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16. Abstract Over the last 15 years, the Texas Department of Transportation (TxDOT) has seen an increase in pavement failures during and immediately after construction on roads designed to last 20 years or more. The cause of many of these failures is sulfate-induced heave where an expansive mineral called ettringite is formed from a calcium-based stabilizer (lime or cement) reacting with clay and sulfate minerals (usually gypsum) in the soil. Traditionally, TxDOT has removed and replaced soils with more than 2000 ppm sulfates. Earlier in this research project, lime was identified as a plausible stabilizer in soils bearing sulfate concentrations up to 7000 ppm. This portion of the research investigates if anything can be used to stabilize soils (reduce swell and increase strength) with sulfate concentrations above 7000 ppm. Three-dimensional swell was measured on laboratory prepared specimens with sulfate concentrations of 0, 10,000, and 20,000 ppm. Twelve stabilizers were selected for the 3-D swell testing based upon positive results obtained by other researchers. Stabilizers that significantly reduced swell in the high-sulfate soils were then subjected to unconfined compressive strength testing. Three stabilizers (Claystar 7, ground granulated blast furnace slag + lime, and class F fly ash + lime) provided significant swell reduction (10 to 12 percent) over the untreated soil; two of the stabilizers were selected for strength testing. The fly ash swell test results were obtained too late to include in strength testing. The Claystar7 showed an improvement of 41 lb/in ² over the untreated sample for retained strength in the unconfined compressive strength after 10 days capillary rise. The ground granulated blast furnace slag showed a 79 lb/in ² retained strength. This project showed that soils with sulfate concentrations up to 20,000 ppm can be treated in a timely manner without having to remove the high-sulfate soil and replace it with a select material.					
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RECOMMENDATIONS FOR STABILIZATION OF HIGH-SULFATE SOILS IN TEXAS

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DISCLAIMER

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The United States government and the State of Texas do not endorse products or manufacturers. Trade or manufacturers' names appear herein solely because they are considered essential to the object of this report.

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CHAPTER 1

INTRODUCTION

Over the last 15 years, the Texas Department of Transportation (TxDOT) has seen an increase in pavement failures during and immediately after construction on roads designed to last 20 years or more. The cause of many of these failures is sulfate-induced heave (Figure 1) where an expansive mineral called ettringite is formed from a calcium-based stabilizer reacting with clay and sulfate minerals (usually gypsum) in the soil.^{1,2}



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

Many investigations have dealt with identifying failure mechanisms in high-sulfate soils.^{1,2,3,4,5} Based on findings from these researchers, four inorganic constituents have been identified as essential for sulfate-induced heave: water, calcium, aluminum, and sulfate. Hunter (1989) explained many of the chemical reactions involved in generating the vertical heaves.⁴ A more recent study using geochemical modeling illustrates the importance of aluminum availability in generating reactions causing sulfate-heave and how the Al/Si ratio may be modified to mitigate sulfate heave reactions.⁶

Previous studies of the various nonstandard (noncalcium-based) stabilizers have been criticized by nonstandard stabilizer manufacturers 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 three-dimensional (3-D) swell.

The stabilizers, in this report, were selected based upon results of previous research and recommendations of the Project Monitoring Committee (PMC). To evaluate these stabilizers, tightly controlled laboratory 3-D swell tests were made using a moderate Plasticity Index (PI) soil. For stabilizers that performed well in the initial laboratory tests, three additional soils with PIs ranging from 14 to 29 were chosen from the Dallas and Laredo Districts for more detailed testing. These two districts vary in climate as well as soil type allowing a more comprehensive evaluation of the proposed stabilizers.

This research identified what alternatives were available for stabilizing the subgrade when sulfate concentrations were too high (using constraints identified by TxDOT) for stabilization with traditional calcium-based stabilizers like lime and cement. More specifically, soils with sulfate concentrations above 10,000 ppm were stabilized with numerous nonstandard stabilizers and evaluated with respect to 3-D swell and unconfined compressive strength (UCS) because TxDOT is primarily concerned with swell reduction in plastic soils and strengthening the subgrade enough to serve as a working platform for construction.

CHAPTER 2 BACKGROUND

UPDATED SULFATE MAP

Initially, failures due to sulfate-induced heave were reported in the Dallas/Fort Worth area,^{1,2} but as this project has progressed, failures due to sulfate-induced heave have been discovered over the entire state. The map in [Figure 2](#) highlights all of the counties where sulfates have been observed (in yellow). The Eagle Ford formation, which caused many of the early failures, is also depicted on this map in green.

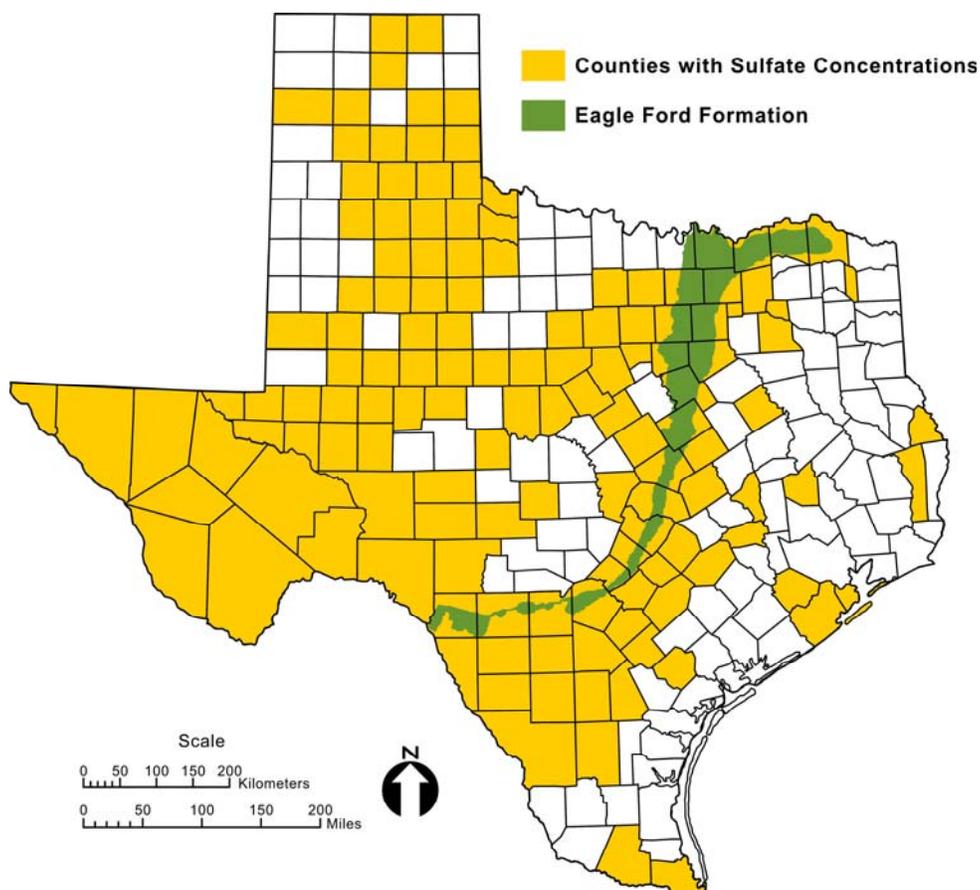


Figure 2. Map Showing Areas in Texas where Sulfates Have Been Identified.

The map in [Figure 2](#) is ever-changing because new counties are continuously being added as sulfates are discovered using the quick field test kits developed earlier in this project. A word of caution regarding this map:

- A county labeled as containing sulfates does not necessarily mean that traditional calcium-based stabilizers should be abandoned since many of the sulfate concentrations are low enough to be treated with traditional stabilizers.
- If a county does not appear to have sulfates according to this map, do not assume that lime-stabilization is without risk because it may be that sulfates have not yet been identified. The researchers recommend testing all soils for sulfates before stabilization to prevent any sulfate-induced heave before it occurs.

PREVIOUS INVESTIGATIONS

In recent years, there have been several studies focused on stabilization of soils high in sulfates. Many of these studies have focused on modifying calcium-based stabilizers with supplementary cementing materials like ground-granulated blast furnace slag (GGBFS), silica fume (SF), amorphous silica (AS), and fly ash (FA). Other investigations have concentrated on alternatives to traditional soil stabilizers like cation exchange products, enzymes, acids, emulsions, and polymers. Following is a review of the literature dealing with remediation of soils high in sulfates.

Modification with Calcium-Based Stabilizers

[Kota et al. \(1996\)](#) reviewed problematic soils in Texas and offered different stabilization options to reduce the swell caused by calcium-based stabilizers in sulfate rich soils.⁷ They recommended double application of lime for low levels of soluble sulfates, and they recommended geotextiles and select fill for higher levels of sulfates in high PI soils.

Substituting GGBFS for a portion of the lime used to stabilize high sulfate clay bearing soils has been shown to work by researchers in the UK.^{8,9,10,11,12} They reported significant swell reduction and strength gain in soils containing either kaolinite or

smectite as the dominant clay mineral where 5% GGBFS and 1% lime by weight was used as the stabilizer.

[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, 19% stabilizer was required to stabilize the base due to a very high moisture content in the degraded base.¹³

Researchers in Louisiana identified four materials that can be added to cement or lime to alleviate swell generated by stabilizing a gravel composed predominantly of anhydrite (CaSO_4).¹⁴ These materials include ground-granulated blast furnace slag, Class C fly ash, silica fume, and amorphous silica.

Four stabilizer treatments were tried on high-sulfate soils in Arlington, Texas.¹⁵ Sulfate-resistant Type V cement performed the best, lime mixed with fibrillated polypropylene fibers was second, GGBFS was third, and the low-calcium Class F fly ash exhibited the lowest performance. Very high stabilizer levels (ranging from 8 to 20%) were used in these soils.

Stabilization with Non-Calcium-Based Stabilizers

In recent years, there has been a proliferation of literature regarding the effectiveness of non-calcium-based stabilizers for subgrade soil stabilization. Many of these stabilizers started as dust control agents for low volume roads. The U.S. Forest Service, the U.S. Army Corps of Engineers, and researchers in Australia have studied many of the additives listed in this report.^{16,17} In the paragraphs that follow, researchers have reviewed some of the literature relating to this topic.

