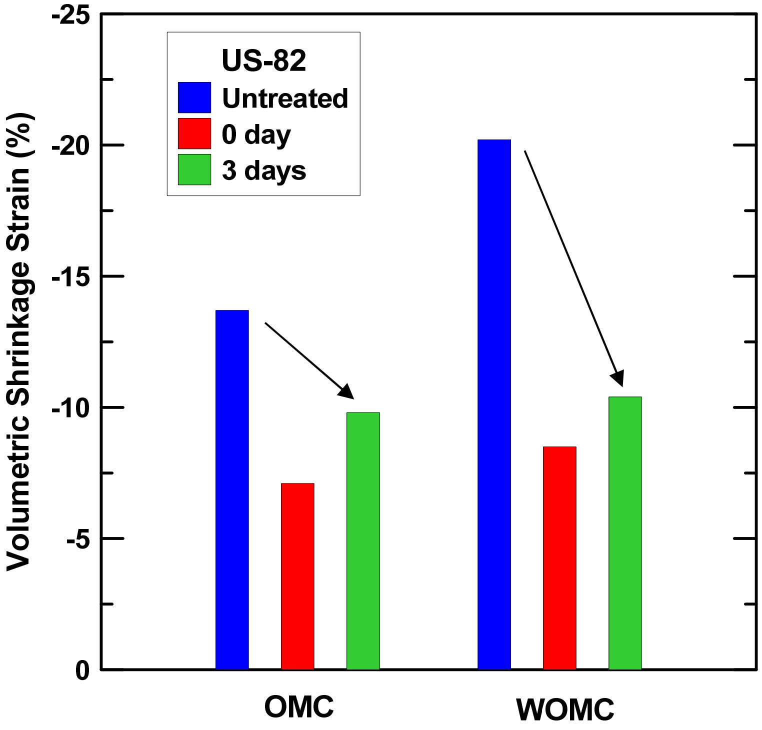

Figure 4.18 Volumetric Shrinkage, US-82 Soil (12,000 ppm sulfates)

It can be seen that the volumetric shrinkage strain is higher at wet of optimum moisture content than at optimum. The reason for this can be attributed to the presence of a higher percentage of moisture at wet of optimum when compared to optimum moisture content condition. Also, the soils which shrank more are high plasticity clays, with plasticity index (PI) greater than 40. These soils are capable of high swelling during the rainy season, as well as shrinkage during dry summer-like conditions.

In lime treated soils, volumetric shrinkage invariably decreased. In four of the six soils, there was slight increase in shrinkage with 3 day mellowing compared to the 0 day mellowing. The reason for this behavior could be due to higher moisture provided (additional 3% to cater for moisture loss during mellowing) in case of 3-day mellowed soils. Except for the Riverside soil, in all the test soils the volumetric shrinkage was higher at wet of optimum moisture content compared to optimum moisture content, owing to more moisture at wet of optimum. Overall, it can be concluded that presence of sulfates do not have any significant effect on shrinkage behavior of soils since shrinkage reduced in all the cases upon lime treatment.

4.1.2 Strength Characteristics

Unconfined Compression Strength (UCS) tests were conducted on untreated soils and used as a reference for comparing the strength changes in treated high sulfate soils at different periods of mellowing and the onset of sulfate and stabilization reactions. The following section describes the results of the testing program.

4.1.2.1 Unconfined Compression Strength (UCS) Tests

The Unconfined Compression Strength (UCS) tests were conducted as per ASTM D 2166 method. The tests were conducted on compacted soil specimens of 2.8 inches in diameter and 5.6 inches in height. UCS tests were conducted on treated soils with different mellowing periods at two moisture conditions (OMC and WOMC) on two identical specimens. Lime-treated samples were allowed to mellow for 0 and 3 days. After the elapsed time periods, samples were compacted and cured in a 100% humidity room for 7 days. Results of the UCS testing on natural soils are presented in Table 4.11. Table 4.12 and Table 4.13 and show the UCS strengths of treated soils at 0 day and 3 day mellowing. Figures 4.19 to 4.30 show the load versus deformation curve for natural and treated soils at different mellowing periods.

Table 4.11 UCS Tests Results (Natural Soils)

Soil	**Sample 1**		**Sample 2**		**Average UCS (psi)**	
	UCS (psi)		**UCS (psi)**			
	OMC	**WOMC**	**OMC**	**WOMC**	**OMC**	**WOMC**
Austin	25.5	20.6	30.5	21.4	28	21
Childress	21.7	16.5	23.8	15.3	22.8	15.9
Dallas	14.5	9.5	17.5	10.3	16	9.9
FM-1417	31.3	19.3	33.3	17.7	32.3	18.5
Riverside	28.8	16.7	30.2	17.7	29.5	17.2
US-82	28.5	18	31.5	18.4	30	18.2

Table 4.12 UCS Test Results (6% Lime, 0 day mellowing)

Soil	Sample 1		Sample 2		Average UCS (psi)	
	UCS (psi)		UCS (psi)			
	OMC	WOMC	OMC	WOMC	OMC	WOMC
Austin	89.6	54	93.5	58.4	91.5	56.2
Childress	110.7	86.8	115.0	75.4	112.9	81.1
Dallas	94.4	56.0	98.8	61.0	96.6	58.5
FM-1417	80.8	61.8	90.2	64.8	85.5	63.3
Riverside	62.6	49.9	71.0	60.4	66.8	55.1
US-82	72.3	42.7	81.0	50.6	76.6	46.6

Table 4.13 UCS Test Results (6% Lime, 3 day mellowing)

Soil	Sample 1		Sample 2		Average UCS (psi)	
	UCS (psi)		UCS (psi)			
	OMC	WOMC	OMC	WOMC	OMC	WOMC
Austin	83.2	52.8	93.2	55.2	88.2	54.0
Childress	99.8	56.62	114.6	60.1	107.2	58.3
Dallas	70.9	62.4	82.1	67.4	76.5	64.9
FM-1417	78.7	47.9	69.8	49.6	74.2	48.7
Riverside	63.5	46.2	58.0	45.5	60.7	45.9
US-82	67.4	42.9	63.4	37.9	65.4	40.4

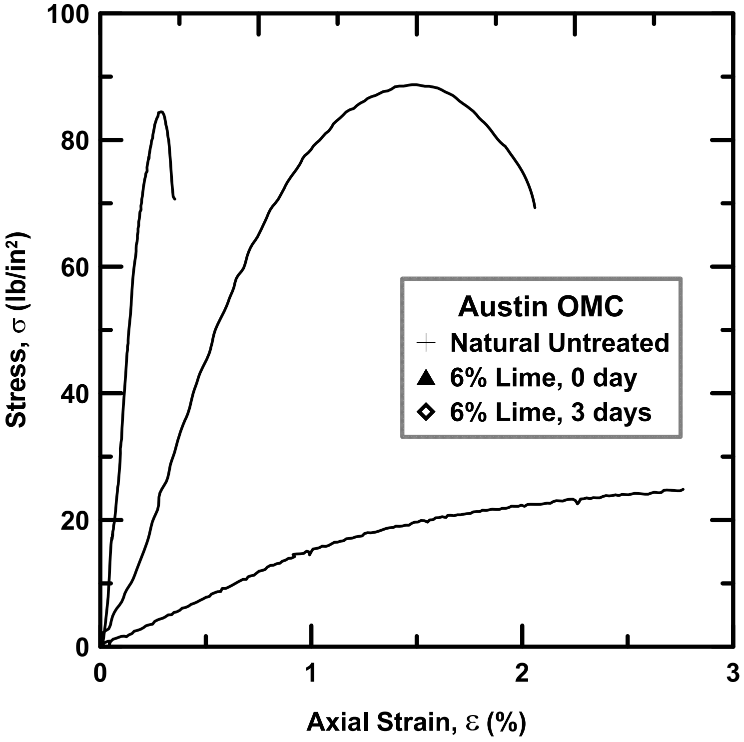

Figure 4.19 UCS Curves for Austin Soil @ OMC

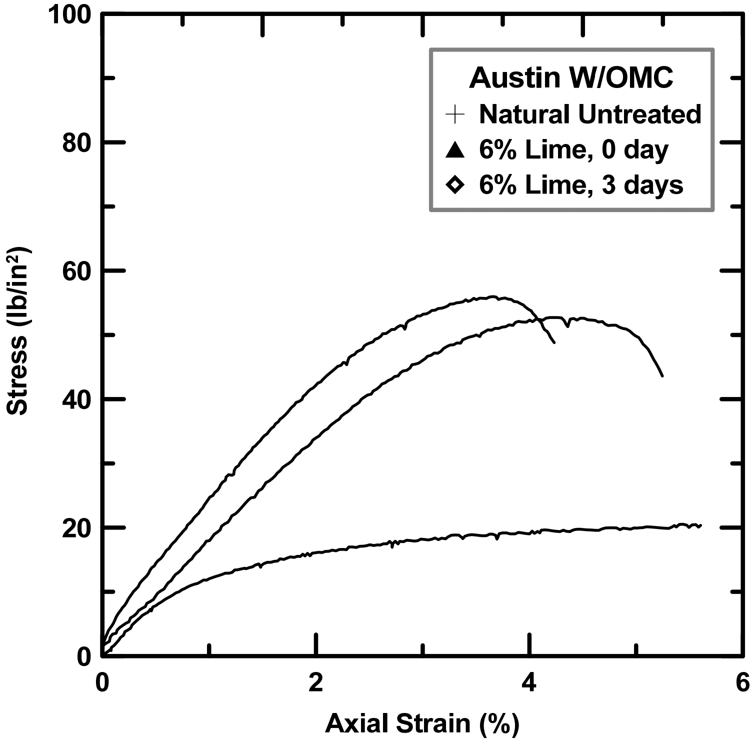

Figure 4.20 UCS Curves for Austin Soil @ w/OMC

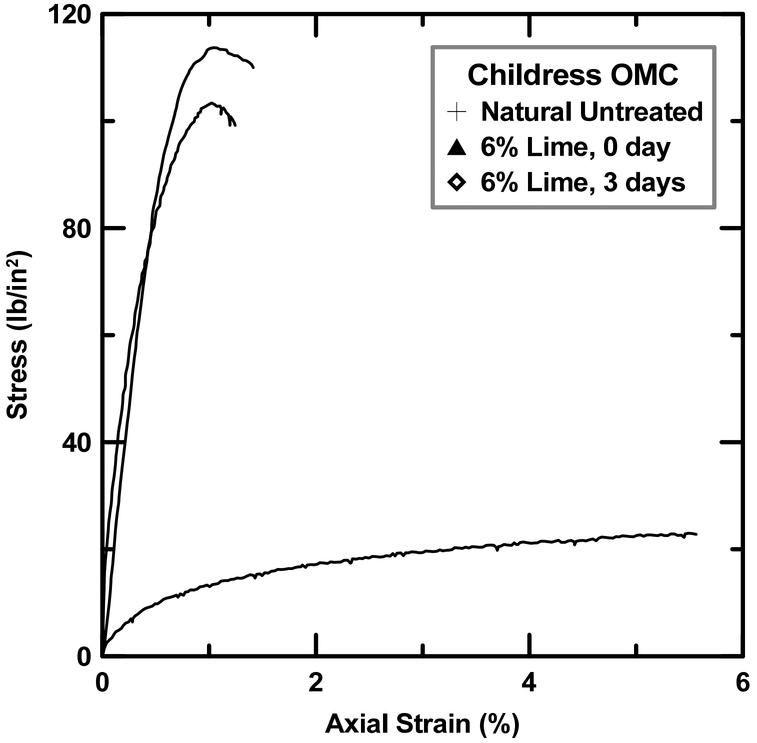

Figure 4.21 UCS Curves for Childress Soil @ OMC

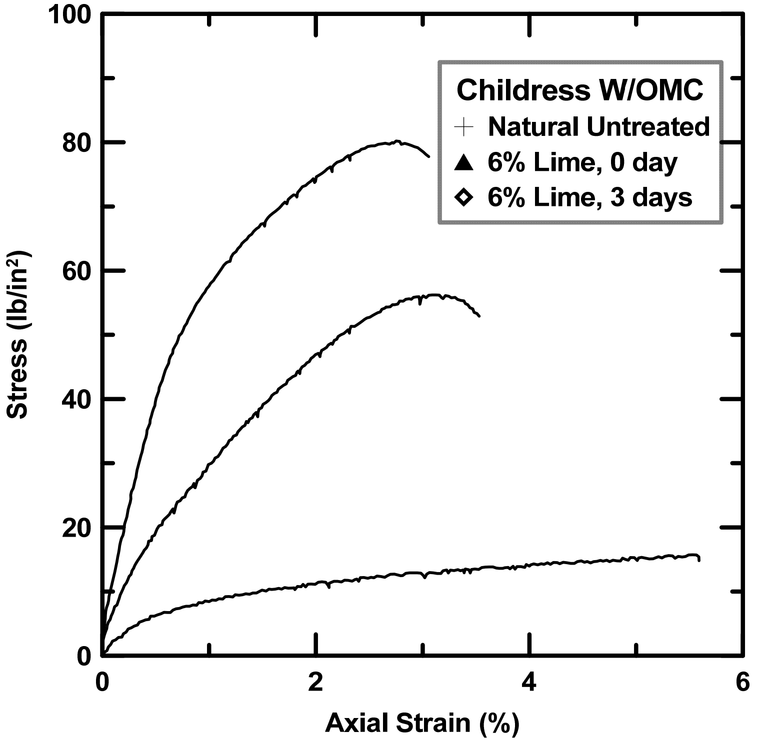

Figure 4.22 UCS Curves for Childress Soil @ w/OMC

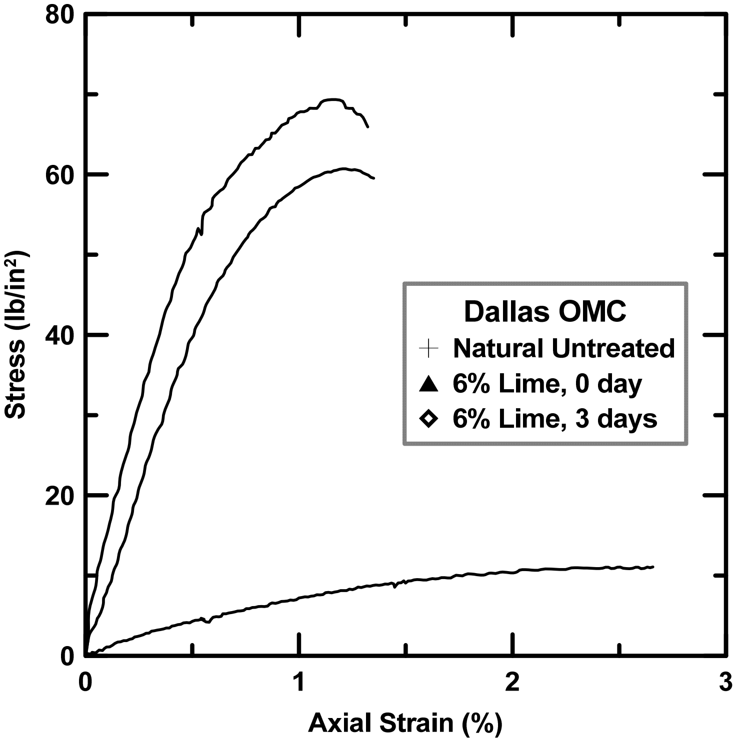

Figure 4.23 UCS Curves for Dallas Soil @ OMC

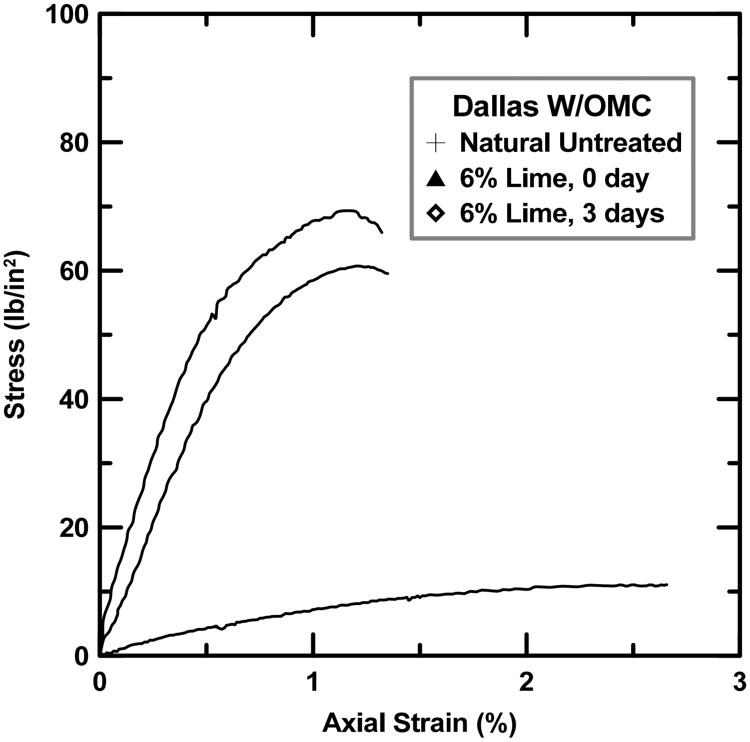

Figure 4.24 UCS Curves for Dallas Soil @ w/OMC

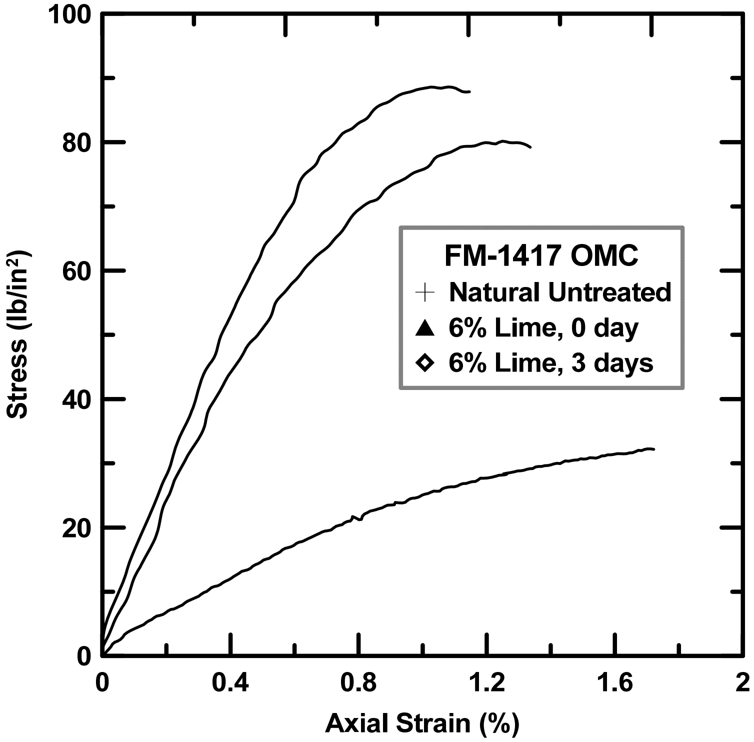

Figure 4.25 UCS Curves for FM-1417 Soil @ OMC

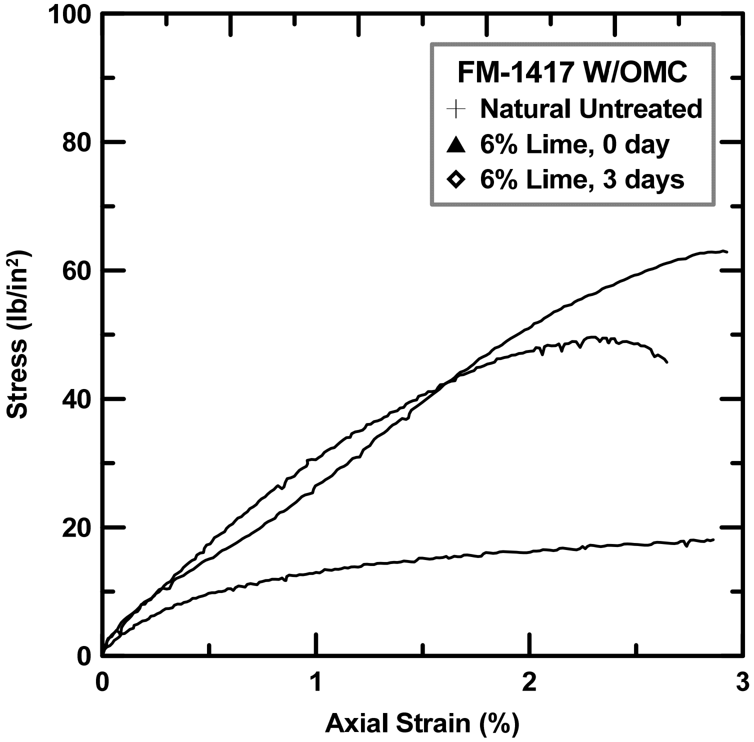

Figure 4.26 UCS Curves for FM-1417 Soil @ w/OMC

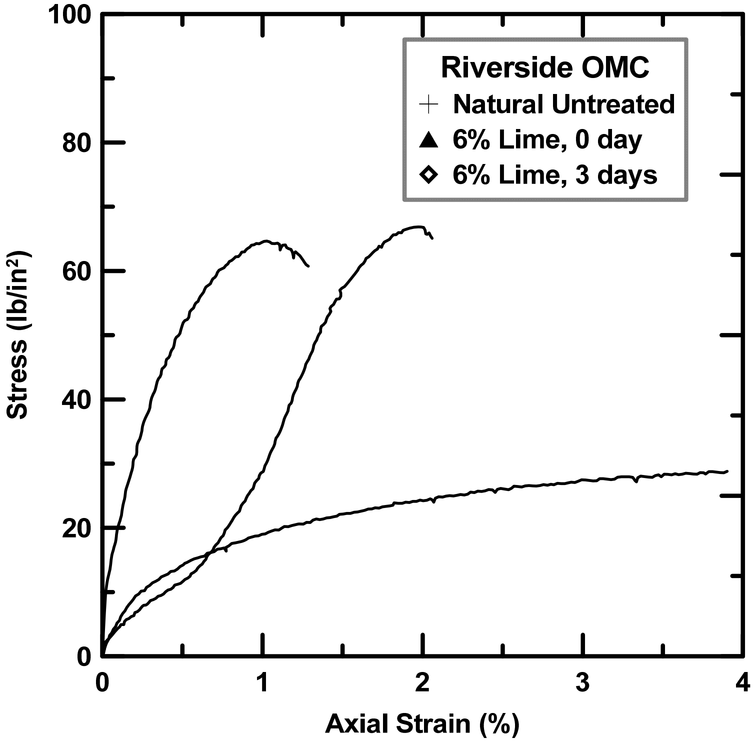

Figure 4.27 UCS Curves for Riverside Soil @ OMC

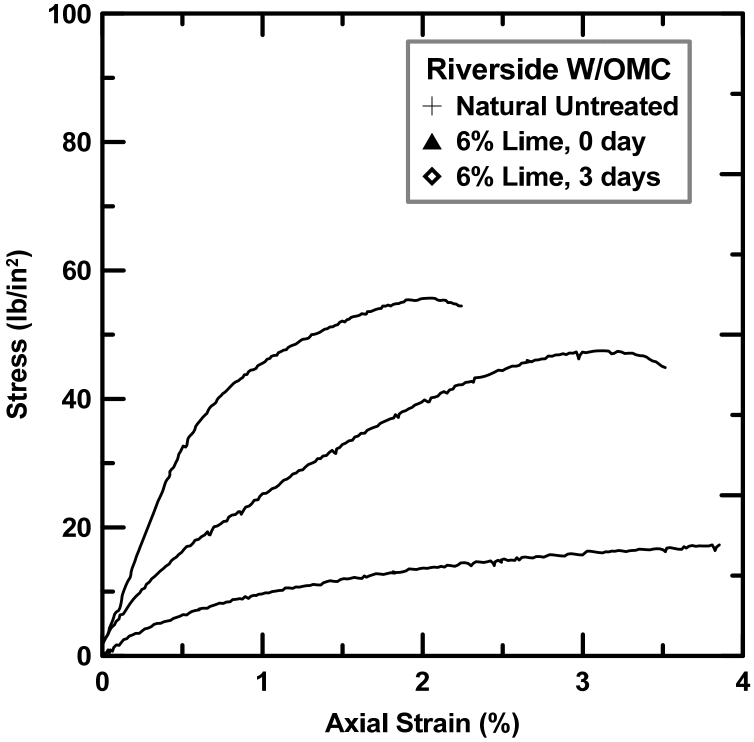

Figure 4.28 UCS Curves for Riverside Soil @ w/OMC

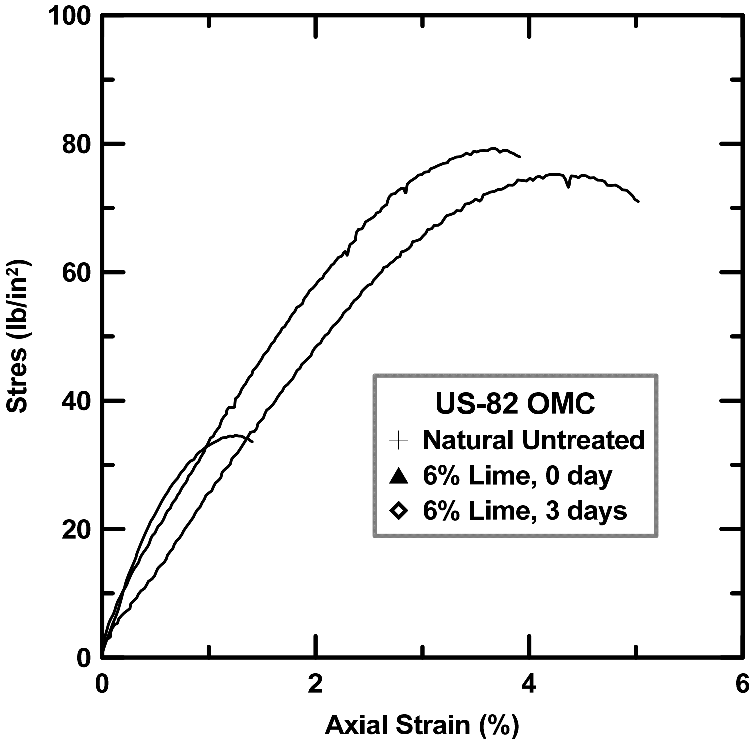

Figure 4.29 UCS Curves for US-82 Soil @ OMC

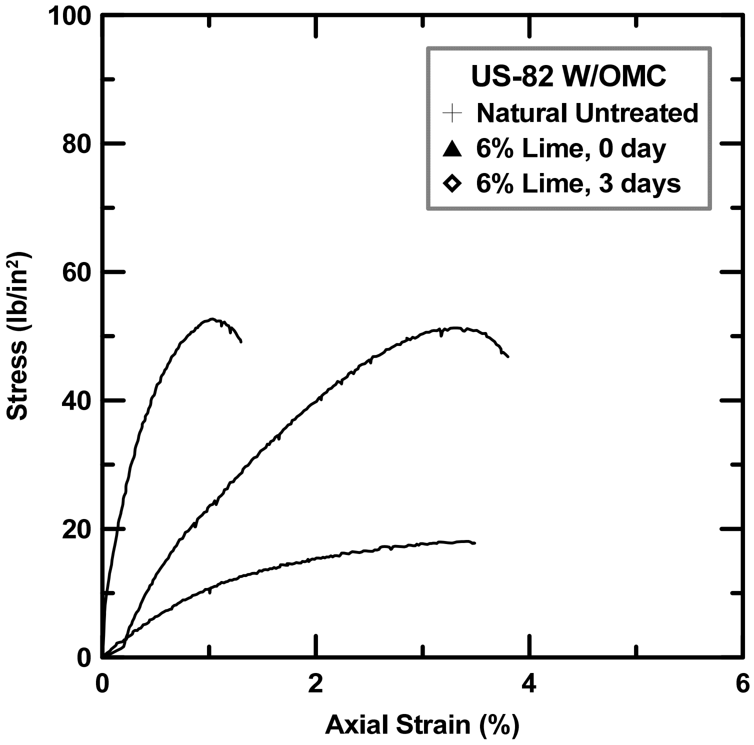

Figure 4.30 UCS Curves for US-82 Soil @ w/OMC

UCS strengths for untreated and treated soils varied from 10 psi to 113 psi. For untreated condition, the highest UCS strength was observed in the FM-1417 soil (32 psi); whereas, the lowest strength was seen in the Dallas soil (16 psi). For 0 day mellowed soils, Childress showed the highest strength (113 psi) and the Riverside soil showed the lowest strength (67 psi) is observed in Riverside soil. At 3-day mellowing, the Childress soil showed the highest strength (107 psi) and Riverside showed the lowest strength (61 psi).

From the UCS test results, it can be seen that the UCS strengths of lime-treated soils are two to five times higher than the untreated soils at 0 day and 3 day mellowing period. Another important observation is that the UCS values of the 3-day mellowed samples are slightly lower than those of the 0 day mellowed samples. This strength loss can be attributed to the higher moisture content provided in mellowed samples and maximum dry density changes (low compaction density) during the mellowing process. Similar trends of strength loss were observed in studies conducted at Purdue University (Jung et. al., 2009). The loss in strength due to mellowing is insignificant since the mellowed strength is considerably higher than the untreated soils strength.

4.1.2.2 1-D Swell Pressure Tests

The 1-D swell pressure test was conducted using the 1-D consolidation setup as per ASTM method (D4546-08 Standard Test Methods for One-Dimensional Swell or Collapse of Cohesive Soils). 1-D swell pressure tests were conducted on all six natural and lime-treated soils at different mellowing periods and optimum and wet of optimum moisture conditions. Results of swell pressure testing are reported in Table 4.14 and Table 4.15 respectively for 0-day and 3-day mellowed soils. Table 4.16 shows the summary of swell pressures for natural and lime-treated soils.

Table 4.14 Swell Pressure of 6% Lime Treated Soils @ 0 day mellowing

Soil Type	Sample 1		Sample 2		Average Swell Pressure (psi)	
	Swell Pressure (psi)		Swell Pressure (psi)			
	OMC	WOMC	OMC	WOMC	OMC	WOMC
Austin	35.8	12.8	36.6	13.3	36.2	13.0
Childress	39.2	16.0	37.6	16.2	38.4	16.1
Dallas	67.9	33.8	70.7	38.6	69.3	36.2
FM-1417	88.6	27.4	93.2	26.2	90.9	26.8
Riverside	58.3	28.2	53.1	30.6	55.7	29.4
US-82	98.0	62.4	97.4	66.2	97.7	64.3

Table 4.15 Swell Pressure of 6% Lime Treated Soils @ 3 day mellowing

Soil Type	Sample 1		Sample 2		Average Swell Pressure (psi)	
	Swell Pressure (psi)		Swell Pressure (psi)			
	OMC	WOMC	OMC	WOMC	OMC	WOMC
Austin	39.8	18.2	42.4	19.0	41.1	18.6
Childress	32.5	17.8	31.1	16.8	31.8	17.3
Dallas	34.2	9.8	33.6	10.6	33.9	10.2
FM-1417	24.5	9.7	20.3	8.9	22.4	9.3
Riverside	7.4	1.8	7.5	2.0	7.4	1.9
US-82	25.2	12.0	28.0	11.6	26.6	11.8

Table 4.16 Summary of Swell Pressures (Natural, 0 day and 3 day mellowed soils)

Soil Type	Swell Pressure (psi)					
	Untreated		0 day mellowing		3 day mellowing	
	OMC	WOMC	OMC	WOMC	OMC	WOMC
Austin	46.2	22.4	36.2	13.0	41.1	18.6
Childress	6.7	2.2	38.4	16.1	31.8	17.3
Dallas	40.7	15.1	69.3	36.2	33.9	10.2
FM-1417	37.1	16.2	90.9	26.8	22.4	9.3
Riverside	8.3	1.8	55.7	29.4	7.4	1.9
US-82	48.9	40.1	97.7	64.3	26.6	11.8

From the swell data of the natural soils, it can be seen that except for Childress and Riverside soils, all the soils showed significant swell pressures upon wetting. Childress soil is high compressible silt (MH) and Riverside soil is low plasticity clay (CL). In Austin and Childress soils at 3 day mellowing period, the swell pressures were significantly higher. The swell pressure measurements followed the same trend as three-dimensional free swell values. From the swell pressure data, it can be concluded that with 0-day mellowing, all other soils except Austin soil showed higher swell pressures. Observed swell pressures varied from 36 psi in Austin soil (lowest) to 98 psi in US-82 soil (highest) at 0- day mellowing. The observed swell pressures are in line with the volumetric swell recorded.

With 3-day mellowing, similar to 3-D swell tests, swell pressures decreased significantly in four of the six soils (Dallas, FM-1417, Riverside and US-82). Austin soil showed the highest swell pressure (41psi) and Riverside showed the lowest (7psi). The observed swell pressures in this case were lower than the untreated swell pressure of the soils. Though all the four soils have dominance of clay minerals in them, mellowing effectively reduced the swell pressures below natural level. The reason for this could be due to the plasticity changes occurring during mellowing period. Another cause for this behavior could be attributed to relatively low sulfate contents in these four soils compared with Austin and Childress soils. Austin showed an increase in swell pressure which is consistent with the volumetric swell result. Austin is high plasticity clay with very high sulfate content (36,000 ppm). With mellowing, the entire sulfate could not be consumed and enough sulfate remained in the system for uninterrupted formation and growth of Ettringite. This could be the reason why mellowing was ineffective in Austin soils. In the case of Childress soil, though the swell pressure decreased with 3-day mellowing, it was not significant since the swell pressure at 3day mellowing was considerably higher than the untreated swell pressure. Childress is high plasticity silt with mineral kaolinite dominance, and the untreated swell of the soil is low compared to others (6.7%). But due to very high sulfate content and a reasonable amount of Montmorillonite and Illite (35%), the heave reactions caused swell magnitudes that were higher than the untreated swell magnitude.

4.1.3 Mineralogical Studies

Reactive alumina and silica measurements were conducted on the natural and treated soils at different mellowing periods to study the role of alumina and silica in sulfate heave and stabilization reactions.

4.1.3.1 Reactive Alumina and Silica Measurements

Reactive alumina and silica measurements were conducted using ICP_MS (Inductively Coupled Plasma Mass Spectroscopy) on treated soils at different mellowing periods. Table 4.17 shows the reactive alumina and silica at 0 day and 3 day mellowing periods. A summary of reactive alumina and silica measurements is given in Table 4.18.

Table 4.17 Reactive Alumina and Silica Measurements @ 0 day and 3 day mellowing

Soil	0 day mellowing				3 day mellowing			
	Al* (ppm)	**Si* (ppm)**	**Al loss (%)**	**Si loss (%)**	**Al* (ppm)**	**Si* (ppm)**	**Al loss (%)**	**Si loss (%)**
Austin @ OMC	22.8	6.1	61	63	18.9	5.1	68	68
Austin @ WOMC	23.0	5.8	60	62	19.2	6.0	67	61
Childress @ OMC	28.1	5.9	63	54	32.2	7.2	61	46
Childress @ WOMC	29.2	5.2	62	62	32.1	6.3	58	54
Dallas @ OMC	87.6	68.2	70	71	122.2	69.2	58	70
Dallas @ WOMC	96.1	72.4	67	69	123.8	74.2	57	68
FM-1417 @ OMC	115.9	47.1	58	66	131.9	50.3	53	64
FM-1417 @ WOMC	120.8	49.7	57	64	135.2	50.9	52	63
Riverside @ OMC	108.8	42.8	65	89	183.7	49.4	41	87
Riverside @ WOMC	137.1	44.8	56	88	155.9	47.2	50	88
US-82 @ OMC	94.2	19.9	67	88	135.6	27.3	54	84
US-82 @ WOMC	99.1	19.9	66	88	172.2	23.0	42	87

Table 4.18 Summary of Reactive Alumina and Silica Measurements (at OMC)

Soil	Natural		0 day mellowing		3 day mellowing	
	Al	**Si**	**Al**	**Si**	**Al**	**Si**
Austin	58.9	15.4	22.8	6.1	18.9	5.1
Childress	75.8	12.6	28.1	5.9	32.2	7.2
Dallas	289.9	231.2	87.6	68.2	122.2	69.2
FM-1417	279.2	137.3	115.9	47.1	131.9	50.3
Riverside	297	379.8	108.8	42.8	183.7	49.4
US-82	323.3	187.1	94.2	19.9	135.6	27.3

From Table 18, it can be seen that Austin and Childress soils have the lowest reactive alumina and silica contents among the six soils under study. The percentage loss of alumina and silica due to lime treatment at different mellowing periods were also calculated and compared with the natural alumina and silica contents of the soil. In general, the alumina and silica loss was more at OMC compared to WOMC, with Riverside soil being an exception. It was observed that the highest percentage loss of silica was seen in the Riverside and US-82 soils at both 0- day and 3-day mellowing. In the rest of the soils, silica loss was more or less the same, and no specific trend between losses of silica and mellowing time was observed. Loss of alumina and silica were higher at 3-day mellowing than at 0-day mellowing in all the soils except Austin soil where, the reverse trend was observed.

The observed volumetric swell in Austin soil was more at 3day mellowing than at 0-day mellowing. Although Austin soil could be stabilized with 0-day mellowing, with 3-day and 7-day mellowing, the observed swell magnitudes were higher than previously. In Childress soil, there was no significant difference between loss of alumina and silica with 0-day and 3-day mellowing. Both Austin and Childress soils are high sulfate soils with soluble sulfates greater than 30,000 ppm. With lime treatment, stabilizing reactions and sulfate reactions occur at the same time, which involves dissolution of clay alumina and silica.

In case of 3-day mellowing, due to its low alumina and high sulfate contents, sulfate reactions dominated the stabilizing reactions, leading to more heaving than 0-day mellowed samples. This is true in the case of 7-day mellowing since the volumetric swell in 7-day mellowing is higher than the 0 and 3-day mellowing. For Childress soil, although the alumina loss was higher at 0-days mellowing compared to 3-day mellowing, the difference in loss percentage was not significant. It is reported in the literature that Ettringite formation depends on the amount of reactive alumina present in the system. Low alumina contents in soils favor the trisulfate hydrate (Ettringite) formation; whereas, high alumina contents lead to simultaneous formations of pozzolanic and Ettringite reactions. As a result, attractive forces from pozzolanic reactions will resist the disruptive forces caused by Ettringite formation and hydration reactions. Low alumina and high sulfate contents in Austin and Childress soils led to more heaving with increased mellowing periods.

An interpretation of the test results and the reasons for ineffectiveness of mellowing in two of the six soils are provided in the discussion section.