[Ferris et al. \(1991\)](#) used barium hydroxide and barium chloride on three soils from California, Colorado, and Texas to reduce the soluble sulfate content.¹⁸ The barium reacts with water soluble minerals in the soil, like gypsum, to form barium sulfate, which has an extremely low solubility. After the soluble sulfates have reacted to form insoluble barium sulfate, the soil may be stabilized with traditional stabilizers (lime/cement).¹⁸

Testimonials for how well six nonstandard stabilizers (ranging from sulfonated naphthalene to enzymes and bioenzymes) performed on U.S. Forest Service roads are given in [Scholen \(1995\)](#), but there are no data to corroborate the claims.¹⁹ [Marquart](#)

(1995) stabilized three Texas Vertisols (pH ranged from basic to acidic) with a sulfonated naphthalene. His work showed that more stabilizer was required for basic soils and that there was an optimal stabilizer content for swell reduction.²⁰

The effects of a potassium stabilizer on expansive clay soils were 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.²¹

A hydrogen ion exchange chemical was investigated by Sarkar et al. (2000) to elucidate stabilization mechanisms using the chemical. They concluded that the hydrogen ion exchange chemical reduced the shrink-swell characteristics of the soil.²²

Katz et al. (2001) investigated a low pH solution of sulfonated limonene. They determined that the smectite is weathered to a less expansive mineral if the liquid stabilizer is applied in a high enough concentration.²³

The effectiveness of three liquid soil stabilizers at changing the engineering properties of clay soils was evaluated by researchers at the University of Texas.²⁴ Rauch et al. (2002) concluded that there were no significant changes in soil properties at the recommended stabilizer application rates.²⁴

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.²⁵

Petry and Das (2001) reviewed how the chemical stabilizers should be evaluated in order to obtain some standard protocol so the different stabilizers can be compared and an educated decision can be made regarding the benefits of the different stabilizers.²⁶

LIME STABILIZATION LIMITS

Based upon research reported by others,²⁷ research performed earlier in this project and documented in Research Report 0-4240-2, and experience of TxDOT employees, the following recommendations for lime stabilization in sulfate-bearing soils were used for this phase of the research.

The use of lime to stabilize high-PI clay soils containing sulfates at or below the threshold of 3000 ppm should be safe and sulfate heave should be of little concern. Special construction techniques at this threshold are not indicated; however, it should be emphasized that construction water, streams, drainage water, groundwater, and site conditions should be investigated to ensure that total sulfates do not exceed the sulfate threshold of 3000 ppm. The rapid field test procedures set forth in Research Report 0-4240-1 and established as TxDOT Test Method TEX-146-E are recommended for this verification.

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

Mellowing should be accomplished by mixing a single application of the optimum amount of lime to the soil at 2% above optimum moisture and allowed to stand uncompacted for a period of time appropriate to the concentration and grain size of the sulfate (2 to 10 days). The water content should be maintained at 2% above optimum throughout the mellowing process.

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

There is no substitute for good laboratory testing. The researchers recommend using a 3-D swell procedure similar to the one outlined at the end of this report to identify different stabilization options. Every soil is unique and may respond differently to lime treatment at varying sulfate levels. Using a moisture content greater than 2% above optimum with extended mellowing times may be effective in stabilizing soils with higher sulfate contents.

CHAPTER 3

NONSTANDARD STABILIZERS

This portion of the research was performed to identify soil treatments that could reduce 3-D swell, increase workability in fat clays (reduce PI), and provide a working platform for construction (strength improvement over natural soil) in soils with sulfate concentrations in excess of 7000 ppm. The limit of 7000 ppm was established based on experience of contractors, experience of TxDOT personnel, and research performed earlier in this project. A sulfate concentration of 10,000 ppm was used in this portion of the research because it is well above the 7000 ppm limit identified for calcium-based stabilizers.

NONSTANDARD STABILIZER CLASSES

Several different classes of nonstandard stabilizers were scrutinized for inclusion in this research. Twelve stabilizers were selected based on recommendations of the PMC and positive results obtained in previous research performed by researchers at the United States Army Corps of Engineers, in Europe, and the University of Texas.

Many of the nonstandard stabilizer products on the market were originally developed for dust control while others are industrial waste byproducts. A few examples of nonstandard stabilizers are acids, enzymes, polymers, resins, and electrolytes. In the paragraphs that follow, some of the stabilization mechanisms for these general stabilizer classes will be discussed.

Acids

Acid stabilizers have shown some promise in acidic soil types. The acid stabilizers contain (H^+) ions in solution. The (H^+) ions are exchanged onto the clays releasing (Al^{3+}), which may form hydroxyl aluminum ions ($Al(OH)_2^+$) that can be held in the clay interlayers.²⁰ This process reduces the cation exchange capacity (CEC), which will reduce the shrink-swell characteristics of the clay.

If sulfates are in the soil, then the sulfate ions may react with the hydroxyl aluminum ions to form other precipitates. [Adams and Hajek \(1978\)](#) illustrated that a low

sulfate-to-aluminum ratio promotes gibbsite ($\text{Al}(\text{OH})_3$) formation and a high sulfate-to-aluminum ratio promotes basaluminite ($\text{Al}_4(\text{OH})_{10}\text{SO}_4 \cdot 5\text{H}_2\text{O}$) formation.²⁸ The basaluminite has no charge and will not occupy the interlayer of clay minerals.

Electrolytes (Salts)

The hydration of exchangeable cations like sodium, calcium, and magnesium is believed to cause the swelling in smectites. The amount of water attracted to the cations is directly proportional to the ionic-charge-to-ionic-radius ratio. Therefore, larger cations with a lower charge attract less water. So, addition of potassium and ammonium salts to the soil will cause cation exchange and reduce the shrink-swell when less hydrated ions like (K^+) and (NH_4^+) replace more hydrated ions in the interlayer sites.²⁰ Because the salts travel in the soil solution by diffusion, they are best applied under saturated conditions.²⁰

Enzymes

Enzymes are protein molecules which are made up of amino acids.²⁹ They can speed up a biochemical reaction by orders of magnitude, but the most amazing thing is that they react with specific bonds in compounds. Some enzymes require a higher fraction of silt and clay with associated humus to provide an organic source to react with the enzymes.¹⁹ Bioenzymes create a culture for bacteria when added to the soil. They can react with carbon dioxide, nitrogen, and oxygen in the atmosphere to form additional organics. The large organic molecules react with clay to prevent uptake of water by the clay.²⁰ It is important to compact soils treated with these stabilizers prior to curing. The greater the compacted density, the more effective the stabilization will be.¹⁹

Polymers

Polymers are defined as linked, simple molecules that may be natural or synthetic. Natural polymers include proteins, cellulose, and rubber. Most synthetic polymers are organic compounds such as nylon, Teflon, and Plexiglas. One may use a chain as an analogy. Each link in the chain is a simple molecule that is attached to other links (simple molecules) by strong covalent bonds.

Organic polymers have been used by soil scientists to stabilize soil aggregates. They tend to be medium to heavyweight organic cations or anions that can replace other ions on clay surfaces. They may bridge soil particles and prevent water ingress, thus reducing shrink-swell.

Resins

Resins have been used to stabilize soils in Alaska with poor particle size distribution making it difficult to stabilize with conventional techniques.³⁰ They are adaptable to various climatic conditions and do not react with the soil but coat the soil particles forming a barrier to water. The cost is typically 1.5 to 4 times conventional stabilization.³⁰

Sulfonated Oils

Sulfonated oils are oils that have been chemically treated with sulfuric acid to make them soluble in water. The oils are ionized by the sulfuric acid generating a net negative charge which makes them attract to cations. These stabilizers are reported to pull cations and water from the clay structure and create amorphous silica (SiO_2) and gibbsite ($\text{Al}(\text{OH})_3$) from the tetrahedral and octahedral sheets of the clay minerals in the process.¹⁹

NONSTANDARD STABILIZERS TESTED IN THIS PROJECT

The researchers tested nine different non-calcium-based stabilizers and three modifications of calcium-based stabilizers ([Appendix B](#)) in addition to lime and cement. The primary concern for TxDOT is reducing 3-D swell to levels less than 7 percent, which is equivalent to a 1 inch potential vertical rise (PVR) assuming a 7-ft zone of influence (TxDOT). Therefore, the focus of testing in this project was measuring 3-D swell for the nonstandard stabilizers.

Methods

Evaluating the various nonstandard stabilizers included in this project consisted of testing each product with a naturally occurring low-sulfate soil and adding sulfates at the

desired levels for testing. Each product was initially tested for 3-D swell. Those products judged by the PMC showing an improvement in swell versus the untreated soil were advanced to the next level, which assessed the unconfined compressive strength.

Soil Properties and Processing

The same soil used for the lime stabilization testing, results reported in Research Report 0-4240-2, was used for evaluating the nonstandard stabilizers. The soil is from the Vertisol order in College Station, Texas. This soil contained sulfates below detection limits (i.e., less than 100 ppm), which allowed the researchers to add sulfates at various levels as required for testing.

The soil used for the proprietary nonstandard stabilizers was processed over a #4 sieve by hand in order to keep it as close to field moisture content as possible, unless otherwise instructed by the vendor. The field moisture content of the soil used for 3-D swell varied between 11 to 17 %.

The soil used for testing with GGBFS and lime, Class F Fly Ash, and Class C Fly Ash was dried in a 140°F (60°C) oven to a constant weight and pulverized to pass through a #4 sieve.

A plasticity index of 24 was measured using American Society for Testing and Materials (ASTM) D 4318 and the optimum moisture content determined by standard proctor (Tex 114-E) for the untreated soil ranged between 21 and 24% due to the large volume of soil used during this study. The optimum moisture content and density (Tex 114-E or ASTM D 698) were obtained for each non-calcium-based stabilizer treatment and used to mold 3-D swell samples with the Superpave Gyratory Compactor to accomplish molding the soil in one lift. Samples were molded in one lift because molding samples in multiple lifts with a drop hammer generates permeability barriers. The permeability barriers do not allow the water to rise up through the sample beyond the bottom lift which does not generate uniform 3-D swell. Optimum moisture content and density determined for the nonstandard stabilizers and the actual molded moisture and density are listed in [Table 1](#). 

Table 1. Optimum Moisture Content and Density versus Actual Moisture Content and Density Used for Molding Gyratory Samples.

Product & Application rate	Tex-114-E determined moisture content (%)	Molded moisture content (%)	Tex-114-E determined density (pcf)	Molded density (pcf)
M10+50 Low	23.6	24	89	88.5
M10+50 High	25	24	86.5	88.5
PennzSuppress D Low	24	24	88.5	88.5
PennzSuppress D High	24.4	24	88	88.5
Dustac	24	24	90	90
EMC2/EMS	25.6	25	88.5	88.5
	25	22.5	87.5	88.5
Roadbond EN1	24.4	24	90.5	90
EcSS3000	23.8	24	91	91
Top Seal	23.2	24	91	91
Soil Sement	25	24	90.5	90
ClayStar 7	24.4	24	91	91

For modification of the calcium based stabilizers (GGBFS and fly ash), the moisture/density relationship obtained for lime was used to compact samples in the Superpave Gyratory Compactor. The optimum lime content of 6 percent was determined by ASTM D 6276. The pH was determined using a saturated soil paste similar to that described by the United States Department of Agriculture (USDA)³¹ (Method Numbers 4F2 and 4C1a1a2).

Vendor Procedures

Detailed procedures for each product were requested from the vendors. The researchers followed the application rate, mixing, and curing procedures received from the vendors as closely as possible. [Appendix B](#) discusses vendor procedures for each product and deviations from the procedures. In addition to the vendor’s recommended application rate, selected products were tested at higher application rates. Products were

tested as the products and procedures became available from the vendors and follow no specific sequence.

Samples were molded at 0 ppm and 20,000 ppm $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ (reagent grade) for all of the products tested. The GGBFS and Class F Fly Ash were tested at 0, 10,000, and 20,000 ppm. Initially, the GGBFS, Class F Fly Ash, and lime combinations were tested with Na_2SO_4 due to the high concentrations tested and the method of adding the sulfate to the soil. Testing began by adding Na_2SO_4 to part of the molding water before being added to the soil. Since Na_2SO_4 has a higher solubility than $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, it is best suited to this method. However, the researchers decided to change to gypsum since this is the most common natural form of sulfate in Texas. The sulfate was added to the soil in solid form and left to cure overnight before any addition of the stabilizers. The GGBFS and Class F Fly Ash were retested at selected points using $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$.

Although the list of stabilizers tested did not include cement, since it is not considered a nonstandard stabilizer, 3-D swell tests were run using 4%, 6% and 8% Type I/II cement.

3-D Swell Samples

Each sample was weighed and mixed separately using a method outlined by each stabilizer manufacturer. The samples were then molded in one lift with a Superpave Gyrotory Compactor at the density and moisture content determined by standard proctor. The sample size was restricted to 4 inches diameter by 4.5 inches tall (10.16 cm X 11.43 cm) due to the constraint of molding in one lift. All samples were molded in duplicate. The samples were then cured according to each manufacturer's specifications and placed in a 3-D swell test modeled after Petry (Tom Petry, unpublished data). Each sample was wrapped in a paper membrane saturated with distilled water, porous stones were placed on the bottom and top of each sample, and a latex membrane was placed over the sample. Duplicate samples were placed in a distilled water bath at 100% humidity and $77^\circ \pm 3.6^\circ\text{F}$ ($25^\circ \pm 2^\circ\text{C}$). The distilled water level was maintained just below the top of the porous stone located on the bottom of the sample to allow water to be drawn up into the sample by capillary action. The researchers constructed duplicate samples of unstabilized soil at the moisture content and density of each of the nonstandard stabilizers for comparison.

Three-dimensional swell was measured by determining the height to the nearest 0.01 inch in three places, 120° apart. The circumference was measured with a clear plastic tape to the nearest 0.002 in. (0.5 mm) near the top, middle, and base of each sample. The three height and circumference measurements were averaged and the volume calculated.

Unconfined Compressive Strength

Samples for unconfined compressive strength testing were molded according to Tex-114-E, which has an equivalent compactive effort to ASTM D 698 but molded in four lifts in a 4 x 6 in. mold. The samples were tested after a combination of the following curing regimes:

- 7-day cure (referred to as 7 day);
- 7-day cure, followed by 4-hour soak (referred to as 4hr soak);
- 7-day cure, followed by a 3-day dry back and then a 10-day capillary rise (known as the Tube Suction Test); and
- 28-day moist cure (in sealed bag).

The 7-day initial curing was done as a moist cure in a sealed bag unless otherwise instructed by the vendor. The 4-hour soak has been proposed as an alternative to the tube suction test as a quicker way of assessing moisture susceptibility.

Fourier Transform Infrared (FTIR) Analysis

The Texas Department of Transportation analyzed samples of the nonstandard stabilizers using two different techniques. One technique involved forming a thin film of the stabilizer on a slide and putting it in the instrument to get a pattern from 4000 to 400 waves per centimeter (cm^{-1}). The other technique involved drying the liquids out and mixing the solid residue with potassium bromide (KBr) to form a powder, which is then compressed into a solid pellet. The pellet is placed in a sample holder where it is scanned by Infrared (IR) radiation to yield a pattern of the beam transmitted through the sample from 4000 to 400 waves per centimeter (cm^{-1}).

Suction Measurements

One of the criticisms leveled against using the 3-D swell and strength tests for evaluation of non-standard stabilizers has been that it fails to take into account the “effectiveness of the layer within the pavement system” (Randolph 2004, personal communication). According to the manufacturer of EMC², the emphasis should be on reducing permeability and moisture susceptibility as was the focus in research study TX-98/3929-1. In this research project, the researchers conducted, among other tests, the triaxial strength and stiffness, 3-D swell, permeability and total and matric suction.³² In the present research project, the criteria used for selection of nonstandards into the field section phase of the project concentrated on 3-D swell and unconfined compressive strength. Based on comments submitted (as summarized above) by the manufacturer of the EMC² + EMS dual component system (hereafter referred to as EMC²), the researchers decided to look at the suction of the College Station soil used in testing the nonstandard stabilizers in the untreated state, compared to the suction of the EMC² treated material.

The College Station soil was used for evaluating the suction of the treated and untreated soil. The soil was kept as close as possible to field moisture content during processing over the #4 sieve. Samples were molded for measuring suction in exactly the same way as was used for molding samples for 3-D swell, with the Superpave Gyratory Compactor in one lift with a sample size of 4 x 4.5 in. Two replicates were molded for each condition tested. The vendor procedures were followed as set out in [Appendix B](#). After molding, the samples were cut into three layers as in [Figure 3](#), repositioned, and placed on a porous stone. The samples were air dried for 3 days.

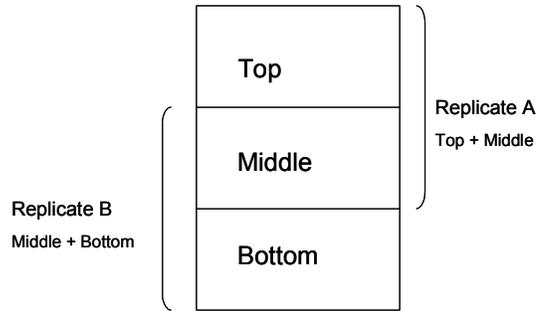


Figure 3. Sample Division for Function Measurements.

After the 3-day air dry, measurements of replicate A were carried out on the top and middle layers and for replicate B on the middle and bottom layers. Total suction was measured using the filter paper method (ASTM D 5298) and a Decagon Devices SC10A/NT-3 Sample Changer Thermocouple Psychrometer/Nanovoltmeter (McInnes 2005, personal communication). Each measured layer of the sample was therefore divided into three pieces, one for the filter paper method, one for measurement with the psychrometer, and one for measuring the water content of the sample itself. Samples analyzed with the psychrometer were sent to the Department of Soil and Crop Sciences at Texas A&M University for testing. The psychrometer measures water potential on subsamples less than 1cm³. Subsamples were taken in multiple locations from the third of the original sample sent for analysis with the psychrometer. Matric suction was not attempted at this stage because the researchers wanted to evaluate the total suction measurements first.

RESULTS

3-D Swell Tests

Three-dimensional volumetric swell is plotted against time in [Figure 4](#) for the EMC² stabilizer. The researchers initially molded samples for the enzyme at two moisture contents, optimum which was 25% and 2.5% below optimum, and the unstabilized soil was molded at 21% optimum moisture for the unstabilized soil. The unstabilized soil was later molded at the optimum moisture content of the enzyme. From [Figure 4](#), one can see that there is very little difference (~1%) in 3-D swell results for

20,000 ppm sulfates between the enzyme (20K, ENZ, 25% MC) and the unstabilized soil (20K, blank, 25% MC) molded at the same moisture content as the enzyme.

With the exception of ClayStar 7, the 3-D swell results for the other noncalcium-based stabilizers are very similar to the results obtained for the EMC² + EMS Dual Component System. Results for the other stabilizers can be found on the CD included in the back cover of this publication.

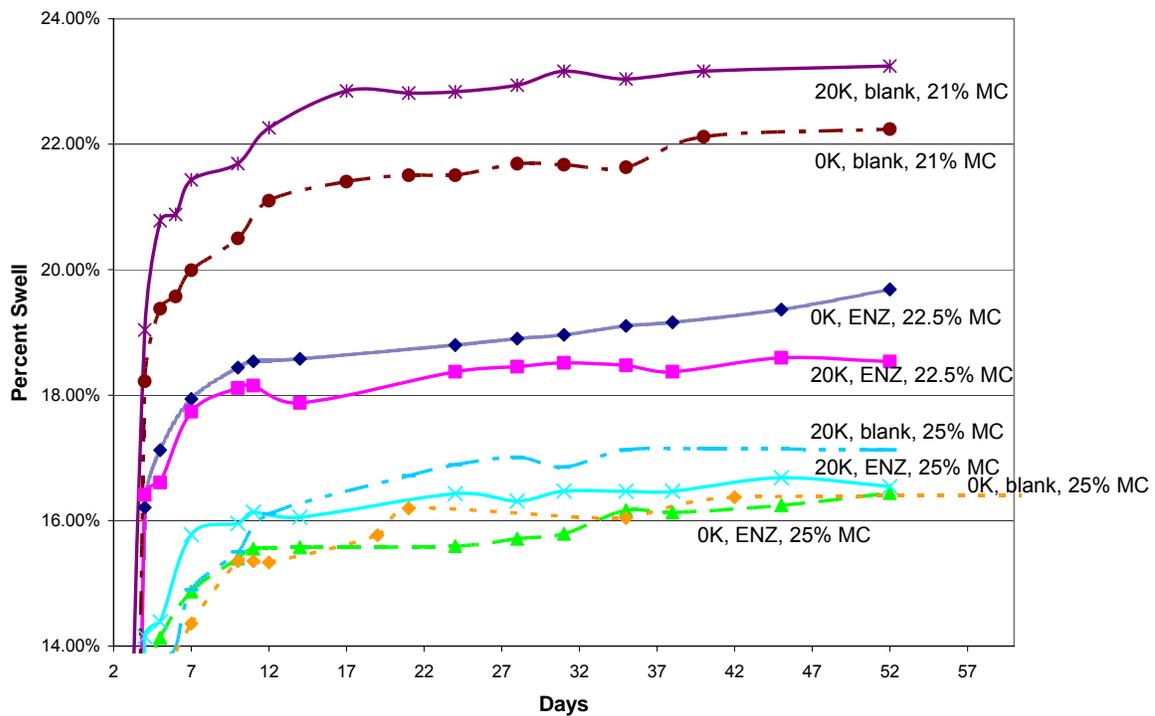


Figure 4. EMC² + EMS Dual Component System Stabilizer Swell Results at Different Moisture Contents.

The ClayStar 7 liquid stabilizer yielded excellent results in the 3-D swell test with the College Station soil (Figure 5). All of these samples were molded at a 24% moisture content and were air cured for 3 days except for one that was air cured to the vendor’s specifications of 7 days, which is labeled (20K ppm, CL 7 improved 3.5 ml/lb, 7-day cure). The extended air cure did not improve the swell results over the standard 3-day air cure. The lowest concentration of the stabilizer (3.5 ml per pound of dry soil) resulted in

the greatest swell reduction (12%) over the unstabilized soil. Increasing the stabilizer concentration did not improve swell results; it actually resulted in an increase in swell. The samples molded at 5 and 10 ml per pound of soil only reduced swell by about 8%.