4.2 Analysis of Test Results

Swell tests conducted on lime treated high sulfate soils indicated that these soils are prone to “Ettringite Induced Heaving” when treated with calcium based stabilizers. In the first part of the research program, pre-compaction mellowing technique with two different mellowing periods (3 days and 7 days) was used to stabilize high sulfate soils (sulfate contents > 8,000 ppm). Results of the testing indicated that mellowing was effective in four of the six soils considered in the current study. The two soils that could not be stabilized even at higher mellowing periods are Austin and Childress soils. The reason for the ineffectiveness of mellowing in these two soils was attributed to very high sulfate amounts (> 30,000 ppm), low reactive alumina and silica contents in the treated soils and low compaction void ratios.

From the on-going discussion it is evident that Ettringite induced heaving is a function of the sulfate content, reactive alumina and silica contents of the native soils and compaction void ratio conditions. Combined effects of all these parameters manifested in the form of observed volumetric swell strains when the treated soils were subjected to hydration. The effects of each of these variables need to be considered separately to understand the mechanisms of swelling in the chemically treated high sulfate soils.

In the analysis of test results section, the reasons for effectiveness of mellowing of the six soils are explained using two approaches. In the first approach, Gibbs free energy for the formation of Ettringite mineral in the treated soil is calculated for the soils at the given sulfate, reactive alumina and silica contents. Also, Gibbs free energy for the formation of pozzolanic compounds is calculated and this energy is compared with that of Ettringite formation. Based on the calculated Gibbs free energy, the favorability of Ettringite reactions over the pozzolanic reactions and vice versa are reported.

In the second approach, mass-volume calculations are performed based on the soil density, specific gravity and sulfate contents to obtain the maximum amount of Ettringite formed in a given compacted soil system. Compaction dry density characteristics of treated soils after sulfate induced soil swelling were used to obtain the void distribution in the treated high sulfate soils. Based on the void ratio changes from initial to heaving conditions, the assessment of Ettringite induced heaving was made.

In the last section of this chapter two swell prediction models are presented. Swell prediction model developed based on the sulfate content value using an approach outlined by Little et al. (2010) is presented and comparisons are made between the predicted volumetric swell and measured volumetric swell strains. Based on the mass volume calculations and initial void ratio of the soils, swell prediction model based on threshold void ratio frame work is presented in the last section of the chapter.

4.2.1 Analysis of Test Results

As mentioned above six different soils from the State of Texas with varying sulfate contents are collected for the research study. Lime is used as the stabilizer for treating these soils. These soils were treated with lime and allowed to mellow for 0, 3 and 7 day followed by volumetric swell testing. Volumetric swell tests indicated that the mellowing is ineffective in two of the six soils considered. The reasons for ineffectiveness of mellowing in two of the six soils are explained using the two approaches presented here.

First approach is based on Gibbs free energy calculations. In this approach, Gibbs free energy for reactants and products are calculated. As the occurrence and direction of chemical reactions are based on the energy exchange between reactants and products, the favorability of a chemical reaction and formation of compounds can be assessed based on the “actual molar volumes” of reactants for the soils considered in this study.

The second approach is based on the mass-volume calculations of Ettringite formation. In this approach the amount of Ettringite is assessed from the native soil dry density and sulfate content. Based on the mass-volume calculations of treated soil swell, the amount of voids, solids and Ettringite per feet cube of soil can be calculated. If the void volume is higher than the Ettringite volume, the formed Ettringite could be accommodated in the soil matrix and no Ettringite induced heave is observed in this case. If the Ettringite volume exceeds void volume, the Ettringite grows out of the void matrix leading to higher swell magnitudes. Based on these calculations the reasons for ineffectiveness of mellowing in two of the six soils considered in the current study were explained.

4.2.1.1 First Approach: Gibbs Free Energy

The occurrence and direction of geochemical reactions are influenced by the exchange of energy between reactants and products (Pauling, 1960; Krauskopf et. al., 1995). This energy is expressed in terms of Gibbs free energy in kJ/mol. A geochemical system with minimum free energy is considered to be in thermodynamic equilibrium. Standard ***Gibbs Free Energy*** of any reaction can be calculated as:

$$\Delta G_r^o = \sum \Delta G_P^o - \sum \Delta G_R^o \qquad (1)$$

Where,

ΔG_r° = Gibbs Free Energy of the Reaction;

ΔG_P° = Gibbs Free Energy of Products;

ΔG_R° = Gibbs Free Energy of Reactants.

In the current analysis, standard Gibbs free energies of both the deleterious (Ettringite formation) and strength giving (pozzolanic) reactions are calculated from the quantities of reaction constituents. Gibbs free energies of both the reactions are compared and the reaction with higher free energy tends to proceed faster than the other. In performing the Gibbs free energy calculations for heave assessment of lime treated high sulfate soils, the following assumptions were made.

1. Heaving is attributed to formation of Ettringite only.
2. Ettringite formation is effected by the concentrations of Aluminum and sulfate only since the soils considered in the current study are treated with same amount of lime (6% by dry weight) and source of water for Ettringite formation is the same in all the cases.
3. Occurrence of deleterious heave is resisted by the formation of strength giving Calcium Silicate Hydrate (CSH) compounds.
4. CSH is considered in the current analysis since it is the most common occurring pozzolanic reaction product. (Taylor, 1964).
5. The formations of calcium aluminum hydrates (CAH) and calcium aluminum silicate hydrate (CASH) compounds are not considered since the portion of alumina entering the Ettringite reaction and CAH and CASH reactions cannot be quantified exactly.

The governing reactions for the Gibbs free energy calculations are given as:

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

(Ettringite Formation Reaction)

$$SiO_2 + 1.7Ca(OH)_2 + 0.917H_2O \rightarrow 1.7CaO.SiO_2\ .2.617H_2O \quad (3)$$

(Calcium Silicate Hydrate, CSH Formation Reaction)

Flow chart showing the Gibbs free energy approach is presented in Figure 4.31. From the Ettringite formation reactions it can be seen that 2 moles of aluminum hydroxide and 3 moles of sulfate react to form 1 mole of Ettringite. Similarly, 1 mole of silicon dioxide reacts to form 1.7 mole of Calcium Silicate Hydrate. Gibbs free energy of reactants and products for Ettringite and CSH reaction are obtained (Fujii et al., 1983; Moschner et al., 2009) and presented in Tables 4.19 and 4.20, respectively.

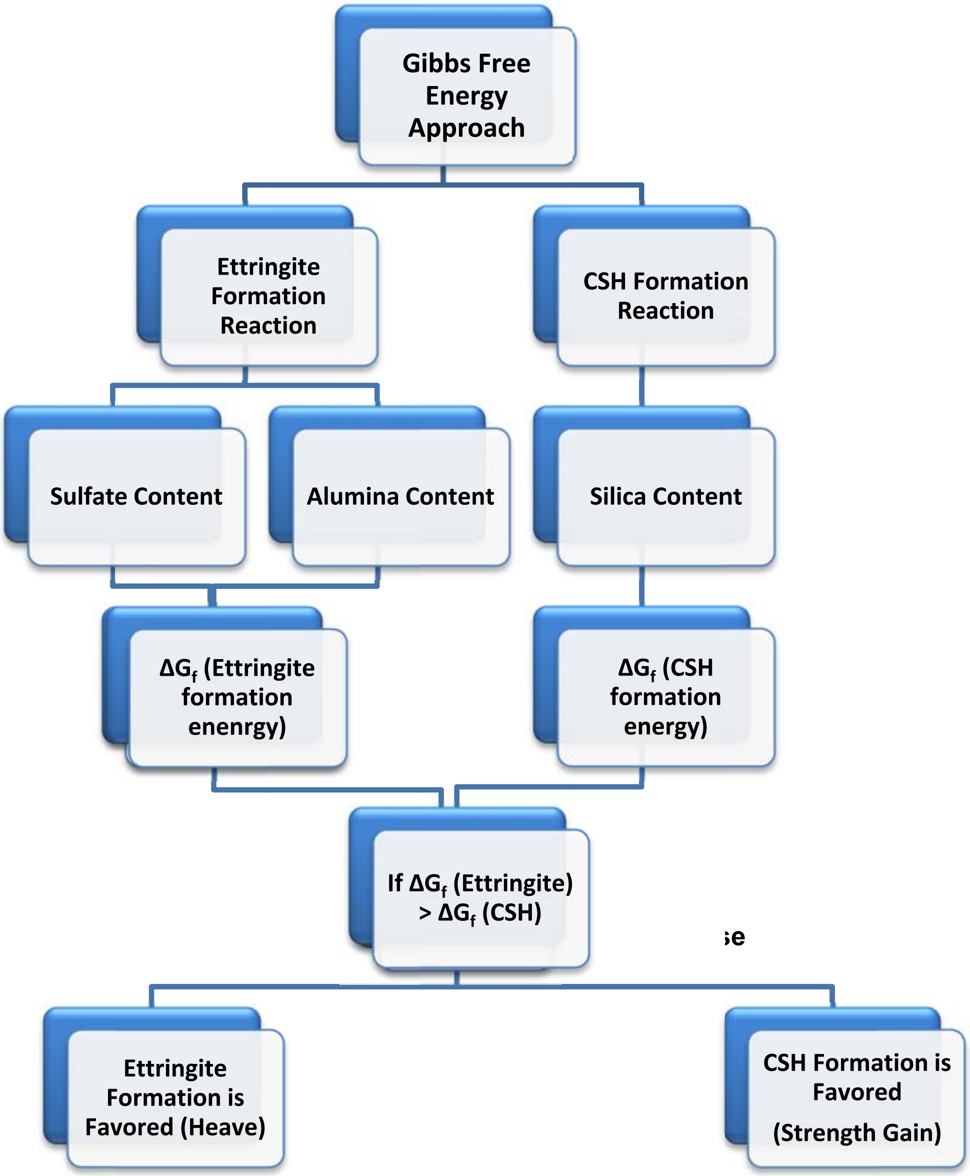

Figure 4.31 Gibbs Free Energy Approach

Gibbs free energy values reported in Table 4.19 and 4.20 are based on standard molar volume of reactants and products for Ettringite and CSH reactions presented in Equations 2 and 3. For Gibbs free energy calculations of Ettringite and CSH reactions, natural alumina and silica contents are obtained from ICP analysis and sulfate contents are measured using Modified UTA method and the same are presented Table 4.21.

Table 4.19 Gibbs free energy of Reactants and Products at 20°C (Ettringite Reaction)

Ettringite Formation Reaction	
Species	**$\Delta_a G^{o}$ (kJ/mol)**
H_2O	-236.84
$(OH)^-$	-157.32
Ca^{2+}	-553.07
$Al(OH)_4^-$	-1301.29
SO_4^{2-}	-744.35
Ettringite	-15196.53

Note: $\Delta_a G^{o}$=Standard Gibbs Free Energy

Table 4.20 Gibbs free energy of Reactants and Products at 20ºC (CSH Reaction)

CSH Formation Reaction	
Species	**$\Delta_a G^{o}$ (kJ/mol)**
H_2O	-236.83
$Ca(OH)_2$	-897.5
SiO_2	-856.3
$CaO \cdot SiO_2 \cdot 2.617H_2O$	-2630

Note: $\Delta_a G^{o}$=Standard Gibbs Free Energy

Table 4.21 Alumina, Silica and Sulfate contents of the Test Soils in ppm

Soil	**Al (ppm)**	**Si (ppm)**	**SO_4(ppm)**
Austin	58.9	15.4	36,000
Childress	75.8	12.6	44,000
Dallas	289.9	231.2	12,000
FM-1417	279.2	137.3	24,000
Riverside	297	379.8	20,000
US-82	323.3	187.1	12,000

Note: Al= Alumina Content; Si= Silica Content; SO_4=Sulfate Content

Based on the alumina, silica and sulfate concentrations in natural soils, the molar volume of alumina, silica and sulfates in mol/L are calculated for Austin soil as per the above chemical reactions. The molar volume amounts of sulfate, alumina and silica are determined and presented in Table 4.22 for the soils considered. Sample calculation of molar volumes of sulfate, alumina and silica for Austin soil is presented below:

$$(SO_4^{2-}) = \frac{Conc.\,in\,\frac{mg}{L}}{MW\ of\ \ SO4} = \frac{\frac{36000mg}{L}X\frac{g}{1000mg}}{96\frac{g}{mol}} = \frac{3}{8}\ mol/L$$

$$Al(OH)_4^- = \frac{Conc.\,in\ mg/L}{MW\ of\ Al(OH)_4} = \frac{\frac{58.9mg}{L}X\ g/1000mg}{95.01\ g/mol} = 6.2\ X\ 10^{-4}\ mol/L$$

$$SiO_2 = \frac{Conc.\,in\ mg/L}{MW\ of\ SiO_2} = \frac{\frac{15.4mg}{L}X\ g/1000mg}{60.09\ g/mol} = 2.56\ X\ 10^{-4}\ mol/L$$

Where Conc.= Concentration; MW= Molecular weight

Table 4.22 Balanced Amounts of Sulfate, Alumina and Silica in mol/L

Soil	**SO_4 (mol/L)**	**$Al(OH)_4$ (mol/L)**	**SiO_2 (mol/L)**
Austin	0.3750	0.0006	0.0002
Childress	0.4583	0.0008	0.0002
Dallas	0.1250	0.0030	0.0038
FM-1417	0.2500	0.0029	0.0022
Riverside	0.2083	0.0031	0.0063
US-82	0.1250	0.0034	0.0031

Note: SO_4 = Sulfate; Al $(OH)_4$ = Aluminum; SiO_2 = Silica

Based on the molar concentrations of reactants shown in Table 4.22, the amount of Ettringite and CSH are calculated by balancing Equations 4 and 5. A balanced Equation for Austin soil is presented below:

For Austin Soil, amount of SO_4, $Al(OH)_4$, SiO_2 in mol/L are 0.3750, 0.0006, 0.0002 respectively. Based on these amounts the final balanced Equation for Ettringite and CSH reactions can be represented as:

$$0.00186Ca^{2+} + 0.0006Al(OH)_4^- + 0.0012(OH)^- + 0.0009SO_4^{2-} + 0.0081H_2O \rightarrow 0.0003Ca_6[Al(OH)_6]_2.(SO_4)_3.26H_2O \tag{4}$$

(Ettringite Formation Reaction)

$$0.00025SiO_2 + 0.0004Ca(OH)_2 + 0.00023H_2O \rightarrow 0.0004CaO.SiO_2 .2.617H_2O \tag{5}$$

(Calcium Silicate Hydrate, CSH Formation Reaction)

From the Equations 4 and 5, the Gibbs free energy of reactants and compounds are calculated separately and the Gibbs free energy of Ettringite and CSH formation reactions are calculated for respective sulfate, alumina and silica concentrations. The calculated final Gibbs free energies for Ettringite and CSH reactions are shown in Table 4.23 for the six soils considered in the current study.

Table 4.23 Final Gibbs Free Energy of Ettringite and CSH Reactions

Soil	**$\Delta_f G^\circ$ (J/mol)**		**Remarks**
	Ettringite Reaction	**CSH Reaction**	
Austin	-800	-480	Ettringite formation is favored
Childress	-104	-40	Ettringite formation is favored
Dallas	-390	-7100	CSH formation is favored
FM-1417	-330	-4260	CSH formation is favored
Riverside	-4070	-11780	CSH formation is favored
US-82	-450	-5820	CSH formation is favored

Note: $\Delta_f G^\circ$= Final Gibbs Free Energy in J/mol

From the Table 4.23, it can be seen that the Gibbs free energy of Ettringite and CSH formation reactions are negative. Negative Gibbs free energy of the reaction indicates energy is released (exothermic) when compounds are formed. Also, since energy is released in the reactions, the reaction can proceed on its own and does not require energy from outside source. Gibbs free energies of Ettringite and CSH reactions are tabulated for six soils. It can be seen from the Table that for Austin and Childress soils, the Gibbs free energy for formation of Ettringite is higher than the CSH reaction implying Ettringite formation reactions (deleterious) are favored when compared to pozzolanic CSH reactions (strength giving reactions).The reason for ineffectiveness of mellowing in Austin and Childress soil can be explained by the free energy calculations since the Ettringite reactions are dominating in these soils when compared to CSH reactions (very high sulfate contents and low reactive alumina silica contents).

In rest of the four soils the CSH free energies are much higher than the Ettringite free energies, indicating CSH reactions are favored in these soils. All these four soils could be stabilized successfully with increased mellowing periods. Higher free energy of Ettringite reactions and lower CSH reactions in Austin and Childress soils are attributed to high sulfate contents and low alumina and silica contents. Converse is true in the rest of the soils owing to comparatively low sulfate contents and high alumina and silica contents. From the on-going discussion it is clear that Ettringite reactions are dependent on the sulfate content and alumina content of soils and the pozzolanic CSH reactions are dependent on the silica content of calcium stabilizer treated soils.

4.2.1.2 Second Approach: Mass-Volume Calculations of Ettringite Formation

In the second approach based on mass-volume calculations, the method outlined by Hunter (1988) is used here. In the first part of the approach the maximum amount of Ettringite that could be formed at certain sulfate concentration is calculated from the stoichiometric transformation of sulfates to Ettringite. In the second part, mechanical calculations are done using the natural dry density of soil as well as the dry density of the treated soils. For a targeted dry density of treated soils, the percentages of soil and Ettringite are obtained from the specific gravities of soil and Ettringite mineral. Once the percentages of Ettringite and soil are obtained, the volume of solids, voids and Ettringite per cubic ft. (ft^3) of the treated soil after elapsed swell are calculated from the mass-volume relationships. A pictorial representation of the Ettringite/Thaumasite growth in the soil matrix and corresponding changes in void volumes are presented in Figure 4.32. Based on the volume of voids, solids and Ettringite, the possibility of deleterious heave in treated soils can be assessed.

The following assumptions are made in performing the mass-volume calculations.

1. Phase diagram comprising of soil solids and voids (at complete saturation or no air phase) is considered in the current analysis.
2. The calculations presented below are based on soils subjected to three (3) day mellowing period.
3. Though 3 day mellowing period is considered in the analysis, the sulfate contents prior to mellowing are used are natural sulfate contents for the analysis. All of sulfate available in the natural soil has participated in the chemical reactions.
4. Since all the soils are lime treated, it is assumed that the observed swell is only due to Ettringite formation and swell due to natural expansiveness of the soil is neglected.
5. Swell due to formation of Thaumasite is neglected since all the swell tests were conducted in laboratory environment at 20°C. At the room temperatures formation of Thaumasite is less favored hence neglected in the analysis.
6. Distressed dry unit weight is obtained from the final moisture content and final sample dimensions.
7. The decrease in dry density of the treated soils after swell is due to formation of Ettringite which has a lower dry density compared to the soil itself.
8. Specific gravity, molar weight and molar volume of Ettringite are taken as 1.77g/cc, 1254 g/mole and 708.5cc/mole respectively.

The data required for mass – volume calculations is presented in Table 4.24 for Austin soil.

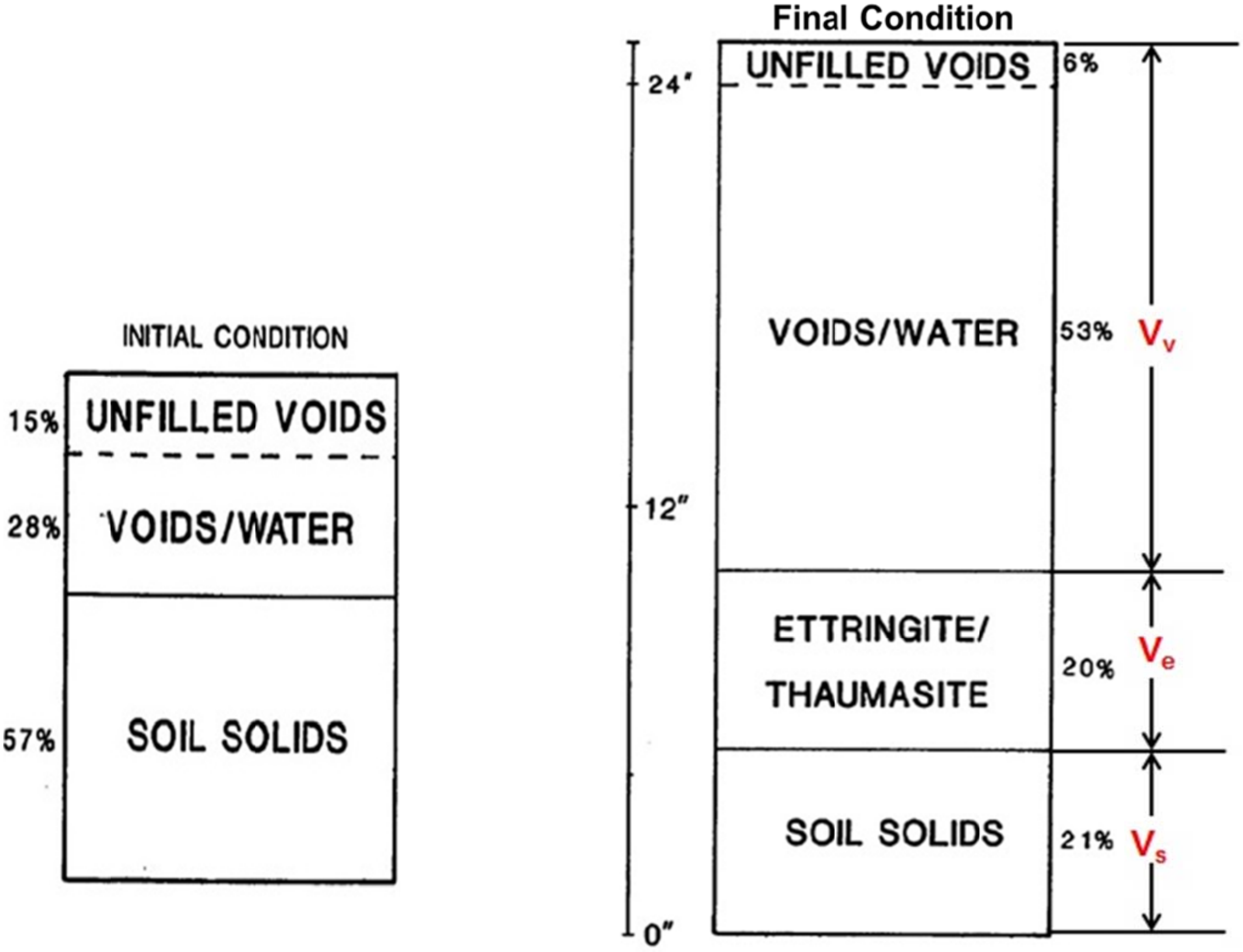

Figure 4.32 Volume Changes in Soil Matrix Due to Formation of Ettringite/Thaumasite (Hunter, 1988)

Table 4.24 Data for Mass-Volume Calculations

Parameter	Value
maximum dry density of untreated soil (γ_{di})	106 pcf
maximum dry density of lime treated soil (before swelling) (γ_{dLi})	95 pcf
Specific Gravity of Soil Solids (G_s)	2.61
Sulfate Content in mg/kg (SO_4)	36,000
Maximum dry density of damaged soil (after swelling) (γ_{dLf})	83.62 pcf
Specific Gravity of Soil Solids (after swelling)(G_{ss})	2.06
Final moisture content (after swelling) ($w_f(\%)$)	33%
Initial volume (V_{iL})	1 ft^3
Final volume (after swelling) (V_{fL})	1.12 ft^3

Chemical calculations are performed first to determine the maximum amount of Ettringite formed using all the sulfate content available. Chemical calculations are given below:

a. Maximum dry density of lime treated soil is $95\,\frac{lb}{ft^3}$

b. Sulfate content of 36,000 mg/Kg is equivalent to

$$\left(95x0.454\ \frac{kg}{ft^3}\right)X\ 36000\ \frac{mg}{kg} = 1552\ grams\ of\ sulfate\ per\ \ ft^3\ \ of\ soil$$

c. 1 mole sulfate is 96 grams, hence total sulfate available is 16.2 moles

$$\frac{1552\ gms}{96\ gm/mole} = 16.2\ moles$$

d. From the Ettringite reactions 3 moles of sulfate reacts to form 1 mole of Ettringite, the amount of Ettringite formed is 5.4 moles.

e. Weight of Ettringite formed is

$$\frac{5.4\ moles\ X\ 1254\frac{gm}{mole}}{\frac{454\ gm}{lb}} = 14.9\ lbs\ of\ Ettringite$$

f. Based on the sulfate contents and soil compaction density the maximum amount of Ettringite that could be formed for Austin soil is 14.9 lbs.

The next step is the calculations to determine the void distribution. Mechanical calculations are given below:

a. From the laboratory data, final dry density of lime treated soil (after swelling) is obtained as $83.6\ pcf$

b. Specific gravity of heaved soil after swelling is $G_{ss} = 2.06$

c. To achieve average specific gravity of 2.06, the percentage of soil and Ettringite are 35% and 65% (weighted average of specific gravities of soil and Ettringite).

d. The system has 65% Ettringite, $0.65\ X\ 83.62\ pcf = 54.35\ pcf\ Ettringite$

e. In a unit volume, Ettringite volume portion per ft^3= $\frac{(0.65\ X\ 83.6\ pcf\ \ X\ 454 gm/lb)}{(1.77\ X\ 28317\frac{cc}{ft^3})} = 0.49$

f. In a unit volume, soil solids per ft^3= $\frac{(0.35\ X\ 83.6\ pcf\ \ X\ 454 gm/lb)}{(2.61\ X\ 28317\frac{cc}{ft^3})} = 0.18$

g. In a unit volume, voids per ft^3= $(1 - 0.49 - 0.18) = 0.33$

h. For every cubic feet of treated soil (after swelling), the approximate percentages of soil, voids and Ettringite are: 0.49 Ettringite, 0.18 soil solids and 0.33 voids.

i. For the soil considered in this example, the void portion is less than the Ettringite portion indicating the swell could not be accommodated in the soil matrix which leads to heaving.

Once the portion of Ettringite formed per ft^3 of soil is obtained it is multiplied with the specific gravity and unit weight of water to obtain the maximum amount of Ettringite that could form in a soil system. The mass-volume calculations performed for all the soils considered in the study are reported in Table 4.25 thru 4.30 at optimum and wet of optimum moisture contents.

Table 4.25 Theoretical Maximum Possible Ettringite in lb/ft^3 of Soil Based on Sulfate Content (@OMC)

oil	**Sulfate Content (ppm)**	**G_s (Natural Soil)**	**Theoretical Maximum Possible Weight of Ettringite (lbs/ft^3 of soil)**
Austin	36,000	2.61	14.9
Childress	44,000	2.51	18.40
Dallas	12,000	2.71	4.54
FM-1417	24,000	2.66	9.08
Riverside	20,000	2.56	8.53
US-82	12,000	2.65	4.65

Table 4.26 Theoretical Maximum Possible Ettringite in lb/ft^3 of Soil Based on Sulfate Content (@WOMC)

Soil	**Sulfate Content (ppm)**	**G_s (Natural Soil)**	**Theoretical Maximum Possible Weight of Ettringite (lbs/ft^3 of soil)**
Austin	36,000	2.61	14.1
Childress	44,000	2.51	17.4
Dallas	12,000	2.71	4.3
FM-1417	24,000	2.66	8.7
Riverside	20,000	2.56	8.1
US-82	12,000	2.65	4.4

Table 4.27 Phase Distribution Based on Mechanical Calculations (@OMC)

Soil	**Sulfate Content (ppm)**	**Per ft^3 of Soil**		
		V_e	**V_S**	**V_V**
Austin	36,000	0.49	0.18	0.33
Childress	44,000	0.58	0.11	0.31
Dallas	12,000	0.35	0.21	0.45
FM-1417	24,000	0.4	0.13	0.47
Riverside	20,000	0.36	0.24	0.4
US-82	12,000	0.3	0.27	0.43

Note: V_e = Volume of Ettringite Per ft^3 of soil; V_s = Volume of solids per ft^3 of soil;
V_v = Volume of voids per ft^3 of soil

Table 4.28 Phase Distribution Based on Mechanical Calculations (@WOMC)

Soil	**Sulfate Content (ppm)**	**Per ft^3 of Soil**		
		V_e	**V_S**	**V_V**
Austin	36,000	0.42	0.21	0.37
Childress	44,000	0.46	0.18	0.36
Dallas	12,000	0.3	0.23	0.47
FM-1417	24,000	0.25	0.27	0.48
Riverside	20,000	0.41	0.16	0.43
US-82	12,000	0.25	0.29	0.46

Note: V_e = Volume of Ettringite Per ft^3 of soil; V_s = Volume of solids per ft^3 of soil;
V_v = Volume of voids per ft^3 of soil

Table 4.29 Mass of Ettringite formed Based on Mechanical Calculations (@OMC)

Soil	Final Volume ft^3 (based on swell data)	Volume of Ettringite (ft^3)	Mass of Ettringite (lbs)
Austin	1.116	0.55	60.40
Childress	1.102	0.64	70.59
Dallas	1.104	0.39	42.68
FM-1417	1.102	0.44	48.69
Riverside	1.090	0.39	43.34
US-82	1.113	0.33	36.88

Table 4.30 Mass of Ettringite formed Based on Mechanical Calculations (@WOMC)

Soil	Final Volume ft^3 (based on swell data)	Volume of Ettringite (ft^3)	Mass of Ettringite (lbs)
Austin	1.098	0.46	46.39
Childress	1.085	0.50	50.81
Dallas	1.064	0.32	33.13
FM-1417	1.070	0.27	27.61
Riverside	1.084	0.44	45.28
US-82	1.067	0.27	27.61

From Table 4.27 and 4.28 it can be seen that the volume occupied by Ettringite formation is very high in Austin and Childress soils. In both the soils Ettringite volume could not be accommodated in the soil matrix which result in Ettringite induced heaving. Ettringite induced heave is of concern in these two soils. In rest of the four soils, the Ettringite volume is lower than the void volume which means even if there is Ettringite formation; the soil can accommodate it in the void space. Similar type of mass-volume calculations are performed on the 7 day mellowed soils. The results indicated that 7 day mellowing is ineffective in Austin and Childress soils. From the on-going discussion it can be seen that, sulfate contents and compaction void ratios are important in assessing the sulfate induced heave in lime treated high sulfate soils.

4.3 Discussion

Four of the six high sulfate soils could be stabilized successfully using pre-compaction mellowing technique. The reasons for ineffectiveness of mellowing in two soils are explained using Gibbs free energy approach and mass-volume calculations. Also, the two soils that could not be stabilized have sulfate contents above 30,000 ppm and contain low alumina and silica contents. The effect of clay mineralogy and compaction void ratios was studied and presented in the following section.

4.3.1 Effect of Compaction Void Ratio on Swell Strains

To study the effect of compaction void ratio (e) on swell behavior, void ratios of the tests soils in compacted state has to be calculated. Using the specific gravity and maximum dry density from proctor curve void ratios of the test soils were calculated at both optimum moisture content (OMC) and wet of optimum moisture content (WOMC). Void ratios of the test soils are presented in Table 4.31.

Table 4.31 Void Ratios of Test Soils

Soil	Sulfate Level, ppm	Compaction Void Ratio	
		OMC	WOMC
Austin	36,000	0.54	0.62
Childress	44,000	0.52	0.60
Dallas	12,000	0.84	0.93
FM-1417	24,000	0.86	0.96
Riverside	20,000	0.61	0.70
US-82	12,000	0.82	0.91

From Table 4.31, it can be seen that both Austin and Childress soils have low void ratios compared to other soils. Low compaction void ratio means more dense soil matrix. It is known that lime treatment decreases the maximum dry density. Though there was a decrease in maximum dry density upon lime treatment it was not significant in these two soils due to low initial compaction void ratios. Due to the low void ratio the Ettringite formation and growth could not be accommodated in the dense soil matrix leading to more heaving. Also, Austin and Childress soils have sulfate contents above 30,000 ppm and the reactive alumina and silica contents in these soils were lower than 100 ppm. Combination of low void ratios, high sulfate contents and low reactive alumina and silica could be the reasons why mellowing was ineffective in these two soils.

Another important observation is that Riverside soil has a lower void ratio (0.61) in comparison to the other soils. Lower void ratio should lead to a denser matrix and hence more swelling similar to the Austin and Childress soils. Though Riverside soil has a lower void ratio, low sulfate contents (compared to Austin and Childress) and high reactive alumina and silica contents (297 ppm, 380 ppm) lead to lower swelling upon mellowing. Due to its low sulfate content all the available sulfate is consumed during the mellowing process and no further sulfate were available for further Ettringite formation. High alumina and silica contents helped in pozzolanic reactions leading to a stabilized material hence less swelling.

4.3.2 Effect of Clay Mineralogy on Swell Strains

Clay mineralogy of the test soils was determined from the cation exchange capacity (CEC), specific surface area and total potassium (TP) measurements. Determination of clay mineralogy gives an insight into what type of clay minerals are present and their reactivity with respect to pozzolanic and sulfate reactions. Clay mineral distribution of the test soils is presented in Table 4.32.

Table 4.32 Percent Clay Minerals Observed for Soils under Study

Soil Region	**% Illite**	**% Kaolinite**	**% Montmorillonite**
Austin	14.5	48.6	36.9
Childress	18.3	65.9	15.8
Dallas	15.2	34.6	50.2
FM-1417	13.2	20.3	66.5
Riverside	21.2	58.7	20.1
US-82	13.7	39.2	47.1

From Table 4.32 it can be seen that both Austin and Childress soils have mineral Kaolinite dominance in them. Also, the reactive alumina and silica contents were lowest in Austin and Childless soils. Dermatas, 1995 reported that mineral Kaolinite releases more alumina during the hydration reactions compared to other clay minerals. Austin is high plasticity clay whereas Childress is high plasticity silt, both with mineral Kaolinite dominance over other minerals. High sulfate content and readily available alumina from mineral Kaolinite could be the reason for ineffectiveness of mellowing in Austin and Childress soils.

4.4 Conclusions

The following conclusions were drawn from the on-going discussion.

1. Volumetric shrinkage is not a concern in chemically treated high sulfate soils since all the lime treated soils exhibited low shrinkage strains compared to natural soils.
2. Effect of strength loss during mellowing is insignificant since the treated soil strengths were significantly higher than the untreated ones.
3. Swell behavior is more important in chemically treated high sulfate soils.

4. Four of the six high sulfate test soils could be stabilized using “pre-compaction” mellowing technique. Swell strains in these soils reduced below natural level with mellowing. Sulfate levels in these soils were below 30,000 ppm.
5. The soils that could not be stabilized by mellowing have sulfate contents above 30,000 ppm.
6. Gibbs free energy calculations indicated higher probability of Ettringite reactions over stabilizing reactions in Austin and Childress soils.
7. The above fact is confirmed by the mass-volume calculations showing Ettringite volume exceeding the void volume.
8. Compaction void ratio has significant influence on the Ettringite growth and subsequent heaving since soils with higher void ratios can accommodate the initial Ettringite growth.
9. Study of clay mineralogy is important since clay minerals like Kaolinite release more alumina to during sulfate reactions contributing to heave.
10. Reactive alumina and silica measurements are important since they help in pozzolanic compounds formation during stabilization reactions.
11. In soils with very high sulfate contents all the sulfate could not be dissolved during mellowing. This is the reason why soils with sulfate contents above 30,000 ppm could not be stabilized by mellowing.
12. Presence of high sulfate content, low reactive alumina and silica and low void ratios are the causes of higher swelling in Austin and Childress soils.

The next section describes combined lime and fly ash treatment to abate heaving in high sulfate soils.

CHAPTER 5

ANALYSIS OF LABORATORY TEST RESULTS (PHASE II)

5.1 Introduction

This chapter is focused on combined lime and fly ash studies on high sulfate soils. Portion of the lime was replaced with fly ash so as to limit the available calcium for Ettringite reactions. Fly ash is a by-product from burning coal, and its properties vary significantly with the source of the coal and the steps followed in burning the coal. Fly ash is broadly classified as class C fly ash and Class F fly ash. The main difference between the two types of fly ash is the availability of calcium. Class F fly ash has low calcium content compared to class C fly ash and needs an activator like lime or cement for effective stabilization. The type of fly ash used in the current study is Class F fly ash.

The Class F fly ash is obtained from Boral Materials technologies and is manufactured in the Monticello plant, Texas. The class F fly ash used in the current study meets ASTM C618 for Class F fly ash specification. Upon literature review, it was planned to use 1:2 lime and fly ash ratio. Different lime and fly ash dosages were tried in the laboratory for optimum performance in terms of strength gain and volume stability. These studies yielded lime and fly ash dosages of 4% and 8%, respectively. Stabilization using 4% lime and 8% fly ash will be referred as 4%L+8%FA in this chapter. A list of variables used in the present experimental program is presented in Table 5.1.

Two soils (Bells and Fm-1417) were chosen for the present combined lime and fly ash stabilization studies. These soils contained sulfate contents of 27,000 ppm and 24,000 ppm respectively and are classified as high sulfate soils. In this chapter, basic, chemical, physical, engineering test results obtained from the entire laboratory testing are provided. Two mellowing periods (0 days and 3 days) were considered in the current study. All the chemical and engineering tests were conducted on duplicate soil specimens for each variable condition. The results were averaged, and average values are reported. The results obtained are repeatable and consistent with minimal variance. The effects of chemical stabilizer, mellowing period, soluble sulfate content, soil clay mineralogy and compaction moisture contents on the swell and shrinkage characteristics of treated high sulfate soils are explained and discussed. The discussion findings are based on the majority of the trends noticed in test results of both untreated (control) soils and treated soils (lime and fly ash) at different mellowing periods. Results of the testing program are discussed in the subsequent sections.

Table 5.1 List of Variables

Description	Variable
Soils	Bells and FM-1417
Sulfate Contents	Two (27,000 ppm and 24,000 ppm)
Stabilizer	One (Lime and Class F fly ash)
Dosage	One (4%L+8%FA)
Moisture Contents	Two (Optimum, OMC and Wet of optimum, WOMC moisture content)
Mellowing Periods	Two (0 days and 3 days)

5.1.1 Basic Properties

The basic tests performed are Atterberg limits, specific gravity, sieve analysis, hydrometer analysis, sulfate content determination and Proctor compaction tests. Untreated and 4%L+8%FA-treated soils and Proctor curves are presented in the appendix. Results of the basic testing are summarized in Tables 5.2 thru 5. 5.

Table 5.2 Atterberg Limits and Classification of Soils

Soil	Atterberg Limits			USCS Classification
	LL	PL	PI	
Bells	68	32	36	CH
FM-1417	72	30	42	CH

Table 5.3 Summary of Basic Tests

Soil	Gradation, %				G_s	Sulfate Content	USCS Classification
	Gravel	Sand	Silt	Clay			
Bells	0	15	18	67	2.65	27,000	CH
FM-1417	0	9	20	71	2.66	24,000	CH

Table 5.4 Sulfate Content Determination

Soil Location	Soluble Sulfates, ppm
Bells	27,000
FM-1417	24,000

Table 5.5 Summary of Proctor Tests on Natural Soils

Soil Type	Natural (Untreated) Soil			
	Moisture Content (%)		Dry Density (lb/ft³)	
	OMC	WOMC	OMC	WOMC
Bells	21	28	93	88.3
FM-1417	27	34	89	84

5.1.2 Chemical Properties

The chemical tests performed were cation exchange capacity (CEC), specific surface area (SSA) and total potassium (TP). Based on these chemical measurements, clay mineral distribution was determined. Chemical properties and mineral distribution is presented in Table 5.6 and 5.7.