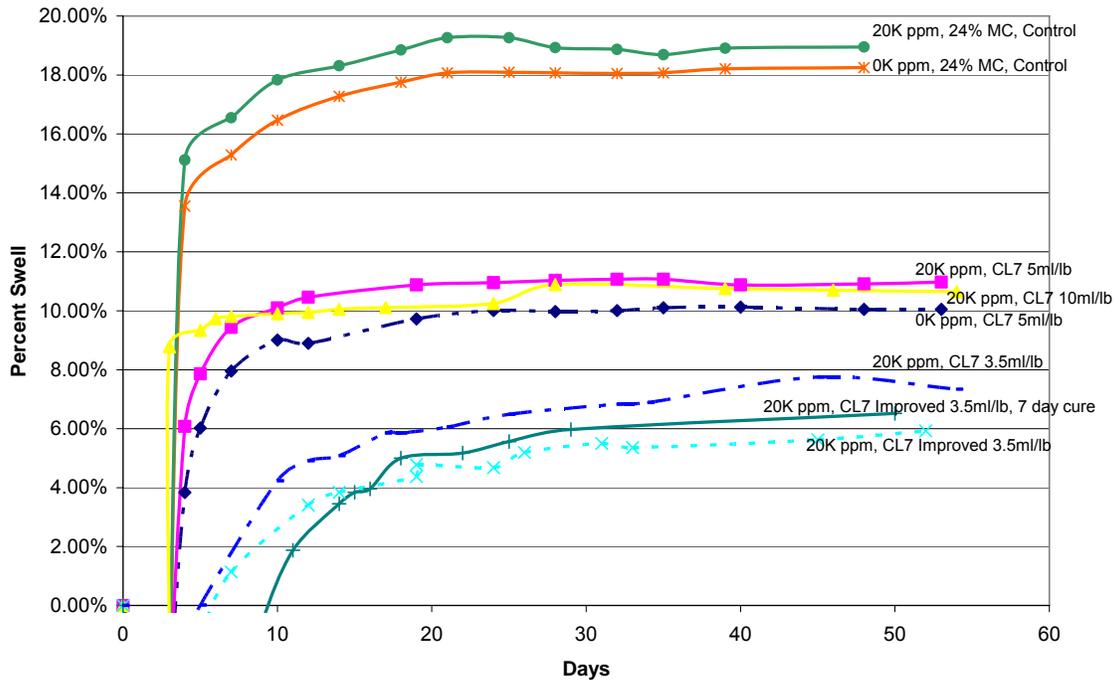


Figure 5. Swell Results for ClayStar 7 with the College Station Soil.

Figure 6 illustrates the effects of GGBFS on stabilization of the College Station soil with zero and 20,000 ppm sulfates. Initially, sodium sulfate (Na_2SO_4) was used as the sulfate mixed with the soil, but later researchers mixed reagent grade gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) with the College Station soil. As shown in Figure 6, there is virtually no difference in the 3-D swell results of the 5% GGBFS and 1% hydrated lime treated samples bearing sodium sulfate (20K ppm, Na_2SO_4 , 1L5S) and gypsum (20K ppm $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, 1L5S) at a concentration of 20,000 ppm sulfates. As the lime concentration increases from 1% to 3% to 6%, in the soil containing 20,000 ppm sulfates, the amount of 3-D swell also increases from 7% to 24% to 42%. It is important to only use enough lime to catalyze the reaction of the GGBFS.

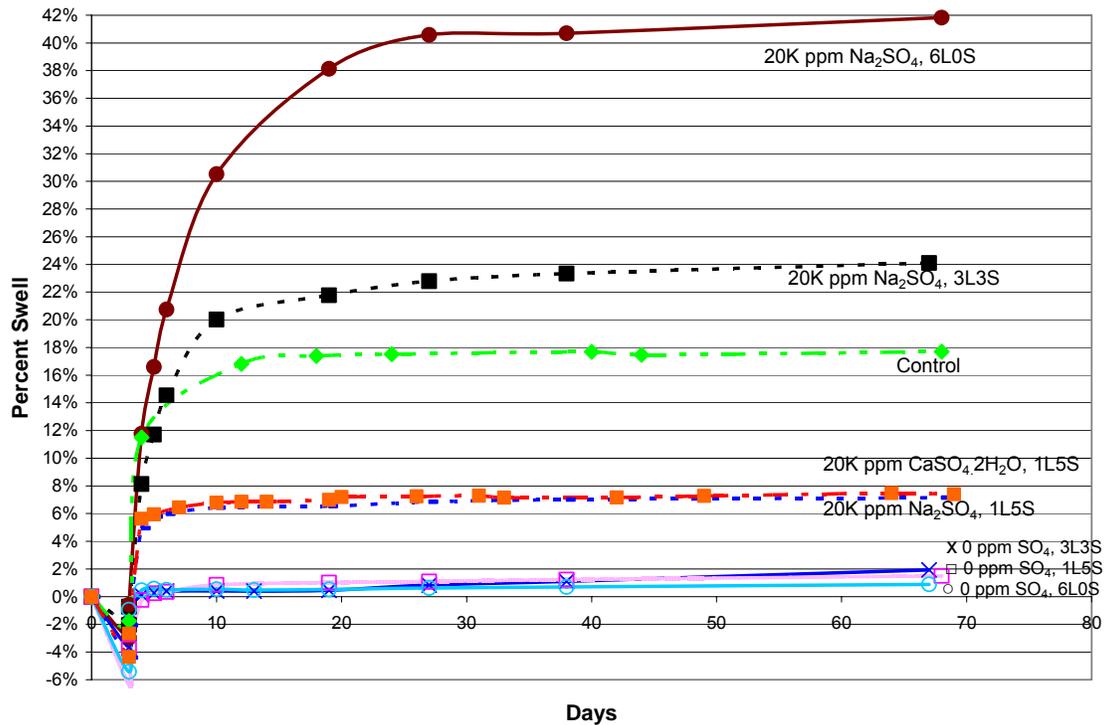


Figure 6. Volumetric Swell of College Station Soil Treated with GGBFS + Lime.

Table 2 shows how much each stabilizer reduced swell in the College Station soil with 20,000 ppm sulfates. The top row in the table lists percent reduction in 3-D swell, and the stabilizers that reduced swell by that percentage are listed in the column below that particular percentage. Products listed in the far left-hand column actually increased 3-D swell over the unstabilized soil. The swell reduction reported in this table is in relation to the unstabilized soil at the moisture content and density of each particular stabilizer. For example, Soil Sement was molded at a moisture content of 24% and a density of 90 lb/ft³. Unstabilized soil samples were molded at the same moisture content and density as the Soil Sement for a direct comparison of how much the polymer actually changed the swell characteristics. In the case of Soil Sement (low), the swell was exactly the same as the unstabilized soil with zero decrease in swell. The Soil Sement (high) was where six times the recommended stabilizer concentration was added to the soil; it actually reduced swell by 8%. All stabilizers labeled with (low) indicate the

manufacturers' recommended stabilizer content for the soil. Samples tested with higher concentrations of some products are denoted by (high). Stabilizers, without concentrations in parentheses like Top Seal, were only tested at the manufacturers' recommended concentrations. The GGBFS and Type F fly ash and lime mixtures were tested at different stabilizer contents, but only the best performing concentrations of 5 weight percent fly ash or GGBFS to 1 weight percent lime are reported here.

Table 2. Reduction in Swell for College Station Soil with 20,000 ppm Sulfates.

Increase	0-2%	3-4%	5-6%	7-8%	9-10%	11-12%
Top Seal	Soil Sement (low)	Roadbond EN 1	5% C FA	Soil Sement (high)X6	GGBFS + lime (5:1)	ClayStar 7 (low)
EcSS3000	EMC ² + EMS	Dustac	F FA + lime (5:1)*	ClayStar 7 (high)	F FA + lime (4.2:12.5)**	
	Enviroseal M10+50/LBS (low)	PennzSuppress D (high)		8% Type I/II Cement		
	Enviroseal M10+50/LBS (high)					
	PennzSuppress D (low)					

*Na₂SO₄

**CaSO₄·2H₂O

Summary

Based upon 3-D swell results using the College Station soil, two stabilizers were selected for further testing for unconfined compressive strength. The two stabilizers were ClayStar 7 and GGBFS + lime. Soil Sement was not selected for further testing because the amount of stabilizer required for stabilization was so high that it was not economical. The Type F fly ash was initially not included in further testing as the swell results conducted with Na₂SO₄ did not look favorable. However, subsequent testing of Type F fly ash done with reagent grade gypsum did seem to produce a favorable reduction in swell.

Soil Properties

TxDOT was interested in how the stabilizers affected soil properties like PI and pH. Table 3 shows that the addition of GGBFS + Lime (5:1) lowers the PI from 24 to 9, therefore modifying the soil sufficiently for construction. The ClayStar 7 had very little if any effect on reducing the PI of the soil. The Claystar 7 did reduce the pH of this soil, but the soil pH was near neutral indicating very little if any influence from carbonate minerals. The GGBFS and lime mixture raised the pH to a value close to that of a saturated lime solution.

Table 3. PI and pH of GGBFS + Lime and ClayStar 7 Treated Soil.

Sample Name	PI	pH
Soil only	24	6.96
GGBFS + Lime (5:1)	9	12.17
ClayStar 7	23	6.03

Unconfined Compressive Strength Results

The GGBFS and lime, mixed in a ratio of 5% GGBFS to 1% lime by dry weight, were cured for different times and subjected to an unconfined compressive strength test. The curing methods include: a 7-day moist cure, being submerged in water for 4 hours, being subjected to a 10-day capillary rise or tube suction test, and a 28-day moist cure test. The results presented in Figure 7 show that the GGBFS treated samples are much stronger than the unstabilized control samples after 7 days moist cure and 10 days capillary rise testing. The GGBFS even increased the strength over the lime stabilized samples with no sulfates: the 28-day moist cured GGBFS showed a significant strength increase over the lime stabilized ones. Each bar on the graph represents an average strength of two samples.

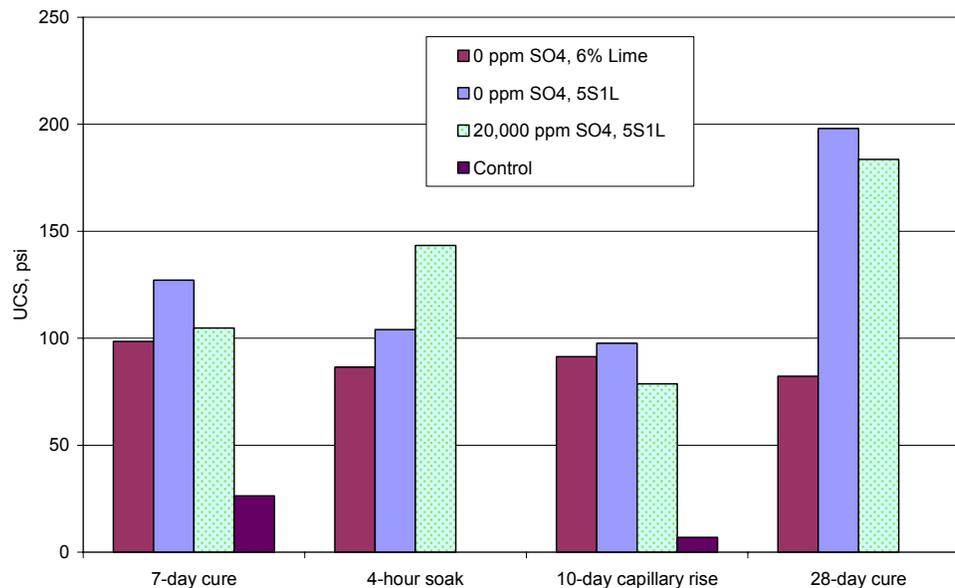


Figure 7. UCS in psi for the GGBFS + Lime Treated College Station Soil.

For the ClayStar 7, the vendor called for a 7-day air cure before starting any testing. For assessing the UCS of the ClayStar 7, samples were molded and cured according to the vendor’s recommendation (7-day air cure) in addition to the conventional cure used for lime and cement testing (7-day moist cure, followed by 3-day dry back for the 10-day capillary rise samples). Two replicates were molded with 20,000 ppm SO₄ for each of the curing methods presented below.

The ClayStar 7 showed significant retained strength over the unstabilized material (Figure 8) in the 4-hour soak (125 lb/in² for vendor cure versus a control that fell apart). For the 10-day capillary rise, the treated samples had some retained strength (41 lb/in² for conventional cure versus the control that fell apart). The control samples for the 4-hour soak and 10-day capillary rise could not be tested because they fell apart.

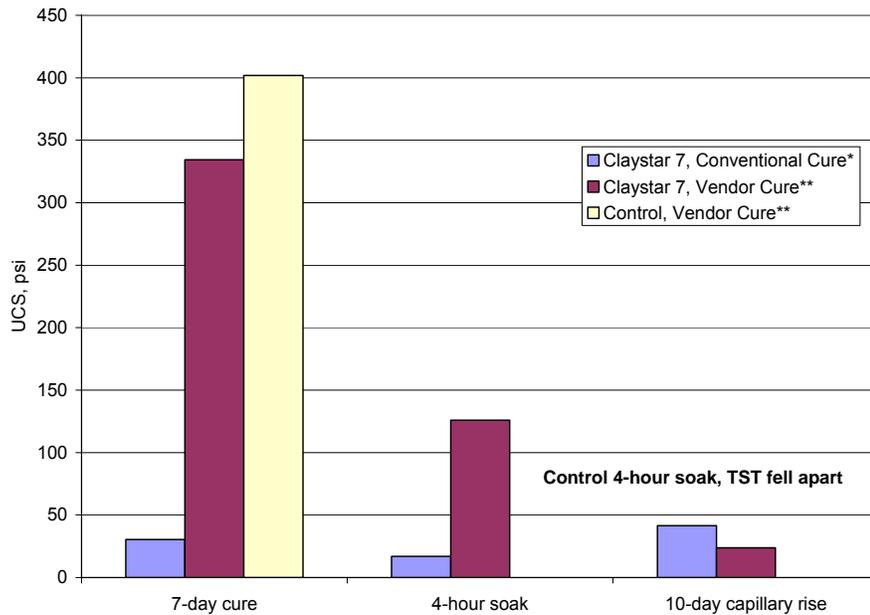
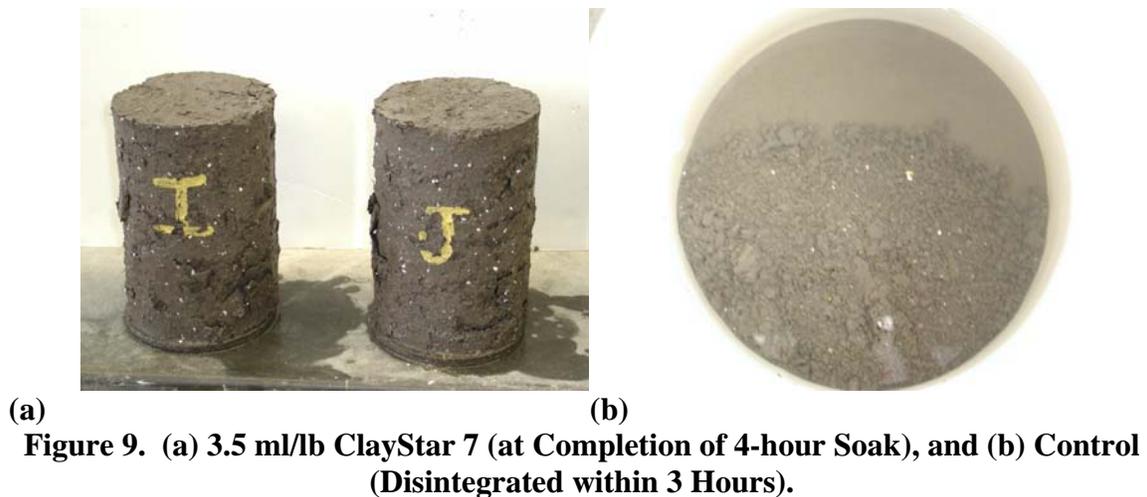


Figure 8. UCS in psi for the ClayStar 7 Product with the College Station Soil.

Figure 9 shows samples after being subjected to a 4-hour soak. The ClayStar 7 treated samples (a), after the 4-hour soak, are still intact while the untreated samples (b) fell apart after 3 hours. Researchers note that the PMC felt that the 4-hour soak was too harsh, but results are nevertheless reported due to the striking difference between the ClayStar 7 treated and untreated samples.



Fourier Transform Infrared Analysis Results

A comparison of the FTIR analyses of one of the stabilizer products is illustrated in [Figure 10](#). This technique of sample preparation involves mixing a solid sample with an alkali metal halide (KBr) and compressing it into a pellet. There are a number of errors that can be associated with this technique.³³ Observations of the two patterns shown in [Figure 10](#) make it very difficult to conclude that these patterns are from the same material. Using this technique also makes it impossible to evaluate the concentration of active ingredients in the liquid stabilizers.

Suction Measurements

Results obtained for both the psychrometer and the filter paper method were highly variable. In both cases, variations within the samples were greater than variations between the samples. Given that the subsample size analyzed in the psychrometer was less than 1 cm³ and the original molded sample size is 4 x 4.5 in., a large number of subsamples would have to be analyzed to obtain accurate results. This was not done. For the filter paper method, less variability within samples would have been obtained if the whole sample (4 x 4.5 in.) were used to obtain a total suction value. There were no trends as far as suction in the top and middle layer compared to suction in the middle and bottom layer were concerned (i.e., the variability could be from the fact that the top layer may have dried out more than the bottom layer).

The mean suction of all the samples measured with the psychrometer was pF=4.61, and the mean suction measured with the filter paper method was pF=4.59.

DISCUSSION

The 3-D swell experiments provide insight into the effectiveness of the various nonstandard stabilizers. As documented by previous investigators, many of these stabilizers show some improvement in soil properties if applied in high enough concentrations with results being inconsistent in different soil types.^{21, 23, 24, 25}

All soils are different, and the stabilizers the researchers evaluated all behave differently in these soils. It is imperative for nonstandard stabilizer suppliers to provide a

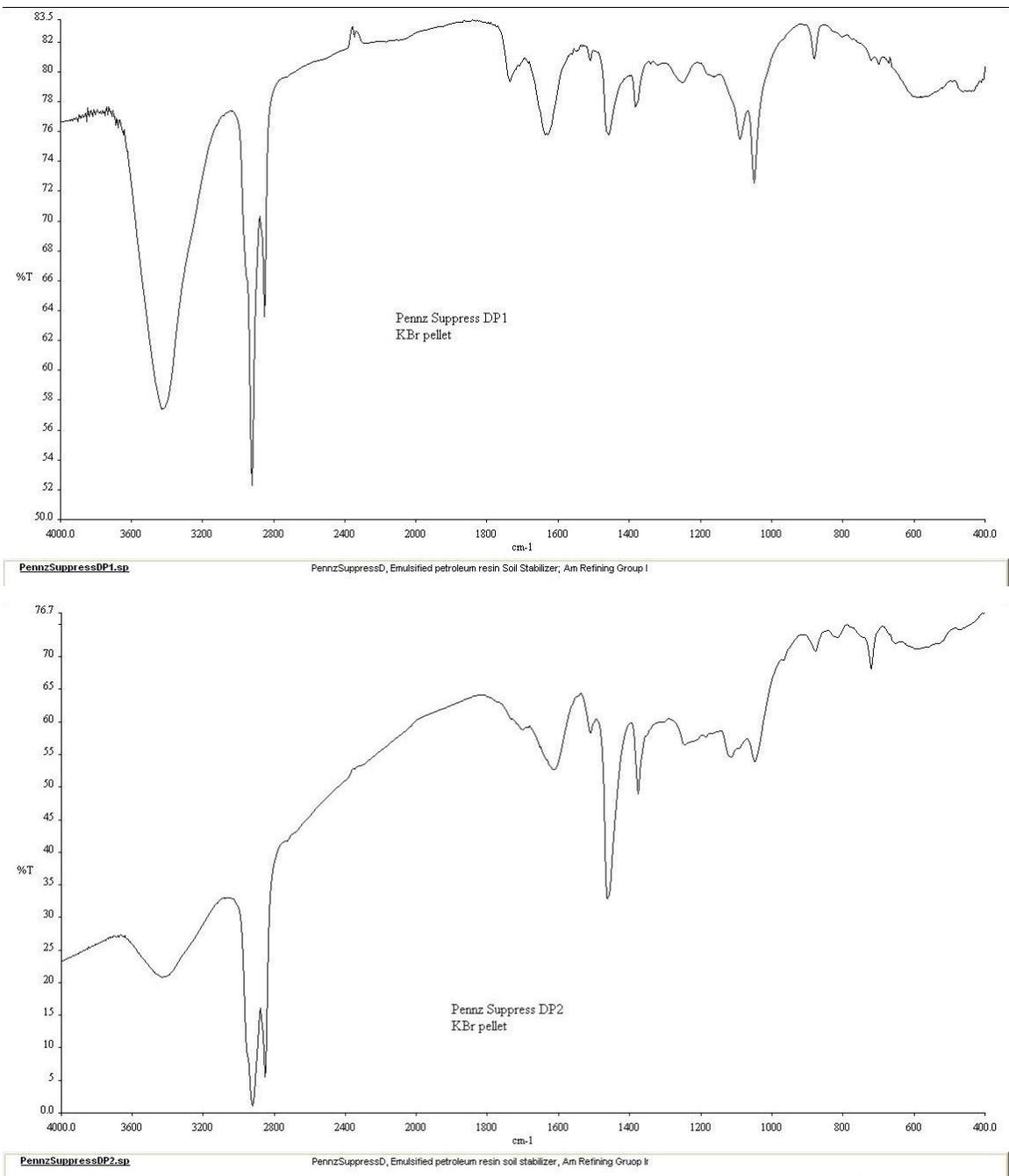


Figure 10. Replicate FTIR Patterns for the PennzSuppress D Stabilizer Using KBr.

mechanism by which their product stabilizes the soil. Some soils may respond well to that mechanism and other soils may not. Following is a short explanation of some of the mechanisms proposed for polymers (Soil Sement and Topseal), acids (Roadbond EN 1 and ClayStar 7), and ground-granulated blast furnace slag.