Table 5.6 Summary of Chemical Tests

Soil	Cationic Exchange Capacity (meq/100g)	Specific Surface Area (m^2/g)	Total Potassium (%)
Bells	76	170	1.4
FM-1417	65	280	0.8

Table 5.7 Percent Clay Minerals Observed for Soils under Study

Soil	% Illite	% Kaolinite	% Montmorillonite
Bells	25	30	45
FM-1417	13	20	67

5.1.3 Volume Change Characteristics

Three dimensional volumetric swell and shrinkage tests were conducted on untreated soils and used as a reference for comparison with volume changes in treated high sulfate soils at different periods of mellowing and onset of sulfate and stabilization reactions. The following section describes the results of the testing program.

5.1.3.1 Three Dimensional Volumetric Swell Test (3-D swell)

The 3-D free swell test measures the potential of the soil to swell in three (3) directions when soaked under water. The three (3) values measured were vertical, radial, and volumetric strains. Vertical swell was measured using a dial gauge placed at the top, and radial measurements were taken using pi tape at the end of test. Two identical specimens are used for each variable condition (OMC and WOMC). Table 8 shows the vertical, radial and volumetric swell strains of natural soils. Tables 9 and 10 show the average vertical, radial and volumetric swell strains of the 4%L+8%FA-treated soils at 0 and 3-day mellowing periods. Volumetric swell strains for lime-treated soils at 0 and 3-day mellowing periods is shown in Tables 5.11 and 5.12 respectively. Table 5.13 shows the summary of the volumetric swell tests on natural and treated soils at different mellowing periods. Vertical strain vs. elapsed time curve for Bells and FM-1417 soils at natural and treated condition (4%L+8%FA) are presented in Figures 5.1 and 5.2 respectively for optimum moisture (OMC) condition. Volumetric swell strains of natural and treated soils at different mellowing periods are shown in Figures 5.3 and 5.4.

Table 5.8 Vertical, Radial and Volumetric Swell Strains-Natural Soils

Soil Type	**Vertical Strain (%)**		**Radial Strain (%)**		**Volumetric Strain (%)**	
	OMC	**WOMC**	**OMC**	**WOMC**	**OMC**	**WOMC**
Bells	8.2	4.8	5.4	2.3	19	9.4
FM-1417	6.0	6.1	5.1	1.3	16.2	8.9

Table 5.9 Vertical, Radial and Volumetric Swell Strains-4%L+8%FA @ 0 days mellowing

Soil Type	Avg. Vertical Strain (%)		Avg. Radial Strain (%)		Avg. Volumetric Strain (%)	
	OMC	**WOMC**	**OMC**	**WOMC**	**OMC**	**WOMC**
Bells	4.7	3.8	2.9	2.4	10.5	8.6
FM-1417	4.6	4.0	2.3	1.9	9.2	7.8

Table 5.10 Vertical, Radial and Volumetric Swell Strains-4%L+8%FA @ 3 days mellowing

Soil Type	Avg. Vertical Strain (%)		Avg. Radial Strain (%)		Avg. Volumetric Strain (%)	
	OMC	**WOMC**	**OMC**	**WOMC**	**OMC**	**WOMC**
Bells	3.6	2.8	2.7	2.0	9	6.80
FM-1417	3.2	2.2	2.0	1.9	7.2	6.0

Table 5.11 Volumetric Swell Strains @ 0 day mellowing (4%L+8%FA)

Soil Type	Sample 1		Sample 2		Average Volumetric Swell Strain (%)	
	Volumetric Strain (%)		Volumetric Strain (%)			
	OMC	**WOMC**	**OMC**	**WOMC**	**OMC**	**WOMC**
Bells	10.7	8.8	10.3	8.4	10.5	8.6
FM-1417	9.2	7.4	9.3	8.2	9.2	7.8

Table 5.12 Volumetric Swell Strains @ 0 day mellowing (4%L+8%FA)

Soil Type	**Sample 1**		**Sample 2**		**Average Volumetric Swell Strain (%)**	
	Volumetric Strain (%)		**Volumetric Strain (%)**			
	OMC	**WOMC**	**OMC**	**WOMC**	**OMC**	**WOMC**
Bells	8.8	6.2	9.2	7.4	9	6.80
FM-1417	7.3	6.6	7.1	5.4	7.2	6.0

Table 5.13 Summary of Volumetric Swell Strains

Soil	**Volumetric Swell (%)**					
	Natural Soil		**4%L+8%FA, 0 days Mellowing**		**4%L+8%FA, 3 days Mellowing**	
	OMC	**WOMC**	**OMC**	**WOMC**	**OMC**	**WOMC**
Bells	19	9.6	10.5	8.6	9	6.80
FM-1417	16.2	8.9	9.2	7.8	7.2	6.0

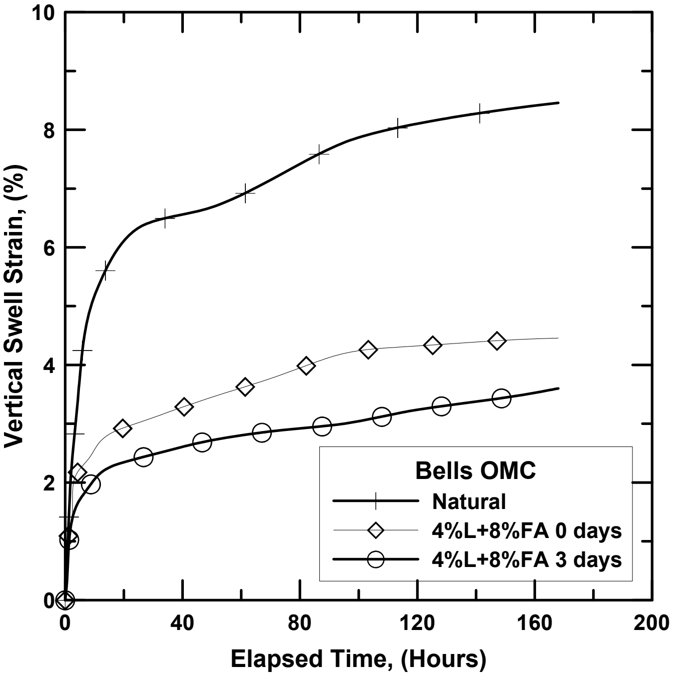

Figure 5.1 Vertical Swell vs. Elapsed Time (Bells Soil - 27,000 ppm)

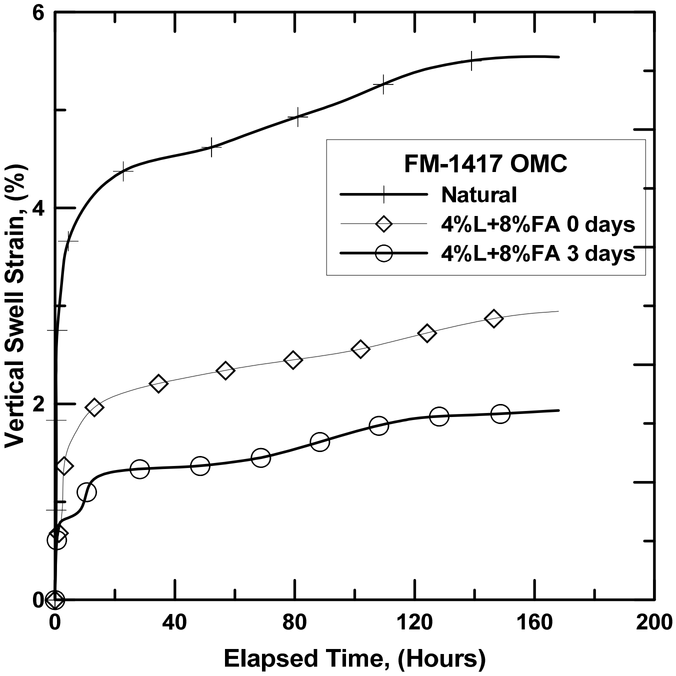

Figure 5.2 Vertical Swell vs. Elapsed Time (FM-1417 Soil - 27,000 ppm)

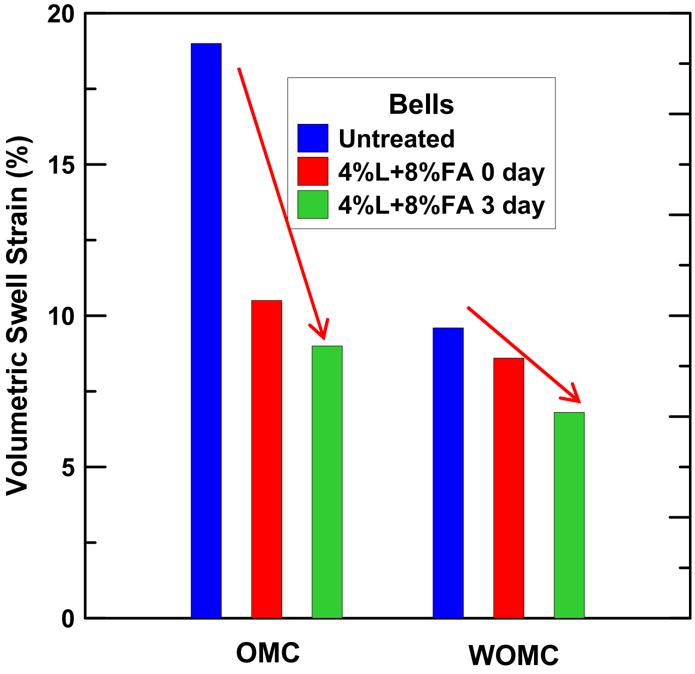

Figure 5.3 Volumetric Swell: Natural and Treated Bells Soil (27,000 ppm)

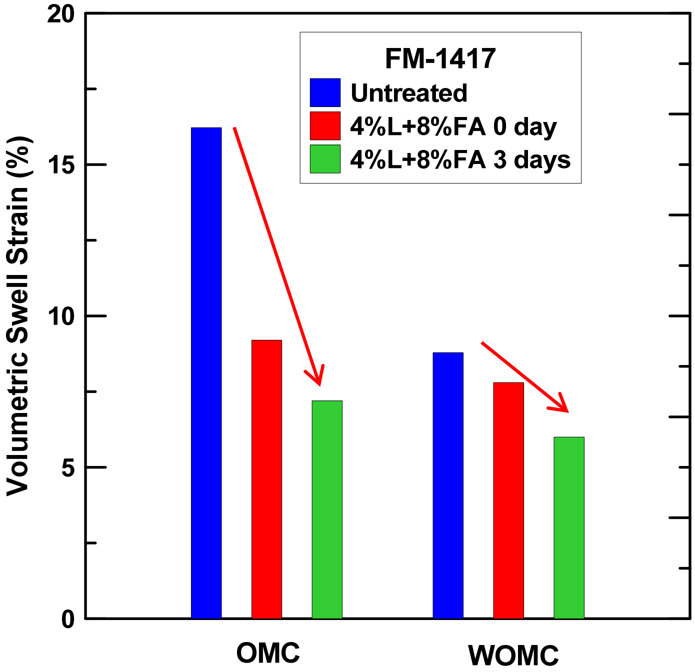

Figure 5.4 Volumetric Swell: Natural and Treated FM-1417 Soil (24,000 ppm)

It can be seen that in untreated condition, Bells and FM-1417 soils exhibited high swelling potential, with volumetric strain close to 20%. These two soils have volumetric swell strains (for the OMC condition) greater than than 10%, and this value indicates a high degree of expansion potential as per the problematic volumetric swell characterizations mentioned by Chen (1988). In addition, these four soils exhibited a PI value greater than 35.

With 0-day mellowing, combined lime and fly ash treatment yielded positive results in both the soils. Observed volumetric swells in Bells and FM-1417 soils were 10.5% and 9.2% respectively. With 3-day mellowing, swell strains further reduced in these soils. Measured swell strain values were 9% and 7.2% for Bells and FM-1417 soils, respectively. These swell strain levels can be considered as non-problematic. Both Bells and FM-1417 soils have sulfate contents below 30,000 ppm and thus responded positively for the lime and fly ash treatment. One of causes of treatment effectiveness is substitution of portion of lime with low calcium fly ash. The effects of clay mineralogy and void ratios will be discussed in the subsequent sections.

5.1.3.2 Three Dimensional Volumetric Shrinkage Test (3-D shrinkage)

Volumetric shrinkage tests were conducted to measure the decrease in the total volumetric shrinkage strain of soil specimens due to the loss of moisture content from the compacted state to a completely dry state. 3-D shrinkage tests were conducted on the 4%L+8%FA treated soils at different mellowing periods. Volumetric shrinkage strains of natural soils are presented in Table 5.14. Volumetric shrinkage strains at 0 day and 3 day mellowing periods are presented in Tables 5.15 and 5.16 respectively. Summary of volumetric shrinkage strains is presented in Table 5.17. Variation of volumetric shrinkage strain with the mellowing period is depicted in Figures 5.5 and 5.6 for Bells and FM-1417 soils respectively.

Table 5.14 Volumetric Shrinkage Strains of Natural Soils

Soil	**Average Volumetric Shrinkage Strain (%)**	
	OMC	**WOMC**
Bells	-14.4	-19.6
FM-1417	-15.6	-20.6

Table 5.15 Volumetric Shrinkage Strains of 4%L+8%FA Treated Soils @ 0 day mellowing

Soil	Sample 1		Sample 2		Average Volumetric Shrinkage Strain (%)	
	Volumetric Shrinkage Strain (%)		Volumetric Shrinkage Strain (%)			
	OMC	WOMC	OMC	WOMC	OMC	WOMC
Bells	-6.9	-8.4	-7.3	-8.6	-5.3	-8.6
FM-1417	-7.5	-8.9	-7.7	-9.0	-5.8	-8.7

Note: Negative sign indicate shrinkage.

Table 5.16 Volumetric Shrinkage Strains of 4%L+8%FA Treated Soils @ 3 day mellowing

Soil	Sample 1		Sample 2		Average Volumetric Shrinkage Strain (%)	
	Volumetric Shrinkage Strain (%)		Volumetric Shrinkage Strain (%)			
	OMC	WOMC	OMC	WOMC	OMC	WOMC
Bells	-9.8	-10.5	-9.7	-10.4	-6	-10.1
FM-1417	-6.4	-9.6	-6.1	-9.3	-6.3	-9.2

Note: Negative sign indicate shrinkage.

Table 5.17 Volumetric Shrinkage Strains Summary

Soil	Volumetric Shrinkage Strain (%)					
	Natural		0 day mellowing		3 day mellowing	
	OMC	WOMC	OMC	WOMC	OMC	WOMC
Bells	-14.4	-19.6	-5.3	-8.6	-6	-10.1
FM-1417	-15.6	-19.9	-5.8	-8.7	-6.3	-9.2

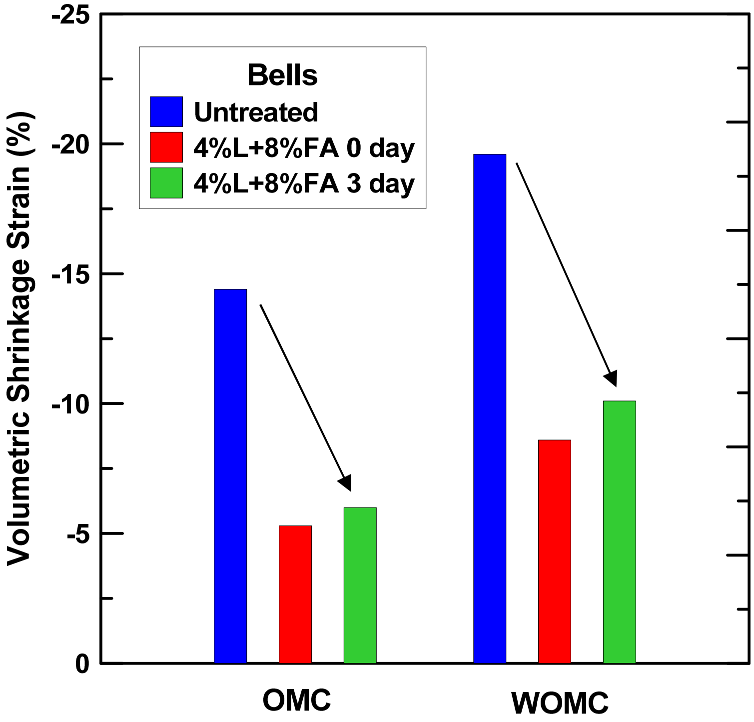

Figure 5.5 Volumetric Shrinkage Natural and Treated Bells Soil (27,000 ppm)

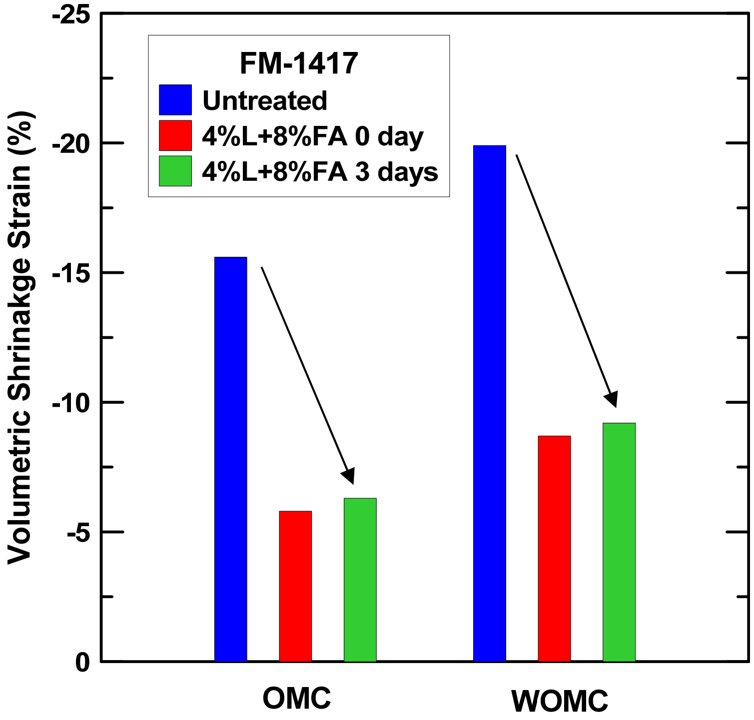

Figure 5.6 Volumetric Shrinkage Natural and Treated FM-1417 Soil (47,000 ppm)

It can be observed from Tables 5.14 thru 5.17 that the volumetric shrinkage strain is higher at wet of optimum moisture content condition than at optimum moisture content condition. The reason for this can be attributed to the presence of higher percentage of moisture availability in the soil specimen at wet of optimum condition when compared to optimum moisture content condition. Also, both the soils in this study are high plasticity clays, whose plasticity index (PI) values are greater than 40. These soils are capable of high swelling during the rainy season, as well as shrinkage during dry, summer-like conditions.

In 4%L+8%FA-treated soils, volumetric shrinkage strains invariably decreased. Shrinkage strains in Bells and FM-1417 soils were 5.3% and 5.8% respectively. Also, in both Bells and FM-1417 soils, there was a slight increase in volumetric shrinkage strain with 3-day mellowing when compared to the same at 0-day mellowing. Shrinkage strains in this case were 6% and 6.3% for Bells and FM-1417 soils respectively. The reasons for higher shrinkage in 3-day mellowed samples could be attributed to higher moisture content provided (additional 3% to cater for moisture loss during mellowing) for the 3-day mellowed soils. Overall, it can be concluded that presence of sulfates do not have any significant effect on volumetric shrinkage strain behavior of soils since shrinkage reduced in all the cases upon combined lime and fly ash treatment.

5.1.4 Strength Characteristics

Unconfined compression strength (UCS) tests were conducted on untreated soils and used as a reference for comparing the strength changes in treated high sulfate soils at different periods of mellowing onset of sulfate and stabilization reactions. Following section describes the results of the testing program.

5.1.4.1 Unconfined Compression Strength (UCS) Tests

The Unconfined Compression Strength (UCS) tests were conducted as per ASTM D 2166 method. The tests were conducted on compacted soil specimens of 2.8 inches in diameter and 5.6 inches in height. UCS tests were conducted on treated soils with different mellowing periods at two moisture conditions (OMC and WOMC) on two identical specimens. 4%L+8%FA-treated samples were allowed to mellow for 0 and 3 days. After the elapsed time periods, samples were compacted and cured in a 100% humidity room for 7 days. Results of the UCS testing on natural soils are presented in Table 5.18. Table 5.19 and Table 5.20 and show the UCS strengths of treated soils at 0-day and 3-day mellowing. Table 5.21 shows the summary of UCS tests. Figures 5.7, 5.8, 5.9 and 5.10 show the load versus deformation curve for natural and treated soils at different mellowing periods.

Table 5.18 UCS Tests Results (Natural Soils)

Soil	**Sample 1**		**Sample 2**		**Average UCS Strength, psi**	
	UCS Strength, psi		**UCS Strength, psi**			
	OMC	**WOMC**	**OMC**	**WOMC**	**OMC**	**WOMC**
FM-1417	31.3	19.3	33.3	17.7	32.3	18.5
Bells	28.5	18	33.1	17.6	30.8	17.8

Table 5.19 UCS Tests Results (4%L+8%FA, 0days mellowing)

Soil	**Sample 1**		**Sample 2**		**Average UCS, psi**	
	UCS , psi		**UCS psi**			
	OMC	**WOMC**	**OMC**	**WOMC**	**OMC**	**WOMC**
FM-1417	82.8	66.4	91.6	64.8	87.2	65.6
Bells	76.8	51	82.8	54.2	79.8	52.6

Table 5.20 UCS Tests Results (4%L+8%FA, 3 days mellowing)

Soil	**Sample 1**		**Sample 2**		**Average UCS, psi**	
	UCS Strength, psi		**UCS Strength, psi**			
	OMC	**WOMC**	**OMC**	**WOMC**	**OMC**	**WOMC**
FM-1417	79.8	49.6	81.8	55.2	80.8	52.4
Bells	77.4	52.3	71.8	50.1	74.6	51.2

Table 5.21 Summary of UCS Test Results

Soil	**UCS Strength, psi**					
	Natural		**4%L+8%FA_0 days**		**4%L+8%FA_3 days**	
	OMC	**WOMC**	**OMC**	**WOMC**	**OMC**	**WOMC**
FM-1417	32.3	18.5	87.2	65.6	80.8	52.4
Bells	30.8	17.8	79.8	52.6	74.6	51.2

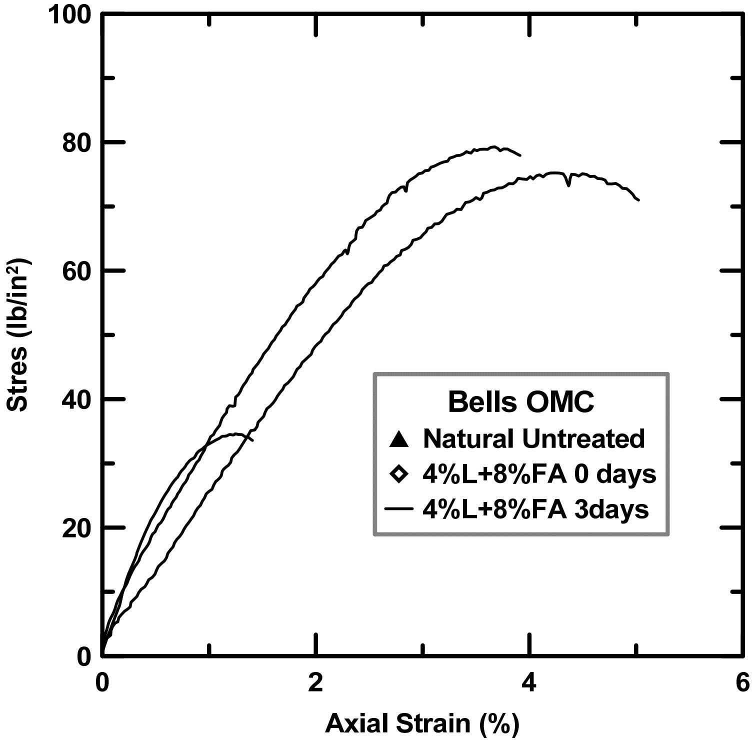

Figure 5.7 UCS Curve for Bells Soil @ OMC (27,000 ppm)

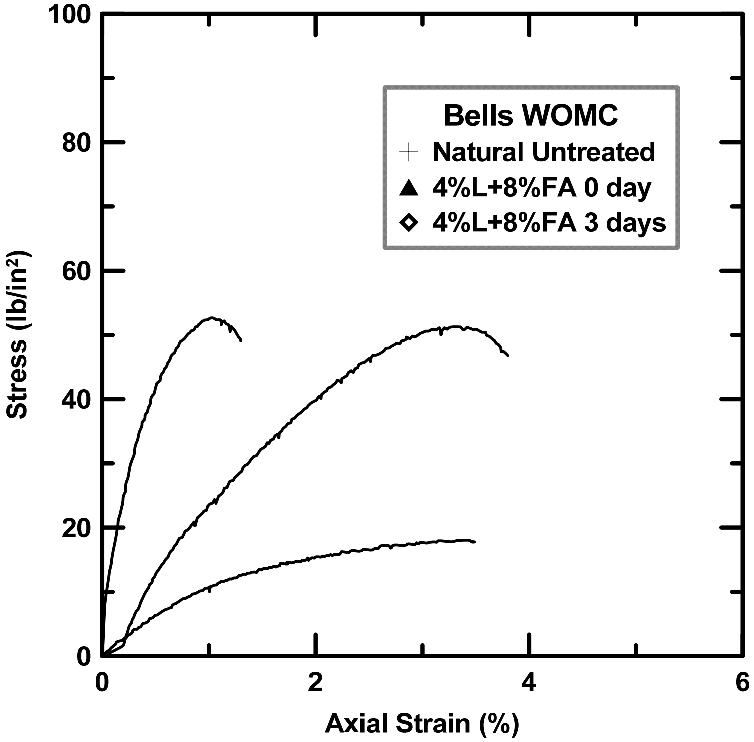

Figure 5.8 UCS Curve for Bells Soil @ wOMC (27,000 ppm)

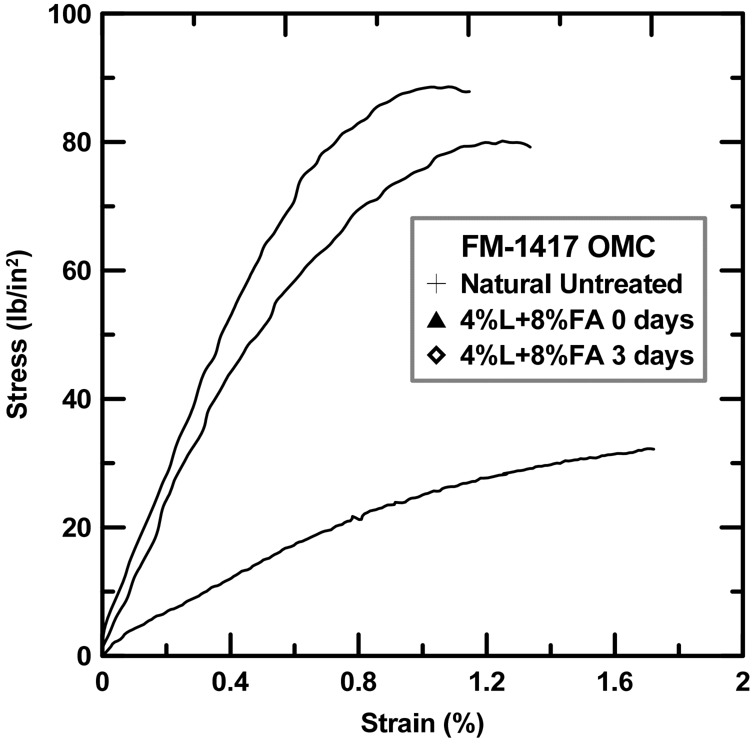

Figure 5.9 UCS Curve for FM-1417 Soil @ OMC (24,000 ppm)

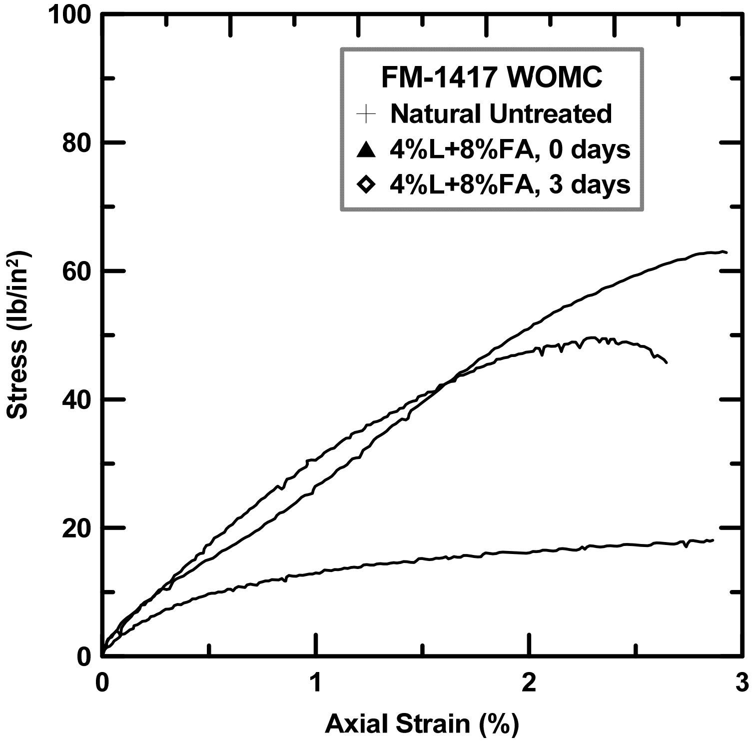

Figure 5.10 UCS Curve for FM-1417 Soil @ WOMC (24,000 ppm)

UCS strengths for untreated and 4%L+8%FA treated soils varied from 30.8 psi to 87.2 psi. For untreated condition, FM-1417 soil (32 psi) showed higher strength compared to Bells soil (30.8 psi). From the UCS test results, it can be seen that the UCS strengths of 4%L+8%FA-treated soils were two to three times higher than the untreated soils. Another important observation is that the UCS values of the 3-day mellowed samples were slightly lower than those of the 0-day mellowed samples. This strength loss can be attributed to the higher moisture content provided in mellowed samples and maximum dry density changes (low compaction density achieved due to mellowing) during the mellowing process. The loss in strength due to mellowing is still insignificant since the mellowed strength is considerably higher than the untreated soils strength.

5.1.4.2 1-D Swell Pressure Tests

The 1-D swell pressure test was conducted using the 1-D consolidation setup as per ASTM method (D4546-08 Standard Test Methods for One-Dimensional Swell or Collapse of Cohesive Soils). 1-D swell pressure tests were conducted on Bells and FM-1417 soils at natural and treated conditions at optimum and wet of optimum moisture conditions. Untreated swell pressures are presented in Table 5.22. Results of swell pressure testing are reported in Table 5.23 and Table 5.24 respectively for 0-day and 3-day mellowed soils. Table 5.25 shows the summary of swell pressures for natural and 4%L+8%FA-treated soils.

Table 5.22 Swell Pressures of Untreated Soils

Soil	**Average Swell Pressure, psi**	
	OMC	**WOMC**
Bells	36.4	22.3
FM-1417	37.1	16.2

Table 5.23 Swell Pressure of 4%L+8%FA Treated Soils @ 0 day mellowing

Soil Type	**Sample 1**		**Sample 2**		**Average Swell Pressure, psi**	
	Swell Pressure, psi		**Swell Pressure, psi**			
	OMC	WOMC	OMC	WOMC	OMC	WOMC
Bells	16.4	7.1	16.5	7.3	16.4	7.2
FM-1417	19.4	9.2	18.7	9.4	19.2	9.3

Table 5.24 Swell Pressure of 4%L+8%FA Treated Soils @ 3 day mellowing

Soil Type	**Sample 1**		**Sample 2**		**Average Swell Pressure, psi**	
	Swell Pressure, psi		**Swell Pressure, psi**			
	OMC	**WOMC**	**OMC**	**WOMC**	**OMC**	**WOMC**
Bells	11.3	3.7	11.1	3.3	11.2	3.5
FM-1417	14.0	5.2	11.6	4.0	12.8	4.6

Table 5.25 Summary of Swell Pressures

Soil Type	**Swell Pressure, psi**					
	Untreated		**4%L+8%FA, 0 day**		**4%L+8%FA, 3 day**	
	OMC	**WOMC**	**OMC**	**WOMC**	**OMC**	**WOMC**
Bells	36.4	22.3	16.4	7.2	11.2	3.5
FM-1417	37.1	16.2	19.2	9.3	12.8	4.6

It can be seen from the untreated swell data that both Bells and FM-1417 soils showed swell pressures which are indicative of expansive soils. Both the soils are high plasticity clays (CH) with plasticity index greater than 35. Bells and FM-1417 soils are dominant in the mineral Montmorillonite, which is a highly expansive mineral. The observed swell pressures were 36.4psi for Bells soil and 37.1psi for FM-1417 soil respectively. The observed swell pressures are in line with the volumetric swells measured from the 3-D swell tests.

In 4%L+8%FA-treated soils, with 0-day mellowing, the swell pressures of both soils were below the natural swell pressure. Combined lime and fly ash treatment successfully reduced the swell pressure in this case. The observed sell pressures were 16.4psi and19.1psi for Bells and FM-1417 soils respectively. With 3-day mellowing, 4%L+8%FA treatment reduced the swell further. The swell pressures recorded were 11.2 psi and 12.8 psi respectively for Bells and FM-1417 soils. The recorded swell pressures were in good agreement with the volumetric swell obtained from the 3-D swell tests. Another important observation is that both Bells and FM-1417 soils have sulfate contents below 30,000 ppm and responded positively to the 4%L+8%FA treatment.

5.1.5 Mineralogical Studies

Reactive alumina and silica measurements were made on both natural and treated soils at different mellowing periods to study the role of alumina and silica presence in sulfate heave and chemical stabilization reactions.

5.1.5.1 Reactive Alumina and Silica Measurements

Reactive alumina and silica measurements were conducted using ICP_MS (Inductively Coupled Plasma Mass Spectroscopy) on treated soils at different mellowing periods. Table 5.26 shows the reactive alumina and silica of the soils in natural condition. Reactive alumina and silica of 4%L+8%FA- treated soils at different mellowing periods is presented in Table 5.27. A summary of reactive alumina and silica measurements is given in Table 5.28.

Table 5.26 Reactive Alumina and Silica Measurements Natural Soils

Soil	Reactive Alumina (ppm)	Reactive Silica (ppm)
Bells	370.2	434.7
FM-1417	279.2	137.3

Table 5.27 Reactive Alumina and Silica Measurements @ 0 day and 3 day mellowing

Soil	0 day mellowing				3 day mellowing			
	Al* (ppm)	Si* (ppm)	Al loss (%)	Si loss (%)	Al* (ppm)	Si* (ppm)	Al loss (%)	Si loss (%)
Bells @ OMC	270.2	126.0	27	71	275.6	130.3	25.6	70.0
Bells @ WOMC	281.0	134.8	24.1	69	289.2	141.3	21.9	67.5
FM-1417 @ OMC	182.0	58.6	34.8	57.3	186	63.1	33.4	54.0
FM-1417 @ WOMC	189.0	67.9	32.3	50.5	194	70.2	30.5	48.9

Table 5.28 Summary of Reactive Alumina and Silica Measurements (at OMC)

Soil	Natural		0 day mellowing		3 day mellowing	
	Al	Si	Al	Si	Al	Si
Bells	370.2	434.7	270.2	126.0	275.6	130.3
FM-1417	279.2	137.3	182.0	58.6	186	63.1

From Table 5.26, it can be observed that both Bells and FM-1417 soils have high alumina and silica contents. Presence of higher alumina and silica indicate higher sulfate and stabilization reactions. Presence of higher silica content in both the soils causes the dominance of pozzolanic reactions over the Ettringite formation reactions. It was reported in the literature that silica released during the hydration reactions consumes the calcium, forming the pozzolanic compounds and thereby robbing the constituents required for Ettringite reactions (Tasong et al., 1999).

The percentage loss of alumina and silica due to lime treatment at different mellowing periods was also calculated and compared with the natural alumina and silica contents. The alumina and silica loss was higher at OMC compared to WOMC for both the soils considered in the study. Also, the loss of alumina and silica was higher at 0-day mellowing period compared to the 3day mellowing period. The observed volumetric swells were higher in 0-day mellowed soils than the 3-day mellowed soils.

Another important observation is that the loss of silica in was higher than the loss of alumina in 4%L+8%FA-treated soils at both 0 day 3-day mellowing periods. Both the soils could be stabilized successfully using the combination of lime and fly ash. In combined lime and fly ash systems, the availability of calcium is limited by the replacement of lime with fly ash. The presence of higher silica and high silica losses in treated soils means the stabilization compounds were formed in the early periods of hydration, leading to the strengthening of the soil matrix in both the soils. This is the reason why both the soils could be stabilized successfully using 4%L+8%FA.

5.2 Discussion

The two high sulfate soils considered in the current study could be stabilized successfully using the pre-compaction mellowing technique. These two soils (Bells and FM-1417) have sulfate contents below 30,000 ppm. The effect of clay mineralogy and compaction void ratios was studied and presented in the following section.

5.2.1 Effect of Compaction Void Ratio on Swell Strains

To study the effect of compaction void ratio (e) on swell behavior, void ratios of the tests soils in compacted state were calculated. Using the specific gravity and maximum dry density from the Proctor curve, void ratios of the test soils were calculated at both optimum moisture content (OMC) and wet of optimum moisture content (WOMC). Void ratios of the test soils are presented in Table 5.29.

Table 5.29 Void Ratios of Test Soils

Soil	**Sulfate Level, ppm**	**Compaction Void Ratio (e)**	
		OMC	**WOMC**
Bells	27,000	0.80	0.90
FM-1417	24,000	0.86	0.96

From Table 25, it can be seen that both the Bells and FM-1417 soils have high void ratios. High void ratio means less dense soil matrix. Also due to mellowing, maximum dry density decreases, which means the soil matrix has more void space to accommodate the Ettringite formation and growth. The sulfate contents in both the soils are below 30,000 ppm. The reason for effectiveness of combined lime and fly ash treatment could be attributed to one of the following: either whole sulfate has been consumed during the mellowing process or combined lime and fly ash treatment resulted in early pozzolanic reactions which dominated the Ettringite formation reactions leading to lower swell magnitudes. Combination of high void ratios, sulfate contents below 30,000 ppm are the reasons why mellowing is effective in these two soils.

5.2.2 Effect of Clay Mineralogy on Swell Strains

Clay mineralogy of the test soils was determined from the cation exchange capacity (CEC), specific surface area (SSA) and total potassium (TP) measurements. Determination of clay mineralogy gives an insight into what types of clay minerals are present and their reactivity with respect to pozzolanic and sulfate reactions. Clay mineral distribution of the test soils is presented in Table 30.

Table 5.30 Percent Clay Minerals Observed for Soils under Study

Soil Region	% Illite	% Kaolinite	% Montmorillonite
Bells	25	30	45
FM-1417	13.2	20.3	66.5

From Table 27, it can be seen that both Bells and FM-1417 soils have mineral Montmorillonite dominance in them. It was reported in the literature that mineral Montmorillonite releases less alumina during hydration reactions compared to mineral Kaolinite (Dermatas, 1995). Dominance of Montmorillonite mineral, high void ratios, low calcium availability (due to class F fly ash addition) and sulfate contents below 30,000 ppm could be the reasons for success of mellowing in Bells and FM-1417 soils.

5.3 Observations

The following observations were drawn from the on-going combined lime and fly ash treatment studies.

1. Volumetric shrinkage decreased with combined lime and fly ash treatment in high sulfate soils and hence shrinkage is not an issue.
2. Effect of strength loss due to mellowing is insignificant since the 4%L+8%FA treated soils exhibited much higher strengths than the untreated samples.
3. Two high sulfate soils considered in the current study could be stabilized using “pre-compaction” mellowing technique. Swell strains in these soils reduced below natural level with mellowing.
4. The two soils (Bells and FM-1417) have sulfate contents below 30,000 ppm and contained high void ratios and these also resulted in lesser sulfate induced heaving after chemical treatment.
5. Montmorillonite dominance is one of the reasons for mellowing effectiveness in both Bells and FM-1417 soils.

CHAPTER 6

SWELL PREDICTION MODELS

6.1 Introduction

Swell tests conducted on lime treated high sulfate soils indicated that these soils are prone to "Ettringite Induced Heaving" when treated with calcium based stabilizers. In the first part of the research program, pre-compaction mellowing technique with two different mellowing periods (3 days and 7 days) was used to stabilize high sulfate soils (sulfate contents > 8,000 ppm). Results of the testing indicated that mellowing was effective in four of the six soils considered in the current study. The two soils that could not be stabilized even at higher mellowing periods are Austin and Childress soils. The applicability of mellowing technique in four of the six soils is explained using the Gibbs free energy calculations and mass volume relationships.

In the second part of the research program, two soils with sulfate contents above 20,000 ppm are considered for the combined lime (L) and fly ash (FA) treatment. These soils were treated with 4%L and 8%FA and subjected to swell tests at 0 day and 3 day mellowing periods. Results of the swell tests indicated that the combined lime and fly ash treatment effectively reduced volumetric swells below natural levels. The two soils considered in this study contained sulfate contents below 30,000 ppm and hence are good candidates for this analysis. In addition to effective treatment by lime-fly ash, high void ratios of these two soils may have contributed to less heaving as such voids could accommodate any Ettringite formation and its growth within the soil void matrix.

In this section two swell prediction models based sulfate contents and compaction void ratios are presented. Swell prediction model developed based on the sulfate content value using an approach outlined by Little et al. (2010) is presented and comparisons are made between the predicted volumetric swell and measured volumetric swell strains. Based on the mass volume calculations and initial compaction void ratio of the soils, swell prediction model based on threshold void ratio frame work and threshold void ratios for 3 day and 7 day mellowing periods are presented in the last section of the chapter.

6.2 Swell Prediction Models

In this section, two models for swell prediction are presented. The first model is based on the sulfate contents. Since sulfate content is the main constituent of Ettringite formation reactions, it is used as the limiting reagent for prediction of swell. In the second model, threshold void ratio and sulfate content of the soil are used to predict the Ettringite-induced heaving in treated high sulfate soils.

6.2.1 Swell Prediction Model Based on Sulfate Content of the Soil

Using sulfates as the limiting reagent, Dallas et al., 2010 calculated maximum possible Ettringite formation in any soil system using stoichiometric calculations. Their calculations were based on 1% soluble sulfates (10,000 ppm or 10,000 mg/Kg) of the natural soil. The calculations were also based on the assumption that the entire available sulfate is consumed during the Ettringite formation reactions, which may not be true. For higher sulfate concentrations, the maximum possible Ettringite can be calculated by expressing the sulfate concentration in the order of 10,000 ppm and multiplying. The following methodology is adopted for calculation of maximum volumetric swell strains: 3 moles of sulfate (SO_4) is consumed in the formation of 1 mole of Ettringite as per the flowing chemical reaction:

$$6Ca^{2+} + 2Al(OH)_4^- + 4(OH)^- + 3SO_4^{2-} + 26H_2O \rightarrow \ Ca_6[Al(OH)_6]_2.(SO_4)_3.26H_2O \qquad (1)$$

Based on the molar volume calculations, the maximum possible Ettringite percent based on the dry weight of soil can be estimated based on the following steps:

Step 1: 1% soluble sulfates is equivalent to 10 g of SO_4/1000 g of soil

Step 2: 1 mole of SO_4 is equivalent to 96 g of SO_4

Step 3: 3 moles of SO_4 participates in the reaction to from 1 mole of Ettringite

Step 4: 1 mole of Ettringite, E weighs 1254 gm

Combining Steps 1 to 4 we have,

$$\frac{10g\ SO_4}{1000\ g\ of\ soil} X \frac{1\ mol\ SO_4}{96\ g\ of\ SO_4} X \frac{1\ mol\ \ E}{3\ mol\ SO_4} X \frac{1254\ g\ of\ E}{1\ mol\ E} = \frac{4.36\ g\ of\ E}{100\ g\ of\ Soil} = 4.36\%\ E$$

Volume change or volumetric swell/shrinkage strain due to formation of Ettringite mineral can be calculated as the ratio of difference between the molar volume of products and reagents divided by the molar volume of reagents and this strain is expressed as a percentage. Based on these calculations it was observed that if the Ettringite formation reaction consumes water from the soil matrix, then the resultant volume change in the soil is usually shrinkage since the molar volume of reagents is higher. If the water required for Ettringite formation is derived from outside the soil matrix then the resultant volume change in the soil is expansion or soil heave. This is explained by the Dallas et al., 2010 as follows:

Ettringite formation reactions can be shown as:

$$C_3A + 3C\bar{S}H_2 + 26H_2O \rightarrow C_3A.3C\bar{S}H_2.H_{26} \qquad (2)$$

Where, C_3A is Tricalcium Aluminate; CSH_2 is Gypsum; H is water and $C_3A.3CSH_2.H_{26}$ is Ettringite.

From Stoichiometry, for 1 mole of Ettringite formation, 1 mole of C_3A (Tricalcium Aluminate), 3 moles of CSH_2 (Gypsum) and 26 moles of H (Water) are required. Molar volume of C_3A is 89.1cm^3/mol, CSH_2 is 74.2cm^3/mol, H_2O is 18.02cm^3/mol and Ettringite is 737.6cm^3/mol. The volume change (Volumetric strain, V %) within the soil matrix when one mole of C_3A combines with 3 moles of Gypsum to form Ettringite by consuming water within the soil matrix can be given as:

$$V = \frac{(MV_p - MV_r)}{(MV_p)} \times 100 \qquad (3)$$

Where, V = Volumetric Strain (%), MV_p = Molar Volume of Products, MV_r = Molar Volume of Reagents.

Now, Equation 8 can be used to calculate the volumetric strain for matrix water source (V_M) and external water source (V_E) as follows:

$$V_M = (737.6 - 311.7 - 468.52/780.22) \times 100 = -5\% \qquad (4)$$

In Equation 9, volumetric strain in negative sign indicates potential shrinkage strain of Ettringite. However, when the water is consumed, in the Ettringite formation, which is derived from outside the soil matrix, the molar calculations indicate volume increase (swell) instead of shrinkage. This is represented in Equation 5.

$$V_E = ((737.6 - 311.7)/311.7) \times 100 = 137\% \qquad (5)$$

In Equation 5, volumetric strain in positive sign indicates swelling of Ettringite. It can be observed that the Ettringite formation is accompanied by the expansion of the soil system in which it forms since the molar volume of products (Ettringite) is greater than the molar volume of reactants. This expansion is observed as most of the sulfate heave case histories in the literature reported heaving following a continuous rainfall event after the construction.

Based on this assumption Dallas et al. (2010) calculated the maximum possible volume change in soil due to the formation of Ettringite mineral as 137% (expansion). For a soil with 10,000 ppm of sulfates from Equation 7 we have the maximum possible Ettringite that can be formed as 4.36%E. From volume change calculations it is known that Ettringite mineral formation is accompanied by 137% expansion. Hence the maximum possible soil swelling from Ettringite mineral formation and expansion is calculated as = 4.36% X 1.37= 5.97% (10,000 ppm of sulfates lead to formation of 4.36% E and 1 mole of Ettringite causes 137% expansion). As mentioned above, swell calculations based on sulfate content alone assuming the entire available sulfates are consumed in the Ettringite formation reaction may not be practical. With this objective in the current swell prediction model "effective sulfate" content rather than the actual sulfate content is used. For idealized condition, the maximum possible Ettringite formation and the corresponding shrinkage/swell strains for the soils considered in the current study are presented in Table 7.13. The following assumptions are made in the calculation of maximum possible swell strains using the current model:

1. Sulfate content is the only factor influencing the formation of Ettringite.
2. It is assumed that the amount of sulfates lost during the swelling process is the amount of sulfates that contributed towards the Ettringite induced soil swelling. Sulfate measurements were conducted on the treated soil samples after they were subjected to swell testing. The final sulfate content is deducted from the natural sulfate content to obtain the "effective sulfate content" that actually participated in the Ettringite formation reaction.
3. In the current swell prediction model based on the "effective sulfate content" approach, sulfate contents at 0 day mellowing were not considered since the treated volumetric

swell strains at 0 day mellowing (or no mellowing) are higher than the natural swell in all the soils considered except Austin soil.

Natural sulfate contents and effective sulfate contents at 3 day and 7 day mellowing periods are presented in Table 6.1and 6.2 at optimum (OMC) and wet of optimum moisture contents (WOMC) and these results are used in the current modeling analysis.

Based on the effective sulfate contents the maximum possible volumetric swell due to Ettringite formation is calculated at 3 day and 7 day mellowing periods. Table 6.3 and 6.4 shows the measured swell strains (from lab studies) and predicted swell strains based on the swell prediction model for 3 day and 7 day mellowed soils respectively. Comparison of measured and predicted volumetric swell for 0 and 7 day mellowing periods is presented in Figures 6.1 and 6.2.

Table 6.1 Swell/Shrinkage Predictions based on Idealized Model

Soil	Natural Sulfate Content (ppm)	Ettringite Formed (%)	Water from the matrix		Water outside the matrix	
			Unit Volume Change (%)	Total Shrinkage Strain (%)	Unit Volume Change (%)	Total Swell Strain (%)
Austin	36,000	15.70	-0.05	-0.78	1.37	21.50
Childress	44,000	19.18	-0.05	-0.96	1.37	26.28
Dallas	12,000	5.23	-0.05	-0.26	1.37	7.17
FM-1417	24,000	10.46	-0.05	-0.52	1.37	14.34
Riverside	20,000	8.72	-0.05	-0.44	1.37	11.95
US-82	12,000	5.23	-0.05	-0.26	1.37	7.17

Table 6.2 Effective Sulfate Contents of Treated Soils

(@ 3 and 7 day mellowing periods)

Soil	**Natural Sulfate Content (ppm)**	**Effective Sulfate Content (ppm)**			
		3 day mellowing		**7 day mellowing**	
		OMC	**WOMC**	**OMC**	**WOMC**
Austin	36,000	24480	24520	18720	18900
Childress	44,000	19800	20000	15840	15960
Dallas	12,000	9600	10000	9000	9400
FM-1417	24,000	17280	17500	12000	12400
Riverside	20,000	13800	14000	11600	11800
US-82	12,000	9480	9600	8520	8700

Table 6.3 Measured vs. Predicted Swell Strains

(3 day mellowing period)

Soil	**3 day mellowing**			
	Measured Swell (%)		**Predicted Swell (%)**	
	OMC	**WOMC**	**OMC**	**WOMC**
Austin	11.6	9.8	14.6	14.6
Childress	10.2	8.5	11.8	11.9
Dallas	10.4	6.4	5.7	6.0
FM-1417	10.2	7	10.3	10.5
Riverside	9	8.4	8.2	8.4
US-82	11.3	6.7	5.7	5.7

Table 6.4 Measured vs. Predicted Swell Strains
(7 day mellowing period)

Soil	**7 day mellowing**			
	Measured Swell (%)		**Predicted Swell (%)**	
	OMC	**WOMC**	**OMC**	**WOMC**
Austin	18.2	12.1	11.2	11.3
Childress	11.2	9.9	9.5	9.5
Dallas	9	6.9	5.4	5.6
FM-1417	6.7	5.4	7.2	7.4
Riverside	7.2	5.3	6.9	7.0
US-82	9.8	6.3	5.1	5.2

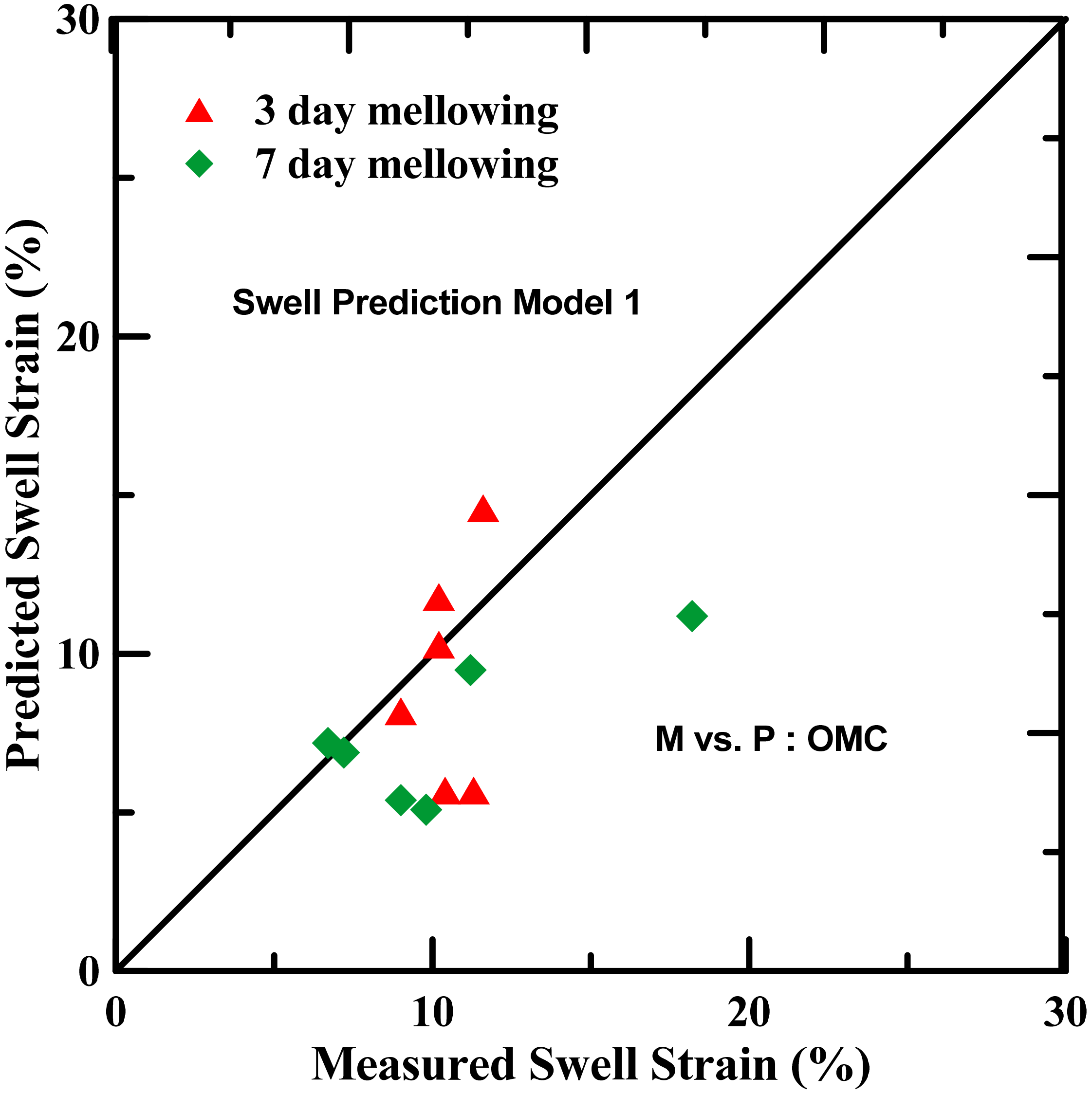

Figure 6.1 Measured vs. Predicted Swell Strains (OMC); Sulfate Content Based Model

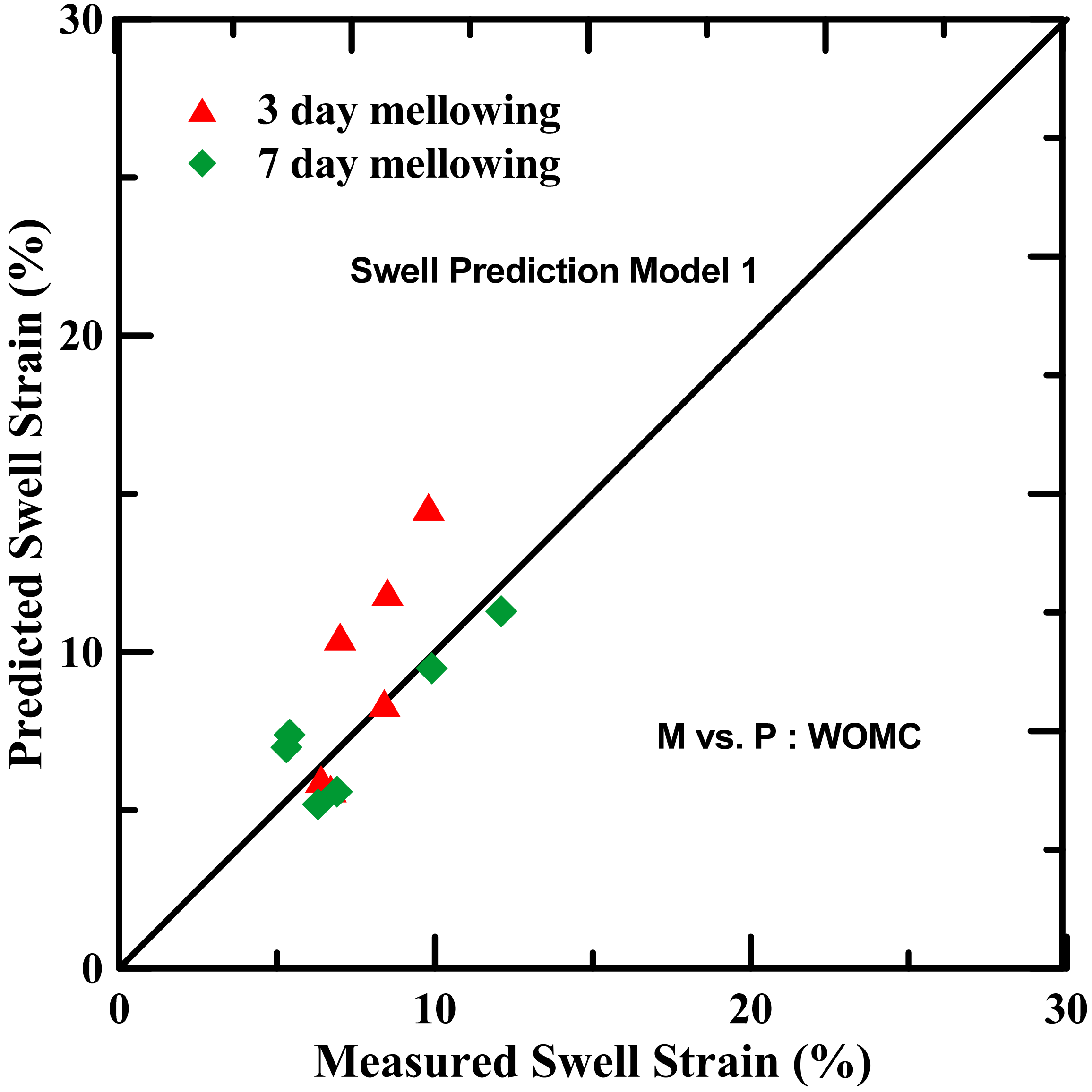

Figure 6.2 Measured vs. Predicted Swell Strains (WOMC); Sulfate Content Based Model

From the Figures 6.1 and 6.2 it can be seen that at optimum moisture content (OMC) the data points are scattered with respect to the control 1:1 line whereas at wet of optimum moisture content, the data points are slightly closer to the control line. Also, in all the test soils at 7 day mellowing the predicted swell strains decreased when compared to 3 day mellowing periods since the predicted swell strain is directly related to the sulfate content present in the soil. This is not true in case of measured swell values since in Austin and Childress soils the measured swell values increased with 7 day mellowing. To address all the six soils considered for the current study, these two soils are also included in the current analysis.

Another important observation is that the predicted swell is higher at wet of optimum compared to optimum moisture content due to higher sulfate contents which is converse to the actual observations. From the analysis above it can be concluded that the swell prediction based on sulfate contents is only an indicator but not actual representation of the real field swell.

The above mentioned analysis for prediction of maximum possible swell strain is based on the reactive sulfate content only. It can be seen from the Equation 1 that Ettringite formation is dependent on the alumina concentration in the treated soil. To study the effects of alumina content alone on the Ettringite formation reaction, similar type of analysis is performed in the current study. For the soils considered in the current study, the maximum amount of Ettringite that could be formed is calculated as 0.196% which is 20 times lower than amount of Ettringite formed based on sulfate content (4.36%). Hence, it is understood that Ettringite swell strain is more dependent on the sulfate contents than alumina content. Other important factors such as compaction void ratio that influence Ettringite induced heave are discussed in the heave assessment studies.

6.2.2 Swell Prediction Model Based on Threshold Void Ratio Frame Work

Based on the experimental data and mass volume calculations the ‘Threshold’ void ratios for Ettringite induced heaving are derived and presented in the current model. The two most important factors that influence the Ettringite induced heave are sulfate contents and compaction void ratios. When the sulfate contents are high and void space is low it is likely that Ettringite induced heave occurs. Hence the effect of both parameters needs to be included in the analysis. Effect of mellowing on Ettringite induced heaving in lime treated high sulfate soils is included in the current model. The following steps are followed in establishing the threshold void ratio frame work for treated soils.

Step 1: Sulfate content of the soil in ppm or mg/Kg is converted as the ratios of volume of sulfate ($V_{sulfate}$) and volume of soil (V_{soil}) as:

$$\frac{V_{Sulfate}}{V_{Soil}} = \frac{Sulfate\ Conc.in\ ppm * 10^{-6}}{\gamma_{Gypsum}\ in\ {}^{kg}/_{m^3}} * \gamma_{Soil}\ in\ {}^{kg}/_{m^3} \tag{6}$$

Step 2: Based on the unit phase diagram approach, initial void volume per ft^3 of soil (V_{vi}) is calculated from the void ratios at optimum (OMC) and wet of optimum (WOMC) moisture contents as follows:

$$Void\ Ratio\ (e) = \frac{V_{vi}}{V_{Solids}} = \left(\frac{V_{vi}}{V - V_{vi}}\right) \tag{7}$$

$$\left(\frac{V - V_{vi}}{V_{vi}}\right) = \frac{1}{e} \tag{8}$$

$$\left(\frac{V}{V_{vi}}\right) = 1 + \frac{1}{e} \tag{9}$$

$$\left(\frac{V_{vi}}{V}\right) = \frac{1}{\left(1 + \frac{1}{e}\right)} \tag{10}$$

$$V_{vi} = \frac{1}{\left(1 + \frac{1}{e}\right)}; (V = 1, unit\ volume) \tag{11}$$

Step 3: From Table 4.27 and 4.28, the volume of Ettringite per ft^3 (V_e) of treated soil at both optimum and wet of optimum moisture contents are obtained. Ratio of the V_e and V_{vi} is calculated. If the ratio is greater than one, the Ettringite cannot be accommodated in the void space of the soil and hence higher swelling is observed. For ratios less than unity the Ettringite swell is not observed. Ratio of V_e and V_{vi} equal to one is considered as the basis for choosing the threshold void ratio.

Step 4: Ratio between V_e/V_{vi} and $V_{sulfate}/V_{soil}$ is plotted and a horizontal line is drawn at the V_e/V_{vi} equal to one and where the horizontal line touches the trend line, a vertical line is dropped from that point to the horizontal axis and the value of $V_{sulfate}/V_{soil}$ is read from the plot.

A sample calculation of $V_{sulfate}/V_{soil}$ and V_e/V_{vi} for Austin soil at 3 day mellowing period is given below:

Sulfate Concentration 36,000 ppm; γ_{soil} = 1696.1 kg/m^3; γ_{Gypsum} = 2300 kg/m^3; e = 0.54; V_e= 0.49 (From Table 12)

$$\frac{V_{Sulfate}}{V_{Soil}} = \frac{36000 * 10^{-6}}{2300} * 1696.1 = 0.0265$$

$$V_{vi} = \frac{1}{\left(1+\frac{1}{e}\right)} = \frac{1}{\left(1+\frac{1}{0.54}\right)} = 0.35$$

$$\frac{V_e}{V_{vi}} = \frac{0.49}{0.35} = 1.397$$

These parameters are presented in Table 6.5 and 6.6 at optimum and wet of optimum moisture contents for 3 day mellowed soils. Plots between $V_{sulfate}/V_{soil}$ and V_e/V_{vi} at optimum and wet of optimum moisture contents are presented in Figures 6.3 and 6.4 respectively. Similar analysis was performed on 7 day mellowed samples. $V_{sulfate}/V_{soil}$ and V_e/V_{vi} are presented in Table 6.7 and 6.8 at optimum and wet of optimum moisture content for 7 day mellowed soils. Plot between $V_{sulfate}/V_{soil}$ and V_e/V_{vi} at optimum and wet of optimum moisture contents are presented in Figures 6.5 and 6.6 respectively for 7 day mellowed soils.

Table 6.5 $V_{sulfate}/V_{soil}$ and V_e/V_{vi} at OMC (3 day mellowing)

Soil	**$V_{sulfate}/V_{soil}$**	**V_e/V_{vi}**
Austin	0.026	1.397
Childress	0.031	1.695
Dallas	0.007	0.767
FM-1417	0.014	0.908
Riverside	0.013	0.950
US-82	0.007	0.666

Note: $V_{sulfate}$ = Volume of Sulfate; V_{soil} = Volume of Soil; V_e = Volume of Ettringite; V_{vi} = Initial Void Volume

Table 6.6 $V_{sulfate}/V_{soil}$ and V_e/ V_{vi} at WOMC (3 day mellowing)

Soil	$V_{sulfate}/V_{soil}$	V_e/V_{vi}
Austin	0.025	1.097
Childress	0.030	1.227
Dallas	0.007	0.623
FM-1417	0.014	0.510
Riverside	0.012	0.980
US-82	0.007	0.525

Note: $V_{sulfate}$ = Volume of Sulfate; V_{soil} = Volume of Soil; V_e = Volume of Ettringite; V_{vi} = Initial Void Volume

Table 6.7 $V_{sulfate}/V_{soil}$ and V_e/ V_{vi} at OMC (7 day mellowing)

Soil	$V_{sulfate}/V_{soil}$	V_e/V_{vi}
Austin	0.026	1.425
Childress	0.031	1.715
Dallas	0.007	0.656
FM-1417	0.014	0.854
Riverside	0.013	0.908
US-82	0.007	0.588

Note: $V_{sulfate}$ = Volume of Sulfate; V_{soil} = Volume of Soil; V_e = Volume of Ettringite; V_{vi} = Initial Void Volume

Table 6.8 $V_{sulfate}/V_{soil}$ and V_e/V_{vi} at WOMC (7 day mellowing)

Soil	$V_{sulfate}/V_{soil}$	V_e/V_{vi}
Austin	0.025	1.250
Childress	0.030	1.305
Dallas	0.007	0.598
FM-1417	0.014	0.490
Riverside	0.012	0.945
US-82	0.007	0.485

Note: $V_{sulfate}$ = Volume of Sulfate; V_{soil} = Volume of Soil; V_e = Volume of Ettringite; V_{vi} = Initial Void Volume

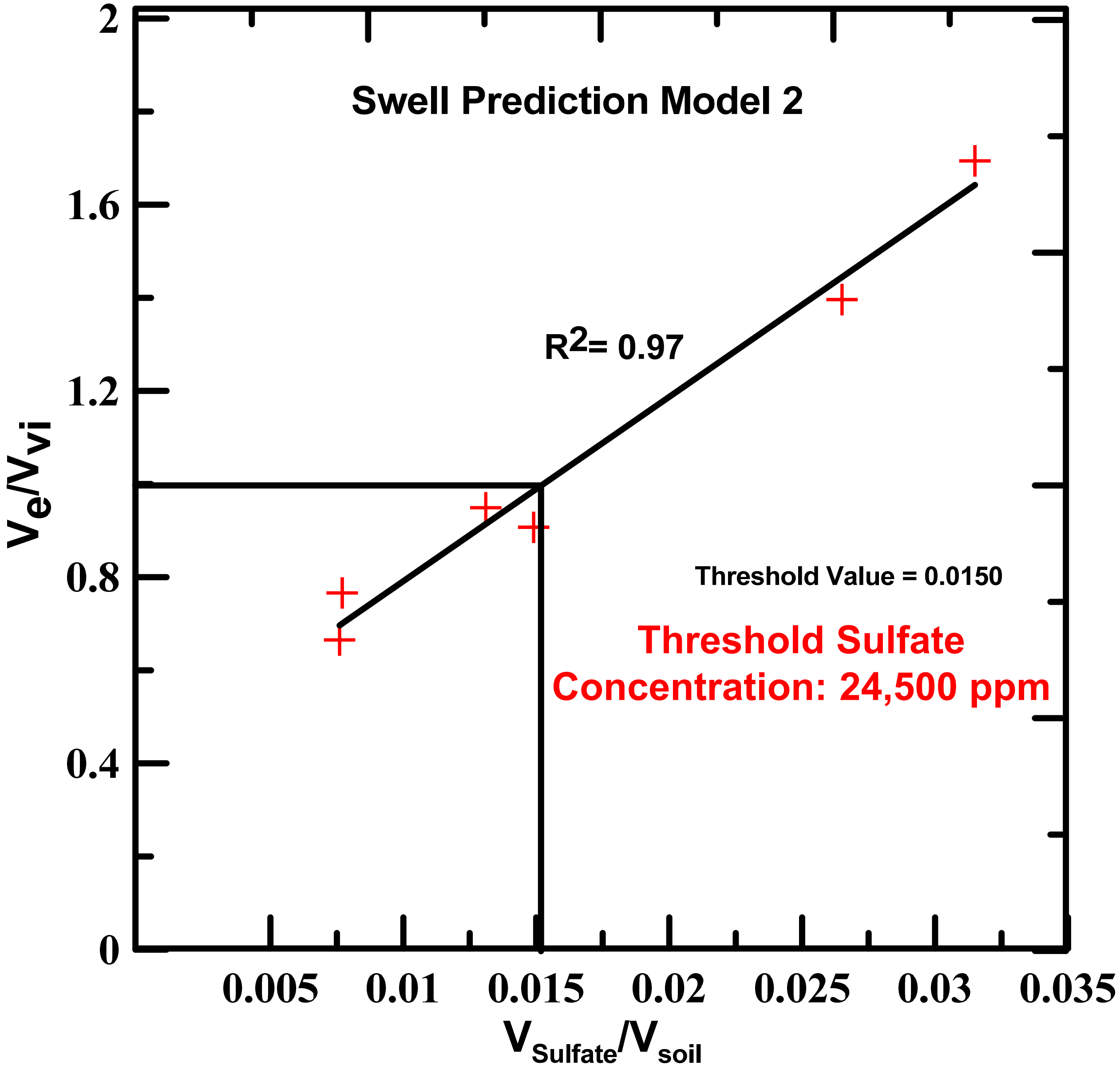

Figure 6.3 $V_{sulfate}/V_{soil}$ vs. V_e/V_{vi} at OMC (3 days mellowing)

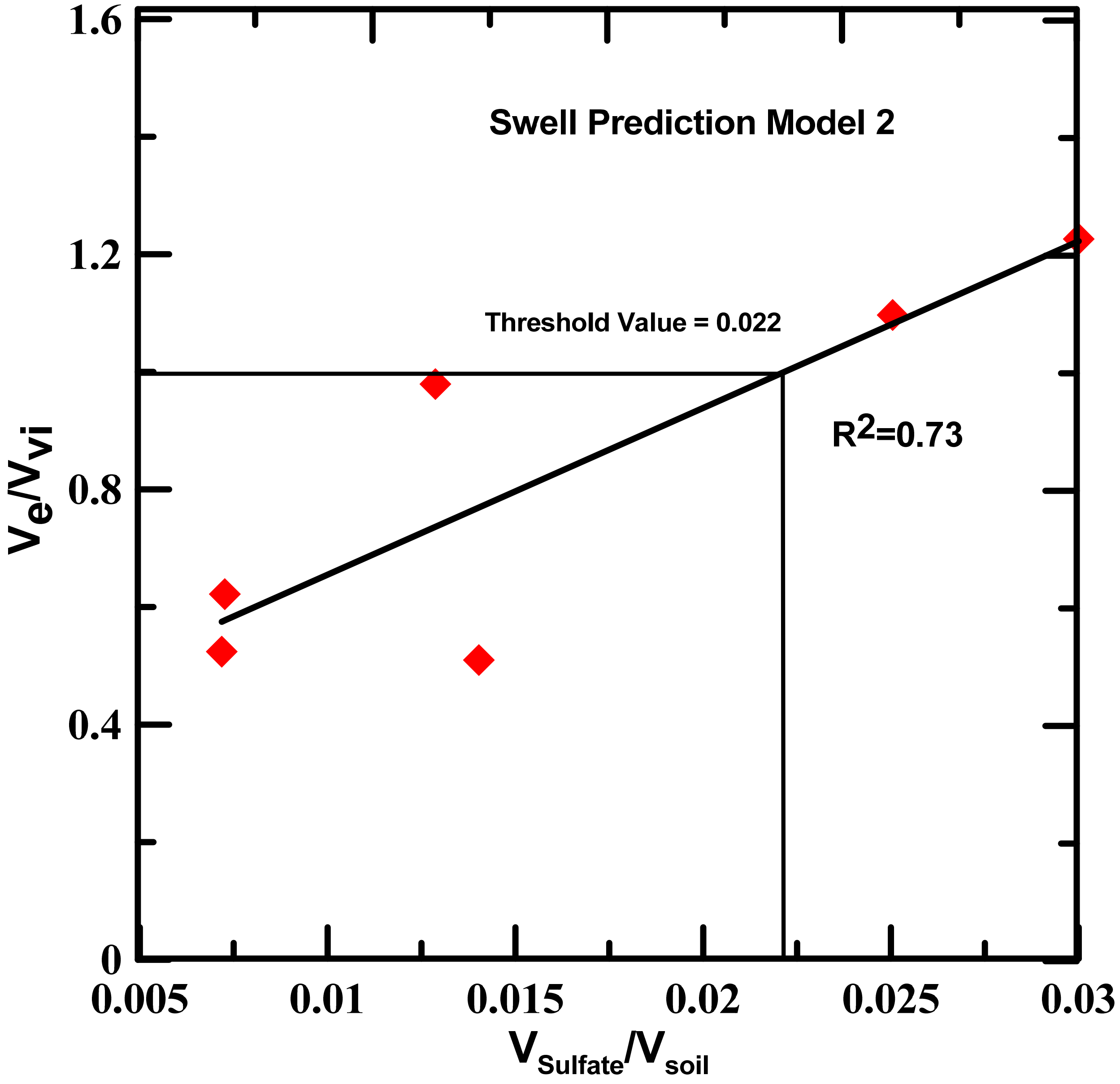

Figure 6.4 $V_{sulfate}/V_{soil}$ vs. V_e/V_{vi} at WOMC (3 days mellowing)

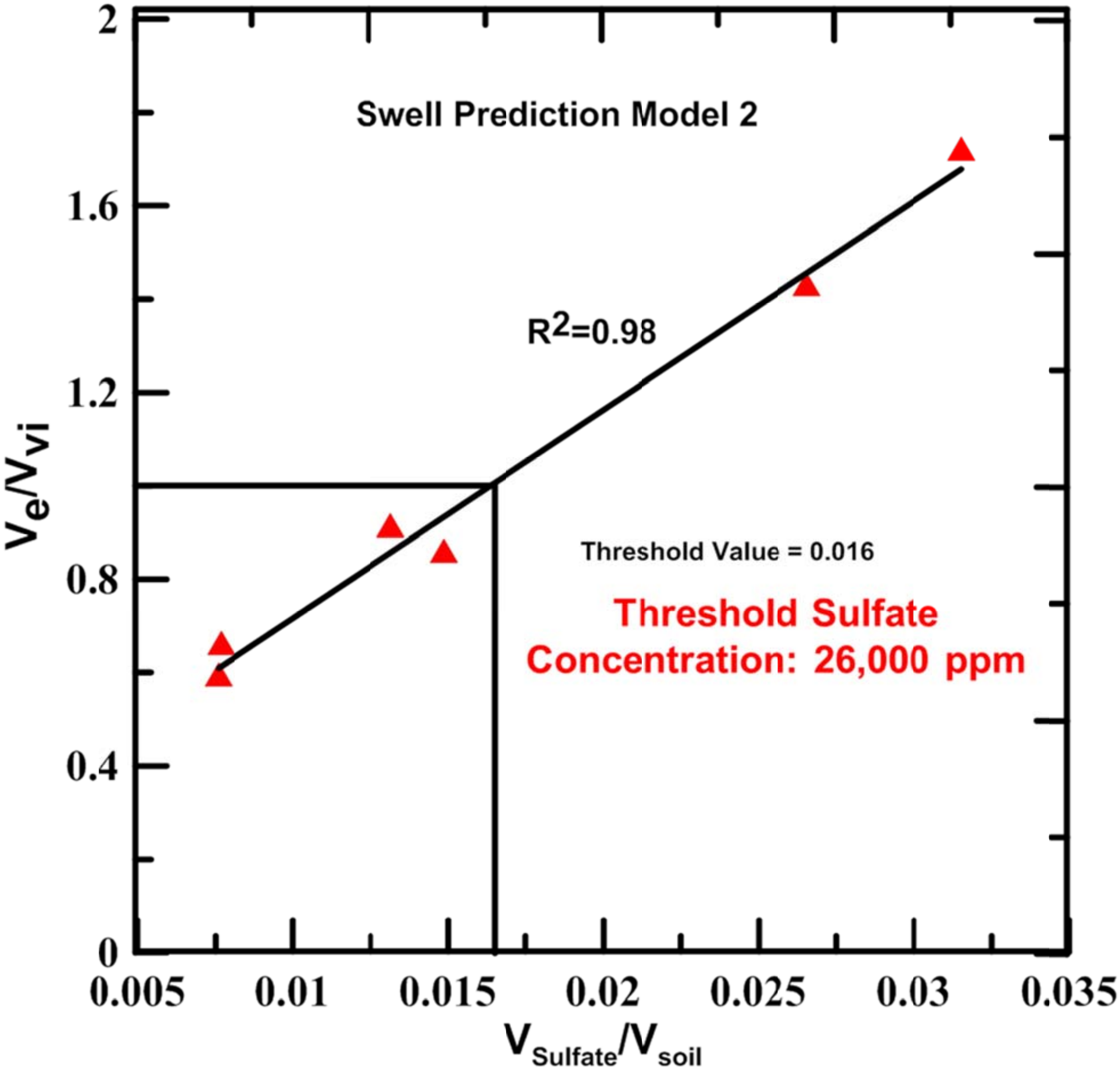

Figure 6.5 $V_{sulfate}/V_{soil}$ vs. V_e/V_{vi} at OMC (7 days mellowing)