Polymers

Based upon the 3-D swell results, it is obvious that the various nonstandard stabilizers tested in this research are vastly different. For example, the researchers tested three stabilizers classified as polymers, which are supposed to stabilize the soil by bridging individual soil particles, preventing expansive clay minerals from swelling by not letting water penetrate the clay structure. If this was the stabilization mechanism, then one would expect this stabilizer to work better in sandy low-PI soils. Since fine clay has a surface area 10,000 times that of sand-size particles, one would expect higher concentrations of the polymer would be needed to coat more soil particles and decrease the swell of a higher-PI soil. For the College Station soil, Topseal and Enviroseal did not work. Soil Sement did show some improvement with a higher stabilizer content.

Acid Stabilizers

The acid stabilizers all performed very differently in treating the College Station soil. ECSS3000 actually increased swell while Roadbond EN1 and ClayStar 7 decreased swell to different degrees. A researcher at Texas A&M University examined one acid stabilizer for a stabilization mechanism.²⁰ For a low-pH soil like the College Station soil (no carbonates), the acid stabilizer reduces swell by formation of hydroxyl aluminum groups which decrease the number of hydrated cations that can exist in the interlayer of clays.²⁰ With higher concentrations of the acid stabilizer, swelling was observed to increase.²⁰ This trend was explained by forming a new mineral, basaluminite, by removing the hydroxyl aluminum groups from the interlayer of the clays which will allow hydrated cations to enter the clays resulting in an increase in swell.²⁰ The ClayStar 7 product showed the exact same trend (Figure 5) with higher concentrations of the stabilizer actually increasing in swell.

Ground-Granulated Blast Furnace Slag

There have been several studies in Europe evaluating the mechanisms that prevent sulfate heave when GGBFS is added as a stabilizer. One mechanism the Europeans suggest is that the calcium ion concentration is reduced by replacing lime with GGBFS resulting in less calcium available for forming ettringite.¹¹ Based on thermodynamic stability diagrams, the Al/Si ratio was reported to be significant in reducing the stability field of ettringite.⁶ Little et al. (in press) stated that GGBFS reduces the stability field of ettringite by changing the Al/Si ratio. Other researchers have suggested that GGBFS alters the pH of the stabilized soil so ettringite will not form. The researchers monitored pH as part of this investigation, but there was no significant difference in pH of the GGBFS and lime-stabilized samples.

There is still much work to be done in this area, but one thing researchers can say is that the GGBFS and lime mixture has reduced swell and increased strength on all high-sulfate soils tested in Texas.

Permanency of Stabilization

There is some concern regarding the permanency of many of the nonstandard stabilizer options. Many of these stabilizers were developed as dust control palliatives and later adapted to soil stabilization. One product evaluated in this research was Dustac, which is a lignosulfonate or tree resin. Initially, the water bath containing the samples was clean (Figure 11a), and the samples performed very well with a reduction in 3-D swell (Figure 12). But over time, the water bath started looking like the Dustac product (Figure 11b) was leaching out of the sample and into the water bath while the swell continued to increase (Figure 12).



(a) After 3 days



(b) After 35 days

**Figure 11. Dustac Stabilized (3.37%) Samples Bearing 20,000 ppm Sulfates
(a) after 3 Days and (b) after 35 Days.**

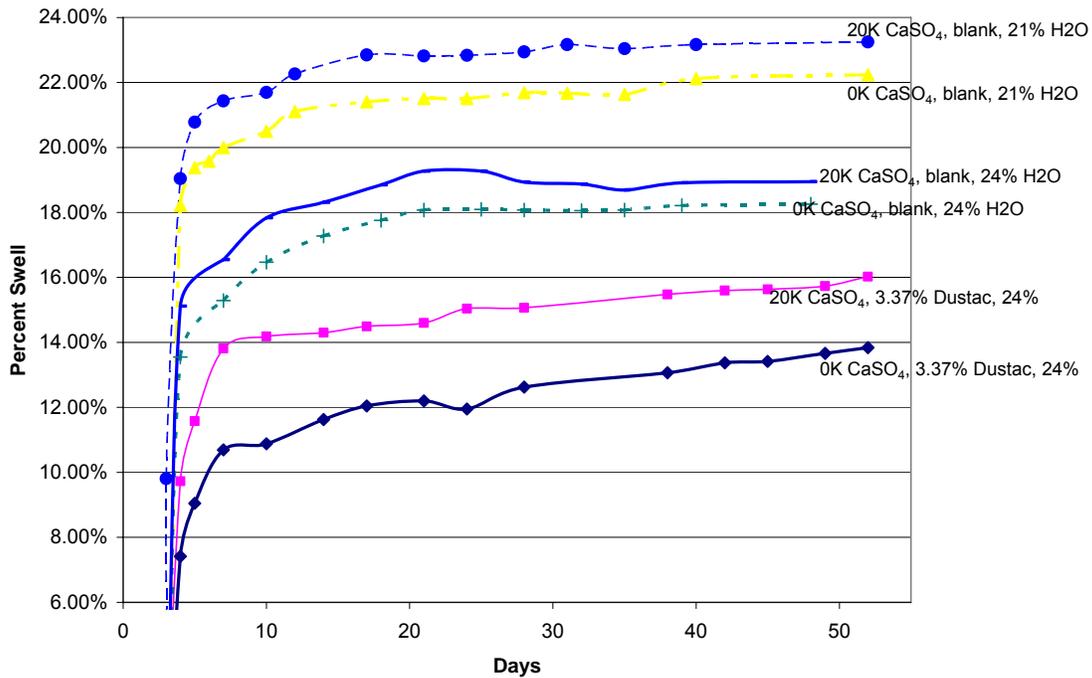


Figure 12. Dustac Treatment Continues to Swell Due to Stabilizer Leaching Out of Soil.

There are some problems with the FTIR analyses of the nonstandard stabilizers (Figure 10). If TxDOT wants to use FTIR as a fingerprinting tool for the nonstandard stabilizers to ensure quality control, then the KBr method probably will not be the best option. There are numerous sources of error with the KBr pellet method including spectral artifacts due to poor sampling, some compounds may react with the KBr, and the act of applying pressure can distort the concentration of different polymorphs.³³ Other problems with this technique include the inability to determine the concentration of active ingredients since the stabilizer is first dried out to evaporate the liquid, and it is critical to consistently use a very small amount of the dried stabilizer in the KBr mixture.³⁴ If the sample is too concentrated in the KBr mixture, then the resultant spectrum will not be very detailed because the sample would absorb too much of the infrared radiation.³⁴

CHAPTER 4

CONCLUSIONS AND RECOMMENDATIONS

CONCLUSIONS

This work was performed to the exact specifications of the nonstandard stabilizer manufacturers. The results obtained are not representative of all soil types, but a general testing protocol ([Appendix A](#)) has been developed to evaluate new stabilizer/soil combinations in the future.

Sulfate problems have been reported from all over Texas. Many other states and countries have reported sulfate heave problems as well. Results of this investigation looking at alternatives to lime and cement in high-sulfate soils (greater than 7000 ppm sulfates) reveal the following:

- The majority of the nonstandard stabilizers tested at the manufacturers' recommended concentrations did not reduce swell any more than just adding water to the soil ([Figure 4](#)).
- One cannot lump all products together ([Table 1](#)). Not all polymers are created equal (i.e., if polymer A yields good results it does not mean polymer B will also yield good results).
- Some of the nonstandard stabilizers initially reduced the swell, but over time they leached out and started swelling again (stabilizer leached out [Figures 11](#) and [12](#)). They could provide a good short-term working platform, but long-term improvement is questionable. Permanency of treatment is a concern.
- It is difficult to keep track of many of these nonstandard stabilizers because the product names frequently change, products are reformulated, and operations get moved overseas.
- Infrared analysis of the dried nonstandard stabilizer products is not a good means of fingerprinting at this point. More work is needed to identify possible sources of error in sample preparation ([Figure 10](#)).

Modification of the calcium-based stabilizers provided much better results in 3-D swell reduction, strength gain, and permanency of stabilization. Listed below are some conclusions drawn from these stabilizer additives.

- The use of 5% GGBFS to 1% dry weight of lime resulted in the least amount of swell ([Table 1](#)).
- Increasing the ratio of lime to GGBFS results in increased swell ([Figure 6](#)).
- The GGBFS and lime produced acceptable strength results in addition to the swell reduction at costs similar to lime stabilization ([Figure 7](#)).
- Class F fly ash also yielded positive results with gypsum-rich soils ([Table 1](#)).
- At this time, the only stabilizer recommended for high-sulfate soils is GGBFS + lime. However, Fly ash may be a reasonable additive in soils deficient in sodium sulfate minerals.

RECOMMENDATIONS

Use the procedures outlined in [Appendix A](#) as a guide to evaluate new stabilizer products in the future (see attached CD). The vendor should include the following information if they want their product to be considered as a stabilizer to be used in Texas. They need to have the cost of their product, application rate, and application procedure (for specific soil types, or certain PI range, etc). They need to explain the stabilization mechanism and show data obtained from an independent lab that illustrates how the stabilizer modified the engineering properties (i.e., PI, 3-D swell, strength, pH, suction, etc.) in accord with their proposed stabilization mechanism.

The researchers recommend looking at coal combustion byproducts in more detail. Conduct some more experiments to identify reaction products. What makes these products effective? The grain size of the pozzolans and the Al to Si ratios may play an important role and are aspects that need to be investigated.

Infrared analysis of the nonstandard stabilizers as a fingerprint to ensure that TxDOT is getting the correct stabilizer is a technique that requires a little more work on sample preparation to obtain consistent results. Sample concentration is key to achieving

good results. The lower the concentration of the material being tested actually yields higher quality patterns with diffuse reflectance.

The researchers also recommend placing some of the stabilizer options in the field to determine how these materials should be handled. Can they be constructed using techniques and equipment like what are currently being used with calcium-based stabilizers?

The researchers recommend long-term monitoring of test sections like ride quality, Dynamic Cone Penetrometer (DCP), and falling weight to monitor the long-term effectiveness of the stabilizer options.