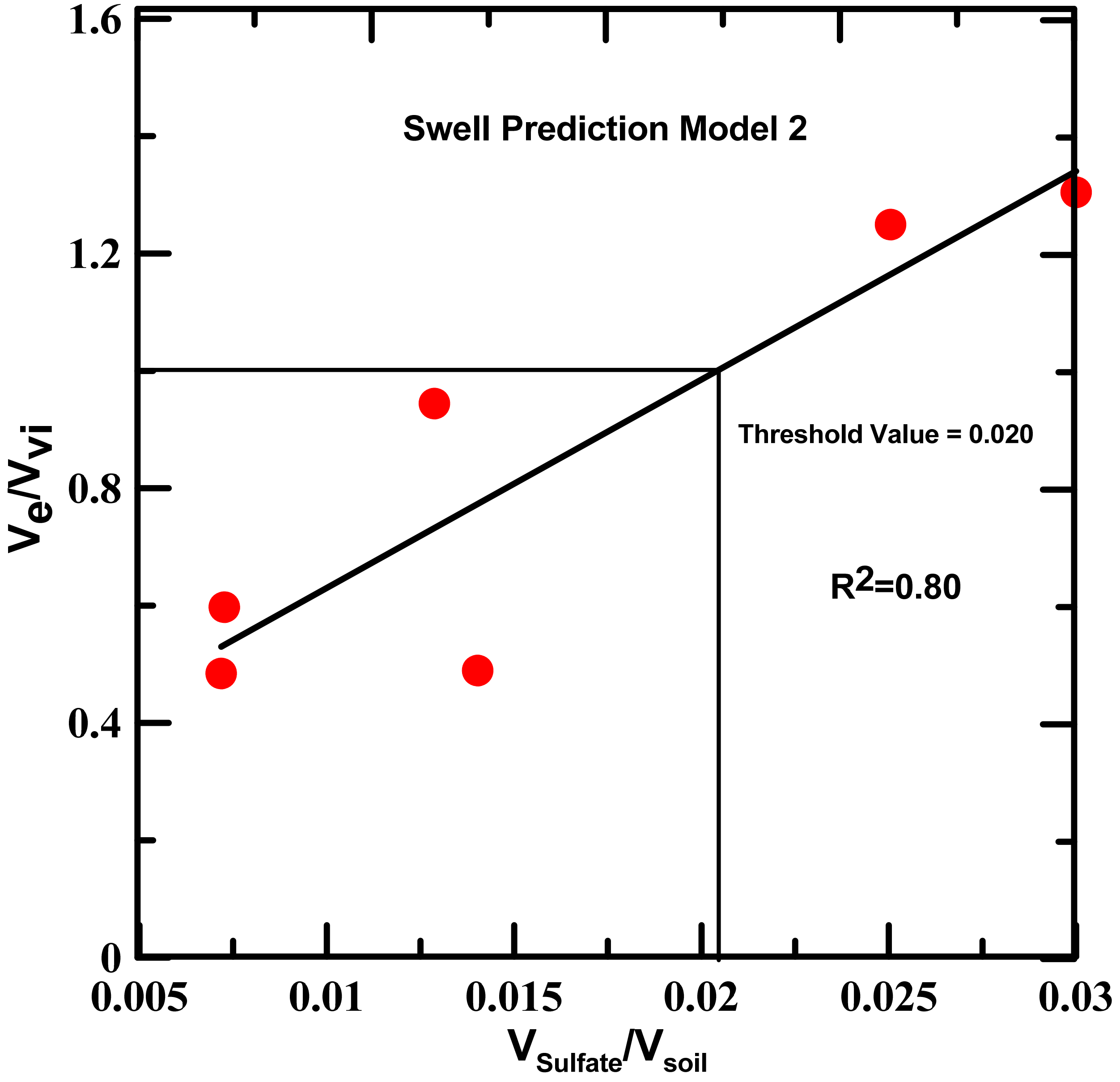

Figure 6.6 $V_{sulfate}/V_{soil}$ vs. V_e/V_{vi} at WOMC (7 days mellowing)

From Figures 6.3 and 6.4 it can be seen that for 3 day mellowing, at $V_{Ettringite}/V_{vintial}$ equal to one, the threshold value of $V_{sulfate}/Vs_{oil}$ is 0.015 and 0.022 at optimum and wet of optimum moisture contents respectively. Any point falling beyond the threshold value (on both axes) indicates the risk of Ettringite induced heaving. The threshold sulfate content is back calculated from the soils unit weight and gypsum unit weight. The threshold sulfate content at OMC for 3 day mellowing is 24,500 ppm.

Also, in case of 7 day mellowed soils, the threshold value of $V_{sulfate}/Vs_{oil}$ is 0.016 and 0.020 at optimum and wet of optimum moisture contents respectively. For 7 day mellowed soils, the threshold sulfate level at OMC is calculated as 26,000 ppm. Based on Figures 6.3, 6.4, 6.5, 6.6 Austin and Childress soils fall beyond the threshold values. These are the two soils that could not be stabilized at both 3 day and 7 day mellowing periods. These Figures are developed based on the experimental studies conducted on six soils collected from Texas. Experimental data comprising of different soil series and sulfate contents can be used to develop generic charts for prediction of Ettringite induced heaving in soils with high sulfate contents.

6.3 Summary & Conclusions

The following conclusions are drawn from the present modeling and analyses.

1. Swell prediction based on sulfate content could be misleading since soils with high sulfate contents give higher swell values and vice versa which is not true in reality.
2. Threshold void ratio frame work can be used to predict Ettringite induced heaving in chemically treated high sulfate soils at different mellowing periods.
3. Using the threshold void ratio framework, the threshold sulfate content at 3 day mellowing is 24,500 ppm.
4. The threshold sulfate content at 7 fay mellowing period is 26,000 ppm.

CHAPTER 7

TIME RATE OF ETTRINGITE FORMATION

7.1 Introduction

In the current research, six different soils with varying sulfate contents were collected from the state of Texas. Soils with sulfate contents below 8,000 ppm were spiked with additional sulfate in the form of gypsum. These soils were treated with lime and allowed to mellow for a period of 0, 3 and 7 days. After mellowing, these soils were subjected to swell tests to witness the volumetric changes in treated sulfate-bearing soils. Swell tests indicated that these soils are prone to Ettringite-induced heaving at 0-day mellowing. At higher mellowing periods, four of the six soils were stabilized effectively. The reasons for ineffectiveness of mellowing in two soils were explained as very high sulfate contents (> 30,000 ppm), low reactive alumina and silica contents and low void ratios. In order to study the time rate of Ettringite formation and subsequent material softening, the following study was considered, and the study details are presented here.

A test methodology was used for assessing the time rate of Ettringite formation and subsequent material property degradation in chemically-treated high sulfate soils. Previous research conducted at The University of Texas at Arlington (UTA) showed that Ettringite formation and its subsequent growth on hydration results in reduction of small strain shear moduli properties (Kadam, 2003; Puppala et al., 2006). Hence, determination of small strain shear moduli (G_{max}) could be one way to assess the destabilizing mechanisms in chemically treated high sulfate soils. Also, the Ettringite reactions were dominant in soils that were cured by submerging in water (complete saturation) compared to the soils cured in a humidity room. In the current study, natural and 6% lime treated soils (without mellowing) were compacted at optimum and wet of optimum moisture contents and small strain shear moduli measurements were conducted at different time periods. Though the Ettringite reactions are absent in untreated soils, shear modulus measurements were conducted to compare the untreated and treated shear modulus values.

For complete saturation and uninhibited Ettringite growth reactions to occur, the test samples were submerged in water and shear moduli measurements were conducted periodically.

7.1.1 Small Strain Shear Moduli (G_{max}) Measurements

In this section, the calculation of small strain shear moduli using the shear wave velocity is presented. Test soils were mixed with the moisture content obtained from the proctor test results and compacted using the Gyratory Compacter Machine. In the lime- treated soils, lime was mixed with soils prior to the addition of water, then compacted. The compacted samples were 4 inch in dia. and 4.6 inch in height. These samples were covered with a rubber later membrane, and porous stones were placed at the top and bottom of the samples and placed in a water bath for complete saturation. Following the saturation, small strain shear modulus measurements were conducted on the natural and treated soil samples.

The soil samples are taken out of the water bath and porous stones were removed. Bender elements were immersed into the soil sample at top and bottom to measure the shear wave velocity and shear moduli. One of the bender elements is connected at the top of the sample and acts as the signal generator. Other bender element is connected to the base of the sample and acts as a signal receiver. Sinusoidal pulse is sent from the transmitting end and the receiving signal is collected and analyzed for the time of travel of the shear wave thru the material (treated high sulfate soil specimen in this case). The transmitted and received signals are collected and displayed on the computer screen by a digital oscilloscope connected parallel to the computer. Small strain shear modulus is calculated from the velocity of shear wave thru the soil sample. Photograph of the set up used in the current research is shown in Figure 7.1. A sample output of the signal id presented in Figure 7.2.

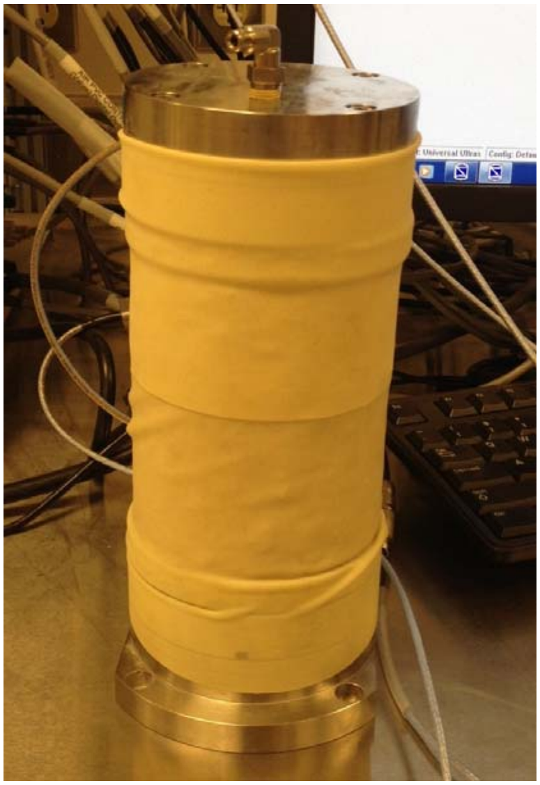

Figure 7.1 Soil Sample with Bender Elements

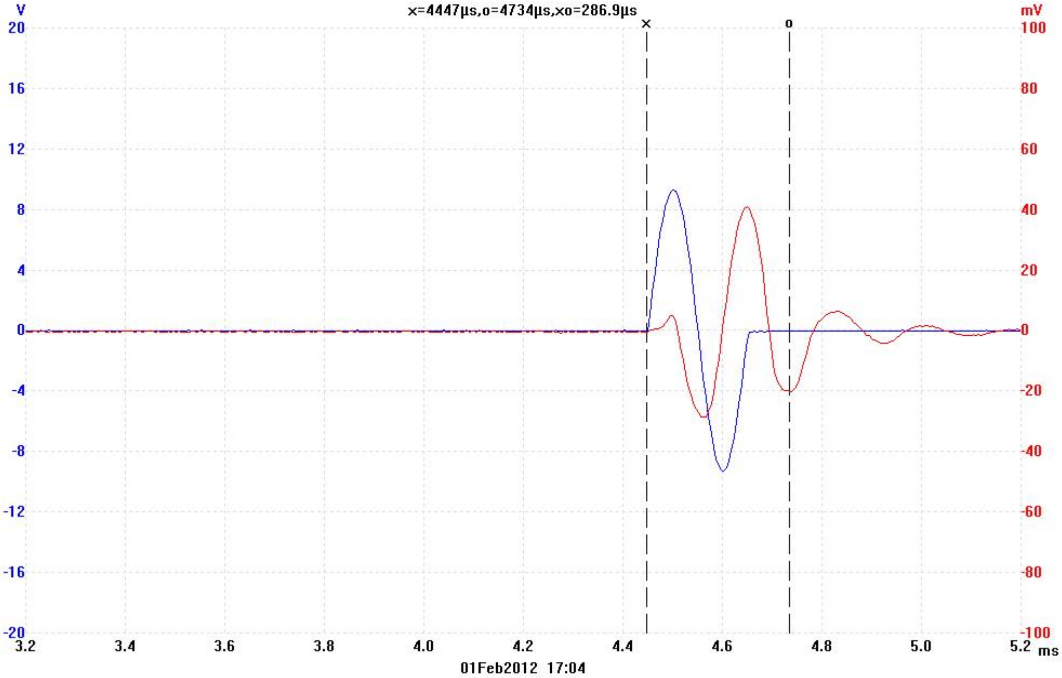

Figure 7.2 Sample Output from the Computer Screen

In general, the first significant inversion of the output signal is considered for calculation of the travel time. It was observed in the current study that first inversion gives slight error in measurement of shear modulus. In order to reduce the error, a second significant inversion of the output signal was considered for calculation of shear modulus in the current study. A sample calculation is presented below for calculation of shear modulus. Since the Bender elements protrude into the sample, the actual travel distance is less than the sample height. The protruding distance of the Bender elements at both ends is deducted from the actual sample height to obtain the true travel distance. Calculation of small strain shear moduli (G_{max}) is presented below.

$$\text{From Figure 7.2, } t = 286.9\ \mu s = 286.9 \times 10^{-6}\ \text{sec}$$

$$\text{Shear Wave Velocity, } V_s = \frac{L}{t}\ (\text{m/sec})$$

$$\text{where } L = \text{Length of Travel} = 4.46\ \text{inch.} = 0.113\ \text{m};$$

$$t = \text{Time of Travel}$$

$$V_s = \left(\frac{0.113}{286.9 \text{x} 10^{-6}}\right) = 394.8 \text{m/s}$$

$$\text{Small Strain Shear Modulus } (G_{max}) = \rho \text{ x } V_s^2$$

$$\text{where } \rho = \text{Mass Density of the Material} = 1700\ {}^{\text{Kg}}/_{\text{m}^3}$$

$$G_{max} = (1700)\text{x}(394.85)^2 = 265.04 \text{MPa}$$

$$G_{max} = 265.04 \text{MPa}$$

In the current study, samples were cured by submerging them in water. As mentioned before, the stiffness measurements were performed after complete saturation of the sample. It was reported in the literature that appearance of Ettringite-induced heave ranged from a few days to several weeks, following the lime treatment (Puppala et. al., 2012). In the current experimental study, the monitoring period was restricted to 9 days. It was observed that the small strain shear modulus (G_{max}) decreased with elapsed time in both natural and lime-treated soils. Shear moduli reduction in natural soils is attributed to the material softening due to moisture absorbance, while in lime-treated soils, formation of Ettringite and subsequent expansion are the causes of material softening. Higher shear modulus values are observed in lime-treated soils compared to natural soils, as pozzolanic reactions occur in lime-treated soils. Initial and final small strain shear modulus values were calculated to study the reduction in shear modulus with time in chemically-treated sulfate bearing soils. Initial and final shear modulus values for natural and lime cement treated soils are presented in Figures 7.3 thru 7.14. Initial and final shear moduli values for the lime=treated high sulfate soils considered in the current study are presented in Figures 7.15 and 7.16 at optimum and wet of optimum moisture contents. The shear modulus variation with time is presented in Figures 7.17 thru 7.40 for natural and lime-treated high sulfate soils considered in the current study.

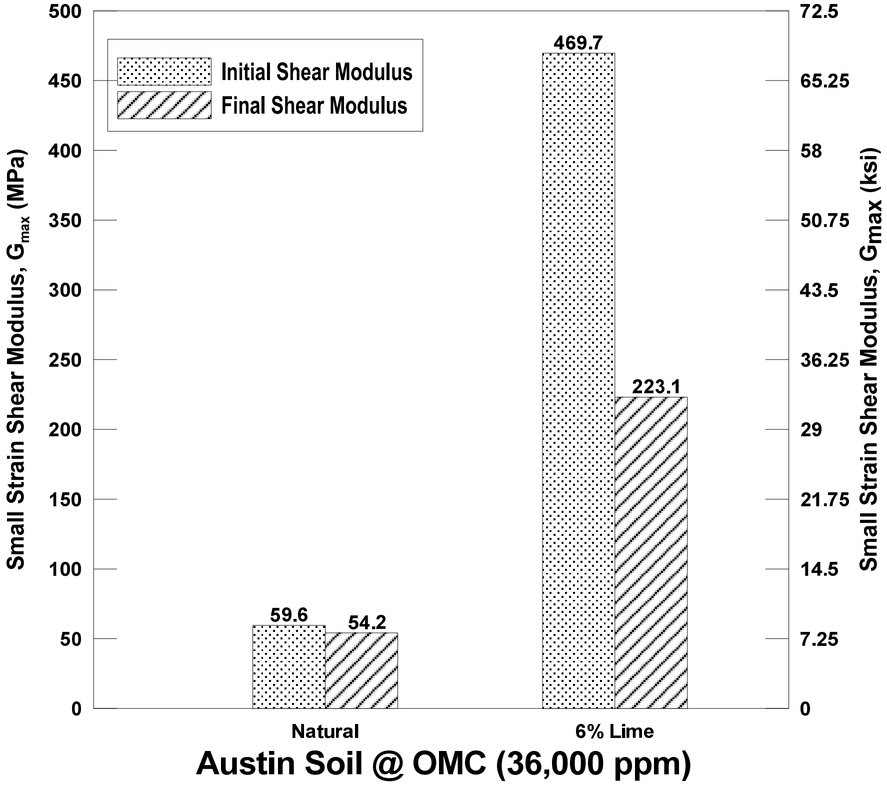

Figure 7.3 Initial and Final Shear Modulus: Austin Soil @ OMC

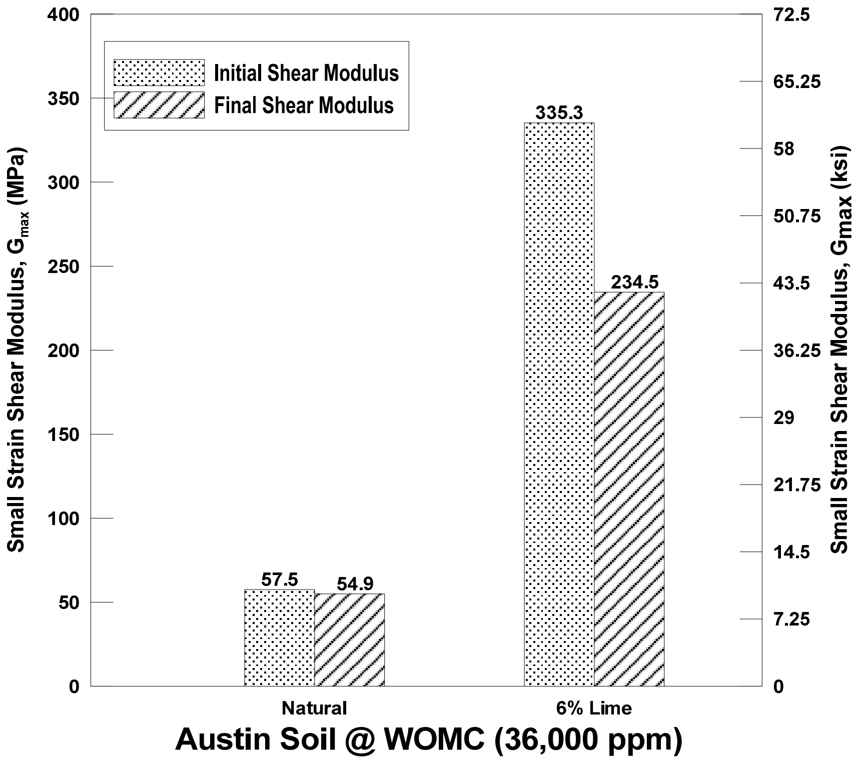

Figure 7.4 Initial and Final Shear Modulus: Austin Soil @ WOMC Soil

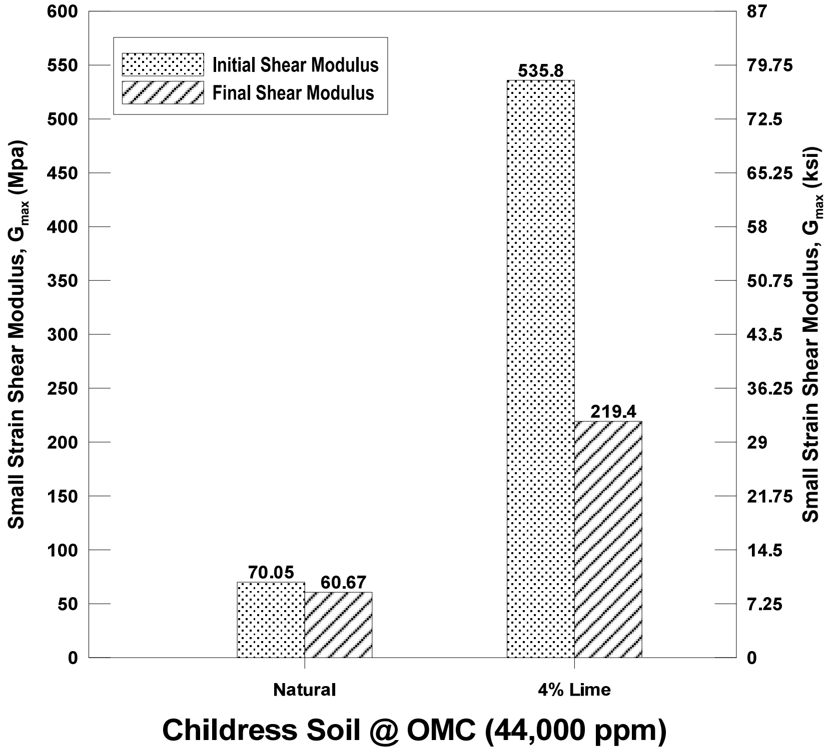

Figure 7.5 Initial and Final Shear Modulus: Childress Soil @ OMC

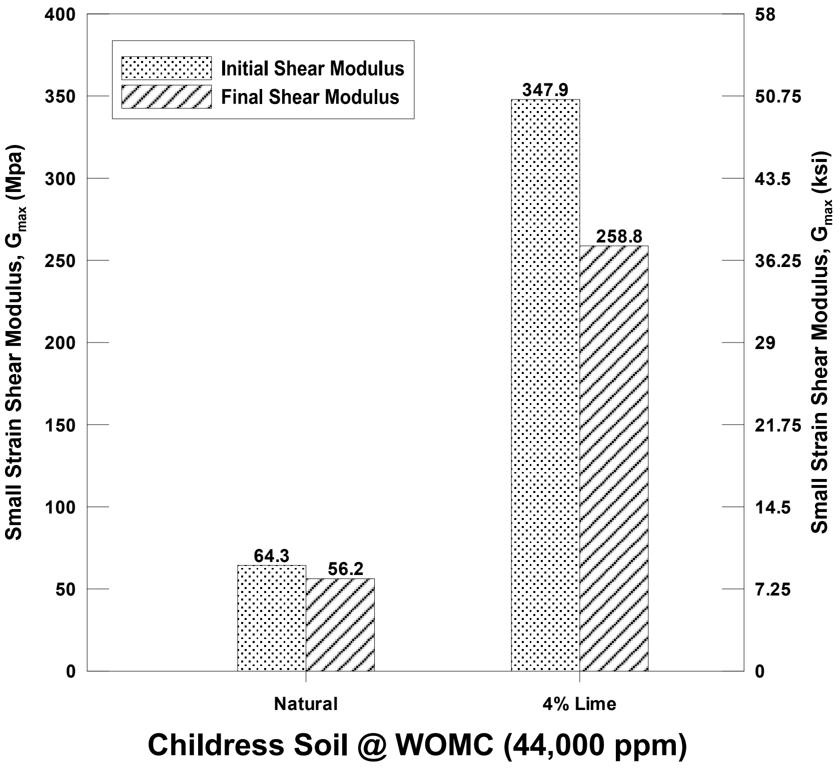

Figure 7.6 Initial and Final Shear Modulus: Childress Soil @ WOMC

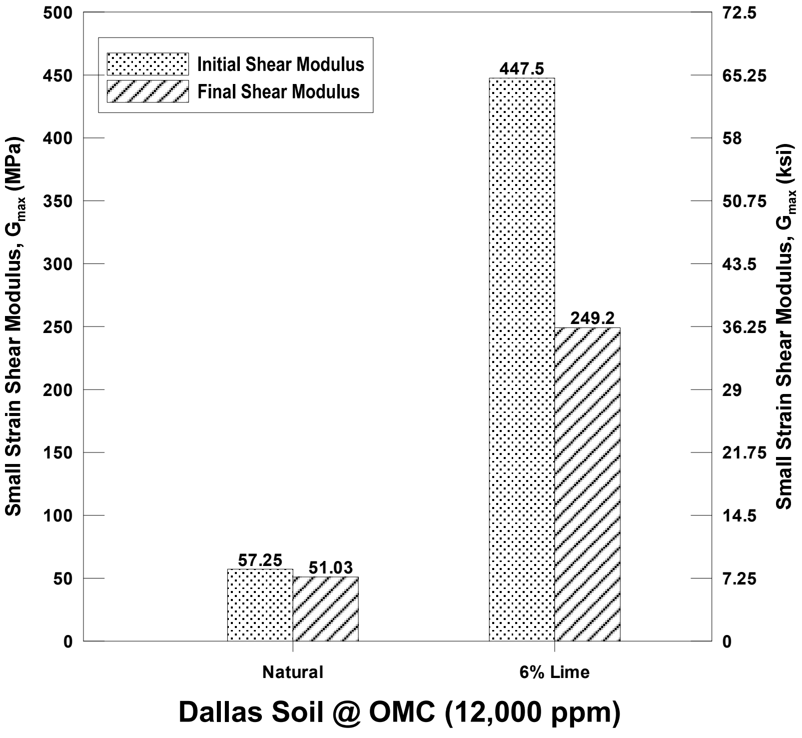

Figure 7.7 Initial and Final Shear Modulus: Dallas Soil @ OMC

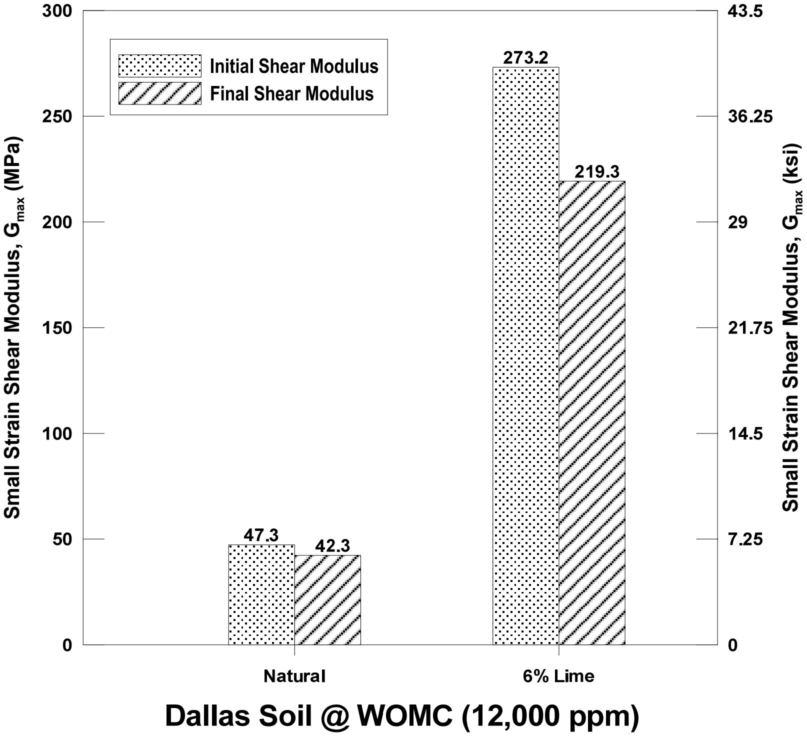

Figure 7.8 Initial and Final Shear Modulus: Dallas Soil @ WOMC

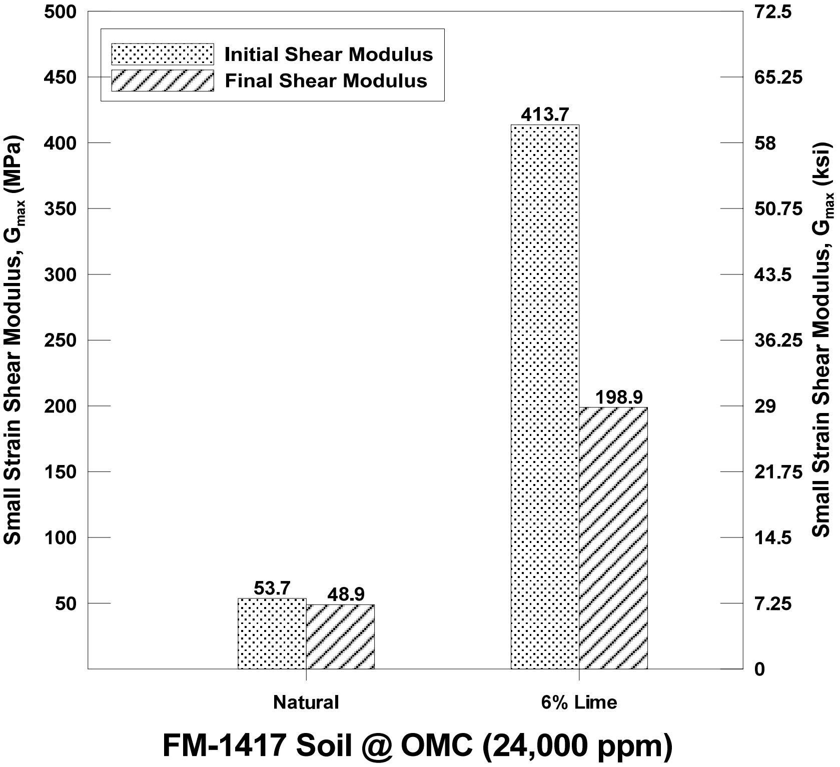

Figure 7.9 Initial and Final Shear Modulus: FM-1417 Soil @ OMC

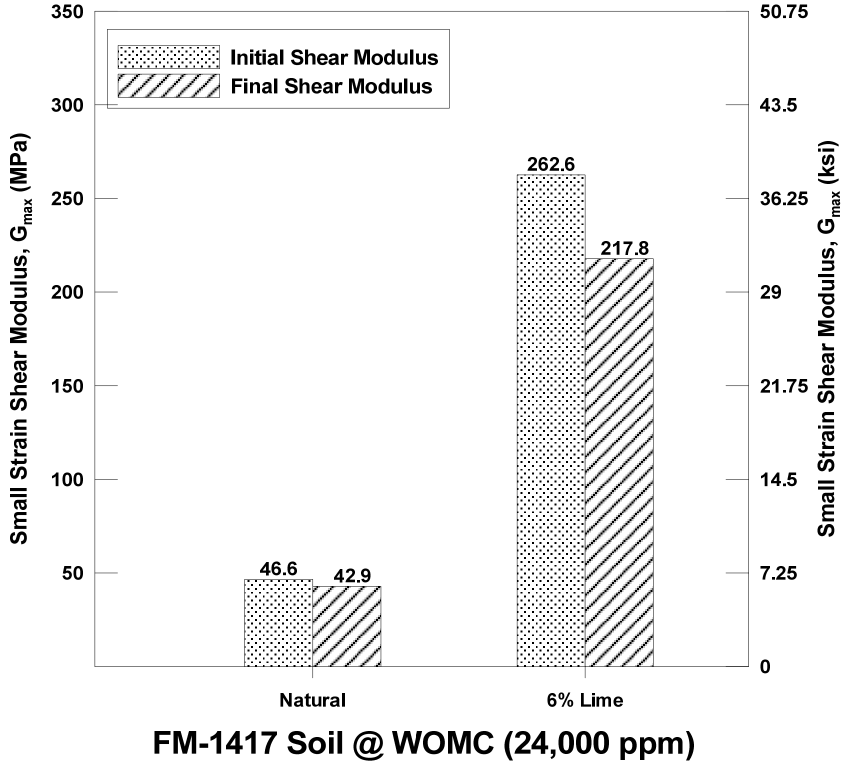

Figure 7.10 Initial and Final Shear Modulus: FM-1417 Soil @ WOMC

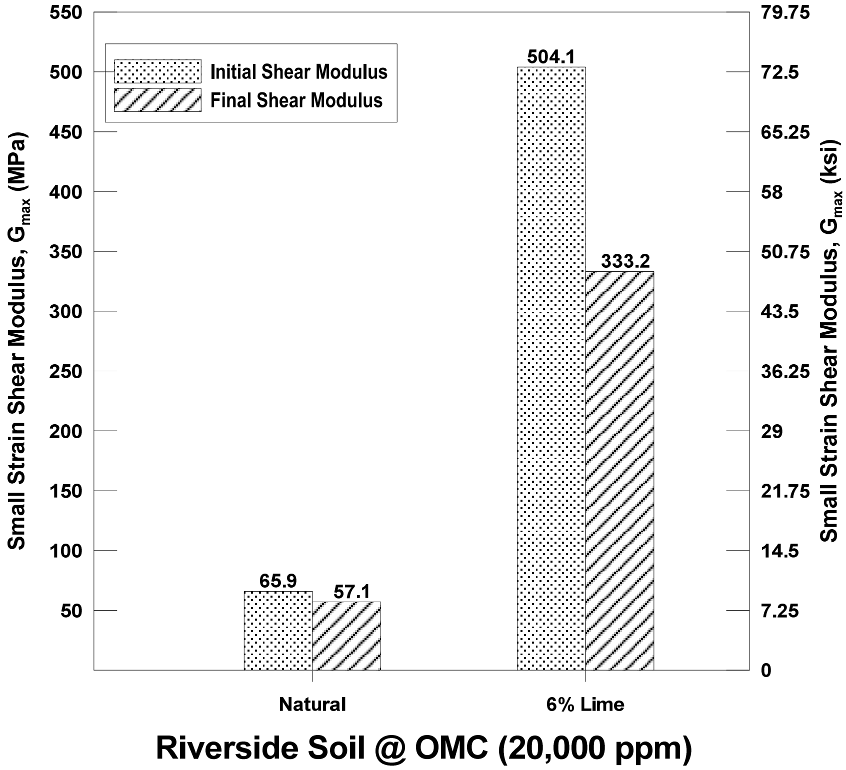

Figure 7.11 Initial and Final Shear Modulus: Riverside Soil @ OMC

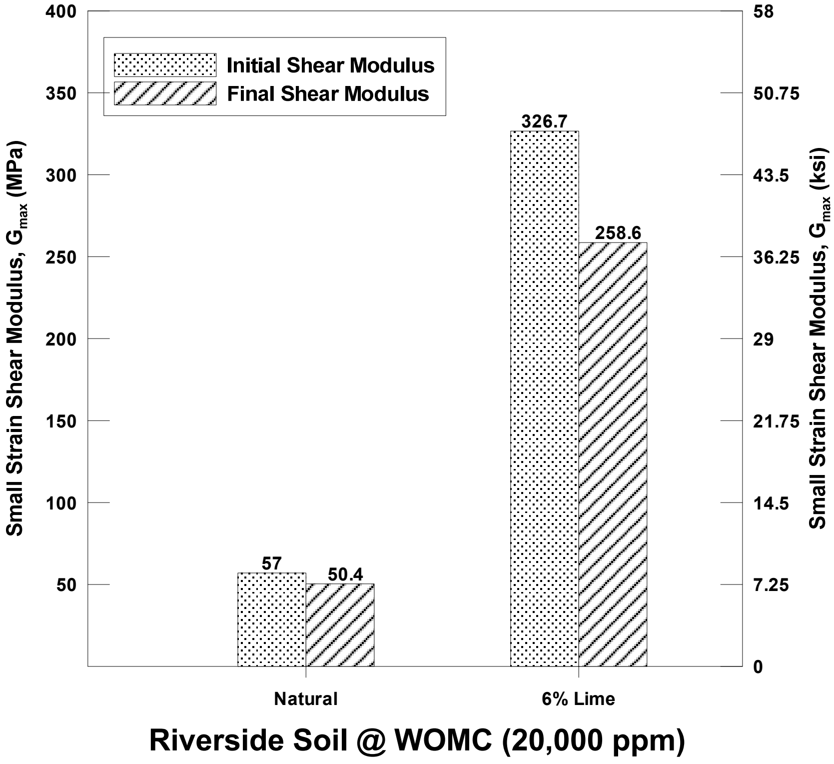

Figure 7.12 Initial and Final Shear Modulus: Riverside Soil @ WOMC

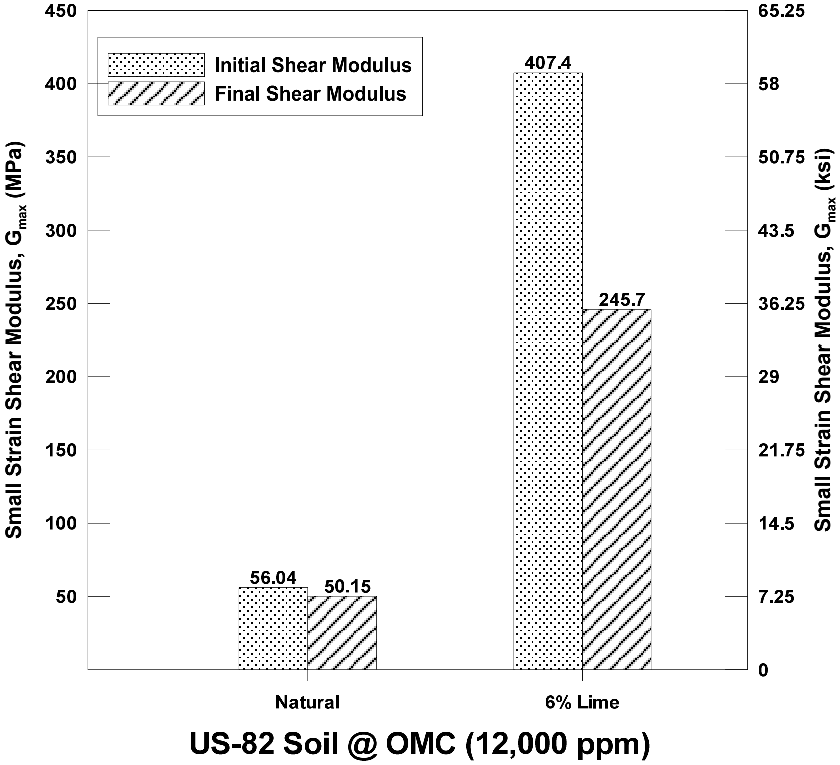

Figure 7.13 Initial and Final Shear Modulus: US-82 Soil @ OMC

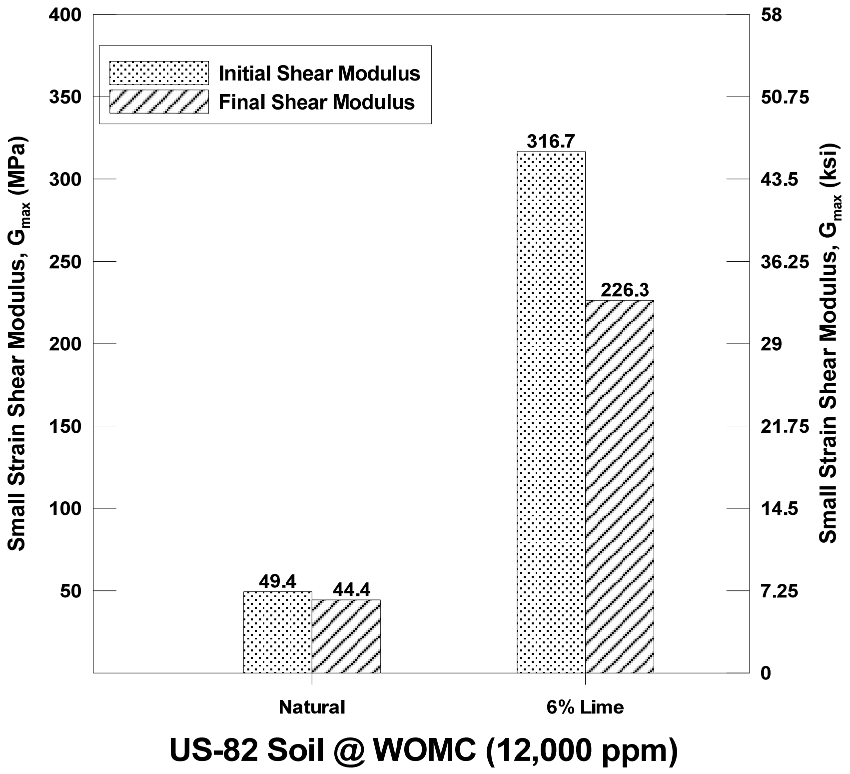

Figure 7.14 Initial and Final Shear Modulus: US-82 Soil @ WOMC

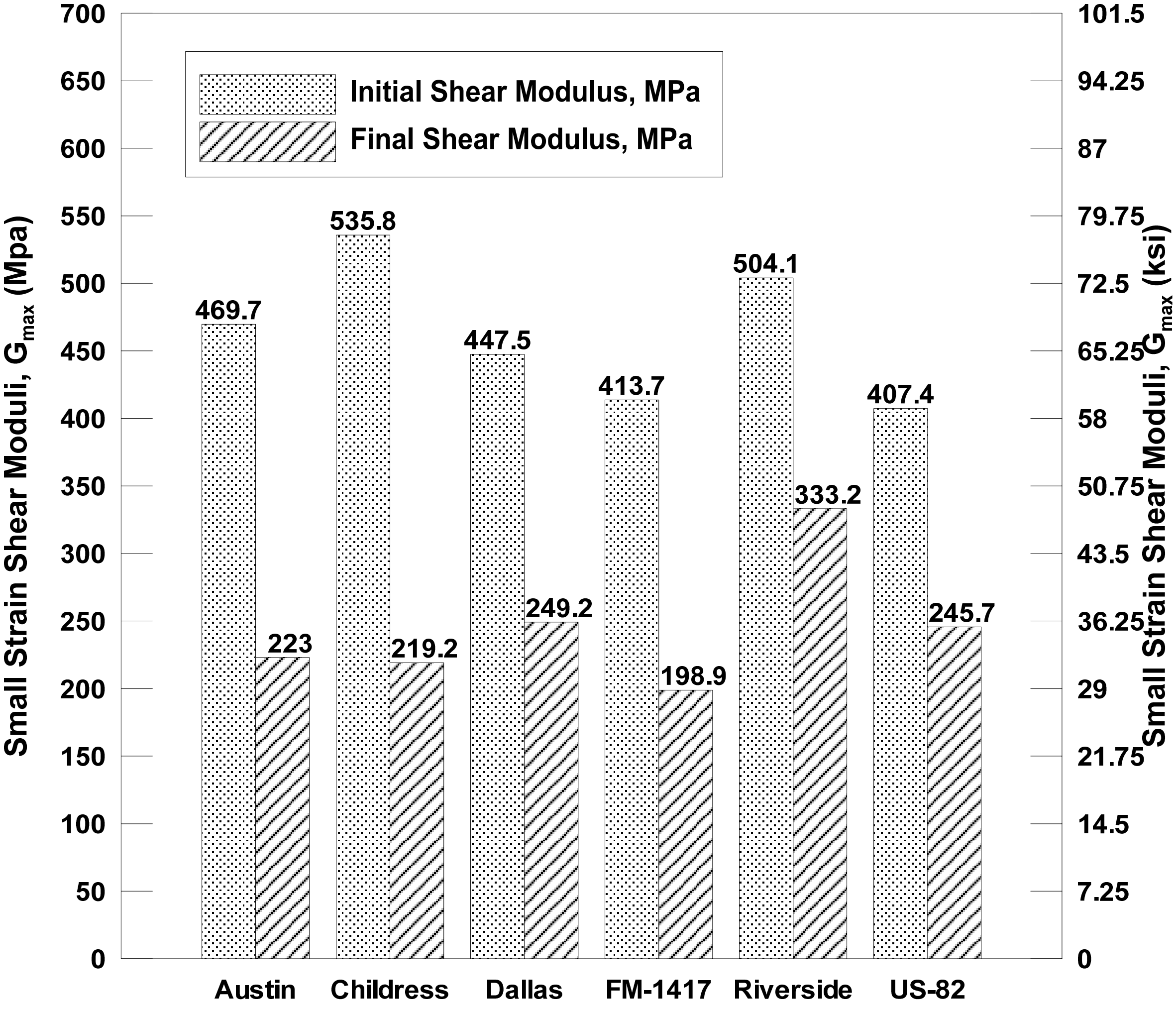

Figure 7.15 Initial and Final Shear Modulus @ OMC

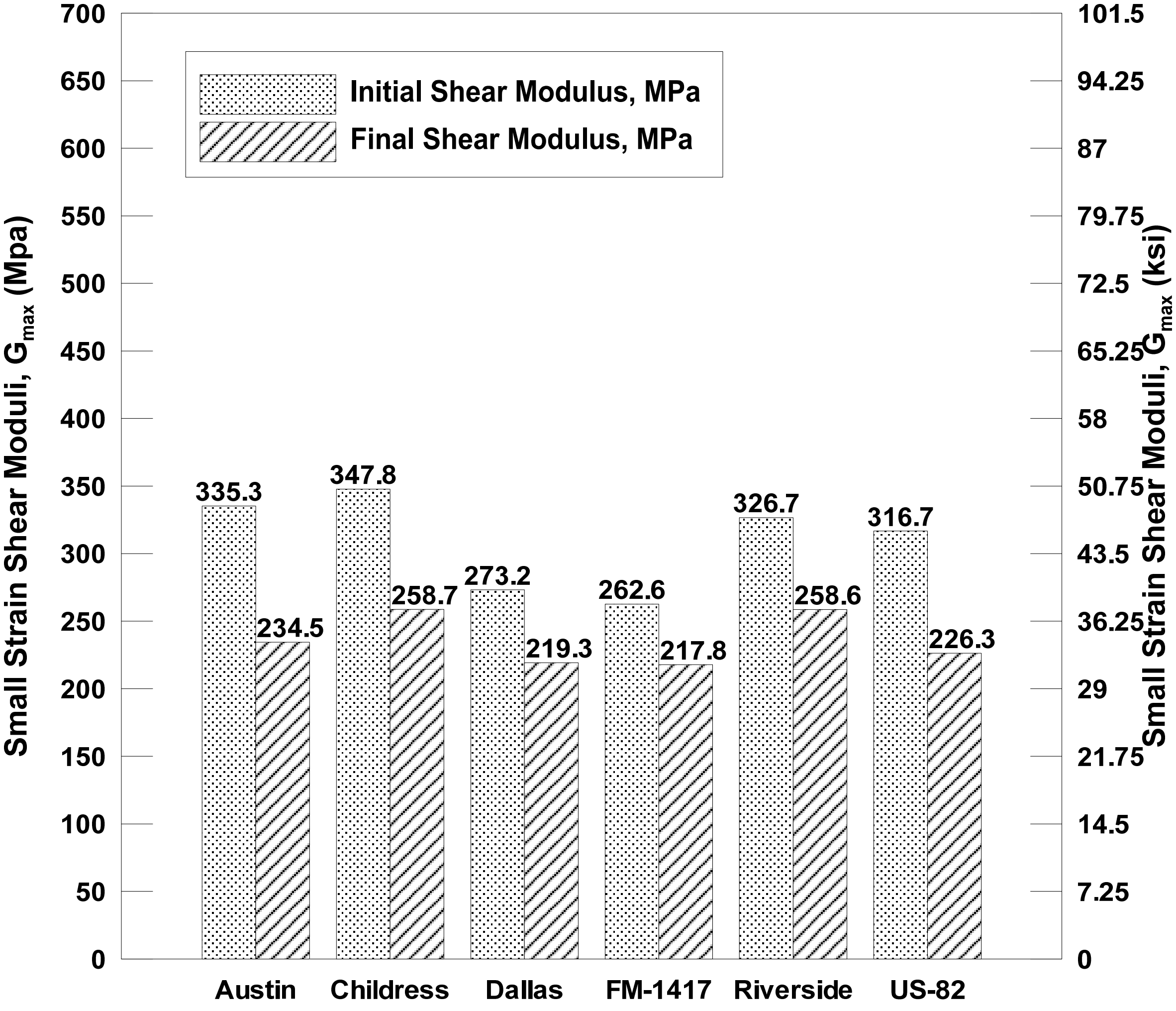

Figure 7.16 Initial and Final Shear Modulus @ WOMC

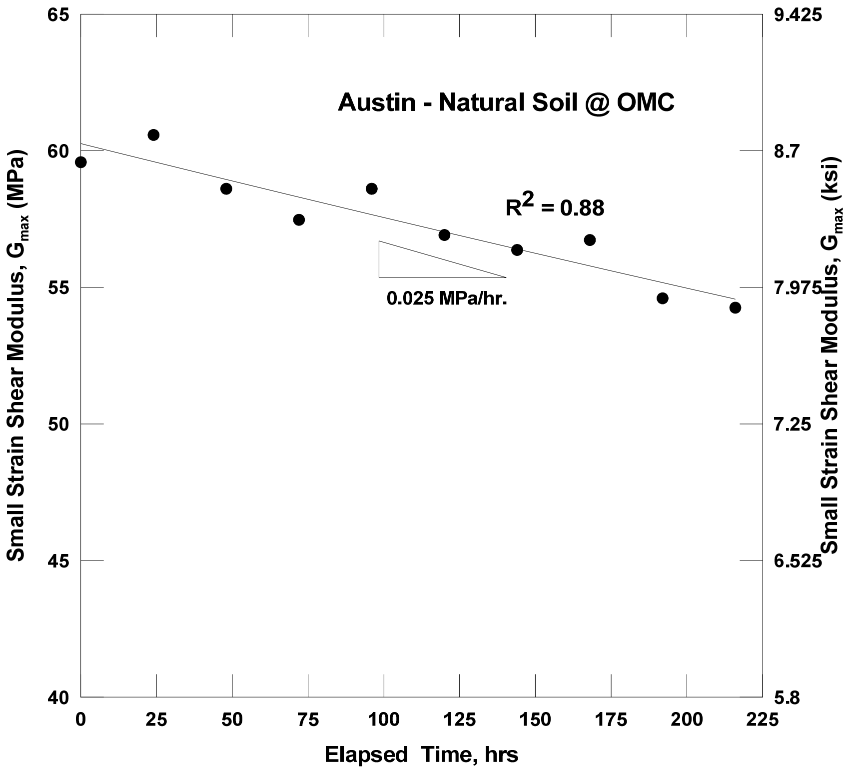

Figure 7.17 Shear Modulus vs. Elapsed Time: Austin Soil (Natural, OMC)

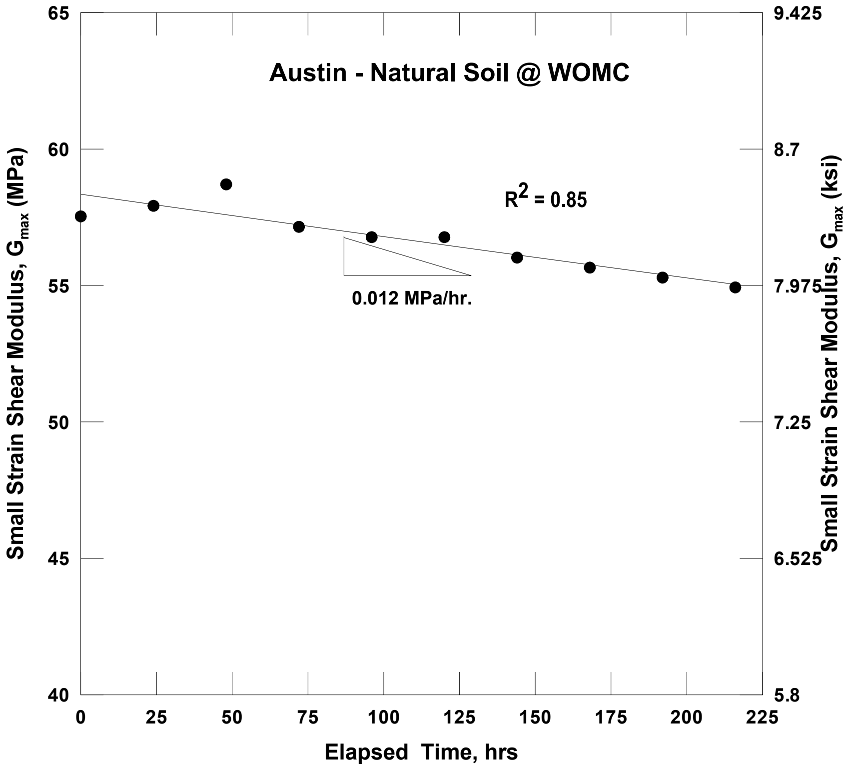

Figure 7.18 Shear Modulus vs. Elapsed Time: Austin Soil (Natural, WOMC)

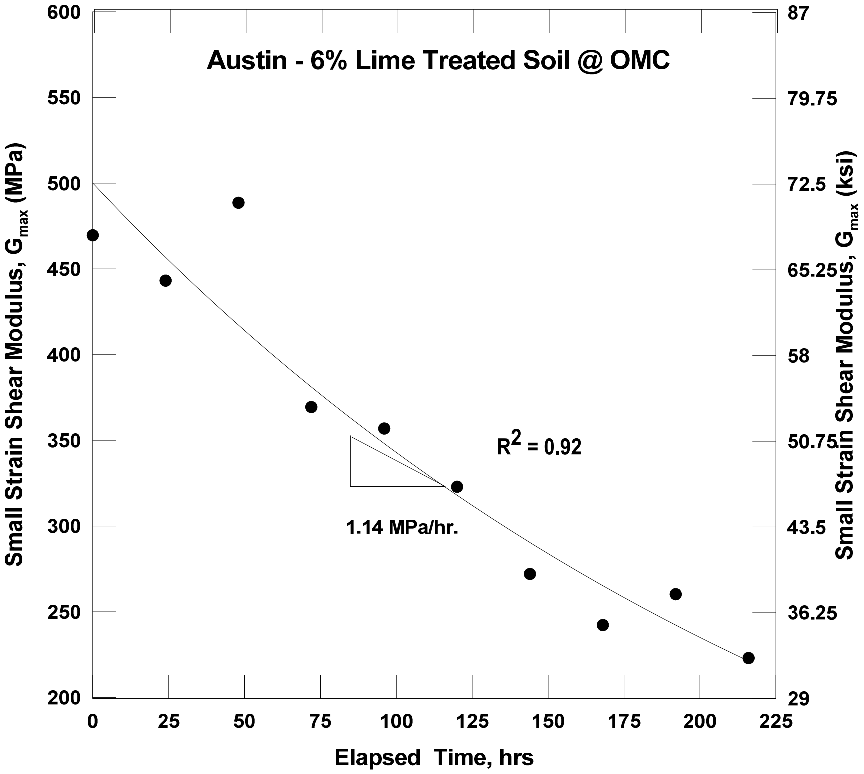

Figure 7.19 Shear Modulus vs. Elapsed Time: Austin Soil (6% Lime Treated, OMC)

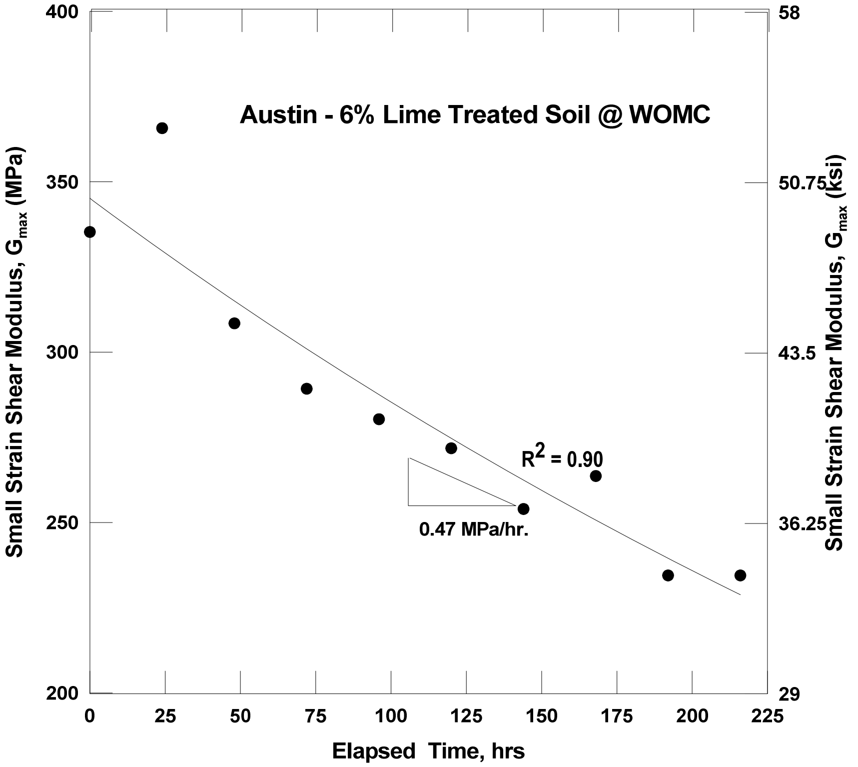

Figure 7.20 Shear Modulus vs. Elapsed Time: Austin Soil (6% Lime Treated, WOMC)

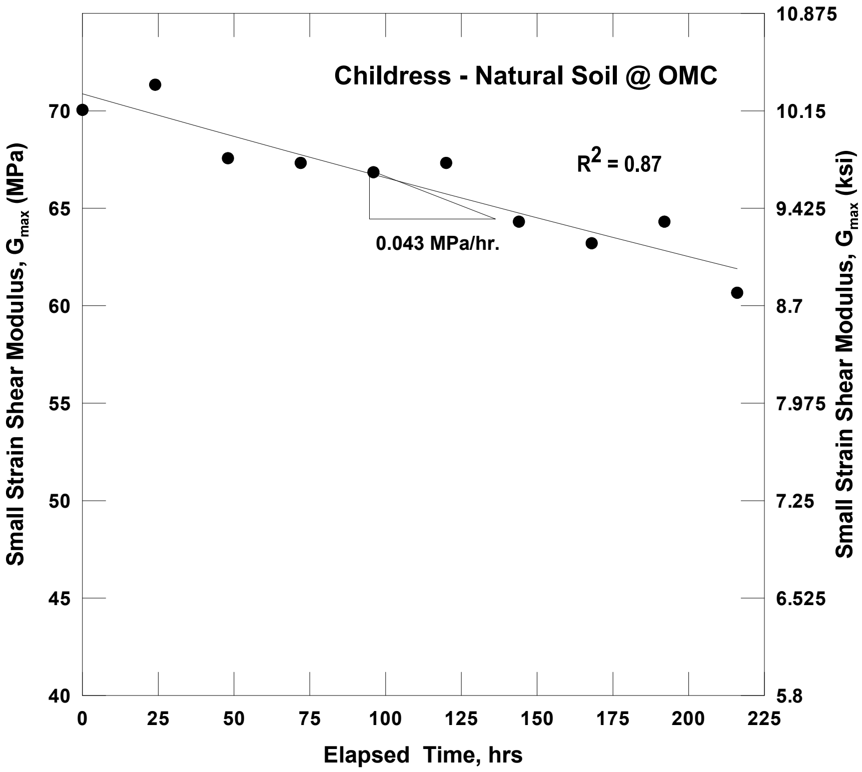

Figure 7.21 Shear Modulus vs. Elapsed Time: Childress Soil (Natural, OMC)

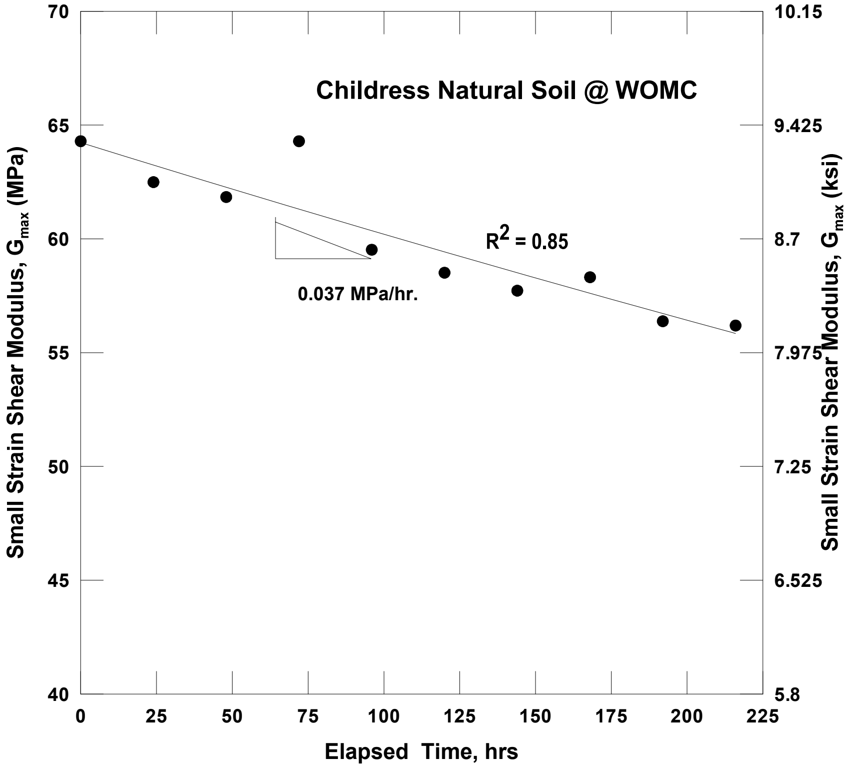

Figure 7.22 Shear Modulus vs. Elapsed Time: Childress Soil (Natural, WOMC)

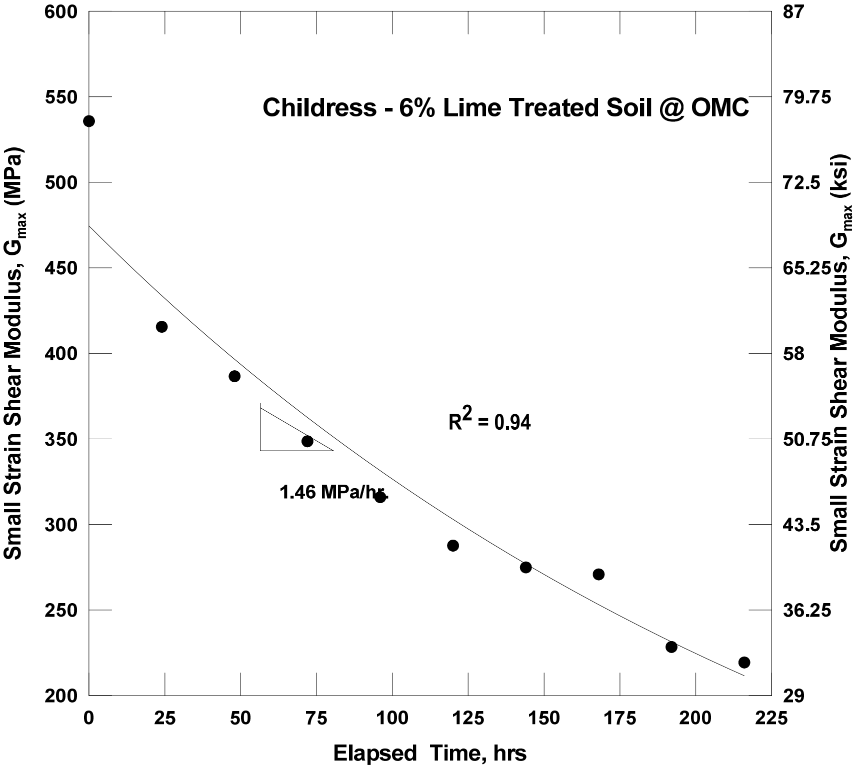

Figure 7.23 Shear Modulus vs. Elapsed Time: Childress Soil (6% Lime Treated, OMC)

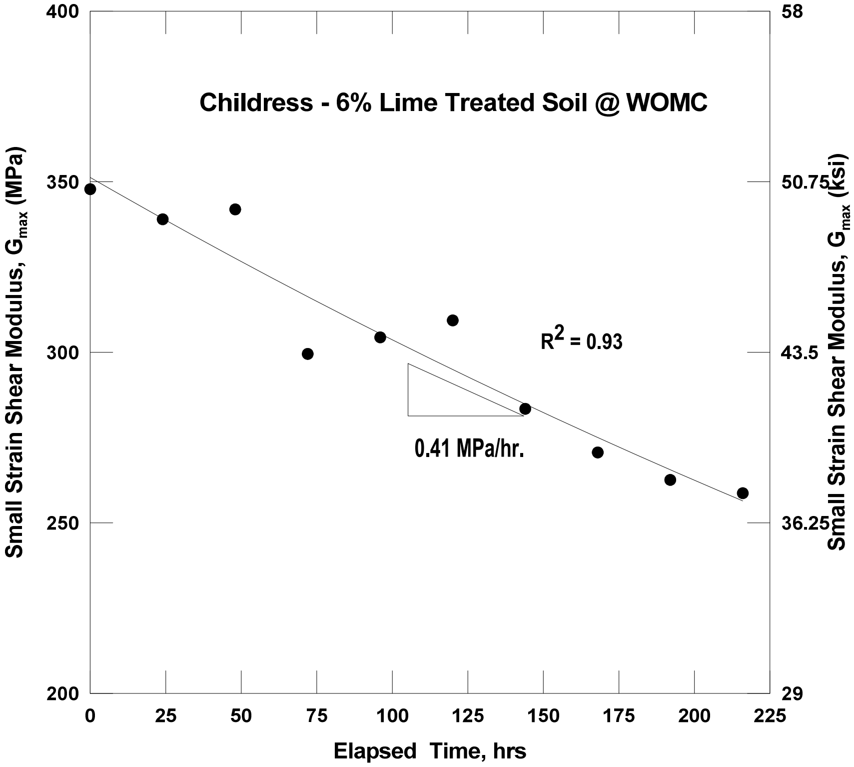

Figure 7.24 Shear Modulus vs. Elapsed Time: Childress Soil (6% Lime Treated, WOMC)

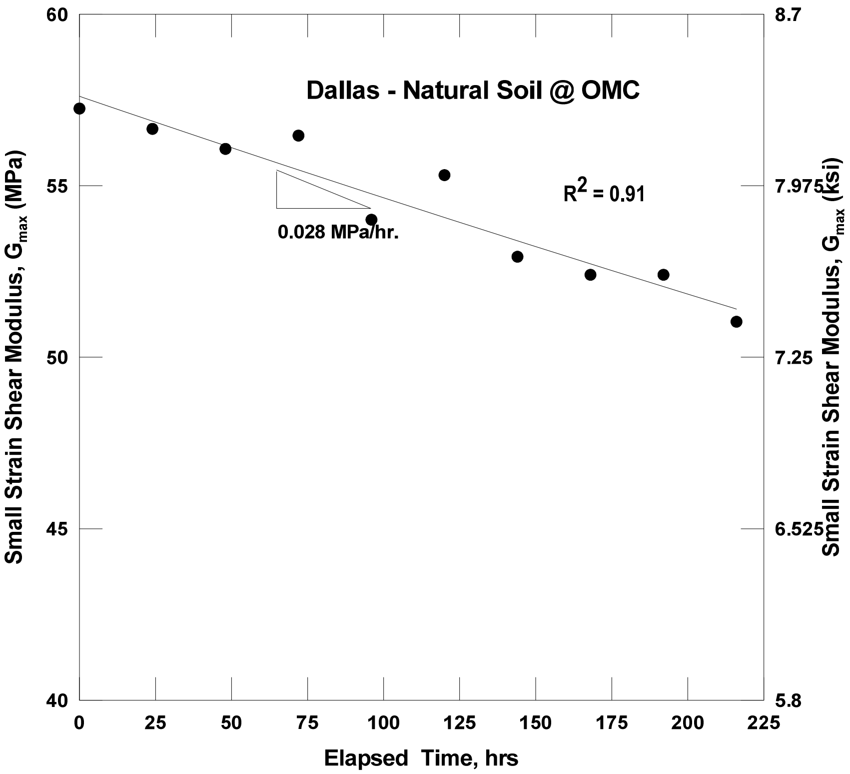

Figure 7.25 Shear Modulus vs. Elapsed Time: Dallas Soil (Natural, OMC)

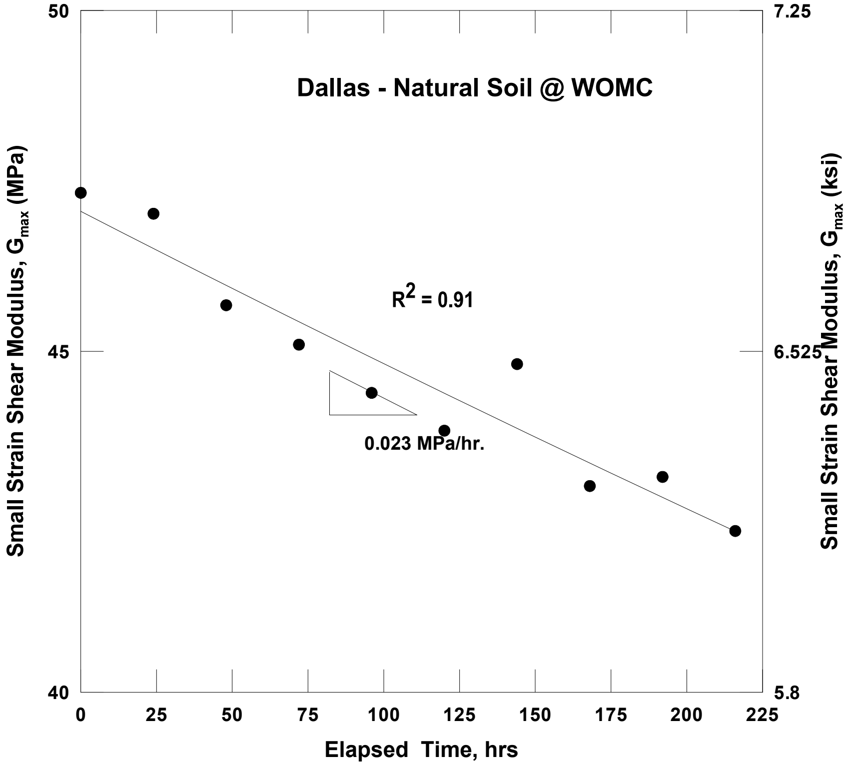

Figure 7.26 Shear Modulus vs. Elapsed Time: Dallas Soil (Natural, WOMC)

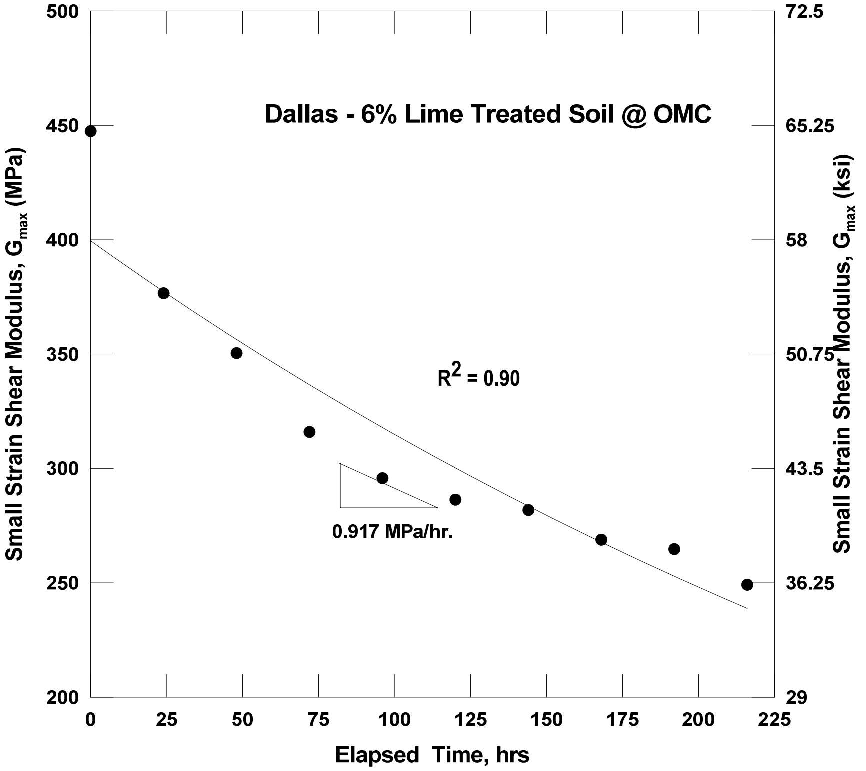

Figure 7.27 Shear Modulus vs. Elapsed Time: Dallas Soil (6% Lime Treated, OMC)

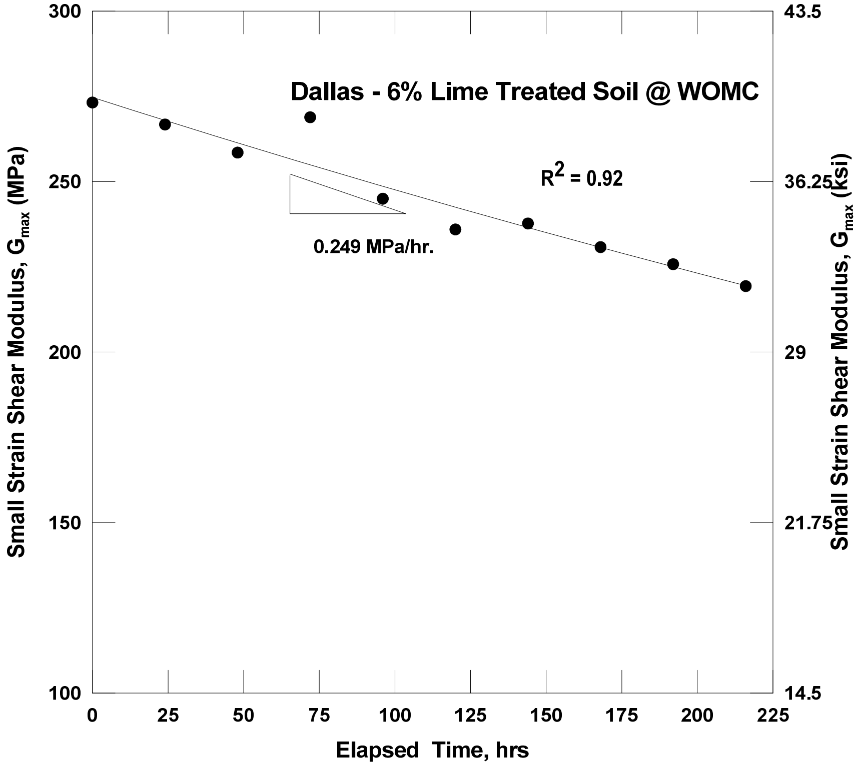

Figure 7.28 Shear Modulus vs. Elapsed Time: Dallas Soil (6% Lime Treated, WOMC)