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APPENDIX A

TEST PROCEDURES FOR CHEMICAL SUBGRADE STABILIZERS

As part of Research Report 0-4240, several nontraditional chemical additives were evaluated as subgrade stabilizers in high-sulfate soils where lime and cement create excessive 3-D swell. Excessive 3-D swell in this project is defined as 7% volumetric swell, which results in a 1-inch PVR assuming a 7-foot zone of influence (TxDOT). The procedures outlined below are a result of that research and are partially adapted from the work of others.^{24,26}

These laboratory procedures are proposed to serve as an indicator of how the chemical additives will or will not perform in the field. If an additive cannot show any improvement in engineering properties under a controlled laboratory setting, then it is believed that it will behave similarly in the field. Most engineers are interested in reducing PI (increasing workability), reducing swell, and/or strength gain (for a working platform). These laboratory procedures are designed with those criteria in mind.

1. Sample a representative portion of the soil to be treated and maintain a gradation of 100% passing the $\frac{3}{4}$ in. sieve and 60% \pm 5% passing the #4 sieve. Enough soil should be obtained to construct moisture density curves for both treated and untreated soils. In addition, there should be enough soil to construct 10 (4 x 4.5 in.) samples of each treatment combination selected for testing: five samples of the treated material and five samples of the untreated material at the moisture content of the treated material. Depending upon the maximum density of the soil, the amount of soil required to construct the samples will be vastly different.
2. Seal the soil in containers to maintain the soil at field moisture contents.
3. Determine the engineering properties (PI, optimum moisture content, maximum density, etc.) of the untreated soil and establish the desired improvement (i.e., swell reduction, strength gain, etc.).
4. Select the treatments that are supposed to provide the desired improvement.

5. Use only distilled or de-ionized water to dilute the chemicals or increase the water content of the soil.
6. Have the vendor supply the recommended application rate, mixing procedures, and curing procedure. The procedure should be the same for laboratory molded samples and field application. The engineer may decide to modify the recommended procedure if they deem it impractical to perform in the field.
7. Use standard or modified proctor compactive effort to determine the optimum moisture content (OMC) and maximum density of the soil treated with the chemical stabilizer using TEX-114-E.
8. For soil to be treated, use distilled or de-ionized water to raise the soil moisture to a water content equal to the OMC of the treated soil minus the moisture content to be added with the chemical stabilizer. Allow the mixture to sit in a sealed container for approximately 16 hours.
9. For untreated control specimens, use distilled or de-ionized water to raise the soil moisture content to the OMC of the treated soil and allow it to sit in a sealed container for approximately 16 hours. It is imperative to construct samples treated with water only at the same level as the chemical treatment in order to determine the benefits of the chemical treatment.
10. For soil to be treated, mix in the vendor's recommended amount of chemical stabilizer using the recommended dilution ratio to bring the soil to the OMC of the treated soil. Mix the samples exactly how they are to be mixed in the field (i.e., dry, slurry, both, etc.).
11. Allow the treated mixture to sit in sealed plastic containers in accord with vendor recommendations, not to exceed 24 hours.
12. Compact specimens in a single lift, to a height of 4.5 in. and diameter of 4 in. with a Superpave Gyrotory Compactor at the moisture content and density determined in Step 7. At the optimum moisture content, there should not be a problem with water getting into the compactor. If water gets into compactor discontinue use of compactor until the moisture/density curve is worked out.

13. Allow the specimens to cure as outlined by the vendor unless the procedure cannot be replicated in the field. If no curing procedure is specified, then allow the specimens to cure for 7 days at room temperature in a sealed plastic bag or container.
14. Test three treated and three untreated specimens for unconfined compressive strength. Follow UCS testing with Atterberg limits (TxDOT Test Method Tex-106-E) and soil pH (TxDOT Test Method Tex-128-E).
15. For the remaining two treated and two untreated specimens, perform a 3-D swell test as follows:
 - A. Weigh each sample immediately after it has been molded, place on a 4 in. diameter x $\frac{1}{4}$ in. high porous stone with a label of the sample name, and record the weight and date on Worksheet 1.
 - B. Measure the circumference of each sample in three places (near the bottom, near the top, and in the middle) using Johnny tape (0.002 in. (0.5 mm) divisions), and record on Worksheet 1.
 - C. Measure the height of each sample at approximately 120° intervals, and record on Worksheet 1.
 - D. Let the samples air dry for 3 days.
 - E. Repeat Steps A, B, and C for each sample immediately after molding.
 - F. Wet a paper towel in distilled water and place around the circumference of the sample.
 - G. Place another 4 in. diameter x $\frac{1}{4}$ in. high porous stone on top of the sample.
 - H. Place a rubber membrane around the sample, and label the membrane with the sample name using a permanent marker.
 - I. Mark the membrane at approximately 120° intervals for height measurements around the circumference of the sample.
 - J. With the porous stones and membrane in place, measure the circumference and height as in Steps B and C.

- K. Place the duplicate samples on a rubber-coated metal rack or a strong plastic rack, and set it in a 20-gallon ice chest. It should be in an airtight ice chest to control humidity ($95 \pm 5\%$) and temperature ($77^\circ \pm 3.6^\circ\text{F}$ or $25^\circ \pm 2^\circ\text{C}$).
- L. Add enough distilled water to the ice chest so the water level is near the top of the porous stones placed on the bottom of the samples, but not high enough to be in direct contact with the soil in the samples.
- M. Monitor swell as in Steps B and C for at least the next 45 days.
- N. Plot the percent swell (on y-axis) over time (in days on x-axis) using a spreadsheet like Excel.
- O. Perform an unconfined compressive strength test on the samples following the swell test to compare with UCS measurements after curing. This test will give an indication of strength retention and permanence of stabilization.

CRITERIA FOR CHEMICAL STABILIZER QUALIFICATION

Using the above test methods, the chemical stabilizers must meet the following criteria to be considered for treatment of subgrade soils:

- The treatment must reduce 3-D swell to 7% or less. (If the 3-D swell for the untreated soil is less than 5%, then the treated soil must not swell any more than the untreated soil).
- The UCS of the treated soil should be at least 50 psi after curing and at least 25 psi greater than the strength of the untreated soil. (Both treated and untreated samples are to be cured exactly the same).
- The UCS of the treated soil following the 3-D swell test should be at least 25 psi greater than the strength of the untreated soil.

APPENDIX B

MOLDING PROCEDURES FOR NONSTANDARD STABILIZERS

Following is a detailed explanation of the molding and curing procedures that the researchers followed to ensure that each product had an opportunity to perform under optimal conditions as specified by the product manufacturers.

NON-CALCIUM-BASED STABILIZERS

Dustac (Lignosulfonate)

At the time of commencement of testing, this product was discontinued; therefore, no specific procedures were submitted by the manufacturer. The product received was part of leftover stock from a facility in Ohio. Researchers decided to continue testing in order to cover the lignosulfonate class of stabilizer. The product was received in powder form.

The researchers adopted the application rate and procedures used in a study done by the U.S. Army Corps of Engineers based on the amount of stabilizer that performed the best.³⁵ An application rate of 3.37% per weight of dry soil was selected. A 30% powder-water solution was prepared with part of the molding water required to reach the optimum moisture content. The remaining water and 30% powder-water solution was mixed thoroughly into the soil by hand. Samples were compacted within an hour of mixing and air dried for 3 days before placing in the 3-D swell test.

PennzSuppress D (Emulsified Petroleum Resin)

At the time the product was received, American Refining Group, Inc. had just bought certain assets from Pennzoil-Quaker State Company, giving it exclusive rights to manufacture PennzSuppress D. Due to the change in ownership, there was not much available in terms of specific procedures. The researchers followed procedures obtained from the PennzSuppress D website.

The researchers tested two application rates. The first application rate was a concentration of 13.75 ml of a 4:1 dilution of PennzSuppress D per 1000 g of soil, which roughly equates to a 1- gallon per square yard treatment and was obtained from the

website. The second application rate of 2.5% PennzSuppress D total solids (66%) per dry weight of soil was obtained from the U.S. Army Corps of Engineers study.³⁵ The PennzSuppress D was mixed with the water required to bring the sample up to the optimum moisture content (from 11% in situ to 24% optimum moisture content). The dilute solution of PennzSuppress D and soil was then mixed thoroughly and compacted.

Enviroseal M10 + 50 and LBS (Polymer)

This stabilizer consists of two components: M10+50, which is an acrylic polymer, and LBS, which is a silicon-salt solution. The vendor's procedures call for an application rate of 2 to 5 liters per m³ soil M10 + 50 and 0.2 to 0.5 liters per m³ soil LBS depending on the amount of clay in the soil. A clay content of 20% requires 0.2 liters LBS per m³ soil, and a clay content of 40% (as was the case with the soil used for testing) requires 0.4 liters LBS per m³ soil. The researchers molded samples for 3-D swell with 5 liters per m³ M10 + 50 and 0.4 liters per m³ soil LBS as per the vendor's recommendation. Samples were also tested at a higher application rate as tested by the U.S. Army Corps of Engineers.³⁵ The LBS was not used in combination with the M10 + 50 in the U.S. Army Corps of Engineers testing.³⁵ Therefore, the application rate for the LBS was increased by the same proportion as the increase in M10 + 50 between vendor and U.S. Army Corps of Engineers application rates.³⁵ The higher application rates were 5% by dry weight of soil M10 + 50 and 0.2507% by dry weight of soil for the LBS.

The vendor's procedure called for the soil moisture content to be 75% or less of the liquid limit of the soil. The soil was at 15% moisture content and the optimum of 24% was reached by adding a mixture of the appropriate quantities of LBS, water, and M10 + 50.

The LBS was mixed with water followed by the M10+50. The LBS/water/M10+50 solution was then thoroughly mixed into the soil and compacted. Samples were air cured for 3 days before commencing the 3-D swell test.

EMC SQUARED 2000/EMS Dual Component Treatment (Enzyme)

This stabilizer consists of an EMC Squared 2000 component used in conjunction with an EMS component. The researchers used an application rate of 9.345 ml per ft³ of

soil for the EMC Squared 2000 and 18.69 ml per ft³ of soil (a standard ratio of 2 to 1) for the EMS.

The soil was maintained as close as possible to the field moisture content during processing. The EMC Squared 2000 was mixed with the water required to bring the soil up to optimum moisture, and the EMS was then added to the diluted EMC Squared 2000 and water solution. This solution was then gradually added to the soil, mixed thoroughly, and allowed to stand in accordance with ASTM D698. Samples were molded and then allowed to cure at room temperature in an unconfined state for 3 days prior to initiating the 3-D swell test.

An optimum moisture content of 25.5% was obtained for the treated material, which was 4.5 percentage points higher than the untreated material (21%). The vendor was concerned with this increase in optimum moisture content and said that a percentage point or two higher is the most they have ever encountered. The researchers agreed to mold a set of samples at 25% moisture content and another at a moisture content of 22.5%.

Roadbond EN 1 (Acid)

The vendor submitted procedures and delivered the product in a 100:1 (water:stabilizer) dilution. The application rate to be used was 3% Roadbond EN 1 solution (in a 300:1 dilution) by dry weight of soil. The soil was pre-wet to optimum moisture content minus 3%, with the 3% Roadbond EN 1 (300:1) solution bringing the soil up to optimum moisture. The procedure called for one-half of the required amount of pre-wet water to be added to the soil and mixed thoroughly. The remainder of the pre-wet water was added and mixed thoroughly. Then one-half of the Roadbond EN 1 (300:1) solution was added and mixed thoroughly for 5 minutes. The sample stood covered for 5 minutes; the remainder of Roadbond EN 1 (300:1) solution was added and mixed for 5 minutes. The sample stood covered for 5 minutes. The sample was remixed for 1 minute and let stand in accordance with ASTM D 698 prior to molding.

The vendor's curing procedure called for the sides of samples to be sealed for 7 days with top and bottom porous stones in place. The samples were then unsealed, porous stones removed, and the samples dried back until one-half of the molding

moisture was removed. The porous stones were then replaced and the sides resealed and placed in a 100% humidity curing room for an additional 10 days. At the end of the 10 days, the samples were ready for 3-D swell testing.

EcSS 3000 Ionic Soil Stabilizer

The researchers received the product in a 300:1 (water:EcSS 3000) dilution ratio along with application and curing procedures from the vendor. The vendor-recommended application rate was 1 gallon per 600 ft³ of soil. The 300:1 EcSS 3000 solution was applied by a spray bottle until optimum moisture was attained, which equated to roughly 1.3 gallons of product per 600 ft³ of soil. The vendor agreed that this would not be a problem given the small volume used for testing in the laboratory.

The procedure called for the treated soil to sit in sealed plastic containers for 7 days and for the material to be mixed at least twice a day during the 7 days. During this testing, the samples were mixed on all weekdays, but not on Saturday and Sunday. The vendor was informed of this and agreed that the sealed treated soil can sit over the weekend without mixing. After the 7-day period, the samples were molded and air cured for 3 days before testing.

The vendor called for the use of ASTM D 4546 Method B with the proper overburden applied. ASTM D 4546 Method B is the standard test method for one-dimensional swell, which was not followed during this research project. Based on observations of sulfate heave in the field, the subgrade swells laterally as well as vertically. The researchers believe that 3-D swell is a more realistic measurement to simulate field conditions.

Top Seal (Vinyl Acrylic Copolymer)

The vendor recommended an application rate corresponding to a dilution of 1 part Top Seal to 3 to 10 parts water. This diluted solution was then used to bring the soil up to optimum moisture content. For this research, a dilution ratio of 1:3 was chosen because it represented the maximum application rate recommended by the vendor.

The dilute solution of Top Seal was mixed in with the soil and compacted following thorough mixing. The vendor's procedure called for an over-coating of the

sample with a dilute solution of Top Seal ranging from 1:1 to 1:20 water to Top Seal approximately 72 hours after extraction from the mold or after a thorough drying of the outer surface. This over-coating was not used because the researchers deemed it impossible to replicate in the field.

The samples were then air cured for 28 days at room temperature, as specified in the vendor's procedure, before starting the 3-D swell test.

Soil Sement (Aqueous Acrylic Vinyl Acetate Polymer Emulsion)

The vendor requested soil for their own testing in order to establish the required application rate of Soil Sement. Based on the vendor's own in-house wet and dry UCS testing, they recommended an application rate of 1 gallon per 10 ft² to a depth of 3 in. The product was mixed in with the required amount of water to bring the soil up to optimum moisture, mixed into the soil, and then compacted. A set of samples as also molded at an application rate six times the recommended vendor's rate. This increased application rate is achieved by using only the product in the as-received state to bring the material up to optimum moisture content. According to the Material Safety Data Sheet (MSDS) received for this product, the percentage weight of active ingredients ranges from 5 to 50%.

The molded samples were cured at 122°F (50°C) until a constant weight was reached. For the vendor's application rate, this was achieved at 20 days and for the increased application rate at 13 days. Following the oven curing, the samples were placed in the 3-D swell test.

ClayStar 7 (Phosphoric Acid and Surfactant)

The application rate submitted by the vendor was 3.5 ml per lb of dry soil. The researchers also decided to test an application rate of 10 ml per lb in order to see if a higher application rate would be beneficial. The amount of water needed above the insitu moisture to achieve optimum moisture was used as the fluid base to which ClayStar 7 was added. The vendor procedure called for aerating the ClayStar 7 and water solution by repetitively pumping in a large beaker with a large syringe to ensure that the ClayStar 7 concentrate is completely dispersed. The dilute solution was allowed to stand for

30 minutes to an hour slightly aerating every 15 minutes to ensure proper mixing. According to the vendor's procedure, the aeration can be accomplished in the field by means of a small truck-mounted or ground-based compressor.

Once the solution hydrolyzed, the dilute solution was applied directly to the soil in a plastic bowl or non-metallic tub or tray. Following thorough mixing, the treated soil stood for 15 to 30 minutes for saturation and penetration. After compaction, the samples were air-cured for 3 and 7 days at room temperature prior to placing in the 3-D swell test.

MODIFICATION OF CALCIUM-BASED STABILIZERS

Ground-Granulated Blast Furnace Slag (GGBFS) *(see [Table B3](#))

Application rates for the GGBFS were established in relation to the optimum lime content as determined by Eades & Grim (ASTM D 6276). Part of the optimum lime (6% for the soil used in this testing) was substituted for GGBFS in the following proportions: 3% GGBFS combined with 3% Lime and 5% GGBFS and 1% hydrated lime. This is similar to the methodology followed by Wild et al. in their testing of GGBFS to prevent sulfate attack in lime-stabilized kaolinite.⁸ The GGBFS was mixed in with the soil followed by the lime and the remaining water to bring the material up to the optimum moisture content and then compacted. After compaction, the samples were air-cured for 3 days at room temperature prior to placing in the 3-D swell test.

The GGBFS used in this project was a Type 100 (ASTM C989) and was supplied by Holcim from their Chicago Skyway Facility. The hydrated lime used was obtained from the Clifton, Texas, plant of the Chemical Lime Company.

Class F Fly Ash *(see [Table B3](#) for Chemical Analysis)

Researchers used hydrated lime as the activator for the Class F fly ash at a ratio of 1:3 (activator:fly ash). The ratio follows the recommendation of the American Coal Ash Association's Flexible Pavement Manual (recommend a 1:3 to 1:4 activator to fly ash ratio).³⁶ To obtain the optimum application rate of lime and fly ash, the unconfined compressive strength of various percentages of lime/fly ash samples as determined following a 7-day moist cure (blue curve in [Figure B1](#)). The UCS was also measured on samples subjected to a 7-day moist cure, followed by a 3-day dry back, and finally a

10-day capillary rise (pink curve in [Figure B1](#)). The application rates represented in [Table B1](#) are as follows:

Table B1. Fly Ash to Lime Ratios Used for Strength Testing.

% Fly Ash	% Lime
0	0
5	1.67
10	3.33
15	5
20	6.67

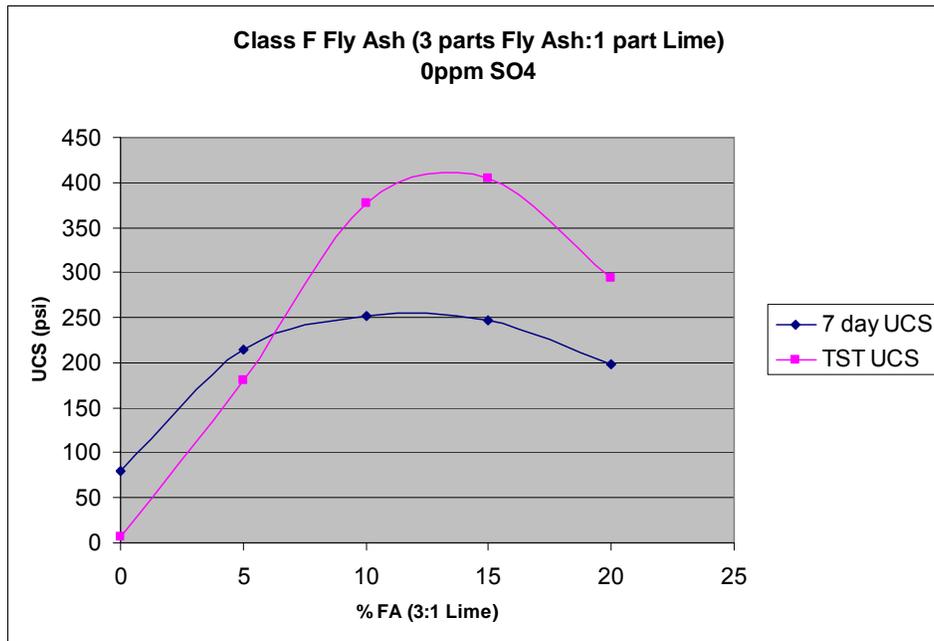


Figure B1. Unconfined Compressive Strength Testing Used to Determine Optimum Stabilizer Content.

The fly ash was supplied by Boral Material Technologies from their Rockdale, Texas, facility and meets specification ASTM C618 for Class F fly ash. The hydrated lime used was obtained from the Clifton, Texas, plant of the Chemical Lime Company.

The researchers subsequently decided to test the following combinations of fly ash and lime ([Table B2](#)).

Table B2. Fly Ash to Lime Ratios Used in Subsequent Testing.

% Fly Ash	% Lime
5	1
10	2
10	4
12.5	4.2

Class C Fly Ash *(see Table B3)

The application rates tested for Class C Fly Ash were 5%, 10%, and 15%. The fly ash was mixed into the soil, followed by the water needed to bring it up to optimum moisture and compacted with the Superpave Gyratory Compactor.

The fly ash was supplied by Boral Material Technologies from their Gibbons Creek, Texas, facility and meets specification ASTM C618 for Class C fly ash.

Type I/II Cement

Samples were molded with 4%, 6%, and 8% Type I/II cement. The cement used was from the TXI Midlothian facility.

***Table B3. Chemical Analysis of GGBFS and Fly Ash Used for This Project.**

Chemical Analysis	GGBFS	Class F FA	Class C FA
Silicon dioxide (SiO ₂) %	36.61	52.55	36.65
Aluminum oxide (Al ₂ O ₃) %	9.08	23.72	20.98
Iron Oxide (Fe ₂ O ₃) %	0.83	4.31	5.51
Sum SiO ₂ , Al ₂ O ₃ , Fe ₂ O ₃ %	46.52	80.58	63.14
Calcium Oxide (CaO) %	38.37	13.08	24.97
Magnesium Oxide (MgO) %	11.02	2.17	4.34
Sulfur Trioxide (SO ₃) %	2.54	0.67	1.25
Sodium Oxide (Na ₂ O) %		0.29	1.66
Potassium Oxide (K ₂ O) %		0.90	0.48
Amount retained on No. 325 Sieve %	1.4	24.96	12.22
Specific gravity	2.88	2.36	2.7