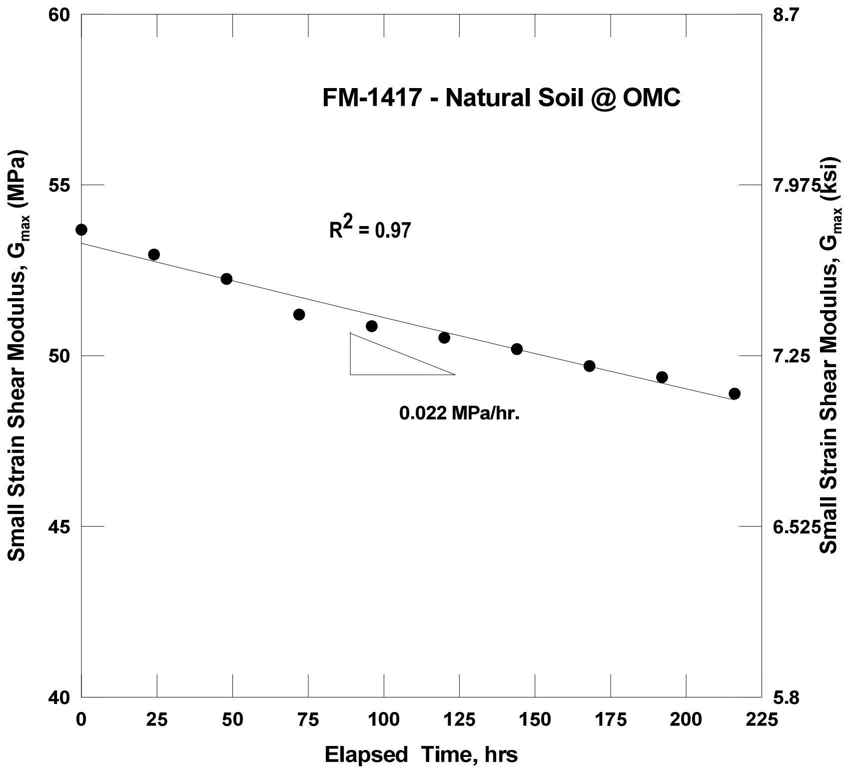

Figure 7.29 Shear Modulus vs. Elapsed Time: FM-1417 Soil (Natural, OMC)

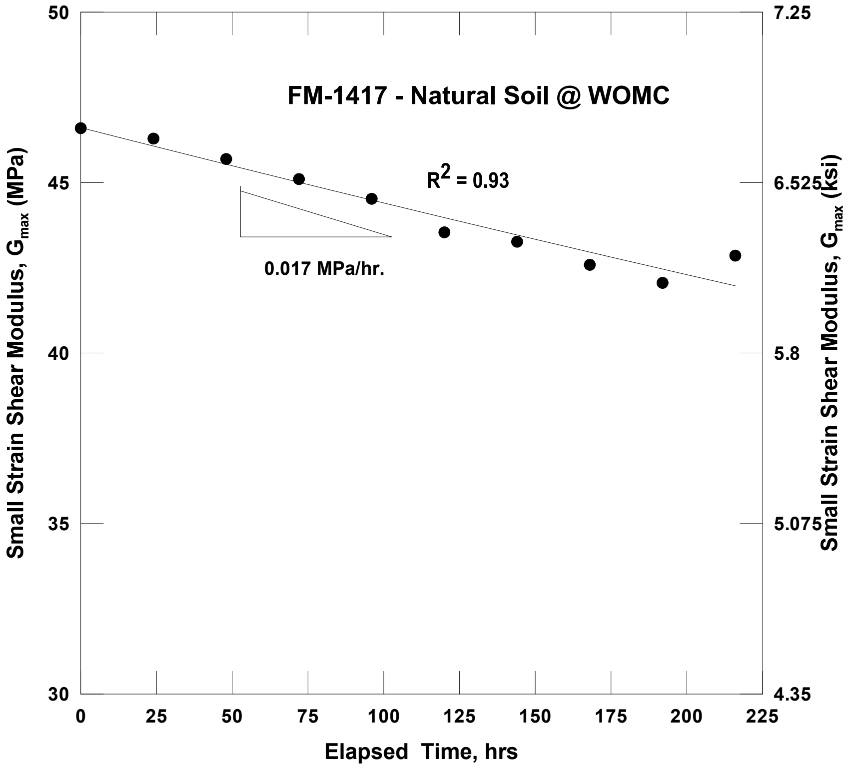

Figure 7.30 Shear Modulus vs. Elapsed Time: FM-1417 Soil (Natural, WOMC)

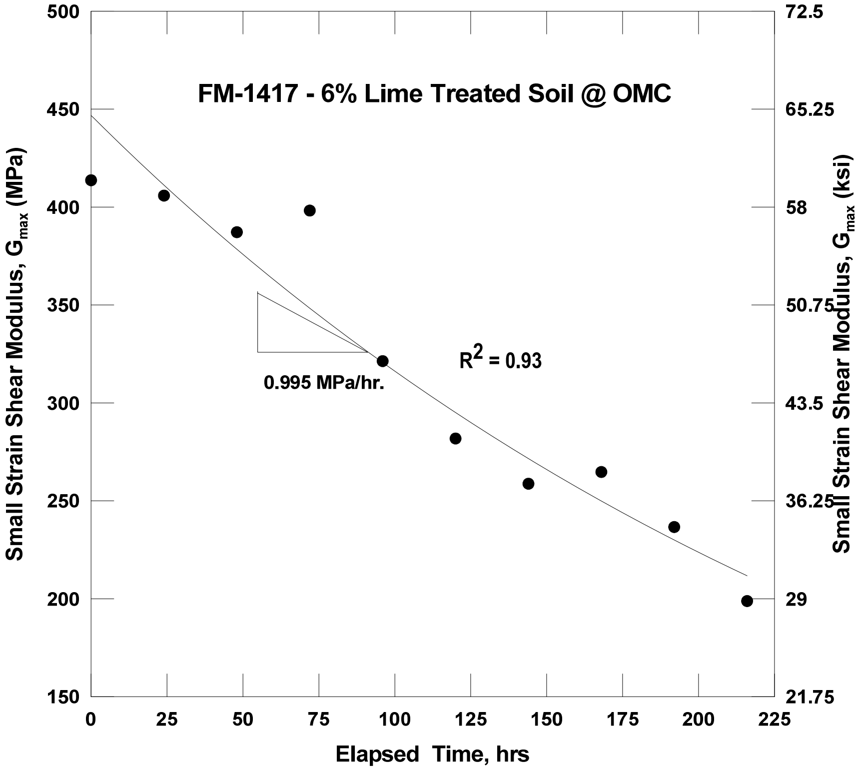

Figure 7.31 Shear Modulus vs. Elapsed Time: FM-1417 Soil (6% Lime Treated, OMC)

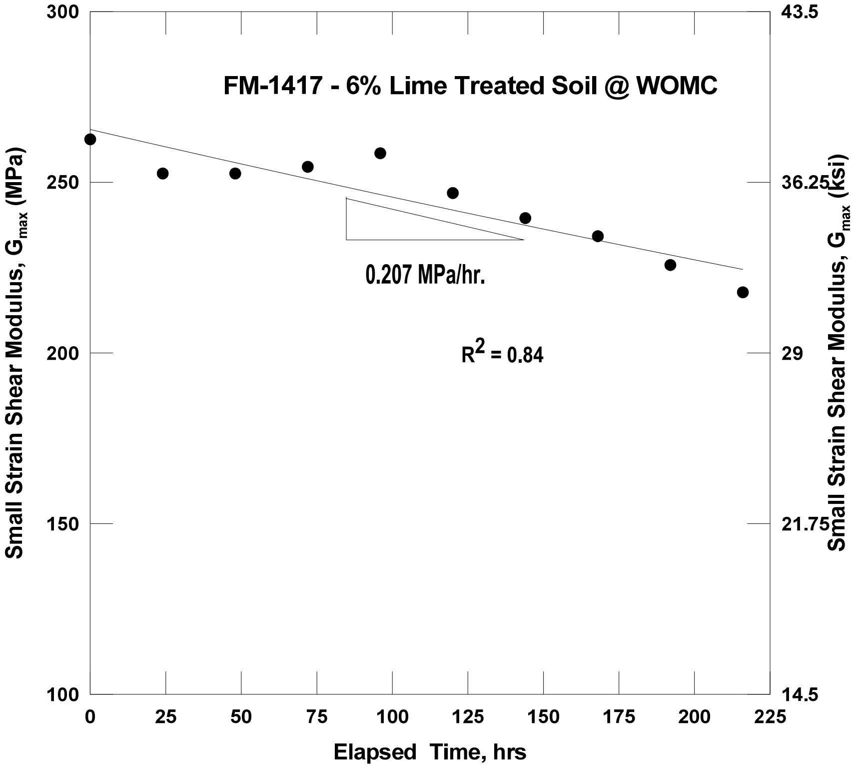

Figure 7.32 Shear Modulus vs. Elapsed Time: FM-1417 Soil (6% Lime Treated, WOMC)

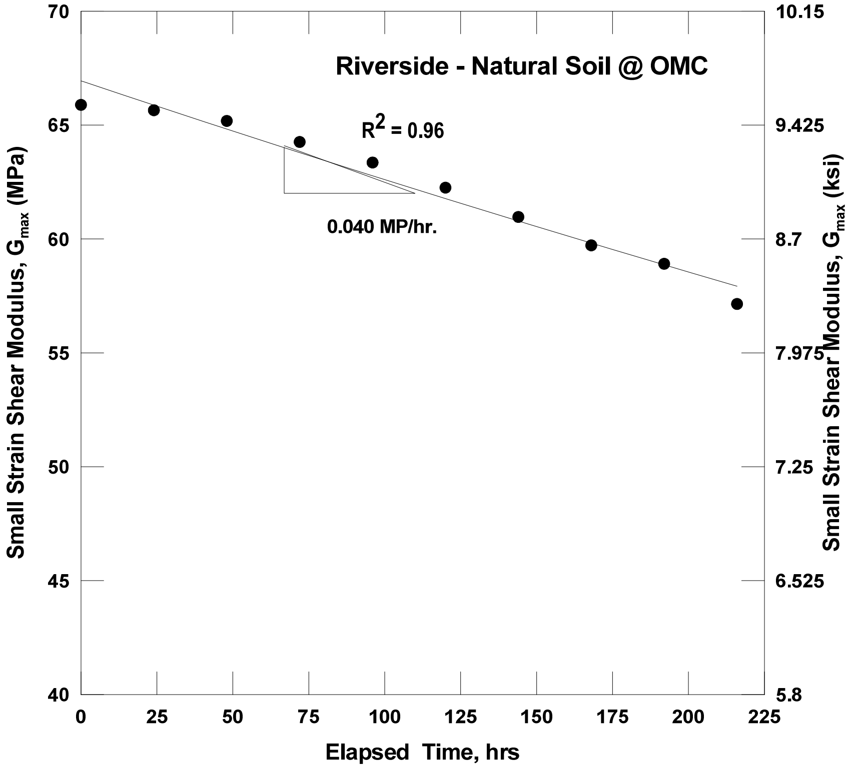

Figure 7.33 Shear Modulus vs. Elapsed Time: Riverside Soil (Natural, OMC)

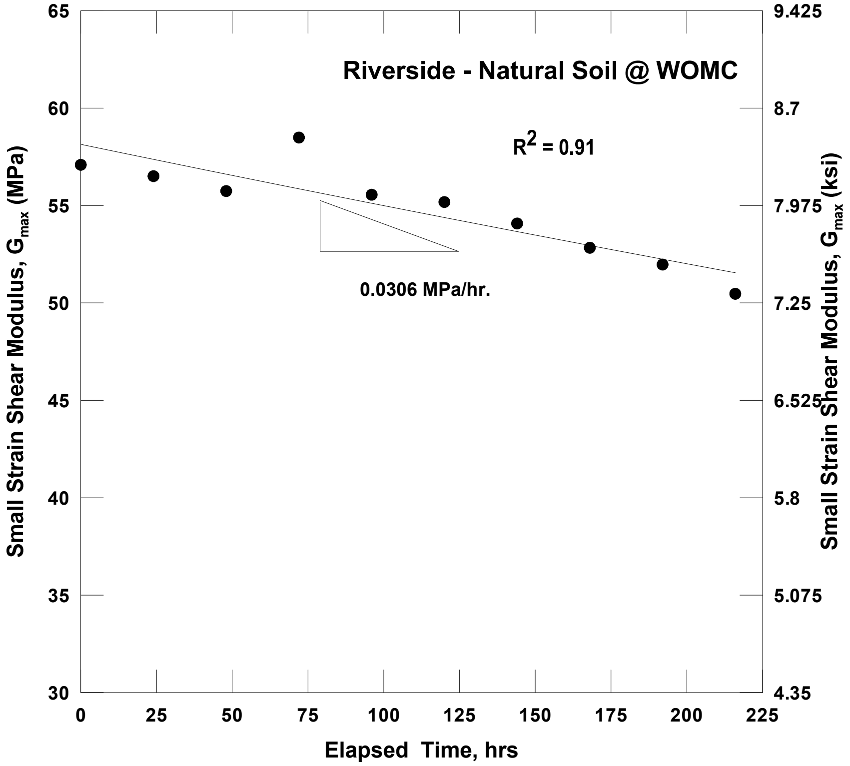

Figure 7.34 Shear Modulus vs. Elapsed Time: Riverside Soil (Natural, WOMC)

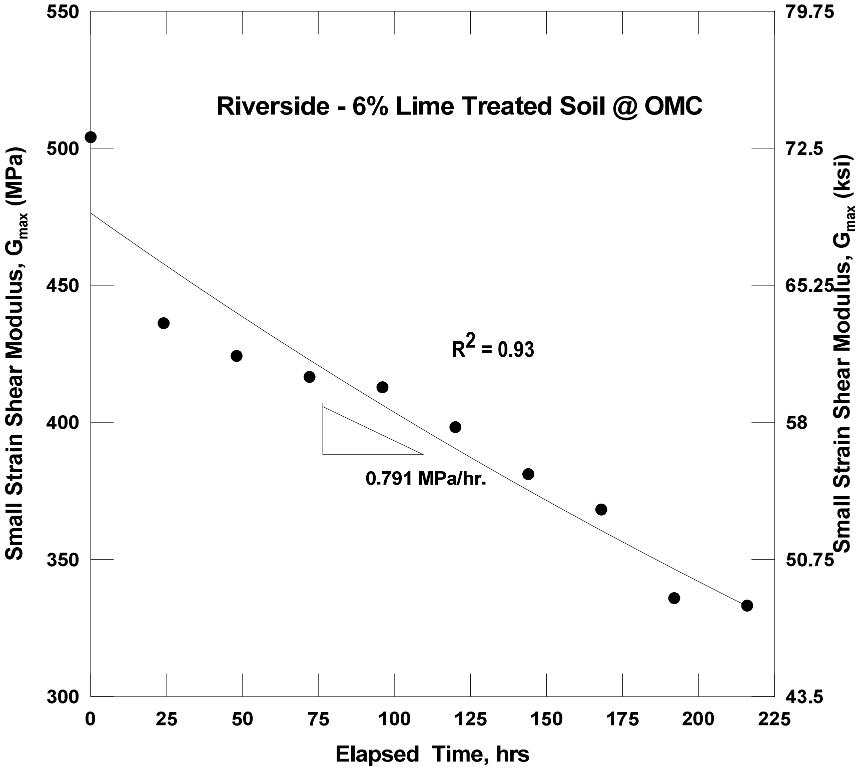

Figure 7.35 Shear Modulus vs. Elapsed Time: Riverside Soil (6% Lime Treated, OMC)

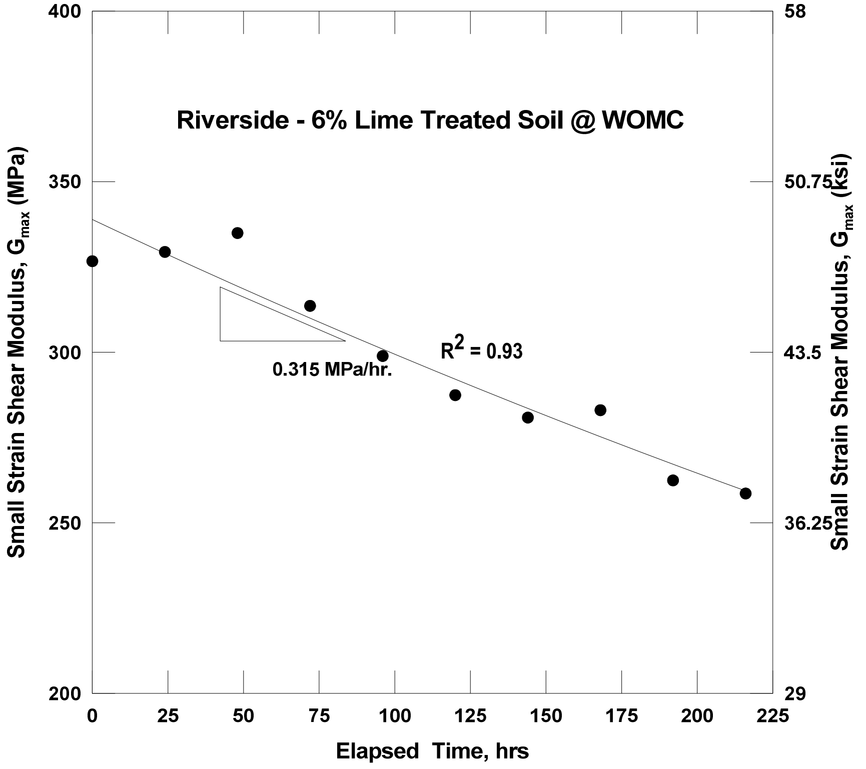

Figure 7.36 Shear Modulus vs. Elapsed Time: Riverside Soil (6% Lime Treated, WOMC)

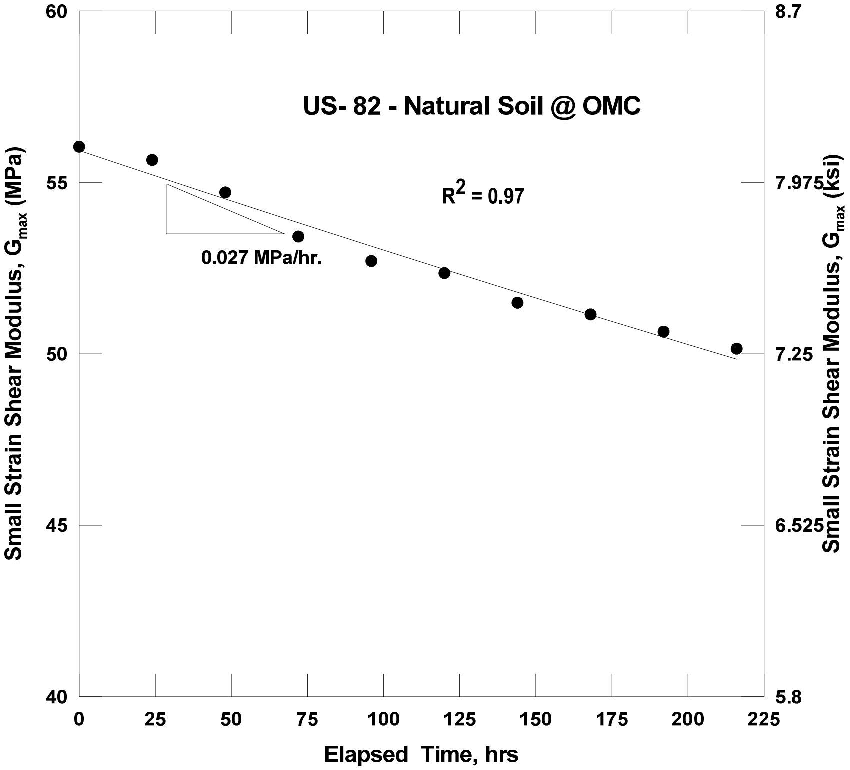

Figure 7.37 Shear Modulus vs. Elapsed Time: US-82 Soil (Natural, OMC)

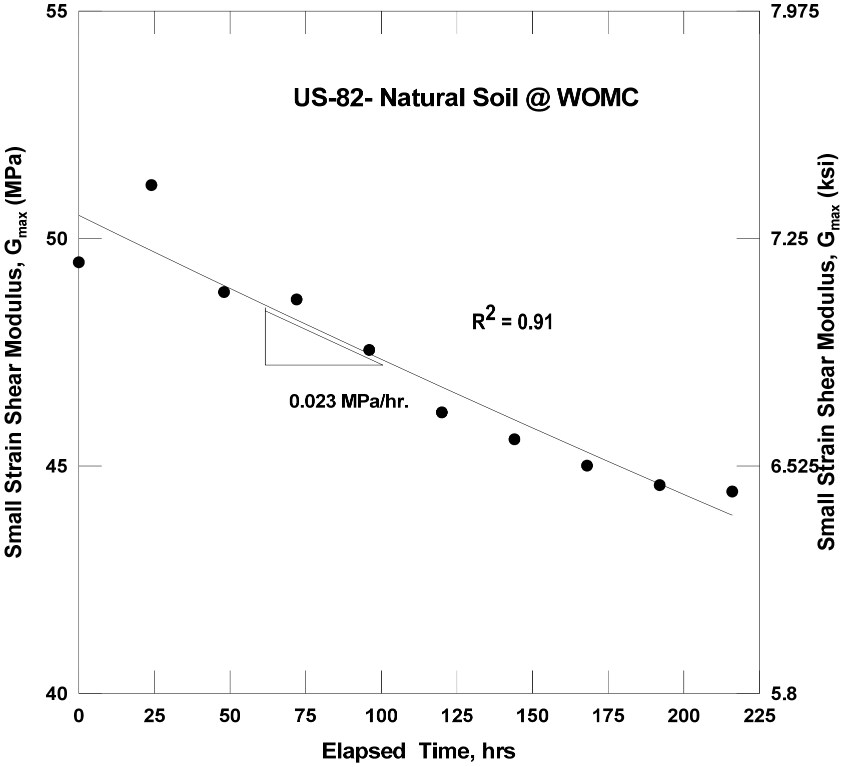

Figure 7.38 Shear Modulus vs. Elapsed Time: US-82 Soil (Natural, WOMC)

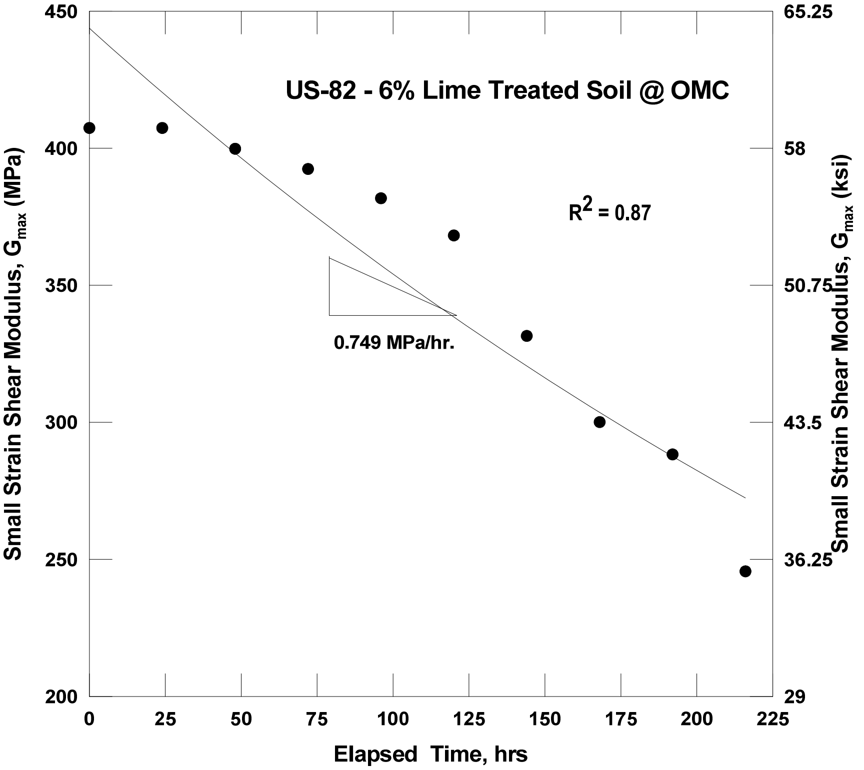

Figure 7.39 Shear Modulus vs. Elapsed Time: US-82 Soil (6% Lime Treated, OMC)

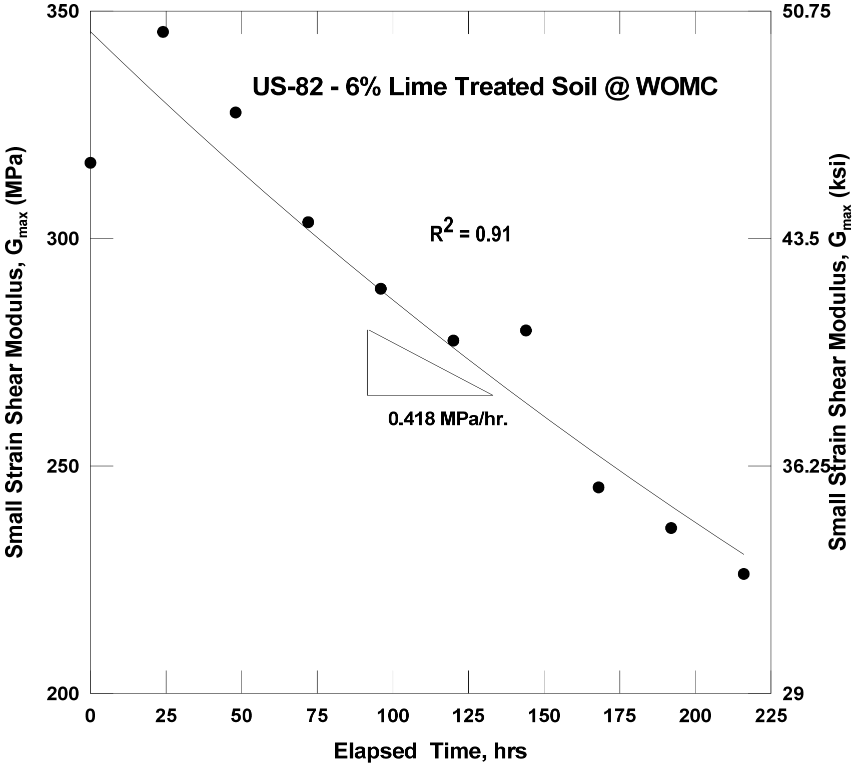

Figure 7.40 Shear Modulus vs. Elapsed Time: US-82 Soil (6% Lime Treated, WOMC)

From Figures 7.3 to 7.40, it can be seen that untreated and lime-treated soil samples experienced major reduction in shear moduli with time. In natural soils, this decrease is attributed to material softening due to presence of moisture. In lime-treated high sulfate soils, formation and growth of Ettringite and the subsequent expansion of the soil matrix are the reasons for shear moduli reductions. In untreated soils at optimum moisture condition (OMC), shear modulus varied from 56 MPa to 70 MPa. The highest shear modulus value was observed in Childress soil (70MPa), which is high plasticity silt; whereas, the FM-1417 soil, which is high plasticity clay at OMC, showed the lowest shear modulus (56 MPa). At wet of optimum moisture content (WOMC), shear modulus varied from 46 MPa to 63 MPa. Highest shear modulus at WOMC is observed in Childress soil (64 MPa) whereas lowest shear modulus is observed in FM-1417 soils.

In 6% lime-treated soils the observed shear modulus values are several times that of the natural soils. In general, the treated shear modulus values are 6-8 times higher than the untreated ones. In 6%-lime treated soils at OMC condition, the highest shear modulus was seen in Childress soil (535 MPa); whereas, the US-82 soil showed the lowest shear modulus (407 MPa). Similarly, at WOMC condition, highest shear modulus was recorded in the Childress soil (348 MPa); whereas, the FM-1417 soil recorded the lowest shear modulus (263 MPa). Analysis of the test results is presented in the following section.

7.2 Analysis of Test Results

It was reported in the literature that in treated soils, Ettringite formation and subsequent heave could take from a few days to several weeks following the chemical treatment. Due to this, in the current study, shear modulus measurements were conducted on treated soils for a period of 9 days. At the end of the 9th day, the final shear moduli values were calculated. The difference of the initial and final shear modulus is termed as the loss of stiffness. For the soils considered in the current study, the highest loss of stiffness was observed in the Childress and Austin soils, in which sulfate contents are above 30,000 ppm. The loss of stiffness followed the same trend as the sulfate content in the soils, with the highest sulfate content soils showing the highest stiffness loss with time. In Childress and Austin soils, the percent loss of stiffness was higher than 50%.

The ratio of the difference between the initial and final shear moduli values divided by the elapsed time period gives the slope of the line in MPa/hr. From the slope of the line, threshold stiffness loss values due to lime treatment are calculated for the high sulfate soils. Initial and final shear moduli values, stiffness loss and slope of the line for natural and 6%-lime treated soils at optimum and wet of optimum moisture contents is presented in Tables 7.1 thru 7.4.

Table 7.1 Stiffness Loss for Natural Soils @ OMC (MPa/Hr.)

Soil	**Sulfate Content, ppm**	**Natural Soils**			
		Initial	**Final**	**Loss**	**Slope of the Line (MPa/Hr.)**
Austin	36,000	59.6	54.2	5.4	0.025
Childress	44,000	70.0	60.7	9.3	0.043
Dallas	12,000	57.2	51.0	6.2	0.028
FM-1417	24,000	53.7	48.9	4.8	0.022
Riverside	20,000	65.9	57.1	8.8	0.041
US-82	12,000	56.0	50.1	5.9	0.027

Table 7.2 Stiffness Loss for Natural Soils @ WOMC (MPa/Hr.)

Soil	**Sulfate Content, ppm**	**Natural Soils**			
		Initial	**Final**	**Loss**	**Slope of the Line (MPa/Hr.)**
Austin	36,000	57.5	54.9	2.6	0.012
Childress	44,000	64.3	56.2	8.1	0.038
Dallas	12,000	47.3	42.4	4.9	0.022
FM-1417	24,000	46.6	42.9	3.7	0.017
Riverside	20,000	57.0	50.4	6.6	0.041
US-82	12,000	49.5	44.4	5.1	0.024

Table 7.3 Stiffness Loss for 6% Lime Treated Soils @ OMC (MPa/Hr.)

Soil	**Sulfate Content, ppm**	**6% Lime Treated Soils**			
		Initial	**Final**	**Loss**	**Slope of the Line (MPa/Hr.)**
Austin	36,000	469.7	223.0	246.7	1.14
Childress	44,000	535.8	219.2	316.6	1.46
Dallas	12,000	447.5	249.2	198.3	0.92
FM-1417	24,000	413.7	198.9	214.8	0.99
Riverside	20,000	504.1	333.2	170.9	0.79
US-82	12,000	407.4	245.7	161.7	0.75

Table 7.4 Stiffness Loss for 6% Lime Treated Soils @ WOMC (MPa/Hr.)

Soil	Sulfate Content, ppm	6% Lime Treated Soils			
		Initial	Final	Loss	Slope of the Line (MPa/Hr.)
Austin	36,000	335.3	234.5	100.8	0.45
Childress	44,000	347.8	258.7	89.1	0.41
Dallas	12,000	273.2	219.3	53.9	0.25
FM-1417	24,000	262.6	217.8	44.8	0.21
Riverside	20,000	326.7	258.6	68.1	0.31
US-82	12,000	316.7	226.3	90.4	0.42

From Table 7.1, it can be seen that highest stiffness loss was observed in the Childress soil (0.043 MPa/Hr.); whereas, FM-1417 soil (0.022 MPa/Hr.) showed lowest loss of stiffness in untreated natural condition at optimum moisture content. At wet of optimum moisture condition, the Riverside soil showed the highest loss of stiffness (0.041 MPa/Hr.); whereas, the Austin soil showed the lowest loss of stiffness (0.012 MPa/Hr.). For the high sulfate soils considered in the current study, the threshold stiffness loss in natural condition was 0.03 MPa/hr. at optimum and 0.025 MPa/hr. at wet of optimum moisture content.

For 6% lime-treated soils at optimum and wet of optimum moisture conditions, the stiffness loss values were much higher than the same of natural soils. In Austin, Childress and FM-1417 soils, the percent loss of stiffness was higher than 50%. At optimum moisture condition, the stiffness loss was the highest in the Childress soil (1.46 MPa/hr.), followed by Austin soil (1.14 MPa/Hr.) and FM-1417 Soil (0.99 MPa/Hr.). At wet of optimum moisture condition, the stiffness loss was highest in Austin soil (0.45 MPa/Hr.) and lowest in FM-1417 soil (0.21 MPa/Hr.). For the high sulfate soils considered in the current study, the threshold stiffness loss at 6% lime treatment was 1 MPa/Hr. at optimum and 0.34 MPa/Hr. at wet of optimum moisture content. The observed threshold stiffness values are in good agreement with the sulfate contents since the soils with highest sulfate content (Childress and Austin) experienced the highest loss of stiffness.

This study clearly demonstrated that stiffness measurements using shear wave velocity-based techniques can be confidently used to evaluate the on-set of soil heaving. This means that the non-destructive technique using shear wave measurements can provide quick assessments of sulfate heaving in the lime-treated soils.

7.3 Observations

In the current research study, six high sulfate soils from the states of Texas in natural and treated conditions were subjected to shear moduli measurements using the bender elements. Following conclusions were made from the current study.

1. Bender element technique is successfully used in the current research to measure the small strain shear modulus (G_{max}) of lime treated high sulfate soils. Measurement of shear modulus in treated sulfate bearing soils is an important indicator of on-going sulfate heave reactions and subsequent material degradation.
2. Small strain shear modulus is higher at optimum moisture content compared to the wet of optimum moisture content in both natural and treated soils due to close packing and denser configuration at optimum moisture content. Small strain shear modulus of lime treated soils is much higher than the natural soils.
3. Measured stiffness loss is higher in lime treated soils compared to natural soils. Threshold stiffness loss for natural soils varied from 0.03 MPa/Hr. at optimum and 0.025 MPa/Hr. at wet of optimum moisture contents. For 6% lime treated soils, threshold stiffness loss is 1 MPa/Hr. at optimum and 0.34 MPa/Hr. at wet of optimum moisture content.

Overall, soils with high sulfate contents exhibited higher stiffness losses with time during the monitoring time period. This shows that non-destructive shear wave based technologies could be good tools to assess sulfate heaving in the treated high sulfate soils.

CHAPTER 8

CONCLUSIONS AND RECOMMENDATIONS

8.1 Summary and Conclusions

The main objectives of this research are to study the heaving mechanisms in chemically treated high sulfate soils (sulfate content > 8,000 ppm) and to develop techniques for stabilization of these soils. Other objectives are to explore the use of non-destructive shear wave velocity measurements on the treated sulfate rich soils to assess the on-set of sulfate heaving in relatively short time period.

In order to achieve these objectives, an experimental program was designed and performed at the geotechnical laboratory of The University of Texas at Arlington. Objectives of the research were accomplished. Test results from the experimental program were analyzed to understand the effects of various physical and chemical compositional factors of soils on the volume change behavior of chemically treated high sulfate soils. Based on the current research, the following conclusions are made:

1. The formation of expansive Ettringite mineral was attributed to the presence of calcium from lime, reactive alumina from soil, soluble sulfates and moisture availability in the treated soils. It was observed in the current study that volumetric shrinkage is not a concern in chemically treated high sulfate soils since all the lime treated soils exhibited low shrinkage strains compared to the natural soils. It is again the swell behavior of chemically treated high sulfate soils that can distress the civil infrastructure. Volumetric swell strains of high sulfate soils varied from 8.8% to 24.4%.
2. High sulfate soils in the current study were stabilized using "pre-compaction" mellowing technique. Mellowing periods considered were 0, 3 and 7 days. It was observed that mellowing is effective in four of the six soils considered in the current study. Sulfate levels in these soils were below 30,000 ppm. The soils that could not be stabilized by mellowing have sulfate contents above 30,000 ppm.
3. Sulfate content has a significant effect on the stabilization of high sulfate soils. The purpose of mellowing is to solubilize the entire available sulfates during mellowing period and which in turn

contribute to the early formation of Ettringite so that no sulfates will be available for further Ettringite formation after re-compaction. In two of the six soils considered in the current study, the entire soluble sulfates could not be dissolved during mellowing period due to large amounts of high sulfate contents in these soils as well as low solubility of sulfates in these soils. These are some of the reasons that has resulted in mellowing not being effective on these two soils.

4. The compaction void ratios also have significant influence on the Ettringite growth and subsequent heaving since soils with higher void ratios can accommodate the initial Ettringite growth. In later stages even if there is Ettringite growth, strength giving pozzolanic reactions impart strength to the soils which resists further swelling of the soil. This is not true in soils with low compaction void ratios as they could not accommodate the initial Ettringite growth leading to weakening of soil and subsequent swelling.
5. Soil compositional factors such as clay mineralogy, reactive alumina and silica contents play an important role in stabilizing chemically treated high sulfate soils. It is observed in the current study that soils with Kaolinite mineral dominance are more prone to Ettringite induced heaving when compared to the soils with other mineral dominance provided all other factors (sulfate content, reactive alumina and silica contents) remain the same. Since reactive alumina and silica are major constituents of the strength related pozzolanic reactions, soils with low reactive alumina, silica and high sulfate contents showed dominance of Ettringite reactions over the pozzolanic reactions which is manifested in the form of excessive heaving.
6. The reasons for ineffectiveness of mellowing in two of the six soils are explained by using the mass-volume calculations and *'Gibb's Free Energy'* approaches. It is observed from the mass-volume calculations that soils with high sulfate contents and low void ratios are the candidates for enhanced Ettringite reactions and higher swell magnitudes. Gibbs free energy calculations indicated that deleterious Ettringite reactions dominate the strength related pozzolanic reactions in soils with low reactive alumina, silica and high sulfate contents.
7. In order to incorporate sustainability principles into soil stabilization, portion of lime is replaced with class f fly ash and combined lime and fly ash treatment studies were hence conducted on two soils

using mellowing technique. The two soils considered for both lime and class f fly ash studies have sulfate contents below 30,000 ppm. Results showed that combined lime and fly ash treatment effectively reduced swell strains in these soils below natural soil swell levels.

8. A swell prediction model is developed based on the 'effective sulfate content' (difference between the initial sulfate content and sulfate content after the swell tests). Results obtained by this model indicated that swell prediction based entirely on effective sulfate contents can give misleading results since the observed swell is a combination of complex interactions among soil sulfates, alumina, silica and compaction moisture content.
9. Threshold void ratio frame work comprising of natural soil void ratio (compaction density) and sulfate content is developed to predict Ettringite induced heaving in chemically treated high sulfate soils at different mellowing periods. This model provided good predictions for the present test soils. This model needs to be further evaluated for outside test soils.
10. In the current study, Ettringite formation and growth were assessed successfully using non-destructive seismic testing. Bender Element tests conducted on chemically treated high sulfate soils inundated completely in water indicated material degradation and subsequent small strain shear modulus reductions indicating these soils are prone to Ettringite induced heaving upon lime treatment.

8.2 Future Research Recommendations

To enhance the knowledge and understanding of heaving mechanisms and to develop sustainable stabilization techniques for chemically treated high sulfate soils, the following recommendations are made:

1. Samples in the current research are mellowed at 100% humidity and control temperature environment. The effectiveness of mellowing technique at higher temperatures as observed in the real field situations needs to be investigated.

2. Method to quantify the amount of alumina and silica participating in the deleterious (Ettringite) and strength giving (pozzolanic) reactions separately has to be developed.

3. Practical method to calculate the amount Ettringite/Thaumasite formed based on the sulfate, reactive alumina and silica contents.

4. Effectiveness of pre-compaction mellowing technique in stabilizing ‘cement’ treated high sulfate soils needs to be assessed.

5. More experimental data comprising of different soil series and mellowing periods can be used to develop generic charts for prediction of Ettringite induced heaving in lime treated high sulfate soils.

6. Stabilization potential of combined lime and ground granulated blast furnace slag (GGBFS) treatment with pre-compaction mellowing technique in stabilizing high sulfates can be assessed.

7. Addition of amorphous alumina and silica and their effects on chemical stabilization of high sulfate soils needs to be studied.

APPENDIX A

PROCTOR COMPACTION CURVES

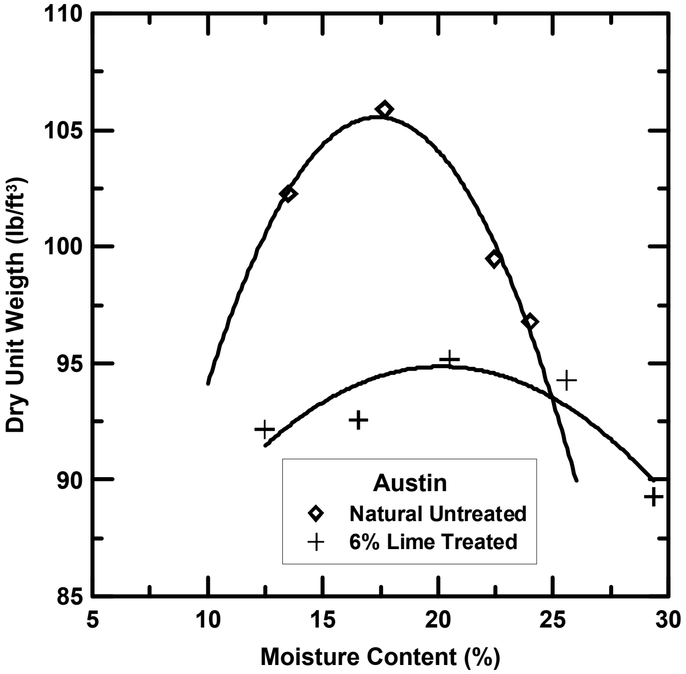

Figure A.1 Standard Proctor Compaction Curves for Untreated and Lime Treated Austin Soil

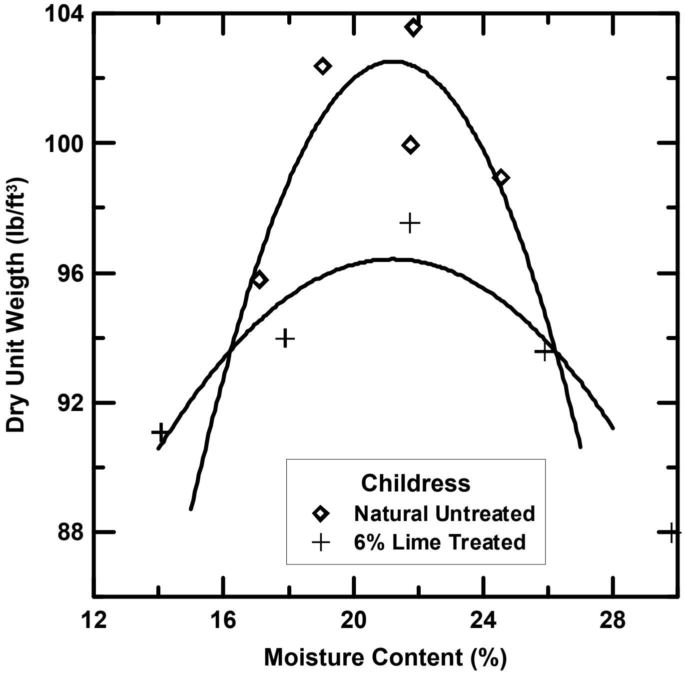

Figure A.2 Standard Proctor Compaction Curves for Untreated and Lime Treated Childress Soil

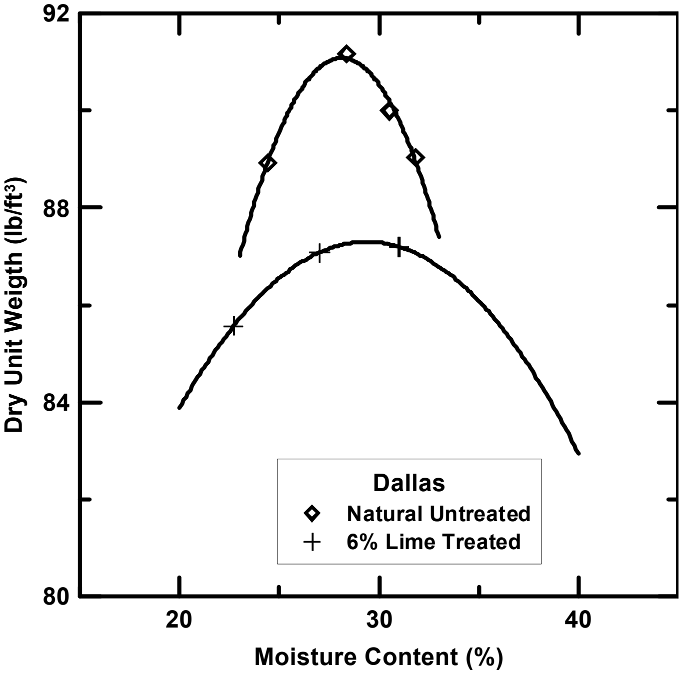

Figure A.3 Standard Proctor Compaction Curves for Untreated and Lime Treated Dallas Soil

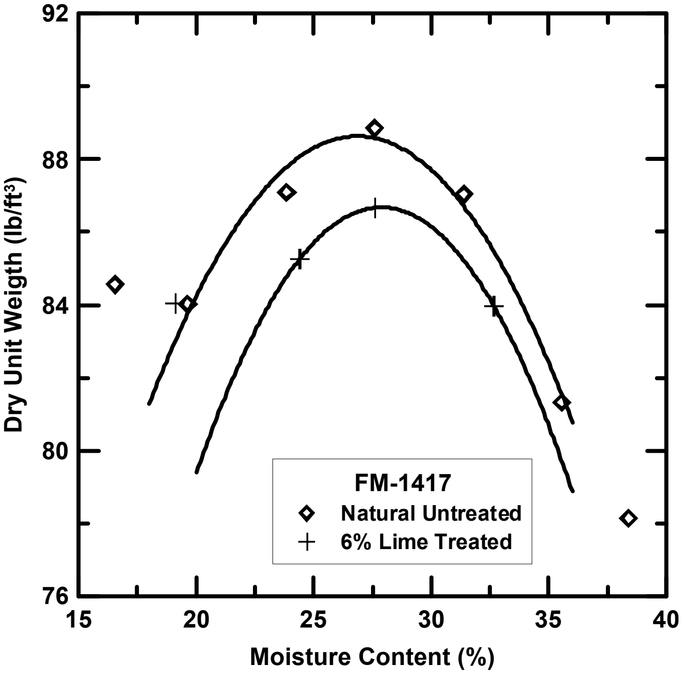

Figure A.4 Standard Proctor Compaction Curves for Untreated and Lime Treated FM-1417 Soil

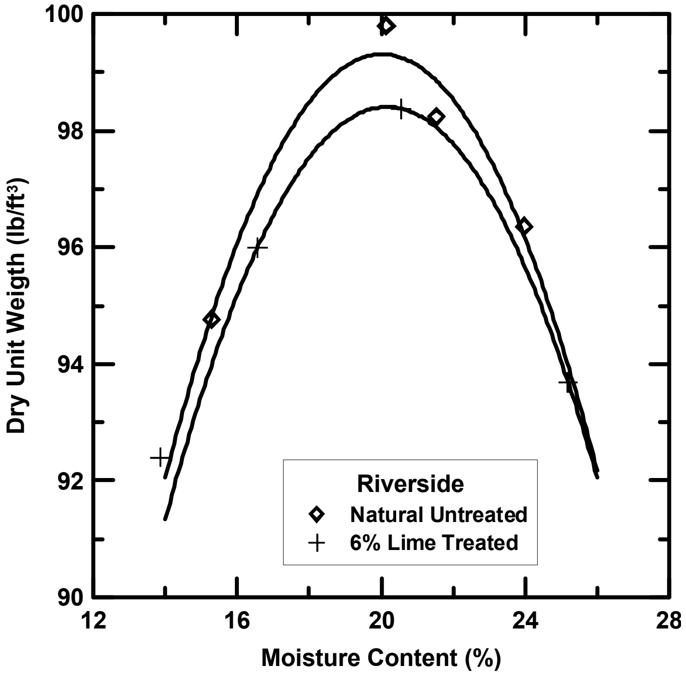

Figure A.5 Standard Proctor Compaction Curves for Untreated and Lime Treated Riverside Soil

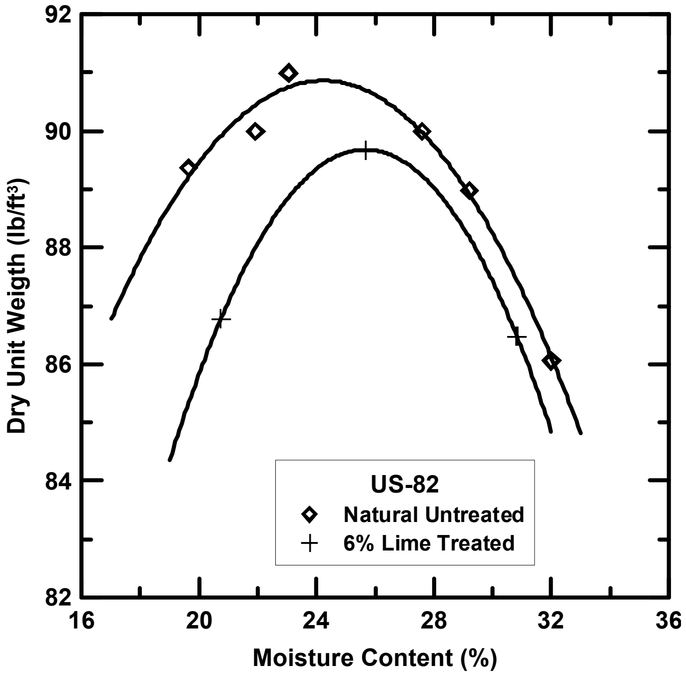

Figure A.6 Standard Proctor Compaction Curves for Untreated and Lime Treated US-82 Soil

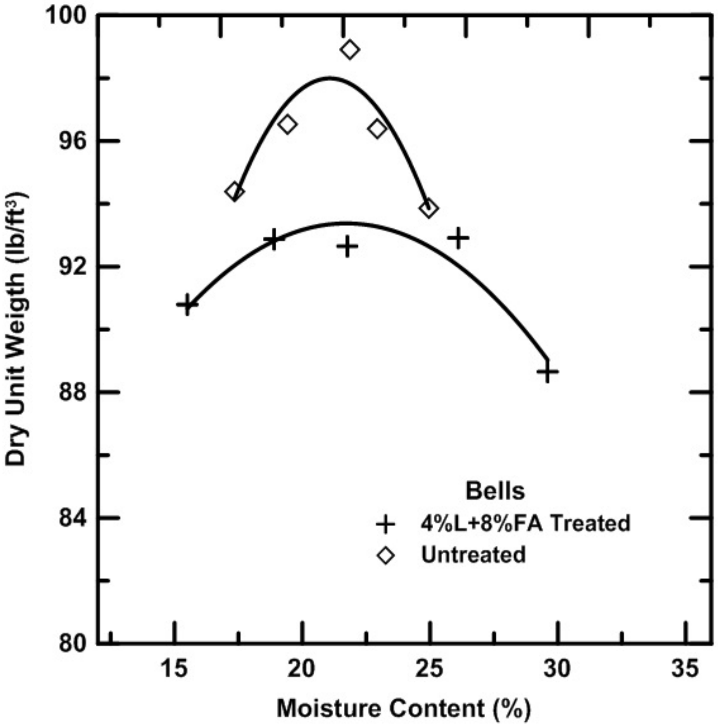

Figure A.7 Standard Proctor Compaction Curves for Untreated and Combined Lime Fly Ash Treated Bells Soil

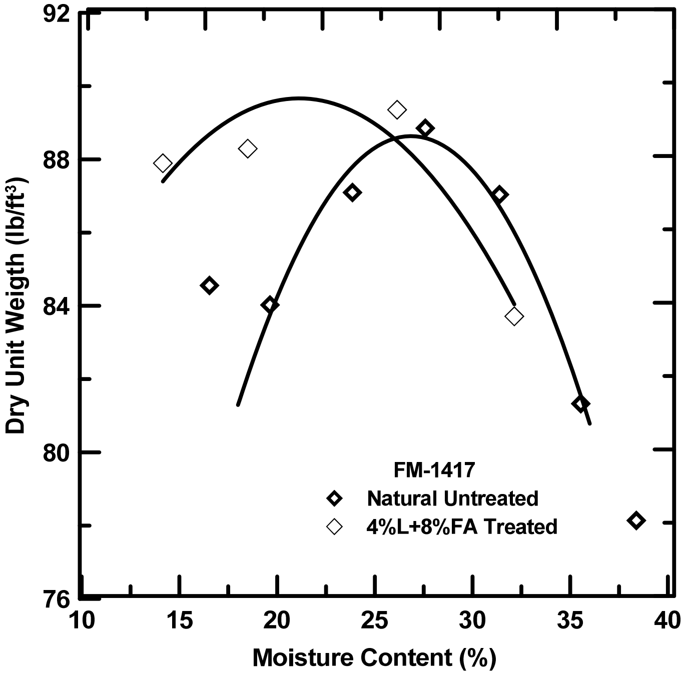

Figure A.7 Standard Proctor Compaction Curves for Untreated and Combined Lime Fly Ash Treated FM-1417 Soil

REFERENCES

1. Adams, A. G., Dukes, O. M., Tabet, W., Cerato, A. B., and Miller, G. A. (2008). “Sulfate induced heave in Oklahoma soils due to lime stabilization”. GeoCongress 2008, ASCE.
2. Addison, M. B., and Petry, T. M. (1998). “Optimizing Multiagent, Multi-Injected Swell Modifier”. Transportation Research Record: Journal of the Transportation Research Board, No. 1611, TRB, National Research Council, Washington, D.C., 1998, pp. 38-45.
3. ASTM (1995). “Annual Book of ASTM Standards,” Section 4, Construction, Vol. 4.08 Soil and Rock; Dimension Stone; Geosynthetics, American Society of Testing and Materials, Philadelphia, Pennsylvania.
4. Bagley, A., Cesare, J. A. (2009). “Case history: sulfate-induced heave of lime treated soils beneath a structure in western Colorado”. Forensic Engineering, 2009.
5. Berger, E., Little, D. N. and Graves, R. (2001). “Technical Memorandum: Guidelines for Stabilization of Soils Containing Sulfates”. http://www.lime.org/publications.html Accessed September, 2010.
6. Bolander, P. (1997). “Chemical Additives for Dust Control What We Have Used and What We Have Learned”. Transportation Research Record: Journal of the Transportation Research Board, No. 1589, TRB, National Research Council, Washington, D.C., 1997, pp. 42-49.
7. Burkart, B., Goss, G. C. and Kern, J. P. (1999). “The Role of Gypsum in Production of Sulfate-Induced Deformation of Lime-Stabilized Soils”. Environmental and Engineering Geoscience, Vol. V, No. 2, 1999, pp. 173-187.
8. Carter, D. L., Mortland, M. M. and Kemper, W. D., (1986). “Specific Surface” In A. Klute(ed), Methods of Soil Analysis, Part 1, 2nd. ed., Agronomy 9:413-423.
9. Chapman, H. D., (1965). “Cation-exchange capacity” In: C. A. Black ed., Methods of soil analysis - Chemical and microbiological properties, Agronomy 9, 891-901.
10. Chavva, P. K., Vanapally, S. K., Puppala, A. J. and Hoyos, L. (2005), “Evaluation of Strength, Resilient Moduli, Swell, and Shrinkage Characteristics of Four Chemically Treated Sulfate Soils from North Texas”, GSP 136, ASCE Special Publication, Geofrontiers, Austin, 2005.
11. Chakkrit, S., Puppala, A. J., Chikyala, V., Saride, S., and Hoyos, L.R. (2008). “Combined Lime and Cement Treatment of Expansive Soils with Low to Medium Soluble Sulfate Levels”. Geocongress, 2008.
12. Chen, F. H. (1988). Foundations on Expansive Soils, Developments in geotechnical engineering, Vol.12, pp.12.

13. Chen, D. H., Harris, P., Scullion, T., and Bilyeu, J. (2005). “Forensic Investigation of a Sulfate-Heaved Project in Texas”. Journal of Performance of Construction Facilities, ASCE, November 2005, pp. 324-330.
14. Chittoori, B. C. S. (2008). “Clay mineralogy effects on long-term performance of chemically treated expansive clays.” Ph.D. dissertation, Univ. of Texas at Arlington, TX, 326.
15. Chittoori, B., and Puppala, A. J., (2011). Quantitative estimation of clay mineralogy in fine-grained soils. Journal of Geotechnical and Geoenvironmental Engineering, v 137, n 11, p 997-1008, 16 December 2011.
16. Clesceri, L. S., Greenberg, A. E., Trussell, R. R. (Eds), 1989. Standard Methods for the Examination of Water and Wastewater. 17th ed., American Public Health Association, American Water Works Association and Water Pollution Control Federation, Washington, D.C., pp.4-75-4-81.
17. Cohen, M. D., (1983). “Theories of expansion in sulfoaluminate–type expansive cements: schools of thought”. Cement and Concrete Research, Vol. 13, pp. 809-818.
18. Deepthi, V., Puppala, A. J., and Chakkrit, S. (2007). “Field studies rigid pavement sections built on stabilized soils”. GSP 169 Soil and Material Inputs for Mechanistic-Empirical Pavement Design, GeoDenver 2007.
19. Deng, M., and Tang, M. (1994). “Formation and Expansion of Ettringite Crystals”. Cement and Concrete Research, Vol. 24, No. 1, 1994, pp. 119-126.
20. Dermatas, D. (1995). “Ettringite-induced Swelling in Soils: State-of-the-art.” Applied Mechanics Rev, Vol. 48, pp. 659-673.
21. Dubbe, D. D., Usman, M. A. and Moulton, L. K. (1997). “Expansive Pyritic Shales”. Transportation Research Record 993, TRB, National Research Council, Washington, D.C., 1997, pp. 19-27.
22. Eades, J., L., and Grim, R., E., “A Quick Test to Determine Lime Requirements for Soil Stabilization.” Highway Research Record 139, HRB, National Research Council, Washington D.C., 1966, pp. 61-72.
23. Ferris, G. A., Eades, J. L., McClellan, G. H., and Graves, R.E. (1991). “Improved characteristics in sulfate soils treated with barium compounds before lime stabilization”. Transportation Research Record 1295, TRB, National Research Council, Washington D.C., 1991.
24. Floyd, M., Czerewko, M., Cripps, J.C., and Spears, D.A, (2003). Pyrite oxidation in lower Lias Clay at concrete highway structures affected by thaumasite, Gloucestershire, UK. Cement and Concrete Composites, 25, pp 1015-1024. ISSN 958-9465(03)00123-9.
25. Foster, M. (1953) Geochemical Studies of Clay Minerals III. The determination of free silica and free alumina in Montmorillonites, Geochemica Cosmochimica Acta, Vol. 3, pp. 143-154.

26. FUJI, K.; KONDO, W. Estimation of thermochemical data for Calcium Silicate Hydrate (C-S-H). Journal of the American Ceramic Society, 1983, vol. 66, p. C220- C221.

27. Gaily, A. H. (2012). “Engineering Behavior of Lime Treated High Sulfate Soils”. M.S. Thesis, The Univ. of Texas at Arlington, Arlington, Texas.

28. Giummarra, G. J., Foley, G. and Cropley, S. (1997). “Dust Control Australian Experiences with Various Chemical Additives”. Transportation Research Record: Journal of the Transportation Research Board, No. 1589, TRB, National Research Council, Washington, D.C., 1997, pp. 50-53.

29. Griffin. (2001). ‘‘Evaluation of Sulfate Resistant and Ordinary Portland Cements For Effective Stabilization Of Natural Sulfate Rich Subgrades”. M.S. Thesis, The Univ. of Texas at Arlington, Arlington, Texas.

30. Harris, P., T. Scullion, S. Sebesta, and G. Carlos, (2003), “Measuring Sulfate in Subgrade Soil - Difficulties and Triumphs.” Transportation Research Record, No. 1837, National Research Council, Washington, D.C., pp. 3-11.

31. Harris, J. P., Sebesta, S., and Scullion, T. (2004). “Hydrated Lime Stabilization of Sulfate-bearing Vertisols in Texas”. Transportation Research Record 1868, TRB, National Research Council, Washington, D.C., 2004, pp. 31-39.

32. Harris, J. P., Scullion, T., and Sebesta, S. (2004). “Hydrated Lime Stabilization of Sulfate-bearing Soils in Texas”. Texas Transportation Institute Research Report 0-4240-2, College Station, Texas, 2004.

33. Harris, P., Voldt, J. V., Sebesta, S., and Scullion, T. (2005). “Recommendations for stabilization of high-sulfate soils in Texas”. Technical report 0-4240-3, Texas Transporation Institute.

34. Harris, J. P., Von Holdt, J., Sebesta, S., and Scullion, T. (2006). “Recommendations for Stabilization of High-Sulfate Soils in Texas”. Transportation Research Record No. 1952, TRB, National Research Council, Washington, D.C., 2006, pp. 71-79.

35. Harris, J. P. (2006). Develop Guidelines and Procedures for Stabilization of Sulfate Soils. Texas Transportation Institute, Project Summary Report 0-4240-S.

36. Hagerty, D. J., Ullrich, C. R., Denton, M. M. (1990). “Microwave drying of soils.” Geotechnical Testing Journal, v 13, n 2, Jun, 1990, p 138-141.

37. Hausmann, M. R. (1990). “Engineering principles of ground modification”, McGraw–Hill, New York.

38. Hunter, D. (1988). “Lime-Induced Heave in Sulfate-Bearing Clay Soils,” Journal of Geotechnical Engineering, ASCE, Vol. 114, pp. 150-167.

39. Hunter, D. (1989). “The Geochemistry of Lime-Induced Heave in Sulfate Bearing Clay Soils”. (Dissertation) University of Nevada, Reno, 1989.

40. Higgins, D. D. (2005). “Soil Stabilization with Ground Granulated Blast furnace Slag”. UK Cementitious Slag Makers Association (CSMA), September 2005.

41. Inthrasombat, N. (2003). ‘‘Ettringite formation in lime treated sulfate soils: Verification by mineralogical and swell testing.’’ M.S. Thesis, The Univ. of Texas at Arlington, Arlington, Texas.

42. Jackson, M. L. (1958). “Soil chemical analysis.” Prentice Hall Inc., Englewood Cliffs, N.J.

43. Joffee, J.S., (1949). Pedology, Pedology Publications, New Brunswick, New Jersey.

44. Jung, S., and M. C. Santagata. (2009). Mitigating the Expansive Behavior of Chemically Treated Soils.Publication FHWA/IN/JTRP-2009/02. Joint Transportation Research Program, Indiana.

45. Kadam, R. (2003). “Evaluation of low strain shear moduli of stabilized sulfate bearing soils using bender elements.” MS thesis, The Univ. of Texas at Arlington, Arlington, Tex, 155.

46. Knudsen D., Peterson G.A. and Pratt P.F. (1982). “Lithium, sodium and potassium.” In: Page A.L. (ed.,) Methods of Soil Analysis, Part 2. American Society of Agronomy, Madison, WI, 229–231.

47. Kollman, H., Strubel, G., and Trost, F. (1977). "Mineralsynthetische unterSuchungen zu treibursachen durch Ca-Al-sulfat und Ca-Si-carbonat-sulfat hydrat." Tonindustrie Zeitung, 101(3), Germany, pp. 63-70.

48. Kollman, H. (1978). "Minerlogische untersuchungen uber ausbluhungs undtreiberscheinungen and baustoffen durch sulfate." Giessener Geologische Schriften, 18, Giessen, Germany, pp.159

49. Kota, P. B. V. S., Hazlett, D., and Perrin, L. (1996) “Sulfate-Bearing Soils: Problems with Calcium Based Stabilizers”. Transportation Research Record 1546, TRB, National Research Council, Washington, D.C., 1996, pp. 62-69.

50. Krauskopf, K. and Bird, D.K., 1995. Introduction to Geochemistry. Third Edition, 647 pp.

51. Lambe, T. W and Whitman R. V. (2000). Soil Mechanics, John Wiley & Sons, New York, pp. 45-51.

52. Little, D. N. (1995). “Stabilization of Pavement Subgrades & Base Courses with Lime.” Kendall/Hunt Publishing Company, Iowa.

53. Little, D. N., Herbert, B., and Kunagalli, S. N. (2005). “Ettringite formation in lime-treated soils–Establishing thermodynamic foundations for engineering practice.” Transportation Research Record. 1936, Transportation Research Board, Washington, D.C., 2005, pp. 51–59.

54. Little, D. N., Nair, S. (2009). “Validation of Sensitivity of Sulfate-Bearing Soils to Ettringite Growth by Differential Scanning Calorimetry”. Transportation Research Record: Journal of the Transportation Research Board, No. 2104, Transportation Research Board of the National Academies, Washington, D.C., 2009, pp. 63–70.

55. Little, D. N., Nair, S., and Herbert, B. (2010). “Addressing Sulfate-Induced Heave in Lime Treated Soils”. Journal of Geotechnical and Geoenvironmental Engineering, ASCE, January 2010, pp. 110-118.

56. Mallat A. (2006). “LMSSMat”, Retrieved from http://www.mssmat.ecp.fr/Axe-de-recherche,1529 , accessed on 11/04/2010.

57. Marquart, D. K. (1995). “Chemical Stabilization of Three Texas Vertisols with Sulfonated Naphthalene”. M.S. Thesis, Texas A&M University, May 1995, 45p.

58. McCarthy, M. J., Laszlo, J. C., Sachdeva, A., Jones, M. R. (2009). “Role of fly ash in mitigation of swelling in lime-stabilised sulfate bearing soils”. 2009 World of coal ash conference, May 4-7, 2009, Lexington, KY, USA.

59. McKennon, Hains, N. L., and Hoffman, D. C. (1993). Method for Preventing the Adverse Effects of Swell in Sulfate Bearing, Expansive Clay Soils. United States Patent-5228808, 1993.

60. Mehta, P. K., and Klein, A. (1966). “Investigation of hydration products in the system 4CaO.3Al2O3.SO3.CaSO4.CaO.H2O”. Highway Research Board, Special report no.90, pp. 328-352.

61. Mehta, P. K., (1973). “Mechanism of expansion associated with Ettringite formation”. Cement and Concrete Research, 3, pp. 1-6.

62. Mehta, P. K., (1973). “Effect of lime on hydration of pastes containing gypsum and calcium aluminate or calcium sulfoaluminate”. Journal of America Ceramic Society, 56, pp. 315 - 319.

63. Mehta, P. K., and Wang, S. (1982). Cement Concrete Research, Vol.12, pp. 121-122.

64. Mitchell, J. K. (1986). “Practical Problems from Surprising Soil Behavior.” Journal of Geotechnical Engineering Division, ASCE, Vol. 112, No. 3, pp. 259-289.

65. Mitchell, J. K., and Dermatas, D. (1992). “Clay Soil Heave Caused By Lime-Sulfate Reactions.” ASTM Special Publication 1135, pp. 41-64.

66. Mitchell, J. K. and Soga, K. (2005). “Fundamentals of soil behavior.” 3rd Ed., Wiley, New York.

67. Moschner, G., Lothenbach, B., Winnefeld, F., Ulrich, A., Figi, R., and Kretzschmar, R. (2009). “Solid solution between Al- Ettringite and Fe-Ettringite”. Cement and Concrete Research, Vol. 39, pp. 482-489.

68. Nelson, D. J. and Miller, J. D. (1992). “Expansive soils: problems and practice in foundation and pavement engineering,” John Wiley & Sons. 259 p.

69. Ogawa, K., and Roy, D. M. (1982). “C4A3S Hydration, Ettringite Formation and its Expansion Mechanism: Part II, Microstructural Observation of Expansion”. Cement and Concrete Research, vol.12, pp. 101-109.

70. Pauling, L., 1960. The Nature of the Chemical Bond. 3rd edition, Cornell University Press, Ithica, NY.

71. Perrin, L. (1992). “Expansion of Lime-Treated Clays Containing Sulfates”. Proceedings of 7th International Conference on Expansive Soils, Vol. 1, ASCE Expansive Soil Research Council, New York, 1992, pp. 409-414.
72. Petry, T. M., 1994, “Studies of Factors Causing and Influencing Localized Heave of Lime Treated Clay Soils (Sulfate Induced Heave),” final report, University of Texas, Department of Civil Engineering, Arlington, TX, September.
73. Petry, T. M. and Little, D. N., (1992), “Update on Sulfate Induced Heave in TreatedClays: Problematic Sulfate Levels,” Transportation Research Board, TRR 1362, Washington, DC.
74. Punthutaecha, K., Puppala, A.J., Vanapalli, S., Inyang, H. “Volume Change Behaviors of Expansive Soils Stabilized with Recycled Ashes and Fibers.” ASCE, Journal of Materials, March/April, 2006, pp. 295-306.
75. Puppala, A. J., Hanchanloet, S., Jadeja, M., Burkart, B. (1999). “Sulfate InducedHeave Distress: A Case Study”. Proceedings, Transportation Research Board Annual Meeting, Washington D.C, USA.
76. Puppala, A. J., and Musenda, C. (2000). “Effects of Fiber Reinforcement on Strength and Volume Change Behavior of Expansive Soils.” Transportation research board. 79thAnnual Meeting, paper No. 00-0716, Washington, D.C.
77. Puppala, A. J., Viyanant, C., Kruzic, A. P., and Perrin, L., (2002). Evaluation of a Modified Soluble Sulfate Determination Method for Fine-Grained Cohesive Soils, Geotechnical Testing Journal, Volume 25, Issue 1, pp. 85–94.
78. Puppala, A. J., Wattanasanticharoen, E. and Punthutaecha, K. (2003). “Experimental Evaluations of Stabilization Methods for Sulphate-rich Expansive Soils.” Ground Improvement Vol. 7, No. 1, 2003. pp. 25-35.
79. Puppala, A. J., Griffin, J. A., Hoyos, L. R. and Chomtid, S. (2004). “Studies on Sulfate-Resistant Cement Stabilization Methods to Address Sulfate-Induced Soil Heave.”Journal of Geotechnical and Geoenvironmental Engineering, 130, pp. 391-402.
80. Puppala, A.J., Katha, B., Hoyos, L.R. (2004). Volumetric Shrinkage Strain Measurements in Expansive Soils Using Digital Imaging Technology. ASTM Geotechnical Testing Journal, Vol. 27, No. 6, November, 2004, pp.547-556.
81. Puppala, A. J., Intharasombat, N., Vempati, R. (2005). "Experimental Studies on Ettringite Induced Heaving in Soils.” ASCE Journal of Geotechnical and Geoenvironmental Engineering, Vol. 31, No. 3, pp. 325-337.
82. Puppala, A. J., Kadam, R., Madhyannapu, R., and Hoyos, L. R. (2006). “Small-Strain Shear Moduli of Chemically Stabilized Sulfate-Bearing Cohesive Soils”. Journal of Geotechnical and Geoenvironmental Engineering, ASCE, March 2006, pp.322-336.

83. Puppala, A. J., Saride, S., Dermatas, D., Al-shamrani, M., and Chikyala, V. (2010). “Forensic Investigations to Evaluate Sulfate-Induced Heave Attack on a Tunnel Shotcrete Liner”. Journal of Materials in Civil Engineering, ASCE, September 2010, pp.914-922.

84. Puppala, A.J., Naga S. Talluri., Bhaskar S. Chittoori and Ahmed Gaily. (2012). Lessons Learned from Sulfate Induced Heaving Studies in Chemically Treated Soils. Proceedings of the International Conference on Ground Improvement and Ground Control. Research Publishing, Vol. 1, November, 2012, pp.85-98.

85. Puppala, A.J., Naga S. Talluri., Ahmed Gaily. And Bhaskar S. Chittoori. (2013). Heaving Mechanisms in High Sulfate Soils. Proceedings of the 18th International Conference on Soil Mechanics and Geotechnical Engineering, Paris 2013.

86. Rajasekaran, G. (2005). “Sulphate attack and Ettringite formation in the lime band cement stabilized marine clays”. Ocean Engineering, 32 (2005), pp. 1133–1159.

87. Raymond, N. Y., and Ouhadi, V. R. (2006). “Experimental study on instability of bases on natural and lime/cement-stabilized clayey soils”. Applied Clay Science, 35 (2007), pp. 238–249.

88. Rollings Jr., R. S., Burkes, J. P., and Rollings, M. P. (1999). “Sulfate Attack on Cement Stabilized Sand.” Journal of Geotechnical and Geoenvironmental Engineering, vol. 125 No.5, pp. 364-372.

89. Rollings, R. S., Rollings, M. P., and Poole, T., Wong, G. S., and Gutierrez, G. (2006). “Investigation of Heaving at Holloman Air Force Base, New Mexico”. Journal of Performance of Constructed Facilities, ASCE, February 2006, pp.54-63.

90. Rauch, A. F., Harmon, J. S., Katz, L. E. and Liljestrand, H. M. (2002). “Measured Effects of Liquid Soil Stabilizers on Engineering Properties of Clay”. Transportation Research Record: Journal of the Transportation Research Board, No. 1787, TRB, National Research Council, Washington, D.C., 2002, pp. 33-41.

91. Schlorholtz, S., and Demirel, T. (1984). “Quick Lime-Gypsum Interactions in Stabilized Soil Bases for Concrete Highways”. Cement and Concrete Research, Vol. 14, No. 4, April 1984, pp. 529-532.

92. Santoni, R. L., Tingle, J. S. and Webster, S. L. (2002). “Stabilization of Silty-Sand with Nontraditional Additives”. Transportation Research Record: Journal of the Transportation Research Board, No. 1787, TRB, National Research Council, Washington, D.C., 2002, pp. 61-72.

93. Sarkar, S., and Little, D. (1998). “Stabilization of Sulfate-Contaminated Crushed Concrete Base with Type V Cement and Fly Ash”. Transportation Research Record No. 1611, TRB, National Research Council, Washington, D.C., 1998, pp. 3-9.

94. Sherwood, P. T. (1962). “Effect of Sulfates on Cement and Lime Treated Soils”. Highway Research Board, Bulletin 353, pp. 98-107.

95. Sherwood, P.T., (1993). Soil Stabilization with Cement and Lime. Transportation Research Laboratory State of the Art Review. HMSO

96. Solanki, P., Khoury, N., and Zaman, M. M. (2009). “A comparative evaluation of various additives used in the stabilization of sulfate bearing lean clay”. Journal of ASTM International, Vol.6, No.8.

97. Talluri, N.S., Gaily, A., Puppala, A.J., Chittoori, B.S. (2012). A Comparative Study of Soluble Sulfate Measurements. ASCE GeoCongress 2012, Oakland, California, pp. 3372-3381.

98. Talluri, N.S., Puppala, Chittoori, B.S., Gaily, A., (2013). Stabilization of High Sulfate Soils by Extended Mellowing. Journal of the Transportation Research Board, National Academy of Science, Transportation Research Board (Accepted).

99. Tasong, W. A., Wild, S. and Tilley, R. J. D. (1999). “Mechanisms by which Ground Granulated Blastfurnace Slag Prevents Sulphate Attack of Lime-Stabilized Kaolinite”. Cement and Concrete Research, Vol. 29, 1999, pp. 975-982.

100. Viyanant, C. (2000). “Laboratory Evaluation of Sulfate Heaving Mechanisms in Artificial Kaolinite Soil”. M.S. Thesis, The University of Texas at Arlington, Arlington, Texas, 2000.

101. Walker, Jr. (1992). Method for improving the characteristics of sulfate bearing soils. United States Patent. 5,122, 012.

102. Wang, L., Roy, A., Seals, R. K., and Metcalf, J. B. (2003). “Stabilization of Sulfate-Containing Soil by Cementitious Mixtures: Mechanical Properties”. Transportation Research Record. 1827, Transportation Research Board, Washington, D.C., 2003, pp. 12-19.

103. Wang, L., Roy, A., Tittsworth, R., and Seals, R. K. (2004). “Mineralogy of Soil Susceptible to Sulfate Attack after Stabilization”. Journal of Materials in Civil Engineering, August 2004, pp. 375-382.

104. Wang, L., Roy, A., Seals, R. K., and Zachary, B. (2005). “Suppression of Sulfate Attack on a Stabilized Soil”. Journal of American Ceramics Society, 88 [6] , pp. 1600–1606.

105. West, G. (1959). A Laboratory Investigation into the Effects of Elapsed Time after Mixing on Compaction and Strength of Soil Cement. Geotechnique 9(1), p22, 1959.

106. Wild, S., Kinuthia, J. M., Jones, G. I. and Higgins, D. D. (1998), “Effects of partial substitution of lime with ground granulated blast furnace slag (ggbs) on the strength properties of lime-stabilised sulphate bearing clay soils”, Engineering Geology.

107. Wild, S., Kinuthia, J. M., Jones, G. I. and Higgins, D. D. (1999), “Suppression of swelling associated with Ettringite formation in lime-stabilised sulphate- bearing clay soils by partial substitution of lime with ground granulated blast furnace slag (ggbs)”, Engineering Geology.

108. Zhiming, S. (2008). “Forensic Investigation of Pavement Premature Failure due to Soil Sulfate-Induced Heave”. Journal of Geotechnical and Geoenvironmental Engineeirng, ASCE, August 2008, pp.120-1204.

BIOGRAPHICAL INFORMATION

Nagasreenivasu Talluri was born in Khammam, Andhra Pradesh, India. He graduated from Jawaharlal Nehru Technological University, Anantapur, India, with a Bachelor' Degree in Civil Engineering in 2006. He joined Master's Program at the Indian Institute of Technology Kanpur, India and graduated in 2008. Subsequently, he worked as a Geotechnical Engineer at Tata Consulting Engineers Ltd., Bangalore, Karnataka, India for a year. During his work as a Geotechnical Engineer, he was involved in the deep and shallow foundation design and recommendation; retaining wall design; slope stability and geotechnical report verification for Thermal Power Plant structures. He decided to go to United States to pursue PhD in Geotechnical Engineering. He started his Doctoral program in the Department of Civil Engineering, University of Texas at Arlington with Geotechnical Engineering as the area of research. At UTA, he performed research in High Sulfate Soil Stabilization under the guidance of Prof. Anand J. Puppala and successfully defended his dissertation in July 2013. During the course of his study, he worked in various research areas related to ground improvement, expansive soils and soluble sulfate measurement.